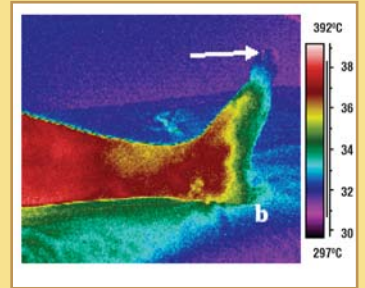
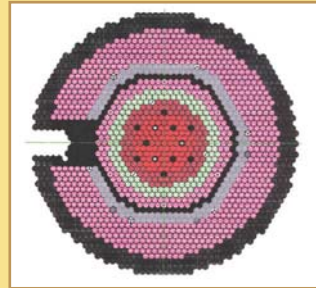
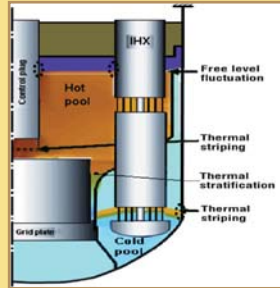


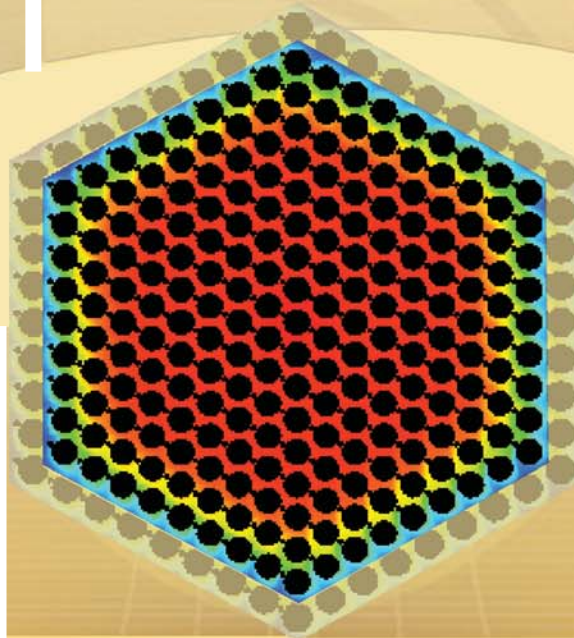
IGCAR

Excellence with Relevance

High Impact Breakthroughs



Significant Achievements 2008-2010



A Mission Centre with Science based Technology

IGCAR

Excellence with Relevance

High Impact Breakthroughs 2008-2010



सत्यमेव जयते

Government of India
Department of Atomic Energy
Indira Gandhi Centre for Atomic Research
Kalpakkam-603102.

FOREWORD



Indira Gandhi Centre for Atomic Research **A Mission Centre with Science based Technology**

High Impact Breakthroughs - January 2008 to March 2010

Mission of IGCAR

- ✓ To conduct a broad based multidisciplinary programme of scientific research and advanced engineering development, directed towards the establishment of the technology of sodium cooled Fast Breeder Reactors (FBR) and associated fuel cycle facilities in the country.
- ✓ The mission includes the development and applications of new and improved materials, techniques, equipment and systems for FBRs.
- ✓ Pursue basic research to achieve breakthroughs in fast reactor technology
- ✓ ISO 9001 certified laboratories and services

Vision Statement

Indira Gandhi Centre for Atomic Research must provide robust R&D products, consultancies and enabling support to realise prototype and commercial sodium cooled Fast Reactors being built by Bharatiya Vidyut Nigam (BHAVINI) and Fast Reactor Fuel Cycle Facility (FRFCF) in time, in cost and with quality to the complete satisfaction of the utilities.

To be a global leader in sodium cooled fast breeder reactors and associated fuel cycle technologies by the year 2020 AD

“Excellence is not a skill. It is an attitude”

-Ralph Marson

Indira Gandhi Centre for Atomic Research was started with a clear mission of developing Fast Breeder Reactor & associated fuel cycle technologies in the country for commercial exploitation. The successful design, construction and criticality of FBTR in 1985 and successful operation for the last 25 years, with a very few incidents, have been an important milestones in demonstrating the indigenous capability of construction and operation of fast spectrum reactors. Achieving a burn-up of 1,65,000 Megawatts day per tonne of the unique high plutonium based carbide fuel, successful reprocessing of this fuel, and mastering the sodium handling technology including extended continuous operation of the sodium pumps without maintenance, are just a few of the major technological highlights of the Indian fast reactor programme.

This success has paved the way for stepping into the commercial phase of the second stage of the nuclear power generation programme. Fast Reactor Fuel Cycle Facility (FRFCF), unique in the world, to close the fuel cycle of sodium cooled fast reactor, co-located with the Prototype Fast Breeder Reactor and Fast Breeder Reactor-I & II, when realised in 2016 shall be another milestone towards attaining leadership in science and technology in this important domain of closing the fuel cycle. We have embarked on design of future reactors with enhanced safety features, reduced cost per unit power production and high breeding with innovative metallic fuels.

The achievements in technology have been possible based on developing rigorous science based technologies for the last forty years in all the relevant disciplines, through multi and interdisciplinary research, attracting and nurturing young men and women of high merit to pursue the cause of national mission with dedication and perseverance. In this journey of excellence, the Centre has achieved several scientific and technological milestones with international benchmarks and high impact on our mission programme. These milestones have high relevance to science based technology being pursued by our Centre.

The compilation of the significant achievements of our Centre during the period 2004-2007 was brought out in the form of a book "Benchmarks". This effort has been appreciated by several eminent persons, peers and colleagues. The appreciation was both for the content and the presentation. I have directed the contributors and editorial team to make a choice of the articles conforming to various criteria like international benchmark, first of its kind, highly relevant to the activities of the Centre or basic research of high impact. Carefully chosen articles highlighting the achievements of the Centre in the last two years (April 2008 to March 2010) have been collated in the form of a compendium. The articles are diverse, for example, describing the codes developed to estimate the performance of fuel, experimental performance assessment of the unique high burnup mixed carbide fuel, studies on structural materials which have the scope of improving the safety and economics of the future reactors, basic research on tunable thermal properties and room temperature ionic liquids. Engineering analysis of optimum plant layout, establishing thermal stripping limits, novel design of reactor components are some examples where innovative R&D was carried out. The technology of reprocessing of the mixed carbide fuel, innovative design of sensors and procedure for developing boron control rods for Prototype Fast Breeder Reactors are some of the significant developments the Centre mastered after putting in considerable effort.

This exercise gives assurance that a path of excellence has become a way of working with the colleagues at the centre.

*"Persistence is the twin sister of excellence.
One is a matter of quality; the other, a matter of time"*

-Anonymous

We firmly believe that quality and commitment of our colleagues is the key to our strategy of accomplishing and sustaining excellence, achieving targets and establishing the Centre among the world leaders in our chosen domains of pursuits. We believe in innovations, enhanced ethics of management, creativity, connectivity and clarity as our toolkit for achieving excellence with relevance. We are pursuing an exciting and vital mission, wherein inspiration is in delivery of quantity with quality.

I would like to compliment the editorial team, under the leadership of Dr. M. Sai Baba, which has worked with dedication and imagination to bring out this unique compendium.



(Baldev Raj)
Director, IGCAR

EDITORIAL

IGCAR- A Mission Centre with Science based Technology

HIGH IMPACT BREAKTHROUGHS

January 2008 to March 2010

Indira Gandhi Centre for Atomic Research is the second largest R&D establishment of the Department of Atomic Energy, next only to Bhabha Atomic Research Centre. Over the years, the Centre has established a comprehensive range of R&D facilities covering the entire spectrum of FBR technology related to sodium purification and monitoring, reactor engineering, structural mechanics, thermal hydraulics and flow induced vibration, component testing in high temperature sodium environment, hydraulic development of sodium pumps, reactor physics, metallurgy and materials, chemistry of fuels and materials, fuel reprocessing, reactor safety, control and instrumentation, computer applications etc. IGCAR has developed a strong base in a variety of disciplines relating to these advanced technologies.

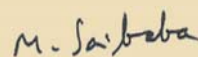
IGCAR has extended valuable and unique expertise and facilities to other vital sectors such as defence, space and Indian industries to develop reliable solutions to specialised and unresolved challenging problems. It has nurtured strong and continuing collaborations with leading educational and R&D institutes like the Indian Institutes of Technology, Indian Institute of Science, National Research Laboratories, Public Sector Organisations, and select institutes from abroad.

At IGCAR, it is our conviction that great heights in technology can be attained only with scientific breakthroughs and innovations. In a multidisciplinary Centre like ours, the achievements are based on robust interface between science and technology. In this journey of excellence, the Centre has achieved several scientific and technological milestones with international benchmarks and high impact on our mission programme. These milestones have high relevance to science based technology as practiced at our Centre. A similar compendium of achievement brought out two years earlier. This is the second in the series for the last two years (April 2008 to March 2010). We have received a large number of contributions from various groups of the Centre and critically reviewed and identified the appropriate contributions for this compendium. Amongst the contributions; 33 articles are categorized as benchmarks in science, 31 in engineering and 23 in Technology.

You would get a flavour of excellence and relevance, when you go through this compendium; we look forward to receive your valuable guidance and suggestions to enhance our approach and motivation. The contents of the compendium are being made available at www.igcar.gov.in/benchmarks. The contributions have resulted in about 150 publications in peer-reviewed journals and several patents. Several of them also formed basis of design, manufacturing, operation and life cycle management decisions.

We are grateful to Dr. Baldev Raj, Director, IGCAR, who has been a constant source of encouragement and inspiration in shaping the structure of this compendium. We thank him for the valuable suggestions and guiding us in our journey of excellence. We take the opportunity to thank all the Group Directors and colleagues of the Centre for their efforts and contributions in bringing out this compendium. It is a pleasure to acknowledge the members of the editorial committee and colleagues from Scientific Information Resource Division for their valuable contributions.

for Editorial Committee

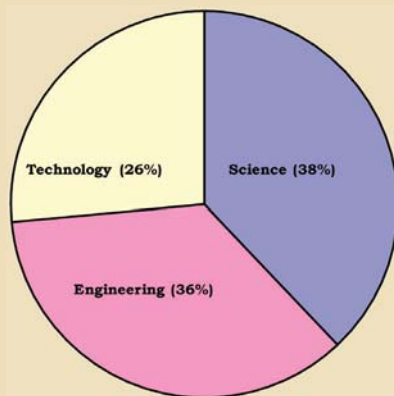


(M. Sai Baba)

EDITORIAL COMMITTEE

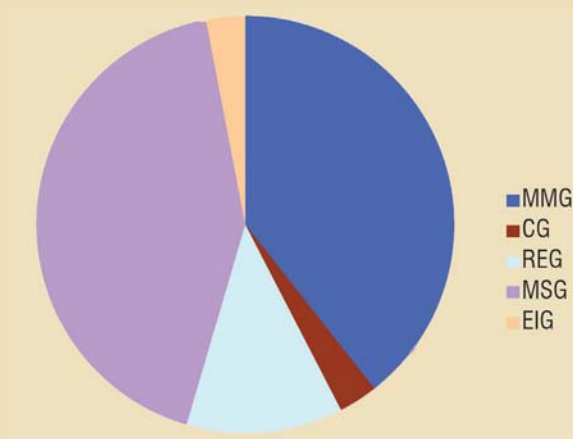
Dr. M. Sai Baba; Dr. Saroja Saibaba; Dr. Vidya Sundararajan;
Dr. T. S. Lakshmi Narasimhan; Dr. N. Subramanian; Shri S. Varadarajan

Contributions

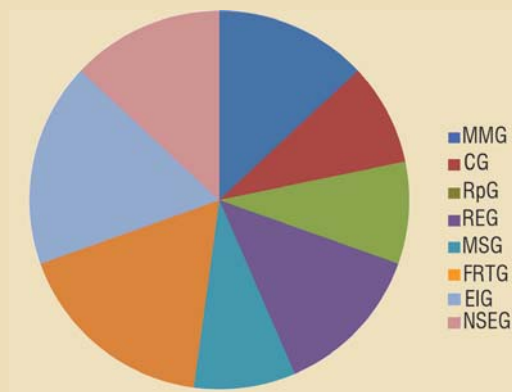


Discipline Wise Contributions

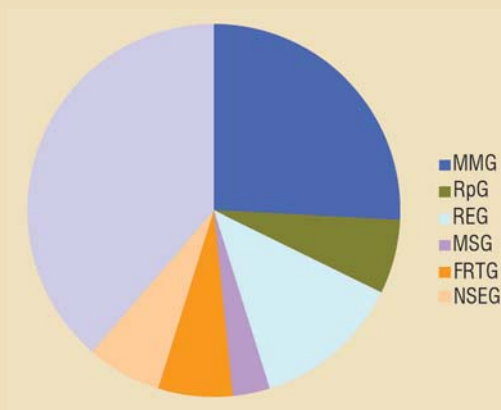
SCIENCE



TECHNOLOGY



ENGINEERING



MMG :Metallurgy & Materials Group
MSG :Materials Science Group
REG :Reactor Engineering Group
RpG :Reprocessing Group
NSEG:Nuclear & Safety Engineering Group
CG :Chemistry Group
FRTG :Fast Reactor Technology Group
EIG :Electronics & Instrumentation Group

IGCAR - A Mission Centre with Science based Technology

High Impact Breakthroughs 2008-2010

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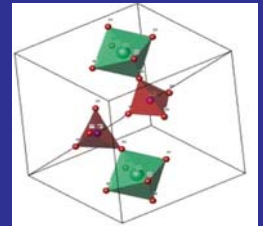
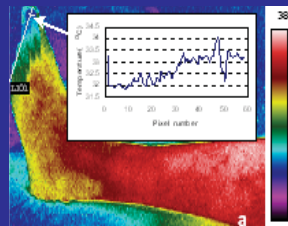
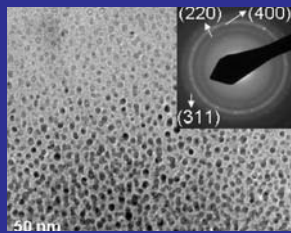
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SCIENCE

Performance Assessment of Mixed Carbide Fuel of Fast Breeder Test Reactor at High Burn-up

EXECUTIVE SUMMARY

Fast Breeder Test Reactor at Kalpakkam uses a unique, indigenously developed mixed plutonium-uranium carbide ($U_{0.3}Pu_{0.7}C$) as the driver fuel. This unique fuel has attained a record burn-up of 165 GWd/t without any failure. This has been possible due to the systematic performance assessment of the FBTR fuel at various stages of burn-up through Post Irradiation Examination (PIE). The examinations to assess the fuel performance included estimation of fuel swelling by destructive and non-destructive methods, metallography, fission gas release measurements etc. The performance evaluation of the FBTR fuel and the extensive thermomechanical modelling and analysis gave the confidence to enhance the burn-up well beyond the conservative design burn-up limit of 50 GWd/t.

OUTLINE

In-pile performance of FBTR mixed carbide fuel was evaluated by non-destructive and destructive examinations. Techniques established inside the hot cells for fuel performance evaluation included X-radiography, neutron radiography, fission gas extraction and analysis system and remote metallography facilities. X-radiography and Neutron radiography techniques were employed to assess the condition of fuel pin internals and to estimate the axial swelling of fuel. Innovative methods were adopted during X-radiography of irradiated fuel pins to overcome the high gamma background. Fuel stack length, pellet to pellet gap and pellet to clad gap were measured from the radiographs. The maximum increase in fuel stack length is around 3.7 % after 155 GWd/t burn-up. Fission gas release measurements at various burn-ups have indicated that the FBTR mixed carbide fuel has very low fission gas release even at high burn-up of 155 GWd/t. At burn-up levels beyond 100 GWd/t, isolation of the top and bottom plenum regions of the fuel pins was observed due to closure of fuel-clad gap. This necessitated innovation in the fission gas extraction system. A new double end fuel pin puncture chamber (Figure 1) was designed and used for fission gas extraction from the high burn-up fuel pins. Gas chromatography of fission gas extracted from the fuel pins after different burn-ups provided valuable information on the volume of fission gas released from the fuel to the plenum, the increase in the internal pressure, Xe/ Kr ratio and helium release due to alpha decay of actinides. Maximum fission gas release estimated on 155 GWd/t burn-up fuel pins was 16 % and the corresponding internal pressure in the fuel pin was measured to be 2.1 MPa is shown in (Figure 2). It can be seen that the trend in the fission gas release in the fuel pins as a function of burn-up follows the trend in swelling behaviour. Swelling, fuel-clad gap evolution, microstructural restructuring, cracking pattern etc were evaluated through metallography of the fuel pin cross-sections. (Figure 3) shows the photomicrographs of fuel pin cross sections at the centre and end of the fuel column at various stages of burn-up. Fuel-clad gap had closed completely along the entire length of fuel column in the case of 155 GWd/t burn-up. Fuel cracking pattern was also found to change from radial mode to circumferential after the closure of fuel-clad gap indicating Fuel Clad Mechanical Interaction (FCMI). Fabrication porosities present in the fuel was found to decrease with increasing burn-up indicating that the swelling of the fuel is being accommodated in the porosities. An outer rim devoid of sinter porosities was observed in the 155 GWd/t burnup fuel pin at the centre of the fuel column indicating that the fuel is undergoing hot pressing/creep deformation due to fuel swelling under clad restraint. This significant observation during post irradiation examination indicated that the fuel is able to accommodate swelling in the available porosities relieving Fuel Clad Mechanical Interaction stress. Microstructural examination and microhardness measurements of the clad did not indicate any carburisation.

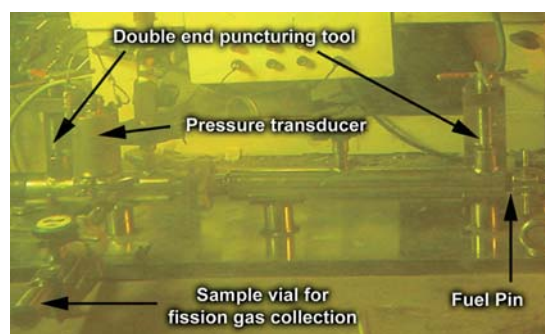


Fig. 1 : Double end fuel pin puncture chamber

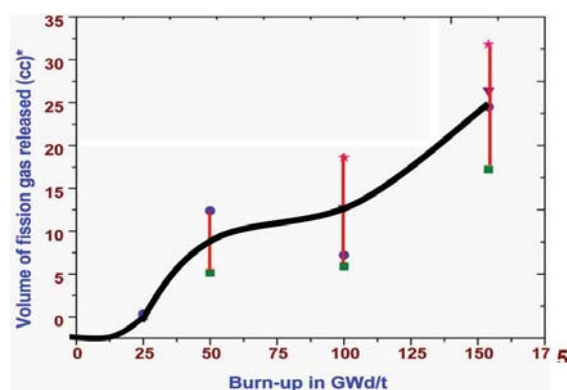


Fig. 2 : Trend in fission gas release as a function of burnup

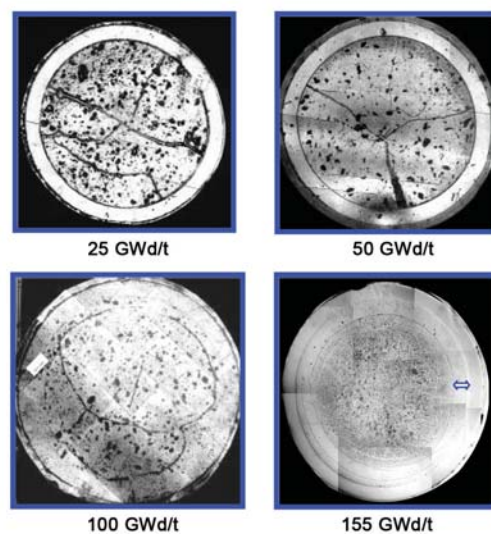


Fig. 3 : Micrographs of fuel pin cross sections at the centre of the fuel column at different burn-ups

ADDITIONAL INFORMATION

Post irradiation examination (PIE) aims at generating and evaluating data that can provide valuable insight into the in-reactor behaviour of fuels and structural materials. Special shielded facilities such as hot cells are employed for examination of spent nuclear fuel elements which are highly radioactive. Hot cells at radiometallurgy laboratory (RML) have facilities for metallurgical examination of irradiated FBTR fuel. The hot cells at radiometallurgy laboratory are maintained under inert gas environment to enable examination of the highly pyrophoric carbide fuel. The cells are equipped with remote handling devices and equipments for carrying out non-destructive and destructive examinations.

GENERAL EXPLANATION RELATED TO DESCRIPTION

With increasing fuel burn-up, fuel-clad gap reduces due to swelling of the fuel. The retention of solid and gaseous fission products in the fuel matrix manifests as swelling which in turn exerts mechanical stress on the cladding. A fuel which exhibits higher plastic deformation due to creep can accommodate the fuel expansion and relieve the stress on the clad. Release of fission gases, on the other hand dilutes the bond gas, reduces the fuel-clad gap conductance and generates stress on the clad due to the build up of pressure. Fission gas release measurements provide valuable data on the inventory of fission gas and the internal pressure in the fuel pins.

The temperature gradients existing in the fuel pin leads to the migration of some fission products which are corrosive in nature and can result in fuel clad chemical interaction. Carbide fuels are fabricated with hyperstoichiometry to prevent metallic phase formation due to reduction in the carbon to metal ratio with increasing burnup. Formation of metallic phase is detrimental due to the possibility of formation of low melting eutectics by the fissile materials with the cladding constituents. However, the higher carbon content in the fuel can lead to carburisation of the cladding by carbon transfer from the fuel to clad. Microstructural examination gives tell tale evidence of the various phenomenon occurring in the fuel.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Fast reactor fuel operates under rigorous environment of high neutron flux and temperature. A host of irradiation effects occur in the fuel and structural materials due to the aggressive environment. These include generation and accumulation of fission products, redistribution of fission products and actinides, changes in the chemical state of the fuel, fuel restructuring, generation of internal pressure in the fuel pin due to release of fission gases etc. These changes will have implications on the fuel performance due to various interactions between the fuel and clad during reactor operation. Study of the various irradiation induced changes and their evaluation are crucial for ensuring safe operation of the reactor, for extending the fuel burnup and to exploit the full potential of the fuel for economic and reliable operation.

ACHIEVEMENTS

The performance assessment of the FBTR fuel at different burnup levels has immensely contributed to comprehensive understanding of the fuel behaviour of the unique plutonium rich carbide fuel. The feedback from post irradiation examination on the irradiation behaviour facilitated the enhancement of linear power and burnup of the fuel to 400 W/cm and 165 GWd/t respectively, well beyond the conservative design estimates without any fuel failure. The role of post irradiation examination, analysis of post irradiation examination data and thermomechanical modelling has been crucial in achieving this remarkable milestone in our endeavour of fast reactor technology development.

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Novel Method for Recovery of Noble Metals using Room Temperature Ionic Liquids

EXECUTIVE SUMMARY

A novel generic approach has been developed for the recovery of fission platinoids from high level liquid wastes using room temperature ionic liquid (RTIL) as medium. The process exploits some fascinating properties such as extracting ability and wide electrochemical window of the ionic liquid, tri-n-octylammonium nitrate, to develop a robust and industrially congenial procedure for platinoids recovery.

OUTLINE

Significant quantities of platinum group metals (ruthenium, rhodium and palladium) are formed as by-products of nuclear fission in a nuclear reactor (Table 1). Most the fission PGMs are non-radioactive except ^{107}Pd isotope, which has a half-life of 6.5×10^6 y. The intrinsic radioactivity of ^{107}Pd (soft β -emitter with E_{max} of 35 keV) is very weak and it can be tolerated for many industrial applications. It is expected that by the year 2030, the amount of noble metals produced by nuclear reactor operation all over the world will be comparable with the amounts of noble metals available in earth crust at that time. Therefore, the spent nuclear fuel is a valuable resource of man-made noble metals.

PUREX process is being adopted for the recovery of uranium and plutonium from spent nuclear fuel. The raffinate rejected after the extraction is known as high-level liquid waste (HLLW), which contains more than 70% of fission noble metals existing in the form of nitrate and nitrosyl complexes. Presently, there is no commercial process available for the recovery of these fission noble metals from this high-level liquid waste.

Room Temperature Ionic Liquids

Room temperature ionic liquids (RTILs) are organic salts, molten at temperatures lower than 373 K. They are generally composed of a bulky organic cation and an inorganic or organic anion. Room temperature ionic liquids have several attractive physical and chemical properties suitable for a wide variety of applications. Properties such as in significant vapour pressure, amazing ability to dissolve organic and inorganic compounds, tunability etc. make room temperature ionic liquids popular for synthetic applications and the wide electrochemical window offers huge scope for electrochemical applications. Room temperature ionic liquids are considered as possible substitutes for conventional high temperature molten salt employed in pyrochemical reprocessing and for traditional molecular diluents employed in aqueous reprocessing for developing novel recovery procedures.

The EX-EL process

A novel generic approach, extraction-electrodeposition (EX-EL) process, has been developed for quantitative recovery of palladium from simple and the most complex form of wastes such as high-level liquid waste (HLLW) arising from nuclear fuel reprocessing. The approach exploits the potentials of room temperature ionic liquid to act both as extractant and electrolytic medium and it involves extraction of palladium(II) from aqueous medium followed by electrodeposition directly from organic phase, which is unknown so far in a traditional solvent extraction process. The illustration is shown in (Figure 1). The process exploits the remarkable properties such as the liquid ion exchange behavior and wide electrochemical window of RTIL, tri-n-octylmethylammonium nitrate (TOMAN), to develop a congenial procedure for quantitative recovery of palladium.

Table 1: Relative abundances of isotopes of fission platinoids present in spent nuclear fuel (burn up 32,300 MWd/Te)

Isotope	Half-life	Relative abundance (wt.%) after	
		1 y cooling	30 y cooling
Ruthenium			
99	Stable	2.4×10 ⁻⁴	3.6×10 ⁻³
100	Stable	4.2	4.37
101	Stable	34.1	35.42
102	Stable	34.0	35.31
103	39.3d	3.6×10 ⁻³	----
104	Stable	23.9	24.9
105	4.4h	Trace	Trace
106	371.6 d	3.8	8.5×10 ⁻¹¹
Rhodium			
102	2.9 y	Trace	-
102m	207 d	Trace	-
103	Stable	100	100
103m	56.1m	1.6×10 ⁻⁵	-
106	29.8 s	1.8×10 ⁻⁶	-
Palladium			
104	Stable	16.9	15.8
105	Stable	29.3	27.4
106	Stable	21.3	26.4
107	6.5 × 10 ⁶ y (0.035Mev)	17.0	15.9
108	Stable	11.7	10.9
110	Stable	3.8	3.6

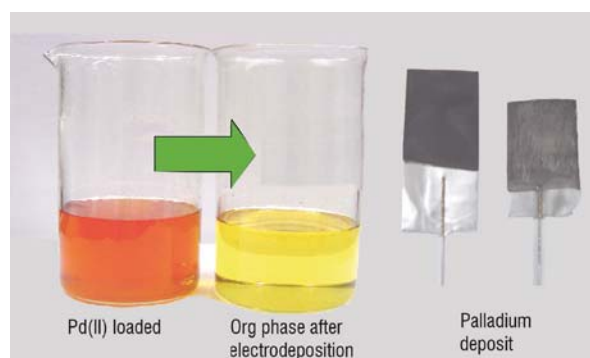


Fig. 1: Illustration of EX-EL method

ADDITIONAL INFORMATION

More than 60% of palladium was extracted using 0.5 M TOMAN/ CHCl_3 in a single contact and complete extraction was achieved in five contacts. The extracted palladium was quantitatively recovered by electrodeposition using a stainless steel cathode. Nearly twenty hours were required for complete recovery of palladium from organic phase which was contacted with a solution of palladium nitrate in 4.0 M nitric acid, while more than thirty five hours were needed for recovery from organic phase, which was contacted with HLLW. The process holds promise of decontaminating fission products by an order of magnitude during extraction of palladium from high-level liquid waste and also provides a large decontamination factor of ~ 1000 during electrodeposition step.

GENERAL INFORMATION ABOUT THE MAIN OBJECT OF EX-EL APPROACH

The EX-EL method is a novel, simple and industrially adaptable method for the recovery of palladium (as well as other noble metals) from various aqueous streams using room temperature ionic liquid. The procedure involves the extraction of palladium from aqueous acidic medium (leach liquor or HLLW) by a solution of tri-n-octylmethylammonium nitrate (TOMAN) in chloroform. In contrast to the conventional stripping procedure, electrolysis of loaded organic phase is carried out to recover the loaded palladium in metallic form. The unique property of RTIL (TOMAN) to act both as extractant and electrolyte medium has been exploited for the development of this new procedure. Quantitative recovery of palladium during electrodeposition is achieved during extraction as well as electrodeposition. The lean organic is recycled back to extraction stage. The present method avoids most of the problems associated with the reported procedures and minimizes the generation of secondary waste, which is indeed desirable for the treatment of radioactive wastes such as HLLW.

BRIEF DESCRIPTION ABOUT THEORETICAL BACKGROUND

Most of the cations and anions have very small ionic radii of the order of 10^{-10}m , which is indeed sufficient for permitting the ions to come in contact with each other and interact efficiently. The ionic solids formed from such cation-anion combinations melt at very high temperature. However, substitution of bulky polynuclear cations such as molecular organic cations in place of inorganic cations, sterically prevents the anion to interact efficiently. As a consequence, the magnitude of $\Delta G_{\text{e.s}}$ becomes less negative and the melting point of the resulting compound is lowered. For example the melting point of NaCl is 800°C and the 1-butyl-3-methylimidazolium chloride (bmimCl) is only 68°C . Other examples, shown in Table 2, indicates that the melting point can be brought down to sub-zero levels by appropriate choice of ions. It is realized that when the energy of coulombic interaction ($\Delta G_{\text{e.s}}$) is lower than the thermal energy of room temperature, the melting point of the compound is lowered below ambient temperature and exists as liquid. These are known as room temperature ionic liquids (RTILs) and exhibit all the properties of liquid state. Thus, RTIL is nothing but ionic liquids that are molten at near ambient conditions. The boiling point of water (i.e. 100°C) is taken as the maximum temperature range for defining RTIL.

ACHIEVEMENT

At present no method is available for the quantitative recovery of man made noble metals, palladium from high level liquid waste. The results flowing out from our studies indicate that they can be quantitatively recovered by adopting novel EX-EL method developed in our laboratory. The technique is novel from hydrometallurgical point of view that the extracted metal is electrodeposited directly from loaded organic phase.

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Development of Nanofluid with Tunable Thermal Properties

EXECUTIVE SUMMARY

We experimentally demonstrate the tunable thermal property of a magnetically polarizable nanofluid that consists of a colloidal suspension of magnetite nanoparticles of average diameter 6.7 nanometer. Controlling the linear aggregation length from nano to micron scales, the thermal conductivity (TC) of the nanofluid has been enhanced up to 216%, using 4.5 vol.% of nanoparticles. Repeated magnetic cycling shows that the thermal conductivity enhancement is reversible. It has been confirmed that the large enhancement in thermal conductivity is due to the efficient transport of heat through percolating nanoparticle paths. Our findings offer promising applications in 'smart' cooling devices.

OUTLINE

Magnetic nanofluid is a unique material that has both the liquid and magnetic properties. Nanofluids are considered to be the future coolants for electronic devices and engines. Very recently it has been demonstrated that manipulation of nanoparticles with carbon nanotubes can result in enhanced TC. Several nanofluids with enhanced TC have been tailored during the past several years. The effective medium theory fails to explain the large enhancement of k in nanofluids and the temperature dependence of k . Two hotly debated mechanisms, for heat transport in nanofluids are the Brownian motion induced convection and local percolation behavior.

We used a stable colloidal suspension of magnetite nanoparticles of average diameter 6.7nm, coated with oleic acid and dispersed in hexadecane. The size distribution of the nanoparticles measured using dynamic light scattering (DLS) experiment is shown in inset (a) of Figure 1. The TEM image of the nanoparticles is shown in the inset (b) of Figure 1 shows the TC ratio (k/k_f) of the nanofluid (NF) as a function of volume fraction of Fe_3O_4 nanoparticles, where k and k_f are the TC of NF and the base fluid respectively. No enhancement in TC is observed up to a volume fraction of 1.5% of Fe_3O_4 nanoparticle. At 2.6 vol.%, the increases in k/k_f was marginal. The highest value of k/k_f observed was 1.23 at 7.1 vol.% that corresponds to an enhancement in TC of 23%. The data fitted with Maxwell model shows good agreement, especially at higher volume fractions. The observation of TC enhancement at low particle loading (<2 vol.%) in presence of magnetic field (no enhancement without field) shows that aggregation is the main cause of TC enhancement within Maxwell's limit. The enhancement above 1.76 vol.% could be due to small clusters (dimers or trimers) formed in the NF due to magnetic dipolar attractions. It appears that the wetting of nanoparticles is enhanced due to the organic sheath, which in turn leads to a lower interfacial thermal resistance. We find that nanofluids with very small particles (~5nm) are also prone to settling when particles are uncoated. (Figure 2) shows the k/k_f of the NF as a function of applied magnetic field, during rise and decay, in 2.6 vol.% of Fe_3O_4 nanoparticles. It can be seen that the enhancement starts above 20 Gauss (G). Further increase in magnetic field leads to drastic enhancement in the TC.

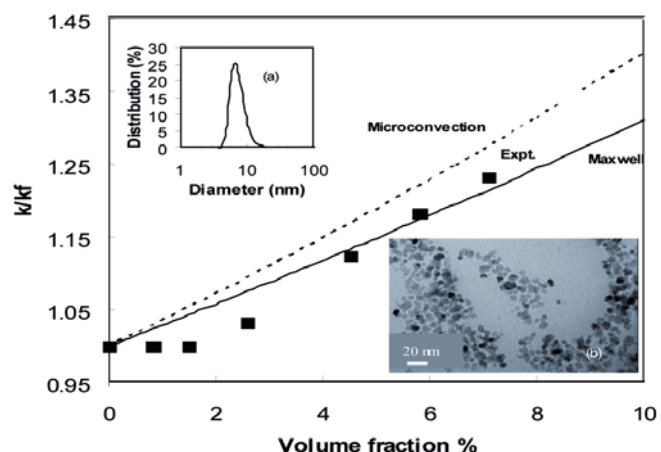


Fig. 1: The TC ratio (k/k_f) of the NF as a function of volume fraction of Fe_3O_4 nanoparticles. The size distribution of Fe_3O_4 nanoparticles (a) and the TEM image of nanoparticles (b) are shown in the inset

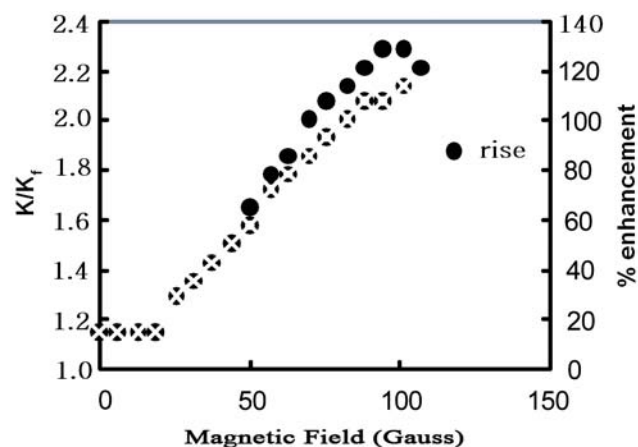


Fig. 2: The TC ratio (k/k_f) of the NF as a function of increasing and decreasing applied magnetic field in 2.6 vol.% of Fe_3O_4 nanoparticles

ADDITIONAL INFORMATION ABOUT MAGNETIC NANOPARTICLES

The observed reversible tunable thermal property of nanofluid may find many technological applications for this fluid in nano-electromechanical systems (NEMS) and micro-electromechanical Systems (MEMS) based devices. For example, depending upon the cooling requirement, the current or magnetic field can be precisely programmed to obtain the desired level of thermal conductivity enhancement or cooling. The mechanism of heat transport from a cylindrical device immersed in such a nanofluid coolant without and with magnetic field is depicted in the inset of Figure 3. When the field is off the nanoparticles behave as a normal fluid with random arrangement of particles. When the field is turned on, the parallel mode conduction leads to drastic enhancement of thermal conductivity.

GENERAL INFORMATION

The maximum enhancement of TC (128%; $k/k_f = 2.23$) is observed at a magnetic field of 94.5 G. While lowering the magnetic field, the TC value shows a small hysteresis but comes back to the original value when the magnetic field is turned off. Repeating the magnetic cycles several times, the TC is restored to its original value. The above experiments repeated in a variety of NF samples with aqueous and non aqueous carrier fluid showed similar results. Figure 3 shows the TC ratio (k/k_f) and the corresponding enhancement with applied magnetic field in the NF with 4.5 vol% of Fe_3O_4 . The inset of figure shows the schematic of the mechanism of heat transport from a cylindrical device immersed in NF without and with magnetic field. The variation of k/k_f at three different magnetic cycles (rise and decay) shows that the enhancement is reversible, with a slight hysteresis. The maximum enhancement was 216%, at an applied magnetic field of 101 Gauss. Increase in magnetic field in a magnetically polarizable nanofluid, leads to the nanoparticles aligning in the direction of magnetic field when the magnetic dipolar interaction energy dominates over the thermal energy. When the dipolar interaction energy becomes sufficiently strong, the magnetic particles form chain like structure. Here, each magnetite particle is single domain super-paramagnetic with a magnetic moment, m . Without any external magnetic field, the magnetic moments of the scatterers are oriented in random direction, which starts to align themselves along the direction of the magnetic field as the magnetic field is increased.

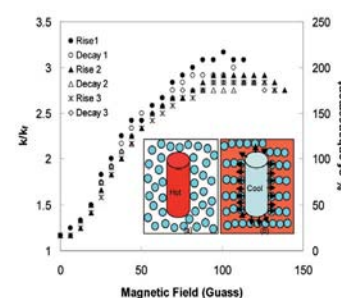


Fig. 3: The TC ratio (k/k_f) and the enhancement as a function applied magnetic field in the NF with 4.5 vol.% of Fe_3O_4 nanoparticles.

BRIEF DESCRIPTION

As nanoparticles start to form chains, the convection velocity drops drastically due to the cube dependence of particle size. Therefore, the Brownian Motion is severely arrested as the chain length increases. As the convection velocity decreases with increasing magnetic field, the observed enhancement in thermal conductivity can not be due to the microconvection mechanism. In a nanofluid, series and parallel modes of thermal conduction through the base fluid and the nanoparticles can be visualized within the mean-field models. The parallel mode has the geometric configuration that allows the most efficient means of heat propagation. Extremely large thermal conductivity can be expected from such parallel modes. Using the experimental data, the expected values of enhancement for the upper HS and parallel modes are 150 and 225% respectively. Strikingly, the observed enhancement of 216% is close to the predicted value for parallel mode conduction.

ACHIEVEMENT

We have developed magnetically polarizable nanofluids with tunable thermal properties. We demonstrate that by controlling the linear aggregation length from nano to micron scales, the thermal conductivity of the nanofluid can be tuned from a low to very high value. Further, it has been found that under repeated magnetic cycling, the thermal conductivity is reversible. Our findings offer promising applications in thermal management.

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Assessment of Multiple Recycling of Fuel in Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

A thermal neutron reactor does not support multiple recycle of uranium-plutonium fuel due to the degradation of fissile content of plutonium in just one cycle. In the FBR closed fuel cycle, possibility of multi-recycle has been recognised. The present study, using ^{239}Pu equivalence approach, demonstrates the feasibility of achieving near constant input inventory of plutonium and near stable plutonium isotopic composition after a few recycles of the PFBR fuel. After about five recycles, the cycle to cycle variation in the above parameters is below 1%.

OUTLINE

An FBR fuel cycle involves recycling of the discharge fuel, after reprocessing and refabrication, in order to effectively utilize the unburnt fuel remains and the freshly bred fissile material. Fuel gets burnt and transmuted in the core as well as in the blankets. Fissile burning is prominent in the core and the rate as well as the extent of loss in fissile content limits the fuel cycle length. The fissile breeding in the core is usually inadequate to replenish the lost fissile content. The total fissile breeding including that in the axial and radial blankets of an FBR would generally be more than adequate to make up for the fissile loss. Further, the fuel burnt during a cycle in the core is a small fraction of the fuel fed initially and a large portion of it is still unburnt at discharge. Thus extracting the unburnt part of the fuel in addition to the fuel bred in the core and in the blankets, is a major step in the FBR fuel cycle. After refabrication, suitably compensated for loss of fuel worth, could be re-fed to the reactor for its next cycle of operation. Such a fuel cycle is called as closed fuel cycle. Multiple recycle is not possible in thermal neutron reactors due to the deterioration of fissile content in plutonium just after one cycle. The present study is mainly aimed at finding the feasibility of achieving near stable plutonium inventory and plutonium composition after a few recycles in an FBR.

The fuel recovered from the spent-fuel is different from the original fuel in composition and hence in its effectiveness. Such changes are brought about due to transmutation of the fuel nuclides during the irradiation period, and also due to radioactive decay. It is mandatory that the mass of the feed fuel in each subassembly (SA) and its reactivity worth are ensured to be the same at the beginning of each cycle, which necessitates accepting variation in the fissile enrichment. The concept of " ^{239}Pu equivalence" is employed to judge the quality of the recycled fuel as against the initial fuel. The ^{239}Pu equivalence values used in the present work are given Table 1.

Therefore, given the initial feed composition, a study of fuel recycling involves (i) computation of the composition of the unburnt portion of the core fuel at the end of a cycle, (ii) computation of the composition of fissile mass produced in the blankets, (iii) computation of the fuel composition needed at the beginning of the next cycle, taking into account the recovery fraction and the decay due to delay times. Such a series of computations is repeated as many times as the desired number of cycles, to study the fuel evolution. Figure 1 shows one cycle of operations considered in the present study with a PFBR subassembly.

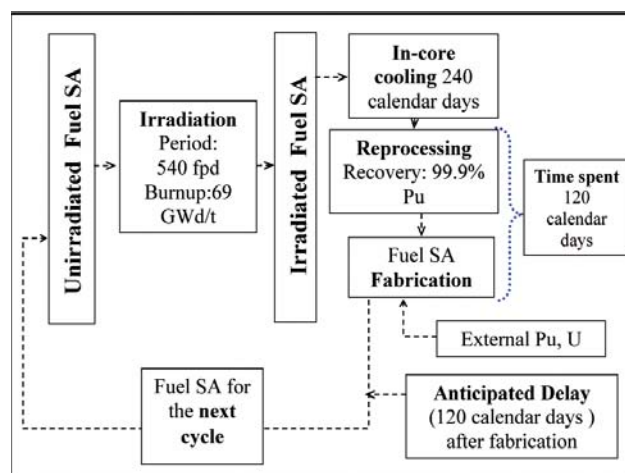


Fig. 1 : Fuel Cycle for PFBR

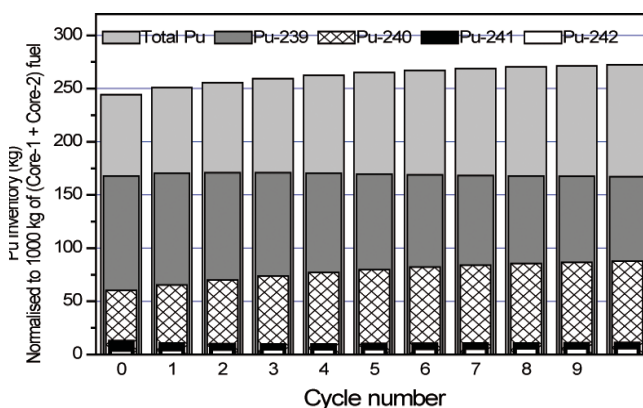


Fig. 2: Plutonium Inventory over Consecutive Cycles

Table 1: ^{239}Pu Reactivity Equivalence

Nuclide	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu	^{241}Am
Equivalence	1.0	0.1	1.5	0.04	-0.24

ADDITIONAL INFORMATION ABOUT EFFECT OF LONGER OUT-OF-PILE DELAY

The calculations were repeated with an out-of-pile delay time of four hundred and eighty calendar days. The observations are practically the same as above, except that the ^{241}Am production in each cycle, is doubled. Though the longer delay time must be significant with respect to management of the fuel cycles, the differences are not pronounced with respect to changes in the fuel composition.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

In the present work, periodic recycling of the fuel has been studied in the context of prototype fast breeder reactor (PFBR), for ten reprocessing cycles. The computer code ORIGEN2 is used to compute nuclide compositions for different times of irradiation or cooling. The core is irradiated for a burn-up of 69 GWd/t, which corresponds to an irradiation time of five hundred and forty full power days (fpd) at a specific power of 130 MW/t. At 75% capacity factor this corresponds to seven hundred and twenty calendar days. A net out-of-pile delay time of two hundred and forty calendar days has also been considered (Figure 1).

The inventories of plutonium isotopes per 1000 kg of total fuel, combined for core-1 and core-2, over the 10 cycles are given in Figure 2. It is seen in Figure 2 that the replenished plutonium has almost the same amount of ^{239}Pu as when the cycle begins. There is a marked buildup of ^{240}Pu , which practically accounts for the increase in total plutonium inventory. The ^{239}Pu equivalent of the total plutonium inventories in core-1 and core-2 respectively are maintained at about 164 kg and 219 kg per ton of the respective fuel.

However, the plutonium inventories increase from 207 kg per ton to 232 kg for core-1 and from 277 kg to 308 kg per ton for core-2 during the ten cycles. The ratio between the ^{239}Pu equivalence of the total plutonium to total plutonium inventory varies from 79.2% to 70.7% in core-1 and 79.2% to 71.2% in core-2. These variations show a tendency for saturation. Similar ratio for the plutonium produced in the radial blanket is 96% indicating its high worth. The fertile buildup is subsequently compensated for by a corresponding reduction in ^{238}U . Small changes in ^{239}Pu as well as in ^{241}Pu do not show up in the graph, but are not ignorable, because of their high worths. The inventories of ^{238}Pu and ^{241}Am are too small to be visible in Figure 2. The important point to note from Figure 2 is the saturation tendency of the inventory after a few cycles, indicating the possibility of recycling with little change in the fuel composition.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The ^{239}Pu equivalence method:

If q^i is the equivalence of an isotope i , M^i being its inventory, the ^{239}Pu equivalent mass of this inventory is $m^i = M^i q^i$. The elemental equivalents are obtained by summation. The inventory of a Pu isotope in the refabricated fuel is given by $M_N^i = \frac{m_N^i}{q^i}$, where $m_N^i = \frac{m_{\text{L}}^{\text{Pu}}}{m_{\text{B}}^{\text{Pu}}} \times m_{\text{B}}^i + m_{\text{R}}^i$, with L indicating loss in the core, R the quantum recovered, and B the quantum produced in the blanket.

ACHIEVEMENT

It is found that multiple recycling of the same fuel is possible for PFBR, keeping the isotopic content nearly constant. Concept of ^{239}Pu reactivity equivalence has been used for finding the variation in plutonium initial inventory between cycles. It is found that plutonium composition change with the cycle become negligible (less than 1%) after a few cycles. The study has also shown that the ^{239}Pu equivalent produced in the blanket is nearly 1.5 times the ^{239}Pu equivalent needed as compensation to start the next cycle. This indicates the self-sufficiency of PFBR in multiple fuel recycling. Further, the tendency of saturation observed in the inventories after a few cycles shows the possibility of recycling with only marginal change in the fuel composition.

PUBLICATIONS

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Superhydrophobic and Superhydrophilic Titanium Surface Prepared by a Novel Method

EXECUTIVE SUMMARY

Surface whose contact angles exceed above 150° are known as superhydrophobic (SHB) surfaces. The present study, describes the procedures involving the preparation of SHB coatings on titanium surfaces by dip coating method using myristic acid as the SHB coating material and the preparation of superhydrophilic surfaces by employing appropriate anodizing conditions. Atomic Force Microscopy (AFM) was used as the imaging tool to characterize these surfaces, contact angles of the surfaces were measured using KRUSS DAS 100 contact angle instrument. The results confirmed that coating with myristic acid on anodized titanium surfaces lead to the formation of superhydrophobic surfaces showing increased corrosion resistance and fouling resistance and ideal anodizing conditions lead to formation of superhydrophilic surfaces.

OUTLINE

Achieving superhydrophobicity on titanium surface has two prerequisites namely (i) nano surface roughness and (ii) a coating of hydrophobic material. The titanium surface was ultrasonically cleaned and pickled in nitric acid: hydrofluoro acid (40:4g/L) bath. Anodization was carried out in an electrolytic mixture of 1M H_2SO_4 and 0.16M HF for 1h in 30V to increase the roughness of the surface. Then 0.5M myristic acid ($CH_3(CH_2)_{12}COOH$, tetradecanoic acid) in ethanol was coated by using a simple dip coating method with an immersion time of 10 days, after which the samples were baked at $130^\circ C$. Superhydrophilic surface of titanium was obtained by anodization using 1M H_3PO_4 +0.5Wt % HF as electrolyte for 2h at 50V. The modified surfaces were characterized using Atomic Force Microscope (AFM) and water contact angle measurements using KRUSS DAS 100 contact angle instrument. The stability of the superhydrophobic coating was studied using Solartron 1287 Electrochemical Interface based electrochemical Impedance Spectroscopy studies (EIS). The microbe-repellant property was evaluated by exposing the samples to seawater observing the surfaces using an epifluorescence microscope.

The AFM studies on the coatings formed at various myristic acid concentrations showed that concentrations of 0.5M led to uniform coating and the particle size of the coating were in the nanometric range (100-200nm). It is possible, by anodization to generate a micro-rough surface and then by means of subsequent fixation of hydrophobic organic compounds to form superhydrophobic surface. EIS results (Figure1) showed that Ti coated myristic acid has high R_p value and low capacitance value than polished Ti which infers the coatings on these surfaces were stable and there was a significant increase in the corrosion resistance of titanium. The SHB surfaces when exposed to seawater and observed using an epifluorescence microscope revealed their ability to resist microbial attachment, exhibiting microbe-repelling property (Figures 2a and 2b). Figure 2c confirms the increased biocompatibility of the superhydrophilic surfaces as shown by the increased bioadhesion on these surfaces. Superhydrophobic coatings are reported to lower the surface free energy (s.f.e.) of a surface. In vivo studies have confirmed the beneficial effect of a low s.f.e. material in controlling microbial adhesion. Similarly increasing the s.f.e. could lead to a reversal of the effect, which is exactly what we observed on the superhydrophilic surfaces which exhibited increased bioadhesion properties.

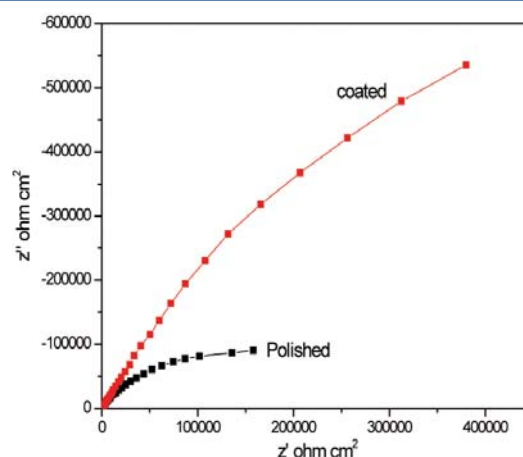


Fig. 1: Electrochemical impedance study of super hydrophobic titanium surface showing high corrosion resistance

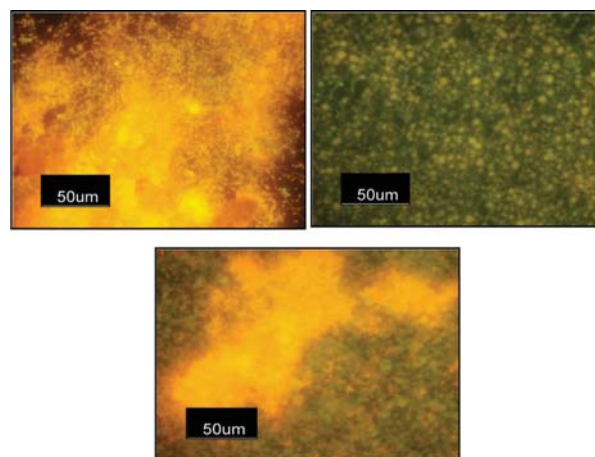


Fig. 2: Microbial attachment on control (uncoated) titanium b) Significant reduction in microbial attachment on superhydrophobic titanium surfaces c) superhydrophilic surfaces showing increased biocompatibility

ADDITIONAL INFORMATION ABOUT SUPERHYDROPHOBICITY AND SUPERHYDROPHILICITY

Hydrophobic means ‘water repelling’ and hydrophilic means ‘water loving’. When a water drop is placed on the solid surface, it may form more dense region with collapsed hydrogen bonded network if the surface is superhydrophobic in nature which gives the Water Contact Angle (WCA) above $140-150^\circ$ or it may form less dense region with open hydrogen bonded network when the surface is superhydrophilic whose water contact angle is usually below $20-50^\circ$.

GENERAL INFORMATION

Titanium is well known for its excellent corrosion properties in various environments. In the nuclear industry titanium is used in the manufacturing of dissolver vessels in nitric acid environments, where there is a constant need to increase its corrosion resistance. In addition, titanium is also proposed as the condenser material for PFBR, owing to its excellent corrosion resistance and total immunity towards microbially influenced corrosion. But titanium faces an unresolved problem of biofouling in such applications. Another application of titanium is in medical industry, where titanium surface is required to show increased bioactivity and biocompatibility when used as implant materials. In order to meet these challenges the present study focuses on manipulation of the surface wettability properties of titanium surfaces, by an innovative and simple approach which could make the surface either superhydrophobic or superhydrophilic. Surface energy and surface roughness are the two dominant factors for wettability which determines the water contact angle of the surface. When the surface energy is lowered, the hydrophobicity is enhanced. The lotus leaf is a famous example of a naturally occurring superhydrophobic surface, where water droplets falling onto them bead up and roll off. Recently, a lot of attention has been attributed to superhydrophobic coating on material surfaces. Surface whose contact angles exceed above 150° are known as superhydrophobic surfaces. The requisites for superhydrophobic surface include a combination of enhancing the surface roughness and lowering the surface free energy by incorporation of apolar groups on the metal surface. Figure 3 gives the spread of a typical water drop on the superhydrophobic surface (148°) and superhydrophilic (22°) surface respectively.

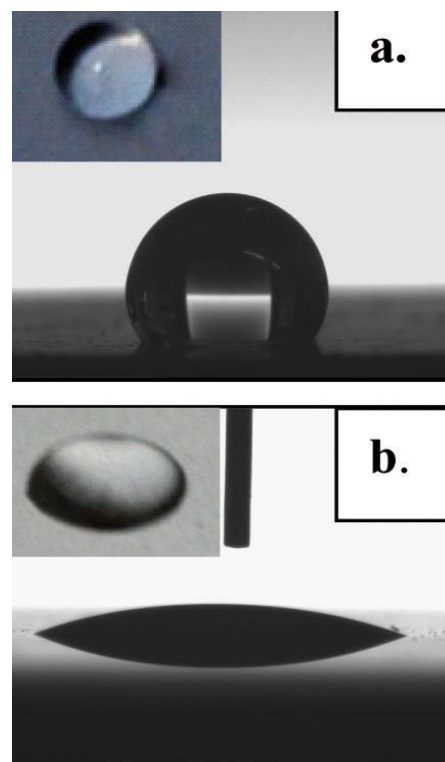


Fig. 3 : (a) Water droplet on superhydrophobic surface of titanium (inset showing photograph of water droplet on test surface) and (b) water droplet on the superhydrophilic surface of titanium

BRIEF DESCRIPTION

The lotus leaf has WCA $\sim 168^\circ$ whose surface is structured on two length scales by micron and nano-sized wax protrusions which predominantly contains $-\text{CH}_2-$ groups on the surface. The water droplets falling onto lotus leaf bead up and roll off. These rolling droplets pick up small particles of dirt so that the lotus leaves are self-cleaning, which is called “lotus effect.” Self-cleaning glasses, clothes, for protection against corrosion of concrete constructions (e.g. bridges, marines, under water constructions etc), anti-snow sticking are some of the applications of superhydrophobic surface. This technology could be applied in to aircraft to ensure that they are able to de-ice. The superhydrophobic coatings could also reduce the buildup of moisture on turbines, wherein less maintenance would be required. The superhydrophobic titanium surfaces possessed the ability to repel microbial attachment property and hence can be used to combat microbial fouling.

ACHIEVEMENT

Using a relatively simple, economic and novel method, superhydrophobic and superhydrophilic titanium surfaces were prepared. The superhydrophobic surfaces exhibited increased corrosion resistance and fouling resistance. The superhydrophilic surfaces showed increased biocompatibility. The study showed that the surface wettability properties of titanium can be manipulated to suit its respective applications.

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A Study of Diffusion Behaviour of Silicon in D9 Steel

EXECUTIVE SUMMARY

The effectiveness of silicon in extending the transient regime of void swelling is well known but the understanding of the underlying mechanism is still lacking. While experimental evidences of increased diffusion of Fe, Cr, Ni on silicon additions has been observed, through the annals of literature, no study on the diffusion of silicon in titanium modified steel is available. The study gives a glimpse of the complex diffusion behavior of silicon in steels which hitherto has remained a conjecture.

OUTLINE

The effect of fast diffusing substitutional elements in suppressing void swelling is well known. It has been shown in 316 steels that addition of silicon leads to a reduction in void number density. In thermal environments silicon is thought to migrate by vacancy exchange mechanisms. There is also a parallel view in the radiation effect community that solute migration takes place by the formation of bound interstitial complexes. Therefore it appears that the solute complex effects and the vacancy diffusion mechanisms are not necessarily mutually exclusive. A confirmation of silicon's influence on the overall diffusivity of solvent components of the alloy has been provided by various researchers. However, these groups have not measured the diffusivity of silicon itself due to the lack of a suitable radioisotope of silicon. The presented study aims at exploring the thermal diffusion behavior of silicon in D9 steels which has remained unresolved. There is considerable scope to extend this study to understand the radiation induced distribution of silicon in titanium modified steels.

D9 alloy samples with 0.25% titanium obtained from Valinox are implanted with 500 keV Si^{30} to a fluence of 3×10^{16} silicon atoms cm^{-2} . The depth profiling of Si^{30} was carried out by the Resonance Nuclear Reaction Analysis (RNRA) using $\text{Si}^{30}(\text{p}, \gamma)$ resonance at 942 keV. This involves implanting the sample with protons of 942 keV and above and observing the gamma rays in the energy range 7–9 MeV. The presence of silicon at a depth where the proton energy reaches 942 keV results in the production of gamma rays whose intensity is proportional to the concentration of silicon and the strength of the resonance. The nuclear resonance reaction profiling experiment was standardized by constructing a thick target gamma ray yield from a silicon wafer containing about 3% of Si^{30} . Through the annals of literature researchers have used the $\text{Si}^{30}(\text{p}, \gamma)$ resonance at 620 keV in applications involving depth profiling of silicon. But considering the small concentration of implanted silicon in the samples, we have chosen the 942 keV resonance. The 942 keV resonance with a width of 2.6 eV possesses a cross-section factor of five times more than the 620 keV, making it an ideal candidate for profiling silicon pertinent to our application. The range and straggling of the room temperature implant as determined by the RNRA is in agreement with that computed from the TRIM program. The D9 alloy samples were isochronally annealed for thirty minutes at temperatures of 320, 390, 460, 530, 600, 630 and 660 K. After every annealing step, the sample was quenched and RNRA was carried out to probe the diffusion of silicon atoms. The broadening of the profiles in the temperature range 320 – 660 K is shown in Figure 1. The diffusion constant is obtained from the profile broadening and the Arrhenius plot shows a linear behavior (Figure 2). An activation energy of 0.34 eV for silicon diffusion is obtained in this temperature regime.

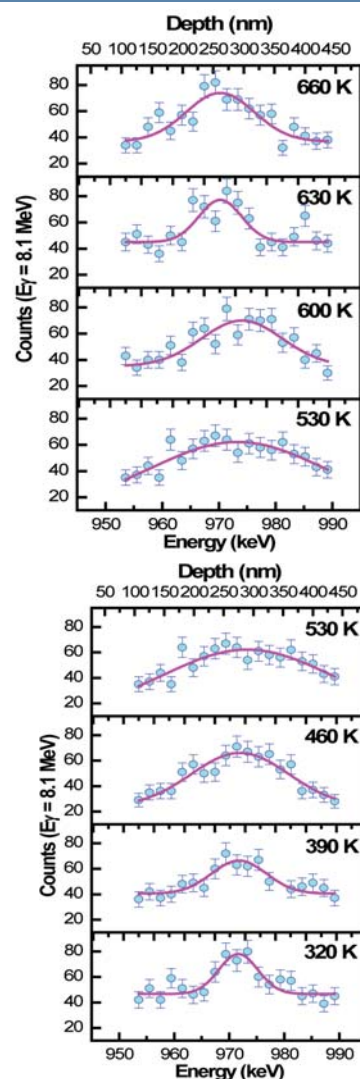


Fig. 1: Depth profiles of Si^{30} in D9 steel in the temperature range 320 – 660 K

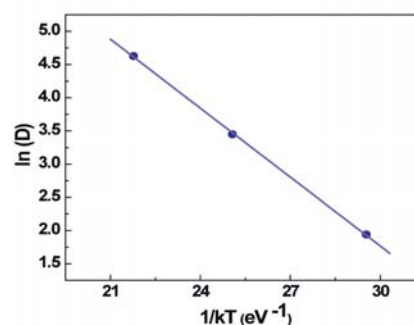


Fig. 2 : Arrhenius plot

ADDITIONAL INFORMATION

The interaction between point defects and solute atoms play a major role in the evolution of irradiation induced microstructure. These solute atoms additions may be deliberate or accidental additions during processing. Addition of certain minor alloying elements even with low concentrations has a direct impact on defect mobility. Hence a fundamental study of diffusion of the solute atoms would result in a better understanding of the mechanism and also would provide estimates of key parameters like activation energies for modeling behavior of the material under irradiation.

AN INSIGHT INTO THE MECHANISM

At temperatures of 600 K and above, an interesting behavior is observed as shown in Figure 1. It is found that there is a tendency for the FWHM of the diffusion profiles to shrink and approach that of the as-implanted profile. Also, as shown in Figure 3, there is a shift in the vertex of the diffusion profile towards the peak damage position. Our previous studies on the annealing behavior of D9 alloy using positron annihilation spectroscopy show that the vacancies start migrating at temperatures above 500 K. Therefore it is conjectured that at temperatures above 600 K silicon atoms are diffusing by a vacancy mechanism. The movement of silicon atoms in a direction opposite to the direction of the vacancy migration has resulted in the shrinking of the diffusion profile. Thus from the study, it is evident that silicon diffuses by both interstitial and vacancy assisted diffusion mechanisms at different temperature regimes. A modeling of silicon diffusion behavior in pure metals or ternary alloys would throw light on the underlying mechanism behind the complex diffusion behavior of silicon in different temperature regimes.

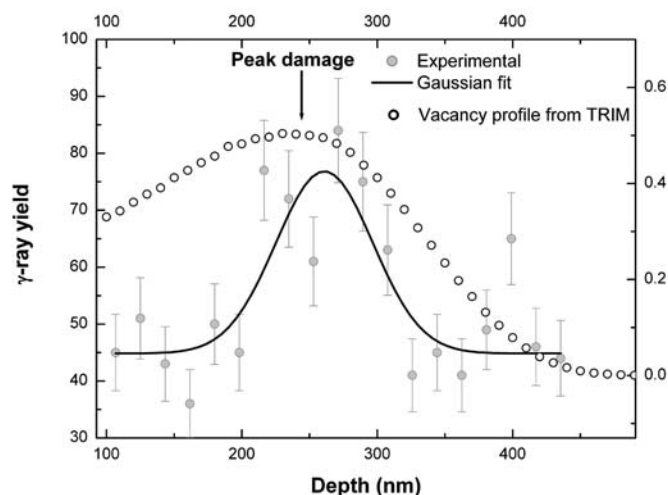


Fig. 3 : Illustration of the depth profile of Si^{30} at 630K. The TRIM profile of the vacancy damage is also shown. It is clearly seen that the vertex of the experimentally determined profile of Si^{30} in D9 alloy annealed at 630K matches with the peak of the vacancy profile

BRIEF DESCRIPTION

The diffusion of silicon in D9 steels was studied by isotopic tracing of Si^{30} using Resonance Nuclear Reaction Analysis technique. Silicon appears to diffuse by different mechanisms in different temperature regimes. A modeling of silicon diffusion behavior in pure metals or ternary alloys would throw light on the underlying mechanism behind the complex diffusion behavior of silicon.

ACHIEVEMENT

The addition of fast diffusing impurity like silicon in steel for suppressing void swelling is well known. Two parallel views exist regarding the diffusion of silicon in steels; by interstitial complex formation the other by inverse Kirkendall effects. The reported study is the first experimental proof to show that silicon diffuses by both interstitial and vacancy assisted diffusion mechanism in two different temperature regimes.

PUBLICATIONS

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Phase Transformations in Ti-5%Ta-1.8%Nb Alloy and Determination of α/β Solvus Lines

EXECUTIVE SUMMARY

The mechanism of phase transformations of the high temperature β phase in Ti-5%Ta-1.8%Nb alloy during cooling has been studied based on the room temperature microstructure that evolved during the transformation. The β phase undergoes a variety of transformations such as Widmanstatten, omega or martensitic depending on its stability as dictated by the content of Nb and Ta, which are β stabilizers. A part of α/β solvus lines of the pseudo binary Ti-Ta-Nb system has also been derived using microstructural methods.

OUTLINE

Ti-5%Ta-1.8%Nb, a $\alpha+\beta$ alloy, is a candidate material for structural applications in highly oxidizing medium, due to its excellent corrosion resistance. The high temperature β phase in this alloy undergoes a variety of phase transformations depending on the solute content and cooling rate. These transformations result in a myriad of microstructures, with characteristic morphology, microchemistry and structure. This has been systematically studied in the alloy subjected to isothermal treatments in β and $\alpha+\beta$ phase fields. It was observed that quenching from β phase field, resulted in a martensite product (α') with a h.c.p structure, which possessed a predominantly lath morphology. The martensitic transformation is attributed to the unstable high temperature β phase due to low amount of β stabilizing elements. However, isothermal treatments high in the $\alpha+\beta$ phase field, resulted in α' with plate morphology with internal twins. This change in morphology is attributed to the comparatively lower instability of β with increase in the solute content with decrease in temperature. Further increase in solute content of β phase with reduction in temperature resulted in a mixture of orthorhombic martensite (α'') and hexagonal ω phase as shown in Figure 1, due to a competition that prevailed between the two types of displacive transformations. Their unique morphology, structure and orientation relationship with the parent phase served as a fingerprint for identification of transformation mechanism. At temperatures lower than 773K, β phase is retained due to the large suppression of the start temperatures for the two types of displacive transformations resulting from high solute enrichment.

Apart from temperature, the decomposition mode of β phase was also sensitive to the cooling rate. A Widmanstatten transformation was always observed during slow cooling, while martensite formed at high cooling rates. The diffusional product consisted of a transformed β structure with alternate lamellae of α and β phase. However, $\beta \rightarrow \omega$ transformation was observed at intermediate cooling rate (air cooling) from $\alpha+\beta$ phase field due to the solute enrichment of β phase. The transformation modes of β phase and the resultant product microstructures have been consolidated into a phase transformation map in Figure 2.

Microchemistry of the coexisting α and β/α' phases at room temperature was experimentally determined and their composition was converted into Nb equivalent. A pseudo binary diagram of this ternary system has been constructed and is shown in Figure 3. Solubility of Ta and Nb in the α phase increased with decrease in temperature from β transus (~ 1043 K) until 1023K, beyond which a decrease was observed. This feature is also observed in the corresponding binary systems. Solubility of Ta and Nb in the α phase continuously increased with decrease in temperature, with a maximum solubility of 28 w% Nb or 35.3w% Ta equivalent at 773K. The volume fraction of α and β phases estimated from the phase diagram showed a good agreement with the values obtained by quantitative image analysis methods. Further studies in this system with alloys of different composition and thermodynamic assessment are in place to establish the phase diagram.

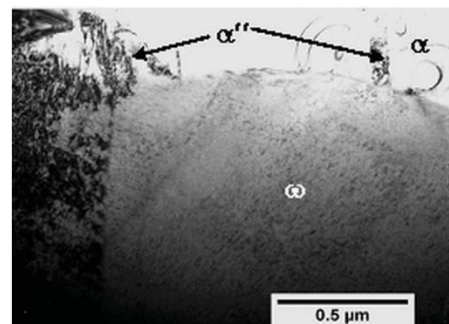


Fig.1: Bright field TEM micrograph showing the coexistence of α'' and ω (region of mottled contrast) phases on quenching the alloy from 1023K

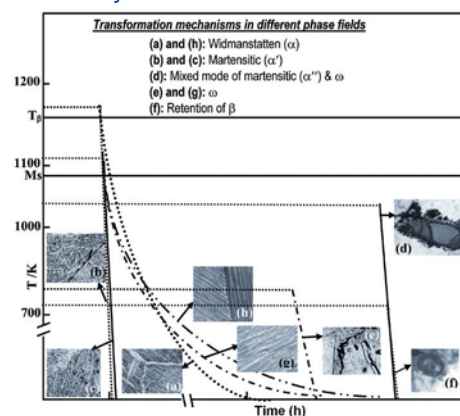


Fig.2 : Schematic phase transformation map depicting microstructural variations due to different transformation mechanisms in (a & b) β and (c to h) $\alpha+\beta$ phase fields

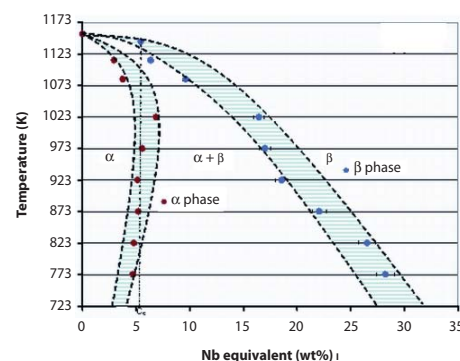


Fig. 3: α/β solvus lines of a pseudo binary diagram of the Ti-Ta-Nb system based on the Nb equivalent estimation from the composition of α and β phases

ADDITIONAL INFORMATION

CONDITION FOR STABILITY OF β PHASE

Since the alloy consists of both Ta and Nb as β stabilisers, the composition of β phase can be expressed in terms of electron/atom ratio to arrive at the criterion for stability of β phase. It is found that an e/a ratio below 4.1 favors a $\beta \rightarrow \alpha'$ transformation, between 4.1 and 4.2 favours the, $\beta \rightarrow \alpha''/\omega$, above which β phase is retained as is evident from Table 1.

Table 1 : Relation between e/a ratio and the stability of β phase

Temperature (K)	Phase	e/a ratio
1143	α'	4.04
1133	$\alpha + \alpha'$	7.5
1113	$\alpha + \alpha'$	4.04
1083	$\alpha + \alpha'$	4.07
1023	$\alpha + \alpha'' + \omega$	4.08
973	$\alpha + \alpha'' + \omega$	4.09
873	$\alpha + \alpha'' + \omega$	4.11
823	$\alpha + \alpha'' + \omega$	4.15
773	β	4.21

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The corrosion resistance and mechanical properties of titanium is well known to be greatly influenced by alloying additions and elements such as Ta and Nb are seen to be beneficial. However, further improvements in performance can be obtained in the Ti-Ta-Nb alloy by tailoring the microstructure, through appropriate treatments, which modify the morphology, amount and size distribution of the coexisting phases. This can only be achieved through the knowledge of the various phase transformation mechanisms and the conditions that lead to the desired microstructures. Information on the phase diagram of the technologically important Ti-Ta-Nb system is scarce in literature. Hence, an attempt has been made to determine the solvus lines based on the estimation of Nb equivalent of α and β phases using microstructural methods.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Various microstructures that control the corrosion and mechanical properties in this alloy can be tailored as an outcome of different phase transformation mechanisms that operate in this alloy system. The instability of the high temperature β phase, understood in terms of the e/a ratio or the solute enrichment of β results in different displacive or diffusional transformations during cooling at different rates

ACHIEVEMENT

Different phase transformation mechanisms of the high temperature β phase in Ti-5Ta-1.8Nb alloy responsible for the formation of variety of microstructures have been identified which is useful to optimize the processing conditions for this alloy. α/β solvus lines of a pseudo binary diagram of the Ti-Ta-Nb system has been arrived at based on microstructural methods.

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Prediction of Diffusion Profiles in Dissimilar Metallic Joints

EXECUTIVE SUMMARY

Mass transport across heterogeneous materials during service leads to undesirable microstructural and microchemical changes at their interface. Formation of a modified interface having different microstructure and property compared to the base material affects the performance of the components in actual service conditions. In this article, prediction of the kinetics of mass transfer, consequent formation of modified zones and validation of the predictions by experimental methods has been highlighted using two case studies namely formation of hard zone in dissimilar weldments of ferritic steels and long term carburization behavior of austenitic stainless steel exposed to flowing sodium.

OUTLINE

Mass transport across heterogeneous materials at their interface during service leads to undesirable microstructural and microchemical changes resulting in material degradation, which is studied in two important class of materials and is illustrated as case studies.

Case study 1: Exposure of dissimilar joints of 9Cr-1Mo and 2¼Cr-1Mo ferritic steels to high temperature leads to microstructural modification at the weld interface. The activity gradient driven carbon diffusion resulted in the formation of a carbon depleted 'soft zone' in the low Cr side and an adjacent carbon enriched 'hard zone' in the high Cr side, with different microstructure and property, which is deleterious to the design life of the weldments. The formation of such zones was predicted by computing the carbon diffusion profiles using finite difference method (FDM) based numerical technique, assuming single phase (without carbide precipitation), which resulted in an underestimation of the width of the zones. More accurate prediction of carbon profiles was carried out using Dictra and Thermocalc software, wherein the effects of simultaneous dissolution and precipitation of carbides in the two zones due to diffusion of carbon were considered. The predicted carbon diffusion profiles (Figure 1a), width of the zones (Figure 1b) as well as the volume fraction of precipitates matched well with experimental results. The model was also extended to determine the role of interlayers such as Ni, Cu and Co which act as effective diffusion barriers and prevent the zone formation. As a typical case, weld joints were fabricated with the predicted thickness of Ni based interlayer which showed the complete suppression of hard zone in the ferritic weldment.

Case study 2: The long term carburization/decarburization behavior of 316LN austenitic stainless steel (SS) on exposure to flowing sodium was studied in a simulated environment of the secondary circuit of PFBR, at 798K for 16000h in the bi-metallic sodium loop. Exposure of SS to sodium at high temperature leads to mass transfer and different forms of corrosion, resulting in carburization or decarburization, depending on the carbon activity difference between sodium and steel. Experimental evidence for the formation of a carburized layer upto 70µm beneath the ferrite layer (~5µm) was obtained from the surface of exposed SS. The carbon content in the carburized layer was determined using the carbon calibration chart generated using EPMA. The effective concentration dependant diffusion coefficients of carbon were also evaluated from the carbon profiles. The carbon diffusion profiles simulated using FDM showed reasonable agreement with experimental results (Figure 2). It was observed that width of the carburized layer increased with time until 10000 h, beyond which the change was small. The model was extended to predict the carbon concentration profiles upto a period of 40 years assuming that the carbon concentration had reached a dynamic equilibrium value of 0.5wt% at the surface and remained constant during further exposure to sodium.

Diffusion in multiphase systems have been evaluated more accurately overcoming the drawbacks/assumptions in the FDM simulation such as low dynamic equilibrium value for carbon concentration, constant stoichiometry of carbides, constant concentration of substitutional alloying elements with precipitation, by carrying out the computations using Dictra and Thermocalc software. Predicted width of the carburized zone, volume fraction of the carbides and carbon concentration matched very well with the experimental results.

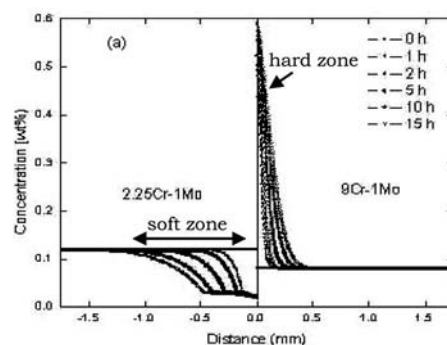


Fig.1a: Simulated carbon diffusion profiles for various durations at a particular temperature of 1023 K

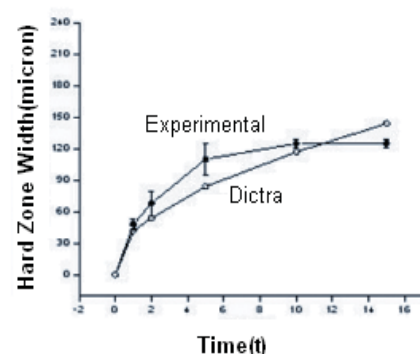


Fig.1b: Comparison of the width of the hardzone obtained using simulation and experiment

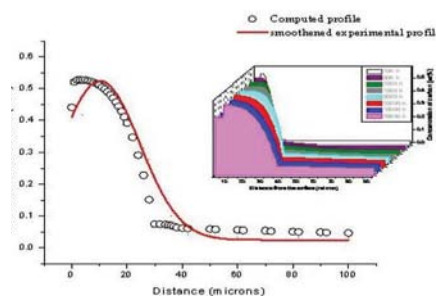


Fig. 2 : Comparison of the experimental and predicted carburization profiles. Inset shows predicted profiles upto exposure time of ~16000 h revealing increase in carburized zone width with time of sodium exposure

ADDITIONAL INFORMATION ABOUT THE ORIGIN OF THE ZONES

When interfaces of dissimilar materials in contact with each other are exposed to high temperature, carbon will diffuse from a region of high activity to a region of low activity, even if the carbon concentration is the same on both sides of the interface. This diffusion phenomenon is attributed to the strong interaction of carbon with other substitutional alloying elements for (e.g.) Cr. The following equation which relates the activity of carbon a_c to concentration of Cr, C_{Cr} and interaction parameter between Cr and C $\epsilon_C^{Cr}: a_c = C_C (\exp(C_C \epsilon_C^C + C_{Cr} \epsilon_C^{Cr}))$ shows that since chromium has attractive interaction with carbon ($\epsilon_C^{Cr} = -72$), an increase in Cr content means a decrease in carbon activity. This activity gradient between the high and low chromium sides acts as the driving force for the diffusion of carbon in both the case studies presented here.

GENERAL EXPLANATION FOR THE ZONE FORMATION

An activity gradient across the heterogeneous material interface results in the diffusion of carbon across the interface. When carbon diffuses to a system where its solubility is less, it precipitates in the form of carbides. In case study 1, diffusion of carbon is from low Cr to high Cr steel. In case study 2, it is from sodium to austenitic stainless steel. In both the structural materials, since Cr is the main substitutional alloying element carbon reacts with it to form Cr rich $M_{23}C_6$ type of carbides. As the temperature or time of high temperature exposure is increased, diffusion of carbon and precipitation of carbides proceeds leading to an increase in the width of the carburized zones. Due to the various constraints on the diffusion of carbon like (1) decrease in the activity difference between the two systems (2) depletion of Cr on the diffusing side and (3) low C level in parent matrix, the width of the zones do not show an increase beyond a specific duration of exposure at a particular temperature.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Using numerical techniques based on Finite difference method, DICTRA and ThermoCalc, the carbon diffusion profiles have been predicted for different experimental conditions in two classes of steels, which are very useful to predict the long term behavior of the components in the service environment.

ACHIEVEMENT

This work demonstrates that numerical techniques combined with limited validating experiments can be used to predict the microstructural modifications across heterogeneous material interfaces which eliminate the necessity to do long term laboratory experiments.

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Mechanical Property Evaluation and Microstructural Characterisation of Highly Irradiated SS316

EXECUTIVE SUMMARY

Type 316 Austenitic Stainless Steel (ASS 316) in 20% cold worked condition is used as the structural material for fuel cladding and duct in Fast Breeder Test Reactor (FBTR). Fast neutron irradiation in these structural materials results in dimensional changes and degradation of mechanical properties which is closely linked to the microstructural changes. The irradiation performance of ASS316 cladding and duct of FBTR has been systematically studied by evaluating the mechanical properties, void swelling and microstructural changes at various displacement damages up to a maximum of 83 dpa (displacement per atom).

OUTLINE

The core materials of fast reactors are subjected to high neutron fluence (10^{22} n/cm²/sec) coupled with high temperatures (400-600°C). Under such irradiation conditions and high temperatures, austenitic stainless steels undergo significant microstructural alterations which induce dimensional changes and alter mechanical properties. These irradiation induced changes are a strong function of irradiation temperature and the neutron fluence or displacement per atom.

The void swelling of the cladding and duct are evaluated from immersion density measurements. The trends of swelling ($\Delta V/V$) with displacement per atom, shown in Figure 1, indicate a faster rate of increase above sixty displacement per atom. Also the extent of swelling in cladding (~11.5%) is higher as compared to duct (~4%) due to the high operating temperatures of the cladding close to the peak swelling temperatures of SS316. These trends are consistent with dimensional increase in the duct and cladding.

The mechanical properties of irradiated cladding are determined by remote tensile tests in the hot cell facility using universal tensile test machine fitted with a resistance heating furnace. These tests have been carried out at various temperatures corresponding to reactor operating conditions (430-490°C); fuel handling conditions (~180°C) and ambient temperatures. Both strength and ductility of the cladding showed a significant decrease at displacement damages > 60 displacement per atom in both high temperature and room temperature tests. The ductilities (uniform elongation) of the cladding were in the range of 2-3 %. The trends in the ultimate tensile strength (UTS) of the cladding as a function of dpa and irradiation temperature are shown in Figure 2.

TEM studies of hexagonal duct showed extensive void formation beyond forty displacement per atom in addition to precipitation and dislocation loops. The void density showed a progressive increase with displacement damage. The precipitates were identified to be mainly of nickel and silicon enriched M₆C type of η phase, whereas radiation induced G phase was also observed at eighty three displacement per atom. The precipitates were associated with the voids which can be understood considering the growth of precipitates being achieved by diffusion of solutes, which in turn brings a vacancy surplus along with it. The retention of cold worked structure in the duct was unambiguously seen after eighty three displacement per atom irradiation which suggests that no recrystallisation has taken place and irradiation hardening is more prominent than the softening effects. The TEM micrographs of SS316 at various displacement per atom superimposed on the swelling curve is shown in Figure 3. The increase in defect density as observed from the dislocation substructure and increase in the void density and size are the sources of irradiation hardening behaviour of duct.

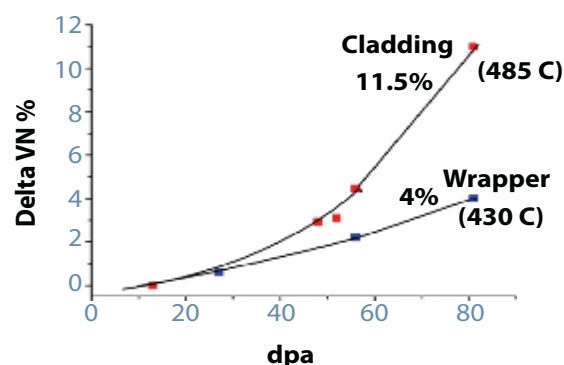


Fig.1: Volumetric Swelling of SS316 cladding and duct with dpa

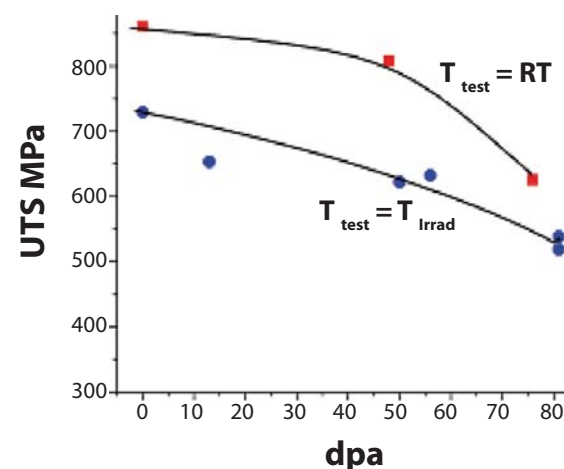


Fig.2: Trends in the variation of UTS in irradiated FBTR cladding as a function of dpa

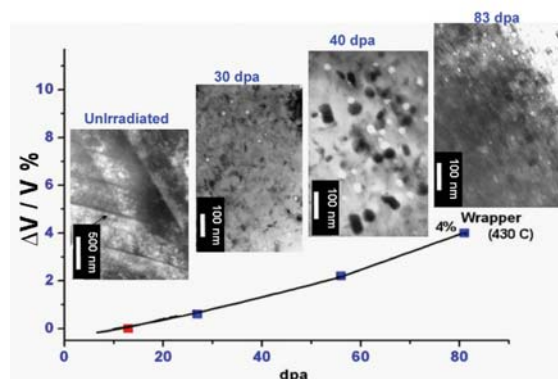


Fig.3: TEM micrographs of SS316 duct at various dpa superimposed on the swelling curve

ADDITIONAL INFORMATION

The choice of the core structural materials for fast reactors has been mostly guided by their resistance to irradiation induced swelling, the related dimensional changes and deterioration of their mechanical properties. The first generation materials belonged to austenitic steels of type AISI 304 and 316 SS. As these steels quickly reached their limits because of unacceptable void swelling, many improvements were made by modifications of the metallurgical structure by suitable thermo-mechanical treatments, addition of stabilising elements, changes in the chemical composition of major and minor elements etc. This led to the development of advanced core structural materials such as D9 and D9I for use up to 100-120 displacement per atom levels. Another class of steels namely the ferritic-martensitic steel and oxide dispersion strengthened (ODS) steels are being considered for exposures beyond one hundred and twenty displacement per atom in FBR core because of their very low rates of void swelling.

GENERAL EXPLANATION RELATED TO DESCRIPTION

The high fast neutron fluence coupled with high temperatures in core materials of fast reactors lead to very high level of atomic displacements and significant microstructural alterations. Defect structures like dislocation loops, vacancy clusters, vacancy loops, voids, helium bubbles etc form as a function of temperature of operation and neutron fluence (quantified by displacement per atom-dpa). Four major phenomena that influence the performance of steels as core components include phase stability, void swelling, irradiation creep and changes in mechanical properties. Voids form in austenitic stainless steels in the temperature range 400-550°C under neutron irradiation resulting from the agglomeration of vacancies. Irradiation creep is caused due to the combined effects of external non-hydrostatic stresses and presence of both interstitial atoms and vacancies at very large supersaturation levels. Fast neutron irradiation strongly affects the mechanical properties of austenitic stainless steel alloys especially the ductility. There are three temperature regimes of importance to embrittlement: A low temperature regime where void swelling is not significant, an intermediate temperature regime where swelling is predominant and a high temperature regime where the mobility of helium is sufficient to cause intergranular failures. Measures to improve swelling resistance of austenitic stainless steels also enhance resistance to embrittlement.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Microstructural alterations are the result of atomic displacements due to collision of energetic neutrons with atomic nuclei. Two basic defects produced are vacancies and interstitials, with typical displacement rates of the order of 10^{-6} dpa/s. The continuous production of point defects during neutron irradiation gives rise to radiation-induced segregation effects. Several microstructural features like grain boundaries, dislocations, twin boundaries, defects and precipitate – matrix interfaces act as sinks for point defects. They act as potential sites for segregation of solutes enhancing the local concentration of solutes. The formation of precipitates removes elements like nickel and silicon which are important for suppressing void swelling and onset of void swelling often coincides with the formation of nickel and silicon rich phases. The correlation between high swelling and ductility loss could be attributed to either the direct effect of voidage in promoting brittle fracture or to the indirect effect of radiation induced segregation due to presence of voids. Radiation induced segregation could lead to instability of austenitic matrix due to nickel segregation at void surfaces facilitating transformation to martensite which would induce embrittlement.

ACHIEVEMENTS

The comprehensive understanding of the irradiation behaviour of 20% cold worked SS316 at high displacement damages up to 83 dpa has facilitated in predicting the residual life of FBTR fuel and structural materials and has ensured safe operation in the reactor, during fuel handling, PIE and subsequent operations. These studies carried out as a part of post-irradiation examination has enabled achieving a burnup of 165 GWd/t in FBTR using plutonium rich carbide as the driver fuel without any failure.

PUBLICATIONS

1. C.N.Venkiteswaran, V.Karthik, P.Parameswaran, N.G.Muralidharan, V.Ananda Raj, S.Saroja, V.Venugopal, M.Vijayalakshmi, K.V.Kasi Viswanathan and Baldev Raj, "Study of Microstructure and Property Changes in Irradiated SS316 Wrapper of Fast Breeder Test Reactor", Journal of ASTM International, 6 (2009) 1.
2. V.Karthik, A. Vijayaraghavan, Shaji Kurien, N.G. Muralidharan, V.Venugopal, Jojo Joseph and K.V. Kasiviswanathan, "High Temperature Remote Tensile Testing of Irradiated Clad tubes of FBTR", Proceedings of Recent Advances in Post Irradiation Examination – 2008.

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Pre-analysis of Phenix End-of-life Neutronics Tests with ERANOS 2.1 and FARCOB

EXECUTIVE SUMMARY

The end-of-life (EOL) experiments in the Phenix reactor are being carried out to demonstrate the safety of fast reactors and also to validate the design codes and data in the field of neutronics and thermal hydraulics. After these experiments, this reactor will be permanently shutdown by the end of this year. Some of these tests are open for international collaboration and are included as a collaborative research project (CRP) under IAEA. Based on CEA-IGCAR collaboration on fast reactor safety, IGCAR has participated in the pre-analysis calculations of EOL neutronics tests with CEA, Cadarache, which were used for finalizing the various test procedures. The code systems used were ERANOS 2.1 (European) and FARCOB (IGCAR). This study enabled us an inter-comparison of IGCAR computer codes used for PFBR design with ERANOS system, which were used for the EFR design. A close agreement is seen for all the neutronics parameters for the Phenix reactor.

OUTLINE

Under CEA-IGCAR collaboration on fast reactor safety, IGCAR has participated in the pre-analysis of various neutronics tests. This collaboration started with the simulation of various neutronics tests using the European state-of-the-art neutronics code system called ERANOS 2.1 with the help of CEA, France. The next phase was the re-analysis of these calculations with IGCAR neutronics code system. The code system used at IGCAR is ABBN93-CONSYST-EFCONSY-COHINT-FARCOB. This exercise was very useful for the CEA team to finalize the various test procedures. The neutronics tests analyzed for the EOL core (Figure 1) were: (i) Worth of fuel and fertile sub-assemblies, (ii) Absorber rod worth, and (iii) Control rod withdrawal test.

The sub-assemblies for the worth measurements are loaded at SAC position. Four types of sub-assemblies are studied, viz. fresh and burnt assemblies of fuel and blanket assemblies. The burnup of SA corresponds to 750 efpd. To study the SCP2 worth change due to fuel burnup, the worth of SCP2 is estimated when fresh and burnt fuel assemblies are loaded around it, by keeping all other 5 SCPs outside the core. For the control rod withdrawal test, the absorber rod SCP2 is withdrawn completely from the critical position and the change in power due to this effect is estimated. Neutronics calculations were done with 3-D diffusion theory. The nuclear data library used in IGCAR code system is the well validated Russian adjusted set called ABBN-93. In ERANOS, the adjusted cross section library ERALIB1 was used. The heterogeneity corrections were made to fuel and absorber rod cross-sections.

The total worth of all rods (6 SCP + SAC) from FARCOB is predicted within 2 % with respect to ERANOS values. It predicts the worth of 6 SCPs within 0.5 %. It is seen that ERANOS predicts 25 % change of absorber rod worth with fuel burnup, whereas it is about 32% with FARCOB. The maximum over power in the neighboring sub-assembly due to SCP2 movement is about 8 % with FARCOB (Figure 2), whereas it is about 10 % with ERANOS. There is a close agreement in the prediction of sub-assembly power (Figure 3) and absorber rod worth between FARCOB and ERANOS. The deviation of maximum sub-assembly peak power is about 1.3 % between these code systems. In general, all the neutronics parameters estimated were closely predicted by FARCOB and ERANOS systems.

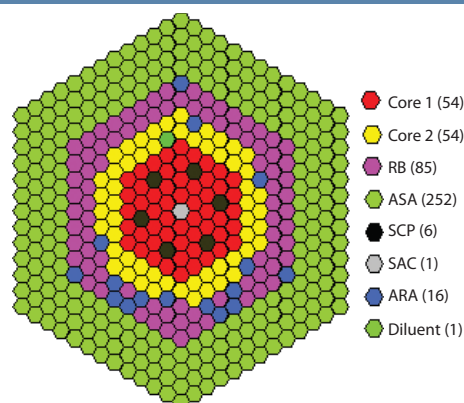


Fig.1: Phenix Core for EOL Tests

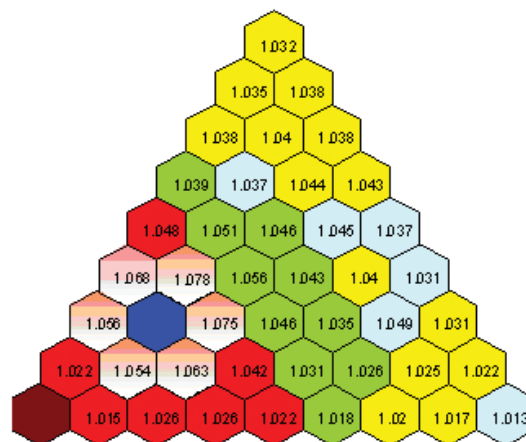


Fig.2: Deviation of SA Power Due to SCP2 (shown in blue colour) Movement from Critical Position with FARCOB

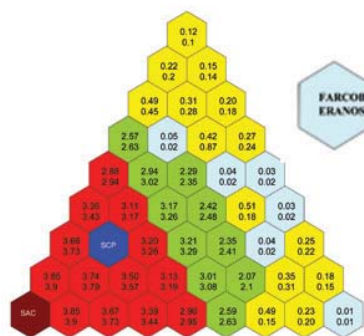


Fig.3: Power Distribution in MWt with ERANOS and FARCOB

ADDITIONAL INFORMATION

The European Reactor Analysis Optimised calculation System, version 2.1, ERANOS 2.1, consists of data libraries, codes and calculation procedures, developed within the European collaboration on fast reactors for more than 20 years. It contains all the functions required for the reference and the design calculations of Liquid Metal Fast Reactor cores (as well as blankets, reflectors and shields), with extended capabilities for Generation-IV type advanced fast reactors, Accelerator Driven Systems (ADS) and Gas Cooled Fast Reactors (GCFR). ERANOS is written with ALOS software, which uses the standard FORTRAN compilers and advanced programming features. It contains the nuclear data libraries in different energy groups (1968 groups for main nuclides, 33 group for fast spectrum applications, 175 groups for shield design and 172 groups for thermal reactor applications) based on JEF-2.2, JEFF-3.1 and ENDF/B-VI (rel.8). It also contains an adjusted library based on JEF-2.2 called ERALIB1. The cell code ECCO is used for preparing the self-shielded cross sections. There are various modules based on diffusion/transport theory to perform 3-D calculations. The S_N transport code BISTRO and its associated perturbation modules are used for preparing the homogenised control rod cross-sections by using the reactivity equivalence method.

GENERAL DESCRIPTION:

The Phenix reactor has demonstrated its design objective of safe and reliable operation of sodium cooled fast reactors. It has been operating successfully since 1973 and its final shutdown was planned by the end of 2009. Before its permanent shutdown, several physics and engineering experiments were planned with the purpose of validating various data and design codes. These End Of Life (EOL) tests are being carried out with international collaboration and IGCAR is also a participant.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Neutronics calculations are performed in 3-D geometry with diffusion theory. The control rod and fuel cross sections are corrected for heterogeneity effects. In IGCAR neutronics code system, the code COHINT is used for preparing heterogeneous cross sections, whereas a procedure based on the reactivity equivalence method is used in ERANOS code system.

ACHIEVEMENTS

The pre-analysis of Phenix end-of-life experiments is a international benchmark on inter-comparison of computer codes used for PFBR design in IGCAR and computer codes used for EFR design in European Union. There is a close agreement between the two sets of predictions.

PUBLICATIONS

1. K. Devan, Laurent Buiron, Jean Tommasi, Pierre Sciora and Frederic Varaine, Analysis of a 3600 MW(t) Mixed Carbide FBR with ERANOS 2.1 and IGCAR Code System, CEA-IGCAR Report (2008).
2. K. Devan, M. Alagan and P. Mohanakrishnan, Pre-Analysis of Phenix End of Life Neutronics Tests with ERANOS2.1 and FARCOB, Report REG/RPD/CPS/81 (2008).

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IGSHIELD: A New Interactive Point Kernel γ -Ray Shielding Code

EXECUTIVE SUMMARY

IGSHIELD is an Interactive Gamma-ray Shielding code (stand-alone) developed in Visual Basic Version 6.0 for windows operating system. The computational methodology is based on the point kernel technique. The new features of the code are: i) handling of multiple source shapes with accompanying energy distributions, ii) incorporation of hexagonal cylinder as a shield body in the combinatorial geometry routine, iii) provision to declare hexagonal cylinder and truncated right circular cone as source bodies, iv) provision to declare point, line and plane as source bodies and v) arbitrary orientation of sources such as cylinder, hexagonal cylinder and truncated cone. The interactive gamma shielding code will cater the needs of a shield designer as well as serve as an excellent educational tool.

OUTLINE

In a point kernel technique, large volume sources are divided into arbitrary predefined smaller volumes, which are considered as point sources with appropriate source weight. The centroid coordinates of such small sources are calculated and these positions are considered, as their locations. From each such source point, the optical path traversed in the source and shield media upto the detector location is estimated to calculate geometrical and material attenuations to get uncollided dose rate. This is subsequently multiplied with the appropriate dose buildup factor (to account for scattered contribution) to get total dose rate. Finally, the dose rate for entire volume source is obtained by summing over all the sampled points. The layout of the main page is shown in Figure 1.

New Features of IGSHIELD code

IGSHIELD is capable of handling multiple source shapes with accompanying gamma energy distributions (Figure 2). For example, a single input file is enough to calculate dose rates for a typical source shield configuration, where there are two volume sources like a cylindrical source with ^{60}Co activity and a slab source tank containing ^{137}Cs activity. The user can also define multiple identical sources using array facility. In a single run, the dose rate at the desired detector locations due to each source is computed and displayed.

The voxel calculational methodology for new source bodies such as hexagonal cylinder and truncated right circular cone has been incorporated. In shielding calculations, sometimes, the volume sources such as a pipeline carrying a radioactive solution may not be parallel to any of the axis and can be inclined depending upon engineering considerations. Since the IGSHIELD is capable of handling multiple source shapes, this feature is also incorporated. The bodies have to be declared as per the input requirements for the geometry. The source points calculated for a cylindrical source parallel to any of the axis is rotated as per the data specified for the body. For the rotation of the sampling points, the standard 3D rotation methodology is adopted.

The input module accepts only the correct data. If the data happens to be an object, the code instantaneously displays the 3D view of all the objects built so far. Thus, the interactive gamma shielding code caters to the needs of a shield designer and also serves as an excellent educational tool.

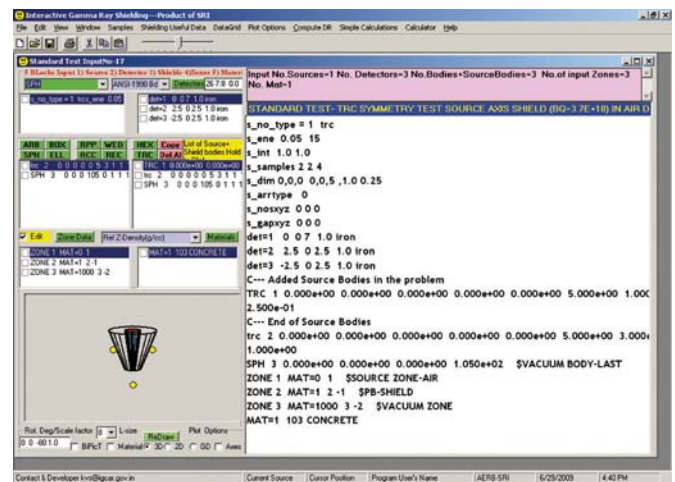


Fig. 1 : Layout of IGSHIELD form

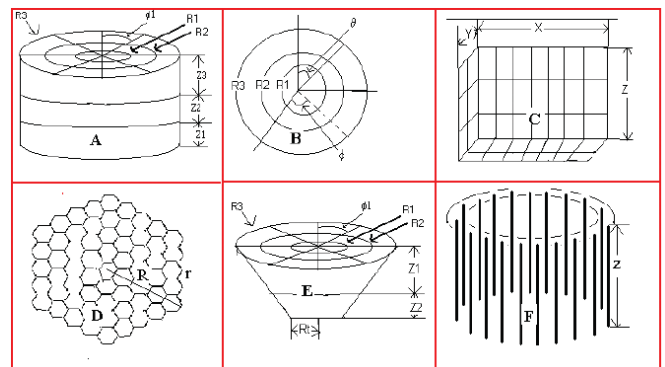


Fig. 2: Source options of IGSHIELD with geometry parameters (A) Cylindrical source, divisions along the R, Z, Φ coordinates (B) Spherical source, divisions along the R, n, Φ coordinates (C) Slab source, divisions along the x, y, z coordinates (D) Hexagonal source, divided into number of hexagonal voxels with side r and height z (E) Truncated right circular cone, divisions along R, Z, Φ coordinates (F) Array of line sources

ADDITIONAL INFORMATION

The information required for shield designers are included in graphical form along with the main code. Some of the useful data included are 1) tenth layer plots for gammas and neutrons, 2) fission product gamma ray intensity, 3) gamma ray and neutron dose conversion factors, 4) gamma and neutron sources, 5) thermal neutron activation calculation, 6) buildup factor curves, 7) derived air concentration for important radio nuclides, 8) gamma ray cross-section for some of the elements 9) gamma energy search, 10) ozone buildup in air and 11) electron dose rates.

GENERAL INFORMATION:

The code is validated by comparing the results for the standard shielding problems with analytical equations and MCNP code. Several sample problems encountered in nuclear industry and other useful information required for shield designers and analysts are the other highlights of the code. The input for any kind of situations can be prepared easily using the forms that flash with appropriate prompts. Since entry of data is the important part of the input, its validity is checked immediately and the user is cautioned against incorrect data. The sample results of the code are given in Table 1.

Limitations of the code

The code can be applied to only those situations where the basic point kernel technique is applicable. The user has to select a medium for the buildup factor calculation based on the source shield configuration and knowledge regarding the selection of medium is essential. The code is capable of handling upto 1000 zones constructed using basic bodies and 100 gamma energies in a single input. The accuracy of the code is governed mainly by the contribution of scattered dose component calculated using infinite medium buildup factor.

Table 1: Calculation of uncollided flux at detector location by analytical equation and IGSFIELD for volume sources with slab shields (Distance, radius and thickness in cm, flux in $\gamma/\text{cm}^2/\text{s}$, Gamma Energy in MeV)

1 Cylindrical with side shield

Strength (Sv): $3.7\text{E}+10 \gamma/\text{cm}^3/\text{s}$ Source material: Iron
 Radius (R): 2, Height (H): 10
 Distance from source axis to p (b): 10
 Thickness of lead shield (t): 5
 Uncollided flux at p

Energy (MeV)	Analytical	IGSHIELD	MCNP
0.5	1.92E+5	1.93E+5	1.25E+5
1.5	9.75E+7	9.73 E+7	9.78E+7

2 Truncated right circular cone with side shield

Strength (Sv): $3.7\text{E}+10 \gamma/\text{cm}^3/\text{s}$
 Base radius (R1): 10
 Height of source region (h): 5
 Source material: Iron
 Thickness of lead shield (t): 5
 Distance from shield H1: 15

Energy (MeV)	Analytical	IGSHIELD	MCNP
0.5	2.72E+5	2.72E+5	1.83E+5
1.5	1.49E+8	1.49E+8	1.53E+9

3 Hexagonal cylindrical source with slab shield

Strength (Sv): $3.7\text{E}+10 \gamma/\text{cm}^3/\text{s}$
 Source material: Iron
 Radius (R): 2
 Height (H): 10
 Distance from source axis to p (b): 10
 Uncollided flux at p

Energy (MeV)	Analytical	IGSHIELD	MCNP
0.5	Not Available	1.70E+5	1.15E+5
1.5	Not Available	8.41E+7	8.60E+7

PUBLICATIONS

K.V. Subbaiah and R. Sarangapani, "IGSHIELD: A New Interactive Point Kernel Gamma Ray Shielding Code", Annals of Nuclear Energy, 35 (2008) 2234.

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Pressure Induced Superconductivity in BaFe₂As₂ Single Crystal

EXECUTIVE SUMMARY

High pressure techniques have been employed to induce superconductivity in single and polycrystalline samples of BaFe₂As₂ - a recently discovered system which otherwise possesses a non-superconducting metallic spin density wave (SDW) ground state. The temperature-dependent electrical resistivity studies in 0–7 GPa pressure range bring about for the first time, a clear pressure-induced superconductivity with zero resistivity in the single-crystal sample. This happens at the expense SDW transition resulting in the highest value of the superconducting transition temperature (T_c) of 35.4 K reported so far. It is surmised that the pressure induced superconductivity in the BaFe₂As₂ system may be linked to a quantum critical point associated with the suppression of the SDW.

OUTLINE

The recent discovery of superconductivity in the iron pnictides has evoked immense response from scientific community which has culminated in synthesis of several new superconductors. One distinguishing feature of these pnictide superconductors is the existence of spin density wave in the parent compounds, which gives way to superconductivity with electron or hole doping. Apart from the ample future scope of enhancing the T_c , as indicated in several doping studies, the occurrence of superconductivity with magnetic Fe in these layered compounds has added further intrigue to the investigation of the role of magnetism in the occurrence of high temperature superconductivity, as in the cuprate superconductors. In the present study, high pressure technique has been successfully employed to induce superconductivity in BaFe₂As₂ system.

Single crystals of BaFe₂As₂ with an average size of 2 mm x 1.5 mm x 0.3 mm have been grown by employing self-flux growth procedure in which excess FeAs is used as a flux. Formation of single crystal was affirmed by recording and analyzing the Laue patterns. The polycrystalline sample was synthesized by adopting the well established procedure in the literature.

In Figure 1 temperature and pressure dependent electrical resistivity $\rho(T, P)$ of a single crystal of BaFe₂As₂ in the 4–300 K temperature and 0–7.2 GPa pressure ranges have been presented. The $\rho(T, P)$ curves reveal a clear superconducting transition with vanishing resistivity at ~ 1.4 GPa pressure with T_c of 35.4 K. It is to be remarked that in previous studies the pressure-induced superconductivity was seen above the critical pressure of 2.5 GPa and the maximum T_c obtained was ~ 29 K in the BaFe₂As₂ system. On the other hand, in the present study we find the largest pressure-induced T_c (35.4 K) at ~ 1.5 GPa. The SDW transition temperature $T < T_{SDW}$ (marked by downward arrows in Figure 1) decreases rapidly with increasing pressure from 135 K at 0.8 GPa to 105 K at 1.4 GPa, which is ultimately not discernible beyond this pressure.

Based on the two equations described in theoretical considerations section, the fits to experimental data in the normal state are indicated in the ρ -vs.- $T^{3/2}$ plot of (Figure 2a). It is seen that the experimental data for pressure $P \leq 1.4$ GPa fit to eq. (1) for $m = 1.5 \pm 0.07$ only in the temperature range above $T < T_{SDW}$. However, $\rho(T, P)$ fits this expression in entire temperature range (40–300 K) above T_c for $P \geq 2$ GPa. From the $T^{3/2}$ -dependence of resistivity, it is inferred that the spin fluctuation dominates the normal-state resistivity. Parameter B, that is indicative of the strength of spin fluctuation, increases systematically with pressure. In $T < T_{SDW}$ regime, at $P = 0.8$ GPa the parameter D of eq. (2) becomes nearly zero and $\rho(T, P)$ is found to exhibit nearly T^2 -dependence indicating a Fermi-liquid (FL) behavior (Figure 2(c)). As the pressure further increases to 1.4 GPa, $\rho(T, P)$ follows a $T^{1.7}$ -dependence indicating the onset of a non-Fermi-liquid (NFL) behavior (Figure 2(d)). When SDW vanishes beyond 2 GPa, $\rho(T, P)$ is seen to follow a $T^{3/2}$ dependence indicating the NFL behavior in all the temperature ranges above T_c as seen in figure 2(a). Such a peculiar evolution of $\rho(T, P)$ may indicate the quantum criticality as observed in several Ce-based heavy-fermion systems.

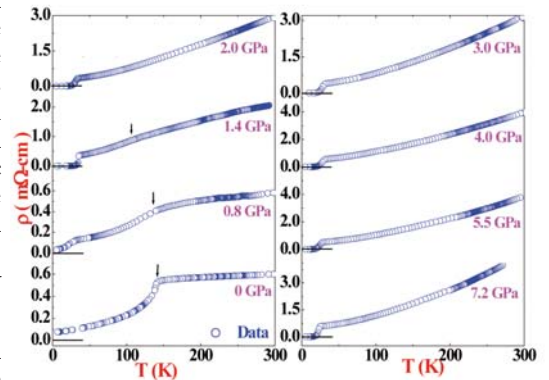


Fig. 1: Temperature-dependent resistivity curves of the single-crystal BaFe₂As₂ samples under pressures indicated in each panel. The zero of each panel is indicated by horizontal thick line. Downward arrows indicate the positions of $T < T_{SDW}$ at various pressures

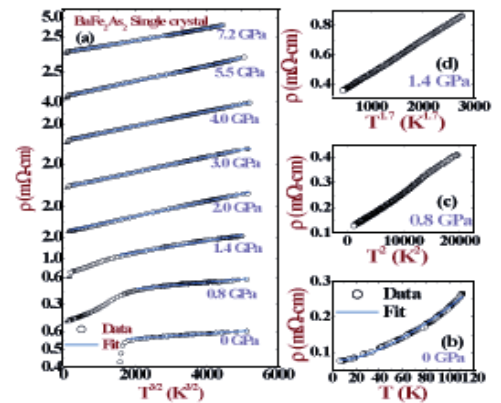


Fig. 2: (a): $\rho(T, P)$ curves of the single-crystal BaFe₂As₂ showing fits to eq. (1) for temperature $T > T_{SDW}$. (b): $\rho(T, P)$ curve at ambient pressure showing fit to eq. (2) for $T < T_{SDW}$. (c) and (d): normal-state $\rho(T, P)$ curves for $T < T_{SDW}$ showing the T^2 - and $T^{1.7}$ -dependence at 0.8 and 1.4 GPa

GENERAL INFORMATION: THEORETICAL CONSIDERATIONS TOWARDS UNDERSTANDING OF THE PRESENT RESULT

In order to gain physical insight on the resistivity behavior, the pressure dependence of the normal-state resistivity of BaFe_2As_2 above and below $T < T_{\text{SDW}}$ have been analyzed. In the case of $T > T_{\text{SDW}}$, Eq. 1 and for $T < T_{\text{SDW}}$ Eq. 2 given below have been used to analyse the $\rho(T, P)$ data:

$$\rho(T) = A + BT^m \text{ ----- (1)}$$

$$\rho = A + CT^2 + DT(1 + 2T/\Delta)\exp(-\Delta/T) \text{ ---(2)}$$

In Eq. [1] & [2], the constant A represents the contribution to the resistivity from electron - impurity scattering, while the term with B accounts for the anti-ferromagnetic spin fluctuation for $m=1.5$, the T^2 term accounts for the Fermi-liquid contribution and the term containing D represents the contribution from antiferromagnetic energy gapped (Δ) spin waves.

The variation of $T < T_{\text{SDW}}$, T_c and the fit parameter B of the single-crystal sample as a function of pressure is shown in Figure 3. $T < T_{\text{SDW}}$ is seen to decrease drastically and vanish beyond $P > 1.4 \text{ GPa}$. On the other hand, T_c shows a non-monotonic variation with pressure, i.e., T_c increases abruptly to 32.8 K at 0.8 GPa , reaches to a maximum of 35.4 K at 1.4 GPa and then decreases monotonically with further increase in pressure. It is worth mentioning that maximum of T_c lies at the border of $T < T_{\text{SDW}}$ instability (cf. Figure 3(a) and (b)). This suggests that magnetic instability may be playing a crucial role in the occurrence of superconductivity. It is noted that while B increases with increasing pressure, T_c decreases beyond 1.4 GPa . This non-monotonic variation in T_c may be due to competition between increase in spin fluctuation (as suggested by parameter B) and a decrease in DOS at the Fermi energy, E_F , with pressure. Recent band structure calculations have shown the gradual reduction of DOS at E_F under pressure up to 10% volume reduction in this system, which is consistent with our above assertion.

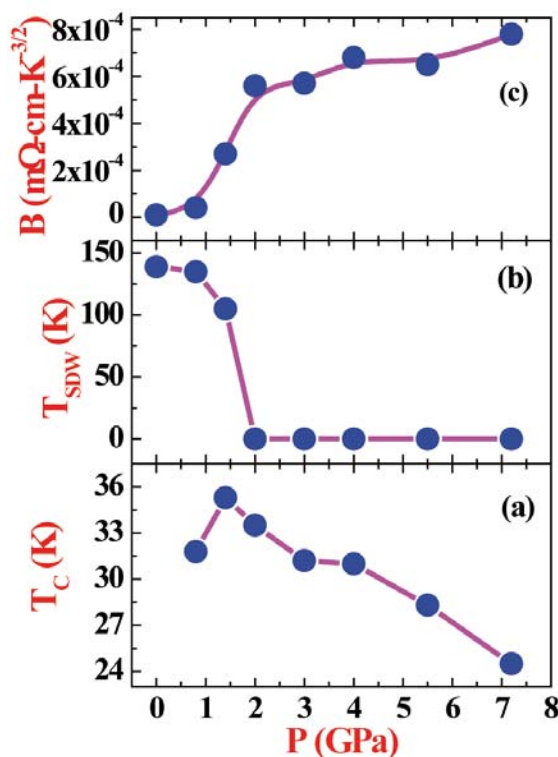


Fig. 3: Variation of T_c , T_{SDW} and fit parameter B, respectively, as a function of pressure for the single-crystal sample

BRIEF DESCRIPTION

To conclude, pressure induced superconductivity is observed in poly and single crystalline samples around $\sim 1.5 \text{ GPa}$ pressure. While superconducting transition remains incomplete in the former, full fledged superconductivity with zero resistivity is observed in the latter sample. The superconducting transition temperature (T_c) exhibits an initial increase followed by decrease at higher pressures, while $T < T_{\text{SDW}}$ decrease rapidly and vanishes beyond 2 GPa pressure. Interestingly, superconductivity and magnetism coexist at moderate pressure of 1.4 GPa . The dominance of $T^{3/2}$ term in $\rho(T, P)$ data indicates the link between spin fluctuation and superconductivity in BaFe_2As_2 . The non-monotonic variation of T_c with pressure is suggestive of the competing effect of the increase of coupling strength and the decrease in DOS at E_F .

ACHIEVEMENTS

The present study is the first report of pressure induced full-fledged resistive superconducting phase transition in single crystalline BaFe_2As_2 with zero resistivity. The maximum value of superconducting transition temperature T_c of 35.4 K obtained at 1.5 GPa pressure is the highest T_c reported so far in this system.

PUBLICATIONS

Awadhesh Mani, Nilotpal Ghosh, S. Paulraj, A. Bharathi and C. S. Sundar, "Pressure-induced superconductivity in BaFe_2As_2 single crystal", Europhys. Lett. 87 (2009) 17001.

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Discovery of Superconductivity in Ruthenium doped BaFe₂As₂

EXECUTIVE SUMMARY

Fe-As superconductors have caught the attention of condensed matter physicists during the last year, reminiscent of the fervour prevalent about two decades ago, aroused by the discovery of the cuprate superconductors. A la the cuprates, several families of FeAs based superconductors have been discovered but highest T_c has been so far been restricted to 55K. The search of newer compounds in the hope of enhancing T_c is occurring at a frenzied pace. In our laboratory we have found superconductivity at 20.8 K for the first time by Ru substitution at the Fe site in the BaFe₂As₂ system. A systematic suppression of the Spin Density Wave (SDW) ground state ushers in the superconducting ground state with increase in Ru concentration in the BaFe_{2-x}Ru_xAs₂ system. The maximum T_c and superconducting volume fraction occurs at x=0.75. Although Ru is iso-electronic to Fe, our Hall co-efficient measurements point out that the compound is electron doped. This is further confirmed by band structure calculations.

OUTLINE

A new era of superconductivity research was ushered in following the discovery of superconductivity in FeAs containing compounds with variety of layered structures, viz., LnOFeAs containing alternate layers of LnO and FeAs (Ln=Rare earth) and MFe₂As₂ (M=alkaline earth), a double FeAs layered compound. Both compounds have a SDW ground state and superconductivity occurs by electron and hole doping respectively. More recently superconductivity at 36 K was discovered in Sr₂VO₃FeAs, containing alternate layers of the Strontium vanadium oxide and FeAs layers. Here superconductivity occurs in the parent compound, without any intentional doping. A spurt of synthesis activities was seen across the world with the aim of increasing T_c, which has maximized at 55 K in F doped SmOFeAs. K doping at the Ba site in Ba_{1-x}K_xFe₂As₂ compound results in maximum T_c of ~38K at x=0.45. Enthused by the fact that isoelectronic substitution of P at As site induced superconductivity in the BaFe₂As₂ system, we ventured on Ru substitution at Fe site, which is yet another isoelectronic dopant. The synthesis was carried out in two steps. At first the FeAs and RuAs were synthesized by pressure locking the volatile constituents in 50 Bar of Argon and subjecting it to heat treatment at 600°C to 800°C. Subsequently Ba was added to the stoichiometric mixtures of the arsenides in a glove box and subjected to a heat treatment at 950°C. Nearly single phase samples were obtained by this procedure.

The Ru substitution at the Fe site is evident from a systematic change in the a and c lattice parameters with increase in Ru. The variation in the temperature dependent resistivity with Ru substitution is shown in Figure 1. It is evident that the SDW anomaly in the resistivity shifts to lower temperature with increase in Ru content and superconductivity occurs at 20.8 K in the sample with x=0.75. Similar large drop in resistivity is seen in the x=1.0 sample as shown in the inset. The normal state resistivity fits well to a T^{1.5} power law, in the superconducting samples, a feature not apparent in the parent compound. To prove that superconductivity has indeed set in, magnetization measurements were carried out as a function of temperature, results in the sample corresponding to x=0.75 is shown in Figure 2. An associated diamagnetism with the drop in resistivity is clear from the figure. Inset shows the zero field cooled (ZFC) and field cooled (FC) magnetisation in another superconducting sample with Ru fraction x=0.5. Inset (b) shows the Hall coefficient in the sample with x=0.75, indicating clearly that superconductivity in the Ru doped samples occur due to the presence of electron carriers. The magnetic field dependence of the superconducting onset in the Ru containing samples, corresponding to fraction x=0.75 were measured in a magneto-resistance cryostat, housed with a 12 Tesla magnet. The T_c shows a small shift with field corresponding to a dH_{c2}/dT_c of 3 T/K, as shown in Figure 3. It can be seen from the figure that there is no significant broadening of the transitions under applied field, indicative of negligible granularity effects and anisotropy in this system.

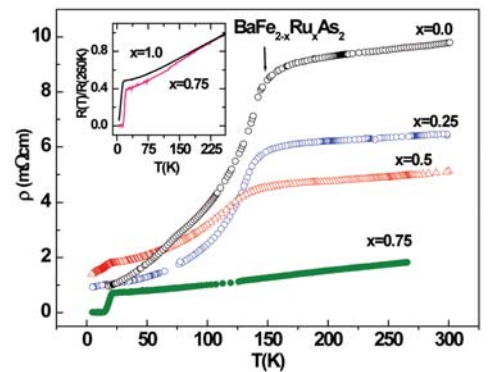


Fig. 1: Temperature-dependent resistivity curves of BaFe_{2-x}Ru_xAs₂ samples. Inset highlights the behavior of the resistivity in the normal state. Downward arrows indicate the positions of T_{SDW} at different Ru concentrations

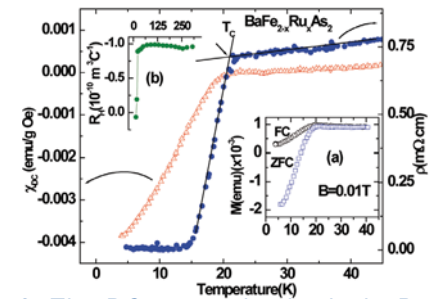


Fig. 2: The DC magnetization in the BaFe_{2-x}Ru_xAs₂ sample with x=0.75 carried out in a measuring field of 0.01T. The coincidence of the diamagnetic drop with the precipitous drop in resistivity is shown in the fig. Inset (a) shows the diamagnetic signal in the sample with x=0.5, inset (b) shows the Hall coefficient measurement in the x=0.75 sample

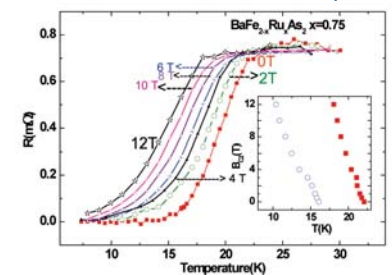


Fig. 3: Superconducting transition vs magnetic field. Inset shows onset and downset vs field

ADDITIONAL INFORMATION

In order to understand how Ru substitution affects the electronic structure of these compounds, we performed density functional calculations for BaFe_2As_2 and $\text{BaFe}_{1.5}\text{Ru}_{0.5}\text{As}_2$ for an appropriate structure, with one of the Fe atoms substituted with the Ru atom, using for the full potential linearized plane wave method plus localized orbitals (FP-LAPW+LO) method with the WIEN2k code. The generalized gradient approximation was used for approximating the exchange interaction. The density of states in BaFe_2As_2 and $\text{BaFe}_{1.5}\text{Ru}_{0.5}\text{As}_2$ are shown in Figure 4. It is clear from the figure that with Ru substitution the density of states contribution from Fe increases, indicating that the Fe 3d electrons get delocalized due to Ru substitution. A small contribution from Ru d levels at EF is also seen. The converged EF in BaFe_2As_2 is 0.60222 Ryd, while that in $\text{BaFe}_{1.5}\text{Ru}_{0.5}\text{As}_2$ is 0.64586 Ryd, clearly indicating an upward shift in EF and therefore electron doping with Ru substitution.

GENERAL INFORMATION

To summarise we have obtained clear evidence for the occurrence of superconductivity in BaFe_2As_2 by an iso-electronic Ru substitution at the Fe site for the first time. The substitution of Ru results in a fairly large expansion of the lattice, which is very different from the behaviour of lattice constant changes seen for all substituents that induce superconductivity in this compound. Band structure calculations carried out in the LDA approximation provide clear evidence for delocalization of the 3d Fe electrons due to Ru substitution. The carrier type turns out to be electrons and measurements of Hall co-efficient confirm this. This study has clearly demonstrated that lattice compression and chemical doping of carriers alone are not the only routes to give rise to superconductivity in the Ba^{122} system.

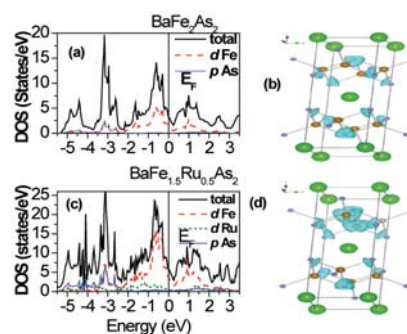


Fig. 4: (a) DOS of BaFe_2As_2 (b) difference charge density in layers. (c) DOS in $\text{BaFe}_{1.5}\text{Ru}_{0.5}\text{As}_2$ (d) the difference charge density

BRIEF DESCRIPTION

We have obtained clear evidence for the occurrence of superconductivity in BaFe_2As_2 by an iso-electronic Ru substitution at the Fe site for the first time. The substitution of Ru results in a fairly large expansion of the lattice, which is very different from the behaviour of lattice constant changes seen for all substituents that induce superconductivity in this compound. Band structure calculations carried out in the LDA approximation provide clear evidence for delocalization of the 3d Fe electrons due to Ru substitution. The carrier type turns out to be electrons and measurements of Hall co-efficient confirm this. This study has clearly demonstrated that lattice compression and chemical doping of carriers alone are not the only routes to give rise to superconductivity in the Ba^{122} system.

ACHIEVEMENT

Ability to synthesise phase pure compounds of $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$, which is both air sensitive and toxic points to the strength of the group in synthesis. Ensuring safe synthesis of the arsenides with the 50 bar set up has enabled the section to continue the program resulting in a miniscule but significant contribution in the area of new superconductors.

PUBLICATIONS

Shilpam Sharma, A. Bharathi, Sharat Chandra, V. Raghavendra Reddy, S. Paulraj, A. T. Satya, V.S. Sastry, Ajay Gupta and C. S. Sundar, "Superconductivity at 20 K in Ru substituted poly crystalline $\text{BaFe}_{2-x}\text{Ru}_x\text{As}_2$ " Phys. Rev B, 81 (2010) 174512.

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Studies on Mechanism of Sodium Fire Attack in Concrete by IR Spectroscopy

EXECUTIVE SUMMARY

Limestone aggregate concrete blocks were subjected to sodium fires conforming to a realistic scenario in order to qualify them as protective sacrificial layers over structural concrete flooring in liquid metal cooled fast breeder reactors. Infrared absorption measurements were carried out on these sodium fire exposed samples as a function of depth from the affected surface. Definite signatures of thermo chemical degradation indicating dehydration and structural modification of the limestone concrete have been obtained for the first time using this technique. Control runs were carried out to delineate the thermal effects of sodium fires from that of the chemical interaction effects. Measurements on limestone aggregate samples treated with fused NaOH provided direct evidence of the exact mechanism of the sodium attack on concrete. Inputs from these studies would have implications on the design and construction of sacrificial concrete layers over the structural concrete flooring in the steam generator building of PFBR.

OUTLINE

The use of limestone aggregate concrete as protective sacrificial layers over structural concrete flooring is a preferred method to limit/eliminate the interaction between sodium and concrete in case of accidental sodium spillage in a fast reactor. It therefore becomes necessary to qualify the limestone aggregate concrete as protective sacrificial layers, by exposing them to a typical hot sodium spillage. Limestone concrete test blocks of composition 48%w limestone as coarse aggregate, 32%w as fine aggregate, 13%w of 53 grade ordinary Portland cement and 7%w of potable water were cast and exposed to hot liquid sodium, simulating a hot sodium leak. It is well known that the key mechanism for the setting and hardening of cement involves hydration of the anhydrous silicate and aluminate phases present to form the corresponding hydrates. Hence, it becomes important to follow the hydration kinetics to understand the extent of setting/degradation in these materials due to sodium fire exposure.

Infrared spectroscopy, a powerful tool to probe the state of hydration in addition to providing useful information of the associated chemical changes, has been used for the first time to obtain direct evidence of the thermochemical degradation in sodium fire exposed limestone concrete samples and generate a depth profile of the degradative effects. Samples removed from various depths were analysed using a DA8 Bomem make FTIR spectrophotometer. Spectrum (Figure 1.) of the reference unexposed sample revealed characteristic absorption features of the major constituent- limestone in the form of calcite. In particular the sharp feature at $\sim 3643 \text{ cm}^{-1}$ riding on the broad water background is known to arise due to the O-H stretching of $\text{Ca}(\text{OH})_2$ - the major hydration product of Portland cement. The broad feature centered around 3500 cm^{-1} corresponds to the absorption features of liquid water (Inset of Figure 2). It is clearly evident from Figure 2 that this feature due to water gets appreciably modified for the sodium exposed samples – a significant softening, accompanied by an increased intensity of the low frequency components is seen. In addition, the sodium exposed samples show splitting of the ν_3 and ν_4 pristine modes. A new sharp feature appears at 1070 cm^{-1} for the sodium exposed sample- presumably the Raman mode of calcite.

Line shape analysis was carried out in the spectral region $3000\text{--}3750 \text{ cm}^{-1}$ for a detailed understanding of the changes associated with the absorption features of water. Figure 3 shows the variation of the stretching modes of water as a function of depth from the affected surface for the test block. It is clear from the figure that these modes exhibit a drastic softening as compared to the reference peak frequencies for depths upto 30 mm from the sodium-concrete interface. Note that the reference values are plotted at a depth of 100 mm for comparison. From the variation, it is concluded that a sacrificial layer thickness of 50 mm is adequate to mitigate the deleterious effects due to sodium exposure.

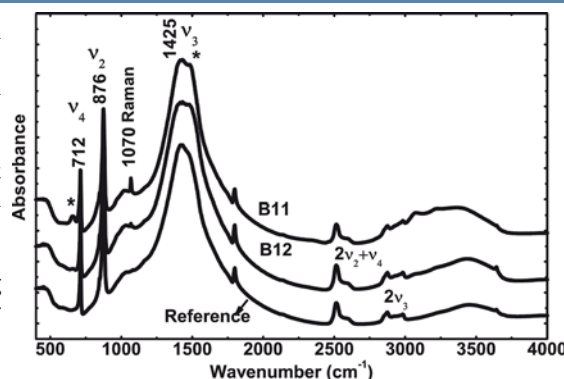


Fig.1: Infrared spectra of unexposed limestone concrete along with sodium exposed samples removed from 10 and 20mm from the exposed surface. ν_2 , ν_3 and $\nu\nu$ are infrared active modes of calcite and ν_1 is the Raman mode activated on exposure to sodium. The broad feature at 3500 cm^{-1} are the absorption feature of bound water in concrete

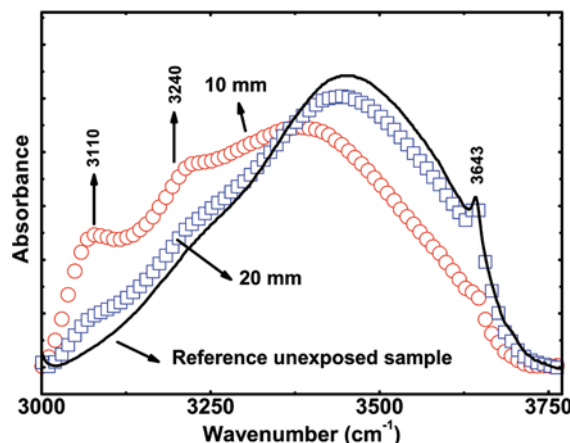


Fig.2: The spectral region corresponding to the absorption features of water. Note the dramatic increase in intensity of the hydrogen bonded components of water at 3110 and 3240 cm^{-1} modes indicating presence of free water. The sharp feature at 3643 cm^{-1} from the O-H stretch of $\text{Ca}(\text{OH})_2$ which is characteristic of set cement is totally absent in the sodium exposed sample removed from 10mm from the affected surface

ADDITIONAL INFORMATION: HOW DO WE UNDERSTAND THE ABOVE CHANGES?

It is well known that water in concrete is held in the following ways: Free water in the capillary pores, physically bound water in the gel pores and chemically bound water in the form of hydration products. On contact with hot sodium, bound water is released from inside the concrete in the form of free water. This would imply an increase in the extent of hydrogen bonding compared to bound water in concrete that is confined. This increased H- bonding leads to a weakening of the O-H stretch modes resulting in the observed drastic softening of the water modes. The new feature at 1070 cm^{-1} can be identified as a Raman active ν_1 mode of calcite that gets activated after interactions with hot liquid sodium. The appearance of this Raman mode point to a lowering of symmetry in calcite caused due interaction with hot sodium signaling a structural transformation of calcite. Both these effects would have deleterious effects on concrete.

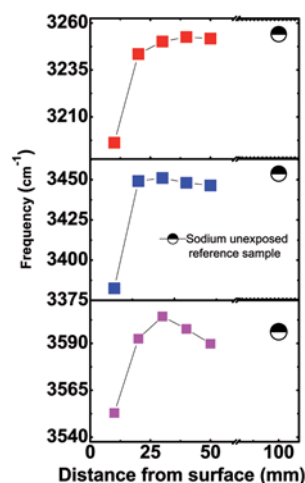


Fig. 3: Variation of stretching modes of water as a function of depth from the sodium concrete interface. The half shaded circles correspond to frequencies of sodium unexposed samples. Note that the values reach the reference frequencies for a depth of 30 mm from the sodium exposed surface

BRIEF DESCRIPTION

Direct evidence of the thermochemical degradation of limestone aggregate concrete on exposure to sodium fires have been obtained using infrared spectroscopy for the first time. Definite signatures of the conversion of bound water in concrete into water in the free state has been obtained. In addition a structural modification of the limestone aggregates has also been observed as a consequence of sodium – concrete interactions, which would lower the strength of concrete. Measurements on limestone aggregate samples treated with fused NaOH provided direct evidence of the exact mechanism of the sodium attack on concrete. From these fundamental studies, it is found that a sacrificial layer thickness of 50 mm concrete is adequate to mitigate the thermochemical effects due to sodium fire.

ACHIEVEMENT

This is the first time that infrared spectroscopy has been used to study the degradative effects of limestone aggregate concrete by exposing them to sodium fires conforming to a realistic accident scenario. Inputs from these studies would have implications on the design and construction of sacrificial concrete layers over the structural concrete flooring in the Steam generator building of PFBR.

PUBLICATIONS

M. Premila, K. Sivasubramanian, G. Amarendra and C.S. Sundar. "Thermochemical degradation of limestone aggregate concrete on exposure to sodium fire," J.Nucl. Mater. 375 (2008) 263.

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Correlation of Magnetism and Hardness in Nickel-base Coatings

EXECUTIVE SUMMARY

Nickel-base Colmonoy 6 is the material chosen for hardfacing of austenitic stainless steel (SS) components used in Prototype Fast Breeder Reactor. Considerable difference in melting points between the stainless steel and Colmonoy 6 results in significant dilution of the hardface deposit from the substrate. Although both austenitic stainless steel and the undiluted Ni-base hardfacing alloy deposit are non-magnetic, the deposit diluted by stainless steel becomes magnetic. In this study, magnetism at different dilution levels of the deposit is measured using Magnegage and Ferritescope and the results are correlated with the dilution of the deposit. As dilution also affects the hardness of the deposit, it was also possible to correlate the magnetic property of the deposit with its hardness. These findings have potential application in using magnetic measurement as a non-destructive method for estimating the hardness of the deposit on actual components not amenable for hardness measurements.

OUTLINE

Colmonoy alloys deposited by Gas Tungsten Arc Welding (GTAW) process suffer from significant dilution by the austenitic SS substrate. Dilution in the first layer is the highest, which decreases progressively in the subsequent layers. Although both Colmonoy and austenitic SS are non-magnetic, it was observed that, the deposit of Colmonoy on austenitic SS is attracted by a magnet, indicating that dilution of the hardfacing alloy by the substrate makes it ferromagnetic. The susceptibility to magnetic attraction is highest in the first layer with highest level of dilution and reduces in the successive layers with reduction of dilution.

In this study, separate deposits of Colmonoy-6 with 1 to 5 layers were produced in five different 316LN SS base plates using GTAW process. To obtain undiluted deposit, Colmonoy-6 rod was melted on a water-cooled copper plate by GTAW process. Magnetic measurements were carried out using Ferritescope and Magnegage. Microstructural analysis was performed using optical and scanning electron microscopes followed by EDS analysis of the matrix. Vicker's hardness measurements and XRD analysis was also carried out for each hardface layer and undiluted deposit.

The susceptibility to magnetic attraction of the Colmonoy-6 deposit on 316LN SS is highest in the first layer and is appreciably low in the second layer; while no magnetism is observed from the third and successive layers. Microstructural examination reveals that the size, morphology and distribution of both boride and carbide precipitate vary widely in different deposit layers (Figure 1). In the first layer, the number and area fraction of the precipitates are small, which increases in second and third layer with less significant change in subsequent layers. Hardness of the first layer is ~ 480 VHN and recovers substantially to 750 and ~ 800 VHN in the second and third layer, respectively, and variation in the hardface deposit is almost negligible from the third layer onwards.

The EDS results show significantly higher Fe content in the first layer (21 wt%) and lower Ni content (59 wt%) than in the undiluted deposit (4.8 wt%, 83 wt%). However, composition of the third layer and the undiluted deposit are similar indicating negligible dilution in the third and subsequent layers (Table 1). Moreover, the first layer contains Mo, an element that is present only in the 316LN SS substrate. The XRD results from undiluted Colmonoy-6 and different deposit layers identify the matrix as f.c.c. Ni phase alloyed with Fe, Cr and Si. The lattice parameter for the matrix phase, estimated from the patterns for each deposit layer, shows that there is a small but definite change for the first and second deposit layers compared to that of the undiluted deposit. This variation in lattice parameter of the matrix with dilution correlated well with corresponding variation of magnetic property measured by Ferritescope (Figure 2).

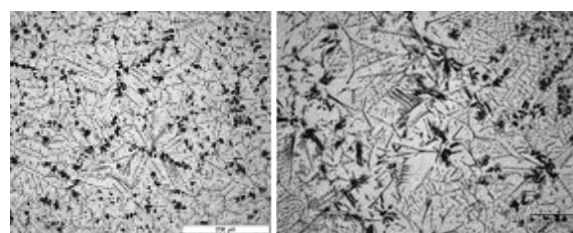


Fig.1: Microstructures of second layer and fifth layer of Colmonoy 6 hardface deposit on 316LN SS

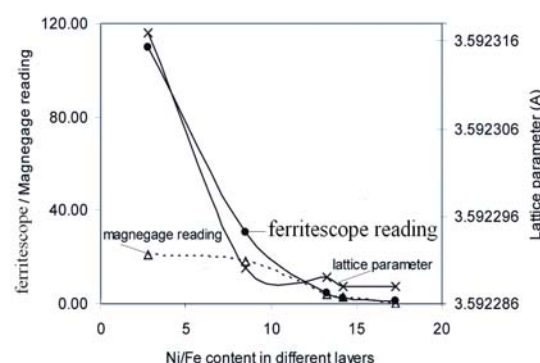


Fig.2: Variation in Ferritescope, Magnigage reading and lattice parameter with dilution of different deposit layers

Table 1: Variation in matrix composition estimated from EDS analysis of Colmonoy 6 in undiluted deposit and in different deposit layers

Element	Layer Deposit on stainless steel					Deposit on Copper
	First	Second	Third	Fourth	Fifth	
Nickel	58.8	80.2	83.6	83.7	83.4	83.5
Iron	21.2	9.46	6.31	5.88	4.82	4.83
Chromium	16.2	6.35	5.67	6.43	6.63	8.01
Silicon	2.82	3.97	4.4	4.02	4.63	3.69
Molybdenum	0.99	-	-	-	-	-

ADDITIONAL INFORMATION ABOUT ORIGIN OF MAGNETISM IN CERTAIN Fe-Ni ALLOY SYSTEM

The magnetism observed in the Ni base hardfacing alloy diluted by stainless steel is similar to magnetism observed in Fe-Ni alloy Invar. Austenitic phase of pure Fe ($\gamma\text{-Fe}$) exists in Low Spin antiferromagnet, with a small atomic magnetic moment of $\sim 0.5 \mu\text{B}$. However, alloying with Ni can result in an Increase in interatomic distance and if it exceeds $3.6 \text{ \AA}$, it leads to appearance of High Spin ferromagnetic state with atomic magnetic moment $\sim 2.8 \mu\text{B}$.

If the magnetism observed in the diluted zone of this Ni-base hardfacing alloy is similar to that observed in Fe-Ni alloys like Invar, then there is yet another property of the deposit that may need to be investigated further; the thermal expansion coefficient (α). Invar alloys have very low α ($1.2 \times 10^{-6} \text{ K}^{-1}$) and hence, it is expected that the diluted zone of the deposit shall also have low α values, which is different from that of the substrate as well as undiluted hardfacing alloy ($8.14 \times 10^{-6} \text{ K}^{-1}$). Austenitic stainless steel is known for its very high α ($17.3 \times 10^{-6} \text{ K}^{-1}$), and if the first deposit layer has very low α , then this can make the deposit prone to cracking during deposition as well as thermal cycling during service. Hence, variation of α with Fe content in Ni-base hardfacing alloy needs to be studied for Ni-base hardfacing alloys which can further improve the understanding of this relatively high cracking susceptibility of Ni-base hardfacing alloys.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

For transition metals, ferromagnetism originates from the d-orbitals containing unpaired electrons and a sharp decrease of atomic magnetic moment is evidenced as the atomic volume reduces below a certain critical value in Ni-Fe alloys, Fe atoms occupy some of the lattice sites resulting in a change in lattice parameters. Other elements present in the alloy also influences the lattice parameters, and hence its magnetic properties. Also, the Mo atom, which is larger in size than the Ni and Fe atom, is known to alter the lattice parameters of Fe-Ni alloys. Mo is present in the first deposit layer, and magnetism observed is also the highest in this layer. In any case, it is clear that change in the lattice parameters of the matrix phase caused by dilution has a major role for the observed magnetism. The correlation between lattice parameter and dilution-magnetic measurements of different layers (Figure 2) is self explanatory. There is almost one-to-one correspondence between variations of Ferritescope reading with lattice parameter with correlation coefficient 0.97897. Though variation of lattice parameter with dilution is not very high its correspondence with magnetic property of the deposit is striking. Figure 3 shows the correlation between hardness and Ferritescope reading. Almost a linear increase in hardness with decrease in Ferritescope readings, with correlation coefficient of -0.99431, is observed. Hence, this correlation can be used to predict the hardness of the deposit simply by measuring the magnetic property of the deposit by Ferritescope.

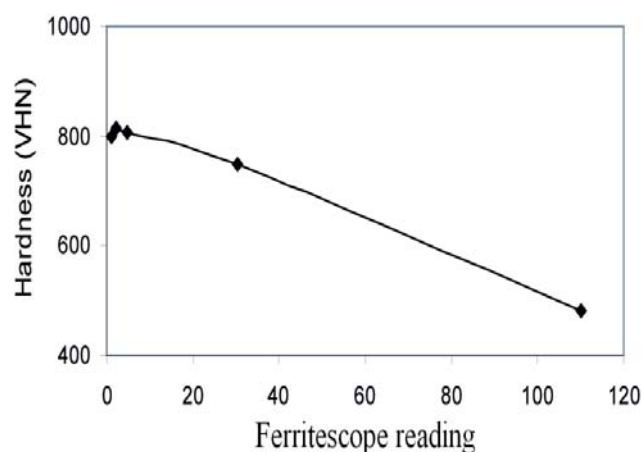


Fig. 3 : Variation in hardness with Ferritescope readings of different deposited layers

ACHIEVEMENT

One of the potential and immediate applications of the results presented here is the use of magnetic measurement as a non-destructive tool to estimate the surface hardness of the Ni-base hardfaced coatings on austenitic stainless steels. Due to reasonably good correlation that exists between Ferritescope reading and hardness, this reading can be used as an index of hardness. Ferritescope is an equipment already used widely in the field for measuring delta-ferrite content in stainless steel welds. The importance of such measurements is significant in hardfacing of actual components because wide variation in hardness can exist in the deposit depending on the dilution of the deposit by substrate material and thickness of the deposit remaining on the component after machining.

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Enhanced Creep Resistance of P91 Steel by Boron Addition

EXECUTIVE SUMMARY

Type IV cracking is a well reported problem in the weldments of ferritic steel including modified 9Cr-1Mo steel (P91). However, modified 9Cr-1Mo steel with controlled addition of boron (P91B) exhibits improved resistance to creep and Type IV cracking. This has been explained based on the stability of the microstructure in P91B base metal as well as the heat affected zone (HAZ). In P91 steel weldment, the intercritical HAZ (ICHAZ) is very fine with substantial recovery while in the ICHAZ of P91B steel, prior austenite boundaries existed before welding retained and within the grain martensite lath boundaries are clearly seen. Thus the results clearly indicate the potential for developing new steels with controlled addition of boron with improved resistance to creep and Type IV cracking.

OUTLINE

Improvement of thermal efficiency and reduction in CO₂ emission from the fossil power plants requires them to be operated at higher temperature; but high Cr ferritic steels currently used in these plants cannot withstand temperatures beyond 600°C. Further, their weldments are susceptible for Type IV cracking, which make the welds weaker than the base metal. During welding the steel close to the weld interface experiences temperatures well above the transformation temperature and the zone of the base metal in which microstructure is affected by the weld thermal cycle is known as heat affected zone (HAZ). HAZ itself is divided into different sub zones and the sub zone that is heated to a temperature range where partial transformation of ferrite to austenite takes place is referred as ICHAZ. After post weld heat treatment (PWHT) and during service precipitates grow rapidly in this zone and preferential recovery of laths and prior austenite grain boundaries (PAGB) reduces creep strength of the ICHAZ. This is known as Type IV cracking and is the main reason for premature failure of the weldment. This study aims to obtain stable microstructures in P91 steel, which is the candidate material for the steam generators of Prototype Fast Breeder Reactor (PFBR), mainly in the HAZ of the weldment by addition of boron. Studies carried out on ferritic steels containing W and Co have indicated that controlled addition of boron does have a significant improvement in the Type IV cracking resistance of the steel.

A special heat of P91 steel with controlled nitrogen and boron was melted by MIDHANI for this study. This steel is used in the normalised (1050°C/1h) and tempered (760°C/3h) condition (N&T). The transformation temperatures for this steel are found to be higher than those of the P91 steel, which is attributed to the slower transformation kinetics due to presence of boron in the steel. Recovery process and coarsening of the precipitate that take place during high temperature exposure is also delayed. As a result under identical conditions of tempering or PWHT, P91B steel has higher dislocation density than P91 steel and hence higher strength. Microstructures of P91 and P91B steels after a heat treatment that simulated the ICHAZ microstructure and PWHT are shown in Figure 1.a-b. It is clear that ICHAZ microstructure of these two steels are vastly different with the former showing a fine microstructure while the latter shows reconstituted prior austenite grain with grain size comparable to that of the base metal and with clear martensite lath inside the grains. Hardness profiles taken across the weld interface (Figure 2) show a dip in the HAZ of P91 weld joint which is absent in P91B weld joint. Further, it can be noted that hardness difference between coarse grain heat affected zone (CGHAZ) and ICHAZ is large in P91 weldment than P91B weldment.

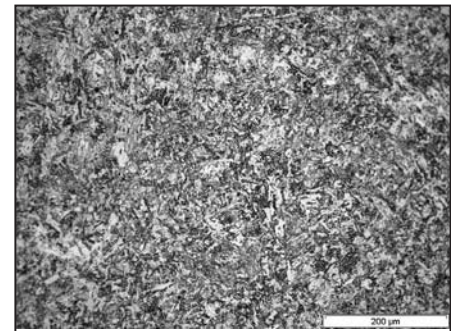


Fig.1a: Microstructure of ICHAZ simulated at 875°C for 5 minutes and tempered at 760°C for 3h of 1150°C normalized and 760°C tempered in P91



Fig.1b: Microstructure of ICHAZ simulated at 875°C for 5 minutes and tempered at 760°C for 3 h of 1150°C normalized and 760°C tempered in P91B steel

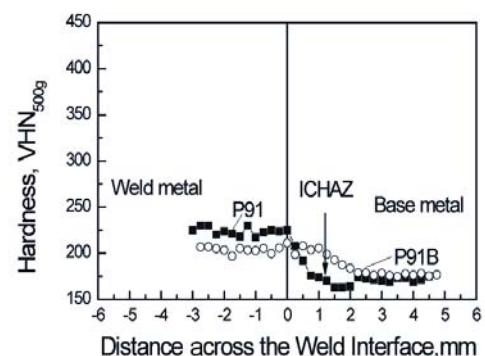


Fig. 2 : Hardness distribution across the weld interface of P91 and P91B weldment

ADDITIONAL INFORMATION

Creep test conducted at 600°C and 120MPa stress on P91 cross weld specimen has failed after 1000 h duration and under the same test condition creep test on P91B cross weld specimen is still continuing with the test duration already crossing three thousand hours. Further, it is also observed that the steady creep rate is much less in P91B steel than in P91 steel. These test results clearly support that the microstructural changes that occur in the heat affected zone due to presence of boron in the steel significantly improves the resistance to creep and Type IV cracking.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Significantly different heat affected zone microstructures found in these two steels are explained based on the different morphologies of austenite formed in these steel during weld thermal cycle. In P91 steel, morphology of austenite formed is globular and austenite nucleation occur along the PAGB that existed in the base metal. In P91B steel, the morphology of the austenite formed is acicular and austenite nucleates within the prior austenite grains maintaining an orientation relationship between the tempered martensitic lath present inside the grains. This is the reason why the prior austenite boundaries present before welding in the base metal is still discernable in the heat affected zone of the P91B steel. Special heat treatments conducted to simulate ICHAZ with different hold time at the peak temperature of simulations 875 and 900°C indicated globular austenite forms also in P91B steel if duration of thermal cycle is long (Figure 3). Thus role of boron in improving the Type IV crack resistance comes from its effect on the ferrite to austenite transformation behaviour.

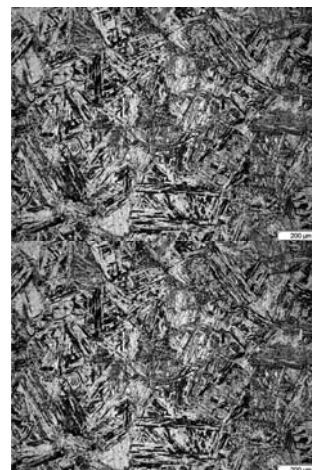


Fig. 3: Microstructure of ICHAZ simulated at 875°C for ten and thirty minutes and tempered at 760°C for 3 hours of P91B steel

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Microstructure of ICHAZ of ferritic steel (including P91) weldment recovered preferentially along the PAGB or lath boundaries due to faster coarsening of precipitates in this zone than the rest of the weld. Strength gradient present across the weldment increases triaxiality in the weldment which, during creep deformation, promotes cavitation at soft ICHAZ leading to Type IV cracking. Addition of boron in P91 improves strength of ICHAZ and most importantly, reduces strength gradient across the weld joint. This is the reason for better creep strength and improved resistance to Type IV cracking of the boron containing ferritic steels.

ACHIEVEMENTS

Previous studies on boron containing ferritic steels have shown improvement in creep and Type IV cracking resistance of these steels; the role of boron in causing these beneficial effects were not clear. The present study has clearly shown the microstructural evolution in the heat affected zone of boron containing steel and how the resultant heat affected zone is stronger than the heat affected zone of the boron free steels.

PUBLICATIONS

1. C.R.Das, S.K.Albert, A.K.Bhaduri, G.Srinivasan, B.S.Murty, "Effect of prior microstructure on microstructure and mechanical properties of modified 9Cr-1Mo steel weld joints", *Materials Science and Engineering A*, 477 (2008) 185.
2. C.R.Das, M.Divya, S.K.Albert, A.K.Bhaduri, B.S.Murty, "Effect of hold time on inter-critical heat affected zone simulation of boron-added modified 9Cr-1Mo steel", *Welding in the World*, 53 (2009) 511.
3. C.R.Das, S.K.Albert, A.K.Bhaduri, B.S.Murty, "Effect of initial prior austenite grain size on heat affected zone microstructure and mechanical properties of modified 9Cr-1Mo steel", *International Institute of Welding – International Congress*, Indian Institute of Welding, Chennai 2008, p.393.
4. A.K.Bhaduri, C.R.Das, S.K.Albert, A.Klenk, B.S.Murty, "Study of deformation behaviour of simulated inter-critical heat affected zones of modified 9Cr-1Mo steel", *Materials and Manufacturing Processes* (in press).

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A Comparative Study of Unprotected Loss of Flow Accidents in Metal and Oxide Core of Fast Breeder Reactor

EXECUTIVE SUMMARY

The main interest in using metal fuels in FBR is to obtain higher breeding ratios compared to ceramic fuel. There is a concern on the safety of such a reactor under loss of flow due to higher coolant void reactivity effect. Studies show that due to lower temperatures and higher negative expansion feed backs, FBR-M behaviour in Unprotected Loss of Flow Accidents is benign even compared to oxide core.

OUTLINE

Most fast reactors use ceramic (oxide) fuels, as there is vast experience in using them. However, there has been growing interest in using metal fuels due to significant advantages over non-metal fuels. The metal fuels also have excellent compatibility with liquid metal coolants, high fissile and fertile densities and high thermal conductivities. The main interest in using metal fuels is to obtain higher breeding ratios and thus shorter fuel doubling times, which arises due to relatively harder spectrum as compared to that of ceramic fuels. It is of interest to study the safety of metal fueled reactors (with varying zirconium content) relative to that of oxide cores. Among the accident scenarios, loss of coolant flow, transient over power are of interest. It is found that the sodium void worth increases from 1.7 \$ to 4.4 \$ as the fuel changes from oxide to U-Pu-Zr fuel for the 500 MWe designs. We examine the safety aspects of the metal cores by analyzing the unprotected loss of flow (ULOF) for two typical FBR designs with metallic fuel, one with U-Pu-6% Zr and another with U-Pu-10% Zr composition, called FBR-M (6%Zr) and FBR-M (10%Zr) respectively. The transient analysis results of these two designs are compared with that of PFBR-oxide design. The coolant coast down is assumed to occur due to pump failure caused by power failure. The flow having time is 8s and reactor shut down system fails.

The ULOF transient is analyzed in the pre-disassembly phase. The analysis is carried out using the code PREDIS which has been validated against the European LOFA benchmark problem and up to onset of sodium boiling in the BN800 benchmark exercise. PREDIS is a multi-channel code, where each flow channel (radial zone) is represented by a fuel pin with its associated coolant flow and surrounding structure. The code uses the point kinetics model for the calculation of reactor power. PREDIS uses a variable time step for advancing the calculations with an upper limit of 5 ms and a lower limit of 0.1 μ s. The code calculates the temperatures in fuel, clad and coolant in all the meshes defined by the radial and axial zones.

It is found that the sodium voiding spreads rapidly in top axial blanket and to below middle of oxide core as shown in the bottom part of Figure 1 in about 70 s (light shading shows the voided region). Core melts and dis-assembles in less than 80 s. In contrast, even after 1400 s the voiding is restricted to top of core in the metal core as shown in the top of Figure 1. The behaviour of the two FBR-M cases are similar with Zr-10% case showing higher times for Na voiding initiation. The net result is that reactivity remains negative in metal core. Reactor power reaches power level corresponding to the capacity of safety grade decay heat removal systems in 900 s. When credit is given for decay heat removal systems, further spreading of Na voiding is prevented and it is inferred that metal core reaches a safe subcritical state at elevated Na temperature.

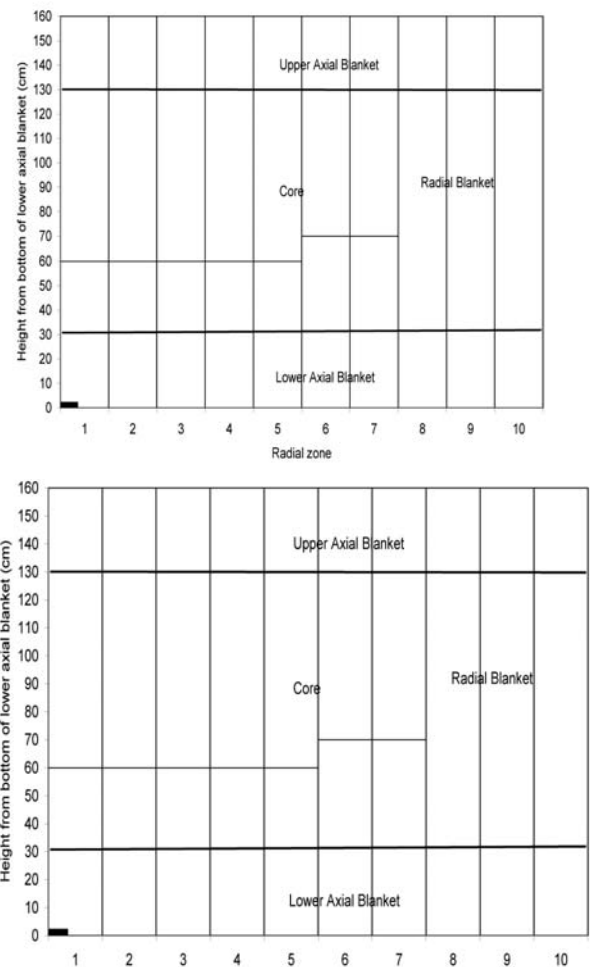


Fig.1: Comparison of Na voiding in FBR-M (6 % Zr) and PFBR

Table 1: Summary Results of Unprotected Loss of Flow Accidents

	FBR-M (Zr-6 %)	PFBR
Reactor power at 75 s (MWt)	231	1306
Net reactivity at 75 s (\$)	-0.26	0.29
Net voiding fraction at 75 s	0.0	0.42
Voiding initiation (s)	900	26
Core damage	Nil	damaged
Reactor power at 1800s (MWt)	17.7	-
Net reactivity at 1800 s	-0.52	-

ADDITIONAL INFORMATION ABOUT SAFETY PARAMETERS OF FBR-M

High breeding ratio of FBR-M results in a low burn up swing and the lower Doppler constant relative to oxide core reduces the positive reactivity to be inserted from startup to full power. It is reported that the high internal conversion ratio in metal fuel makes it possible to achieve near zero burn up swing per cycle. Thus absorber rod reactivity worth requirements are significantly less in FBR-M. The amount of reactivity that can be inserted by uncontrolled withdrawal of a control rod in the metal core is less than half of that of oxide core. As the larger positive coolant sodium void worth is a concern from the safety point of view, there have been proposals to reduce it. Reducing the core height or introducing a sodium plenum instead of the axial blankets are some of the proposals both of which reduce the achievable breeding ratio.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

With the use of metal fuel, the plutonium breeding ratio (BR) increases significantly as shown in the Table 1. The breeding ratio reaches a 1.36 with FBR-M (Zr- 6%). Compared to oxide core, plutonium enrichments are less in FBR-M, but core plutonium inventory is nearly same.

Important physical properties of fuel are also compared in the same Table. Even though fuel melting point is low in FBR-M, the conductivity is an order of magnitude higher than oxide and linear expansion coefficient is double. In addition, effective fuel density is higher in FBR-M.

Table : 1 Physical property of fuel

Parameter	FBR-M (10%Zr)	FBR-M (6%Zr)	PFBR oxide
Breeding ratio	1.24	1.36	1.05
Core plutonium enrichment (%)	15.4/20.6	13.6/18.2	20.7/27.7
Plutonium inventory (t)	1.93	1.97	1.97
Linear expansion coefficient (C ⁻¹)	18.5×10^{-6}	19.7×10^{-6}	11.2×10^{-6}
Thermal conductivity (W/cm/C)	0.22	0.25	0.02
Melting point (C)	1142	1067	2750
Boiling point (C)	3932	3932	3387

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

High conductivity of fuel reduces the operating fuel temperatures in FBR-M. Thus the residual heat transferred to coolant is low after initiation of ULOFA. As reactor also becomes sub-critical due to negative reactivity feedbacks like fuel expansion, Coolant voiding is limited to top axial blanket and top of core. This void reactivity is also negative and reactor reaches decay heat levels in fifteen to twenty minutes.

ACHIEVEMENT

It is seen from the study that despite high Na void reactivity effect in FBR-M, ULOF is benign relative to oxide core. This is due to the low operating temperatures and higher negative reactivity feed backs from fuel expansion. The two FBR-M cases considered with different Zr content in fuel are close to each other and are considerably benign and allow sufficient time for operator action. When safety grade heat removal systems are also considered, it is inferred that FBR-M reaches a safe sub-critical state at elevated Na temperature even without operator action.

PUBLICATIONS

R.Harish, G.S.Srinivasan, A. Riyas and P.Mohanakrishnan, "A comparative Study of Unprotected Loss of Flow Accidents in 500 MWe FBR Metal Cores with PFBR Oxide Core", Annals of Nuclear Energy 36 (2009) 1003.

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Simulation of Plastic Deformation in Oxide Dispersion Strengthened Ferritic Alloys- with 3-Dimensional Dislocation Dynamics

EXECUTIVE SUMMARY

Ferritic alloys strengthened by oxide dispersion strengthening particles are prime candidates for future fast reactor applications. Discrete dislocation dynamics simulations are carried out to analyze plastic deformation of oxide dispersion strengthening and non-ODS ferritic grains. Our simulations show that, before irradiation, particles reduce dislocation mean free path and hence, lead to greater strain localization and significant hardening, by comparison with particle-free grains. After irradiation however, this trend is reversed. Namely, the presence of particles acts against the detrimental effect of the irradiation-induced loops and hence, strain localization and hardening are reduced altogether. These results could help explaining why irradiated oxide dispersion strengthening grains exhibit better deformation characteristics than irradiated, particle-free grains.

OUTLINE

Recently developed Oxide Dispersion Strengthened (ODS) steels exhibit improved post-irradiation ductility, in comparison with conventional, single phased steels. So far, this beneficial characteristic is only partially and qualitatively explained in terms of dislocation based mechanisms. In this work, dislocation-based plastic deformation of oxide dispersion strengthening steels is investigated in more details, using 3D dislocation dynamics (DD) numerical simulations. Calculations are adapted to ferritic grains, accounting for the well-known edge-screw dislocation mobility anisotropy. Implemented cross-slip behavior is based on more recent investigation results, including atomistic molecular dynamics simulations. Introduction of strengthening particles and irradiation effects in dislocation dynamics simulations requires further experimental inputs. In this way, transmission electron microscopy investigations show that oxide dispersion strengthening particles are rigid internal interfaces, due to lattice spacing mismatch between particle and matrix. During irradiation, defect clusters in the form of loops are generated. These loops, having specific dose and temperature dependent sizes and strength act as soft obstacles to post-irradiation dislocation motion.

Adopted grain morphology is equi-axial, with grain diameter set to 1 μm (Figure 1). Grain interfaces are taken as strong obstacles to dislocation motion, as if for highly disordered grain boundaries. Oxide dispersion strengthening particles are introduced as a set of non-shear-able facets (Figure 2). During plastic deformation, mobile dislocations go round the particles, before unpinning. Critical stress corresponding to this mechanism is called the Orowan stress. Simulations representative of irradiated grains include a 1020 m^2 density of 20 nanometer dislocation loops. Selected loop diameter and density correspond to high irradiation doses, performed at 400°C. Loops are immobile, due to their specific geometric characteristics (Burgers vector), as shown by TEM analyses. Implementation of immobile loops in the simulations is described in Figure 2. Each loop is described as a single facet, acting as soft obstacles to mobile dislocations. Dislocation/loop interaction strength between depends on the character of incoming dislocation, as pointed out by independent dislocation dynamics and MD calculations (see also Figure 2 caption).

Then, dislocation dynamics simulations are performed in plastic strain rate controlled conditions, at room temperature. Deformation of both oxide dispersion strengthening and non-ODS grains are simulated before and after introduction of irradiation loops. The results are analyzed in terms of strain localization and stress-strain characteristics, using specially developed post-treatment tools.

Analysis show that, in the absence of irradiation defect clusters, oxide dispersion strengthening particles reduces dislocation mean free path and hence, lead to both material hardening and increased strain localization. After irradiation however, it is seen that the presence of loops in the material reverses these trends. Namely, presence of particles acts against the detrimental effect of irradiation defect structure and helps reducing strain localization. The hardening behavior exhibits the same trend: irradiation induced hardening is significantly reduced by the presence of oxide dispersion strengthening particles.

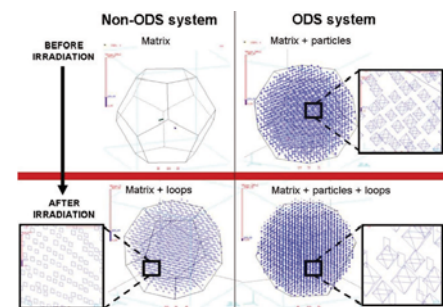


Fig. 1: Plastic deformation of ODS and non-ODS systems are examined for analysis purposes. ODS systems contains hard particles. Non-ODS systems are particle-free (see Figure 2). Irradiated configurations contain loop populations, whereas non-irradiated configurations are loop-free

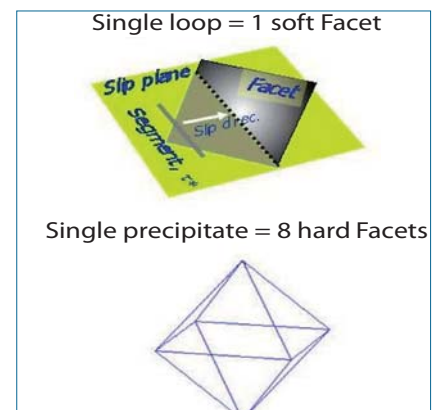


Fig. 2: Particles and irradiation loops are introduced as internal interfaces called facets. Dislocations go through a given facet depending on critical facet strength t_{facet} . Hard facets making up precipitates have $t_{\text{facet}} = \infty$. Soft facets making up loops have $t_{\text{facet}} = 2.7$ times the Orowan stress for screw dislocations and $t_{\text{facet}} = 1.35$ times the Orowan stress for edge dislocation. Particle diameter is 20 nanometer and particle volume fraction is 0.5%

ADDITIONAL INFORMATION ABOUT INTRODUCTION OF IRRADIATION EFFECTS IN DD SIMULATIONS

Irradiation induces the formation of defect clusters in the form of dislocation loops. Introduction of such defects in DD simulations requires specific treatment and inputs. In general, loops are soft obstacles to dislocation motion, which can be overcome by applying a critical shear stress. Nowadays, interaction between mobile dislocation and nanometre sized loops are examined at the atomistic scale, using molecular dynamics simulations. These calculations provide critical loop strength, accounting for both dislocation core effects and long-range elastic effects. Resulting loop strength is introduced as explained in Figure 2, for the sake of diminishing the computational load of massive DD simulations (Figure 1).

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Our DD simulations use the following three modelling ingredients (Figure 3). i- Dislocation lines are discrete, orthogonal edge and screw segments, treated as elastic inclusions moving in an elastic medium. Forces acting on each segment are calculated at ii- discrete time intervals and take iii-discrete spatial positions. The bcc lattice spacing corresponds to ten inter-atomic distances, i.e. the shortest possible dislocation-dislocation annihilation distance.

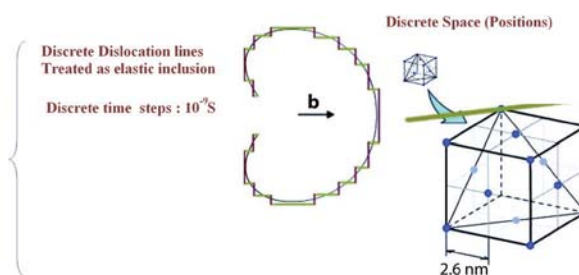


Fig. 3 : Discrete dislocation dynamics framework in three dimensions. Different levels of discretization are used

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Plastic deformation in metals is carried by linear, mobile crystallographic defects called dislocations. Dislocation dynamics simulations are used to model plastic deformation of metallic materials, by explicitly accounting for dislocation motion, multiplication and their mutual interactions. This simulation method links the space and time gaps between discrete and continuum description of metallic materials mechanics.

ACHIEVEMENT

This study is a step forward for prediction of dislocation mobility in complex ferritic ODS alloys, before and after irradiation. Dislocation mobility controls stress-strain characteristics and strongly influences fracture toughness characteristics of future nuclear materials. This work was done under the aegis of Indo-French collaboration “Agreement on basic research and modelling of physical phenomena”, as per the Implementing Agreement on “Investigation of irradiation-induced microstructure in ODS materials using JANNUS facilities and multi-scale modelling”.

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Infrared Thermal Imaging for Diagnosis of Vascular Disorders

EXECUTIVE SUMMARY

Temperature is a very useful parameter for diagnosing diseases. Often there is a definite correlation between body temperature and diseases. We have used Infrared thermography to carry out non-invasive diagnosis of peripheral vascular diseases. Temperature gradients are observed in the affected regions of patients with vascular disorders, which indicate abnormal blood flow in the affected region. Thermal imaging results are well correlated with the clinical findings. Certain areas on the affected limbs showed increased temperature profiles, due to inflammation and underlying venous flow changes. In general, the temperature contrast in the affected regions is about 0.7 to 10°C above the normal regions, due to sluggish blood circulation. The results suggest that the thermal imaging technique is an effective technique for detecting small temperature changes in human body due to vascular disorders.

OUTLINE

Infrared thermography or infrared imaging or thermal imaging is a non contact tool which maps the surface temperature of a body or an object and has a wide range of applications starting from condition monitoring in industries to medical imaging. Medical infrared thermal imaging has been used to study the flow of blood, detection of breast cancer and muscular performance of the human body. Thermal images have been used to quantify sensitive changes in skin temperature in relation to certain diseases. Blood flow can be assessed by many methods including washout technique, Laser Doppler flowmetry and medical infrared thermal imaging. Of these, infrared thermography has the advantages of being non-invasive, fast, reliable, non-contact, capable of producing multiple recordings at short time intervals and absolutely safe for patients and doctors. In these studies, only relative and not absolute temperatures are significant, which have to be measured at many points on the skin, and in this sense, the IR sensing device has many advantages over conventional devices.

Thermal imaging was carried out using Thermovision-550 system. Region of the body which had aggravated pain/puss discharge was chosen as the region of interest based on the patient's medical history. The thermal profile of this area in a patient was compared with the counter part region of the same subject and same region of a healthy volunteer. A 28 years old male, with a history of pain in the left lower limb, which was getting aggravated on prolonged standing, was examined using thermal imaging. He had varicosity of the long saphenous system of the left lower limb and was suffering from complications of varicosity for the past one year. He was using crepe bandages. Local examination of the lower limbs showed dilated veins present in dorsal aspect of foot extending up to lower one-third of the leg on the right lower limb. There were dilated tortuous veins in dorsum of the foot in the left lower limb.

Another subject, a 31 year old male, who had a history of swelling for 5 years in both the lower limbs on prolonged standing, had recurrent ulceration over the left lateral malleolus associated with pain and discharge of puss. The patient underwent treatment and surgery, 4 years back for the same complaint. Local examination of the left lower limb showed tortuous dilated veins, recurrent heeling ulcer on the left lateral malleolus ulcers covered with slough and puss discharge. Figures 1&2 show the thermal images of the left and right leg of the affected patients. The line profile inset in Figure 1 shows the temperature profile along the toe tips.

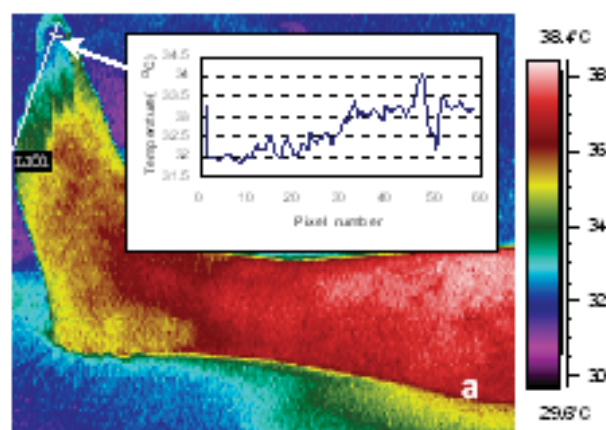


Fig. 1 : Isothermal image of right leg, with varicose veins and line profile along the toes is inset

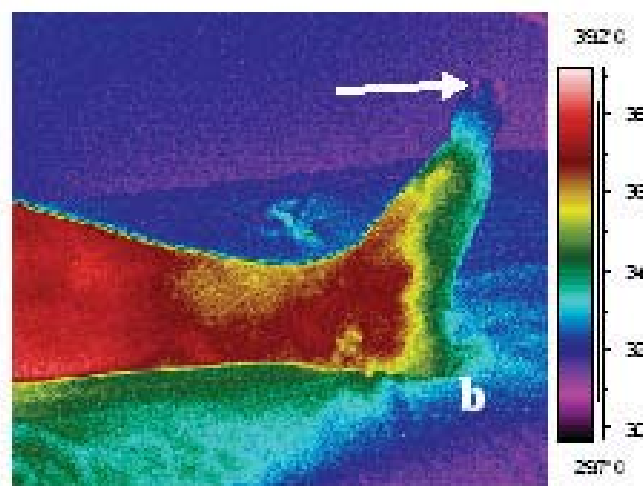


Fig.2: Interior normal view of affected patient left leg

ADDITIONAL INFORMATION ABOUT MEDICAL INFRARED THERMAL IMAGING

The IR radiation covers wavelength that ranges from 0.75 to 1000 μm , among which the human body emissions that are traditionally measured for diagnostic purposes occupy a narrow band of wavelengths ranging from 8 to 12 μm . This region is also referred as the long wave IR (LWIR) or body infrared rays. Another terminology that is widely used in medical IR imaging is thermal infrared (TIR) where the wavelength is beyond 1.4 μm . Within this region, the infrared emission is primarily heat or thermal radiation. The image generated by TIR imaging is referred as thermogram. The near infrared (NIR) region occupies wavelengths between 0.75 and 1.4 μm . Although the NIR and mid-wave IR (MWIR) regions are not traditionally used in human body screening, the new generation detectors enable the use of multi spectral imaging in medicine, in which these regions are observed in different diagnostic cases. The fundamental equations that link the absolute temperature of the object with the intensity and wavelength of emitted radiation are given by Planck's, Stefan Boltzmann and the Wein's Displacement law. The energy radiation after Stefan Boltzman law $W = \epsilon \sigma T^4$, where ϵ is the emissivity and T the absolute temperature. Emissivity depends on factors such as temperature, emission angle, and wavelength. It has been shown that the emissivity of skin (black, white, burnt, male and female) independent of the wavelength and its value is close to 0.98. Therefore human beings can be treated as true black body. For black body the total heat energy radiation is proportional to T^4 . In clinical diagnostics infrared imaging is used as a physiological test that measures the subtle physiological changes that might be caused by many conditions, e.g. contusions, fractures, burns, carcinomas, lymphomas, melanomas, prostate cancer, dermatological diseases, rheumatoid arthritis, diabetes mellitus and associated pathology, deep venous thrombosis (DVT), liver disease, bacterial infections, etc. These conditions are commonly associated with regional vasodilation, hyperthermia, hyperperfusion, hypermetabolism, and hypervascularization which generate higher-temperature heat source. The heat emanating on to the surface from the heat source and the surrounding blood flow can be quantified by using the following Pennes' bio-heat equation:

$$k\Delta^2 T - c_b w_b (T - T_a) + q_m = 0$$

Where k is conductivity, q_m is volumetric metabolic rate of tissue, $c_b w_b$ is the product of the specific heat capacity and the mass flow rate of blood unit per volume of tissue, T is the unknown tissue temperature, and T_a is the arterial temperature.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

From the thermal images, it can be clearly seen that a lower temperature is noted at the distal portion. This is probably due to slow-moving blood circulation in the toes, venous drainage being inadequate due to the varicosity. The temperature in these regions was on an average 0.7 to 10°C above the normal regions. The abnormal temperature is due to varicose veins (Figure 1(a)). The human body creates heat through metabolic activity. The blood in the near-surface vein heats the surface more than normal veins and arteries. Clinically detected areas with varicosity show up as areas of increased warmth in thermal images. From the thermal image (Figure 1(b)), the warm areas are noted on the lateral side of the left leg as well, an unusual finding, because most patients have varicosity located only on the medial side of the leg. The distal region near the toes of one subject is to be less in temperature due to the poor perfusion of blood and is attributed to stasis of circulation due to varicosity. Localized elevated temperatures are easier to discern when the person is in a cool room for at least twenty minutes. A uniform temperature can be seen in the leg of the normal person.

ACHIEVEMENT

Thermal imaging has been successfully used for medical diagnosis of vascular disorders. The temperature in the affected regions of patients with vascular disorders was low in the extremities due to obstructed arteries. However in some areas showed 0.7 to 10°C higher temperature than normal areas due to inflammation and venous flow alteration. In general, the thermal image findings are in good agreement with clinical findings. However, the areas showing higher temperature contrast noted are not obvious in the clinical examination, which needs further studies. This study demonstrates the usefulness of thermal imaging for medical diagnostics.

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3. S. Bagavathiappan, T. Saravanan, John Philip, T. Jayakumar, Baldev Raj, " Infrared thermal imaging for medical diagnosis", Medical Equipment and Automation, 2 (2009) 65.

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Bright Red Emission from Silicon Nanoclusters

EXECUTIVE SUMMARY

Integrating light with Silicon is one of the longstanding problems in the semiconductor technology. A higher quantum efficiency of photoluminescence intensity by one order ($\sim 10\%$) than that for porous-Si (PS $\sim 1\text{--}3\%$) is reported for nanocrystalline (nc)-Si. Formation of direct bandgap in nc-Si, as well as widening of it due to quantum confinement of carriers are well conceived. However, mechanism of luminescence in nc-Si including that of the porous-silicon, is not very well understood. An emission process comprised of an initial non-radiative recombination (time constant $\tau \sim 280\text{--}315$ ps) of excited carriers in the defect states in SiO_2 matrices, to the conduction band minima of nc-Si, followed by a slower process of radiative recombination in the direct band transition for nc-Si along with a non-radiative Auger recombination ($\tau \sim 2.67$ nanosecond) is proposed. A possible population inversion is suggested for the emission process with a competitive Auger recombination process. The results may be encouraging for practical application of Silicon as optical source.

OUTLINE

It is still a daunting task to improve the applicability of light-emitting Silicon micro/nano-structures for successful achievement of stimulated emission and light amplification. Optical gain in the red spectral region is reported in nc-Si embedded in SiO_2 matrix using pulsed laser pumping. Later a stimulated blue emission is reported at extremely high (100 MW.cm^{-2}) excitation density. Most recently, Raman laser is reported in Silicon on insulator waveguide using optical gain in silica matrix. Formation of direct band gap in nc-Si, as well as widening of it due to quantum confinement of carriers are well conceived. However, mechanism of luminescence in the nc-Si system including that of the porous-Si, is not very well understood. According to the present perception, the observed luminescence is explained in terms of electron-hole (e-h) pair recombination with a role of Si-SiO_x interface states. Defect states, created during the growth of nc-Si by ion implantation in silica matrix, is also thought to be responsible for the luminescence process.

A typical XTEM image shows fairly uniform cluster with diameter ~ 5 nanometer (Figure 1) for the annealed sample grown in c- SiO_2 at a fluence of $1 \times 10^{17} \text{ cm}^{-2}$. Electron diffraction pattern [inset in Figure 1] shows the ring pattern corresponding to (220), (311) and (400) planes of nc-Si.

Figure 2a shows the power dependent photoluminescence studies for the samples with peak energy ~ 1.67 eV. Inset in Figure 2a shows a nonlinear log-log plots indicating super-linear power dependence of photoluminescence intensity integrated over area. The estimated power dependence is 1.2. Even though the nc-Si sample is spread $\sim 1 \mu\text{m}$ across the depth, a multiple reflection in the quartz matrix might have increased the active layer thickness affecting the observed intensity. Hardly any spectral change (neither a shift in photoluminescence peak position nor spectral narrowing) is observed even up to the highest continuous wave excitation power density of 5.9 W.cm^{-2} . The bright features of photoluminescence intensities corresponding to the highest incident power density are also shown in the inset of Figure 2a. The decay processes of photoluminescence peak (Figure 2b) can be satisfactorily fitted with a double-exponential function with time constants of τ_1 and τ_2 . The data are collected for different power densities at peak energy ~ 1.67 eV. A pulse shortening is observed when measured at a higher power density of 5 W.cm^{-2} .

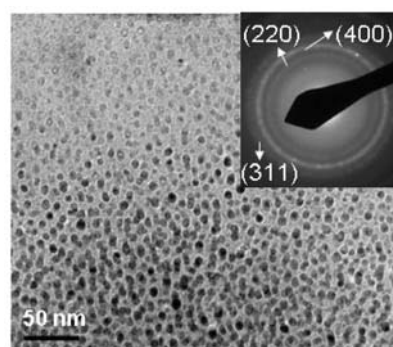


Fig. 1: Cross sectional transmission electron micrograph of nc-Si

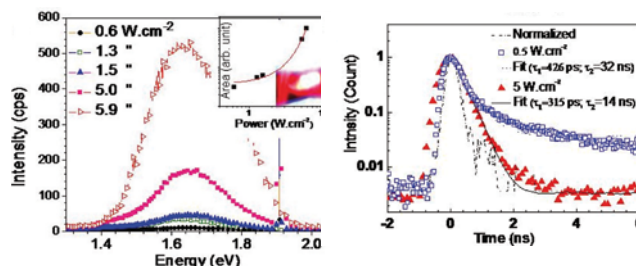


Fig. 2: a) The power dependent PL spectra for samples grown in c- SiO_2 substrates. Insets show the nonlinear log-log plots of PL integrated intensity (area under curves) with varying power density. Pictures with the bright luminescence are also shown. b) Room temperature PL decay processes are examined. The fitting parameters with decay constants are also inscribed

Table: Details of Experiment

Substance	nanocrystalline Si grown by 1MeV Si^{++} implanted in oriented crystalline (c)- SiO_2
Fluences	1×10^{16} - $1 \times 10^{17} \text{ cm}^{-2}$ at 1.7 MV tandetron
Annealing	isothermally at 1373 K
Validation	cross-sectional TEM for nanocluster imaging
Photoluminescence studies	excitation of 375 nm line of He-Cd laser in double subtractive triple monochromator
Luminescence intensity	liquid N_2 cooled 'back-thinned' charged coupled device
Time resolved PL measurements	375 nm pulsed diode laser with pulse width < 70 ps with different power densities at 40 MHz

ADDITIONAL INFORMATION ABOUT NANOCRYSTALLINE SILICON

Various methodologies are thought for improving efficiency of the radiative recombination in the indirect bandgap of bulk nc-Si. An increased quantum efficiency (QE) of $\sim 1\text{-}3\%$ is reported for porous-Si with respect to the measured value of $10^{-4}\text{-}10^{-5}\%$ for c-Si. Recent advances in growth of nc-Si widen the emission range from a blue to a red wavelength region of visible light. The majority of efforts are in the form of nc-Si, along with other systems, e.g., erbium-doped Si-rich oxides, Si/SiO₂ superlattices, Si/SiGe quantum cascade structures, surface-textured bulk Silicon, dislocation engineered strained Silicon.

MECHANISM OF BRIGHT LUMINESCENCE

As a possible mechanism, excited carriers from the valence band of nc-Si are injected into the intermediate defect states, in the silica matrix related to the Si-O interface sates (Figure 3a). These states are produced during the sample preparation process. Absorption peaks $\sim 1.74\text{--}1.8\text{ eV}$ for the nc-Si sample embedded in amorphous and crystalline silica matrices strongly support the presence of such defect states (Figure 3b). This feature of the near-infrared absorption band is caused by a Si=O interface. As predicted by theory and inferred from the experiment, these defect states are formed at the interface between the nc-Si and the SiO₂ matrix. Optically excited carriers thermalize non-radiatively at a very rapid rate (280–315 ps) to the next lower energy state of the conduction band minima corresponding to nc-Si (CB-nc-Si). Finally, the carriers recombine with holes in VB-nc-Si either radiatively and/or non-radiatively (Figure 3a). In case of a broad defect band (as indicated in the absorption spectra (Figure 3b)) each system will emit only in a narrow frequency regime, under the consideration that one or a few e-h pairs can recombine radiatively (14–22 ns at highest power density) in a simultaneous fashion within each individual system. As a consequence, the spectral line will be broadened due to the different neighborhood of each system and PL emission spectrum is not likely to change even with the increase in the excitation intensity. However, amount of defects created in the irradiation process may influence upon the spectral narrowing. The observed spectral narrowing effect, as described earlier (Figure 2) can be well conceived in this model with an expected less degree of defects or defect states in the crystalline matrix than those in the amorphous matrix.

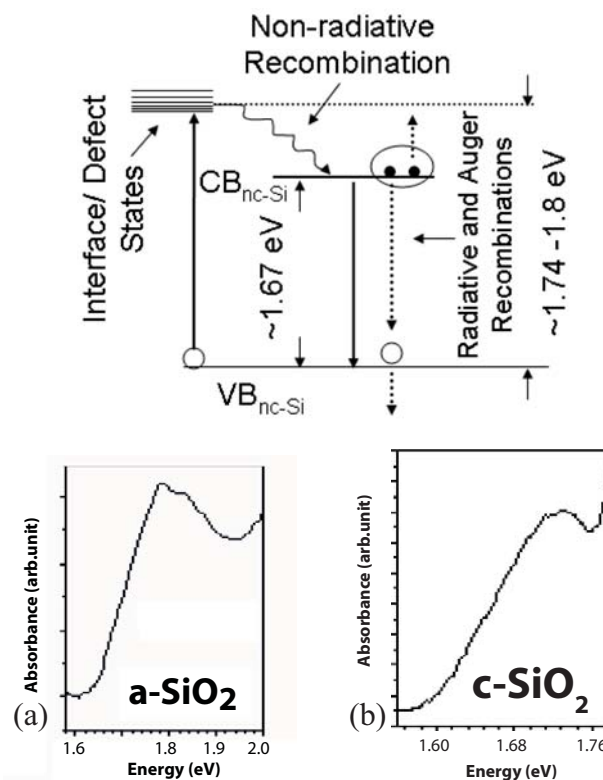


Fig. 3: (a) A schematic band diagram of the overall luminescence process. (b) Typical absorbance spectra for the sample implanted in different SiO₂ matrices at a fluence of $1 \times 10^{17}\text{ cm}^{-2}$ and annealed at 10 hours

BRIEF DESCRIPTION

In conclusion, a super-linear power dependence of the bright red emission $\sim 1.67\text{ eV}$ for nc-Si accompanied with a pulse shortening is observed under a continuous wave excitation. However, the nonlinearity fails to show any significant spectral narrowing. The luminescence process is proposed to be based on the competition between radiative and non-radiative relaxation processes in isolated luminescence centers. The proposed mechanism is supported with the energetic and different recombination time constants derived from experimental observations.

ACHIEVEMENT

A super-linear power dependence of photoluminescence under continuous wave excitation is observed, for the first time, in the radiative recombination process accompanied by pulse shortening. A possible population inversion is suggested for the emission process with a competitive Auger recombination process. The results may be encouraging, one tiny step ahead, for practical application of Si laser.

This work is also cited in 'labtalk' forum as the editor's choice <http://nanotechweb.org/cws/article/lab/35583>

PUBLICATIONS

S. Dhara, C.Y. Lu, K.G.M. Nair, K.H. Chen, C.P. Chen, Y.F. Huang, C. David, L.C. Chen and Baldev Raj, "Mechanism of bright red emission in silicon nanoclusters," *Nanotechnology* 19 (2008) 39540.

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Nanoindentation of 2D Nitride Semiconductors

EXECUTIVE SUMMARY

Indentation technique is extremely useful for understanding atomic movement providing insight to the dynamics of defect as well as crystallization process. Blue diode laser using GaN has the enormous potential for printing (1200 dpi having $\sim 17 \mu\text{m}$ spot size), high density DVD memories and white light based compact display devices. However, high dislocation density and related issues in GaN are one among few of the major hindrances for the application as blue laser. Thus, understating the defect dynamics and crystallization process addressing removal of stress and mechanical properties are of immense interest. We report atomistic model for the mechanism of recrystallization and planar defect formation of epitaxial GaN. Role of crystalline orientation of InN film in their mechanical properties are also described in the similar framework, looking at its importance in solar cell applications.

OUTLINE

Growth of epitaxial (epi-) GaN film with low dislocation density either by pre-grooving the buffer layer or by lateral epitaxial overgrowth (LEO) is to reduce residual strain and help crystallization process. A remarkably low threading dislocation (TD) density $\sim 10^7 \text{ cm}^{-2}$ is reported in epi-GaN film adopting the LEO method. A combination of biaxial and hydrostatic stresses due to dislocation related extended defects and point defects, are reported in epi-GaN. Alloying of GaN with In and Al to produce ternary nitride drives the synthesis of materials whose bandgap vary from one end of solar spectrum to another, of which InN is considered for prospective applications in high efficiency solar cells. Mechanical properties play a very vital role during device fabrication.

An undoped epi-layer of $6 \mu\text{m}$ thickness (0001) GaN/ Al_2O_3 grown by MOCVD with threading dislocation $< 5 \times 10^8 \text{ cm}^{-2}$ along with intrinsic carrier concentration of $\sim 3 \times 10^{17} \text{ cm}^{-3}$ is used in this study. InN films were grown on different orientations of r-plane (1-102), and c-plane (0001)/ Al_2O_3 (sapphire) substrates using plasma assisted molecular beam epitaxy. These layers, grown on r-plane Al_2O_3 without and with substrate nitridation pretreatment, initiated growth of heteroepitaxial semi-polar s-plane (1-101) InN // (1-102) Al_2O_3 and non-polar a-plane (11-20) InN // (1-102) Al_2O_3 oriented InN films. Nanohardness tester with a calibrated Berkovich diamond indenter with tip diameter $< 100 \text{ nm}$ was used for evaluation of mechanical properties. Excitation wavelength of 632.8 nm is used for the micro-Raman spectroscopic studies in the back scattering geometry.

Typical loading-unloading curves are shown (Figure 1) for 50 mN load at a loading-unloading rate of 10 mN.s^{-1} . In both cases, 'pop-in' burst in the loading line (encircled in Figure 1) as well as at the apex is noticed. The mechanism responsible appears to be associated with the nucleation and movement of dislocation sources including lattice atoms for the possible crystallization process.

The E_2 (high) mode at 570 cm^{-1} (Figure 2a), outside the indented region resembles the reported value of epi-GaN. Raman area mapping (out set Figure 2a) using spectral part of $568\text{--}573 \text{ cm}^{-1}$ and $562\text{--}568 \text{ cm}^{-1}$ shows red and green regions lying outside and inside, respectively. It clearly shows that the 567 cm^{-1} signal originates from the stress free region inside the indented mark and the 570 cm^{-1} peak originates from the stressed region. The phonons (Figure 2b) corresponding to the E_2 (high), TO modes at 531 and 559 cm^{-1} , belonging to A_1 and E_1 symmetries, are confined mostly to the XY plane (considering all possible scattering configurations in the indented 'V' grooved region). It is known that defects in GaN propagate in the planar direction. The value of strain rate exponent, m (Figure 3a) and activation volume, VA (Figure 3b) are calculated as ~ 0.26 and $0.3b^3$, respectively. The experimental value of $VA \sim 0.7b^3$ considering Ga and N vacancies, is also comparable. The release of stress takes place by dislocation climb due to high hydrostatic stress present in the plastic volume. Vacancy migration is found to equilibrate the recrystallization process by limiting the dislocation motion.

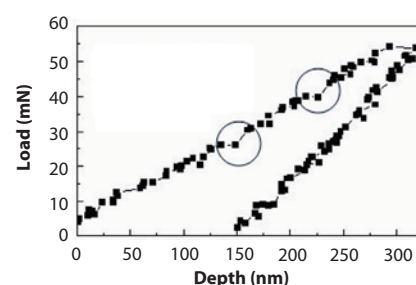


Fig.1: Typical loading-unloading lines at a fixed loading-unloading rate of 10 mN.s^{-1} for 50 mN load

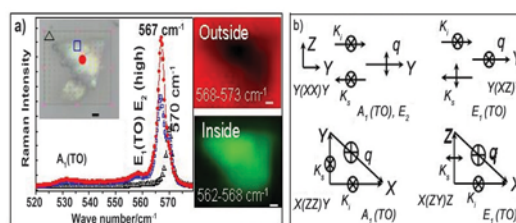


Fig. 2: a) Micro-Raman spectra for epi-GaN. Area mappings of outside and inside of the indentation spot, using different spectral regions indicated in the picture, are shown at the outset. Scale bar is $1 \mu\text{m}$. b) Schematic of different Raman scattering configuration for WZ crystal

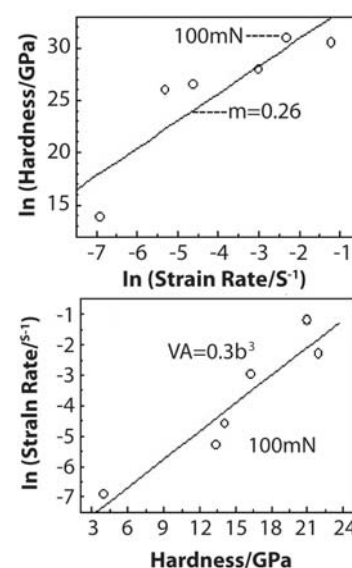


Fig.3: a) log-log plot of hardness vs strain rate, (b) log (indentation strain rate) vs hardness for a load at 100 mN

ADDITIONAL INFORMATION ABOUT NITRIDE SEMICONDUCTORS

Nitride semiconductors are ionic in bonding and grows normally in wurtzite phase. They are chemically inert and have excellent thermal conductivity. Thus, these materials are useful for high power and high temperature electronics. Wide band gap nitride semiconductors are promising materials for room temperature 'spintronic' applications with suitable doping. Recently, InN thin films are also studied for their piezoelectric properties owing to its maximum d_{33} coefficient among group III nitrides.

CRYSTALLOGRAPHIC ORIENTATION INFLUENCING MECHANICAL PROPERTIES

InN sample grown on c-plane Al_2O_3 substrate shows largest hardness values of 8 GPa, which is followed by semi-polar InN ~ 5.5 GPa and a lower value ~4 GPa are recorded for non-polar InN. These results can be understood based on Peierls force (F_{Peierls}) required to initiate the dislocations in a particular crystalline direction. F_{Peierls} decreases with increasing distance between atomic planes (d). Since the distance between planes increases with planar density, slip of the dislocation (related to Burgers vector ' b ') is preferred on closely packed planes. So, the F_{Peierls} has a definite relation with d/b ratio of the slip plane system, which can be used for studying crystalline orientation dependent elastic and plastic (hardness) properties in thin film adhered to substrate.

$$F_{\text{Peierls}} = 2bG_{\text{InN-Al}_2\text{O}_3} \frac{1-\nu \cos^2 \chi}{1-\nu} \omega \exp\left[-2\pi \frac{d}{b} \frac{1-\nu \cos^2 \chi}{1-\nu} \omega\right];$$

$$\omega = \exp\left[4\pi^2/5(nkT/G_{\text{InN-Al}_2\text{O}_3} \nu)\right].$$

In the calculations of F_{Peierls} (Figure 4) the maximum penetration depth per unit load for different samples is considered as l in this calculation. Inset in Figure 4 shows the variations of F_{Peierls} for different χ values as a measure of confidence in this calculation. In case of homoepitaxy (c-plane oriented InN) the ratio of the lattice spacing are used for the InN- Al_2O_3 system. However, lattice spacing of slip plane alone is used for heteroepitaxial cases. An increasingly large F_{Peierls} is observed in a-type, s-type and c-type slip systems with a large Burgers vector. Thus for the c-plane InN film, a large F_{Peierls} is required to initiate plastic deformation and hence the hardness is maximum in this case, followed by s-plane and a-plane samples. The hardness value for c-plane oriented sample is close to the calculated value of ~8 GPa.

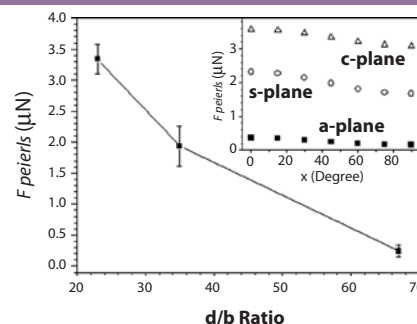


Fig. 4: Peierls force (F_{Peierls}) as a function of the ratio of slip plane separation d and the length of the Burgers vector for the InN- Al_2O_3 slip systems in this experiment. The line for a guide to eye. Inset shows the variation of F_{Peierls} with the angle between the Burgers vector and the line direction of the threading segment (χ) as measure of confidence in the estimation for different slip systems, as inscribed

BRIEF DESCRIPTION

We address an important issue of recrystallization process in GaN under indentation stress. Heterogeneous nucleation of dislocation, originated by shear stress within the plastic zone, is evidenced in the indentation process. The release of stress takes place by climb of dislocation due to high hydrostatic stress present in the plastic volume. A planar defect migration mechanism is evolved. A pivotal role of vacancy migration is pointed out, for the first time, as the rate limiting factor for the dislocation dynamics initiating the recrystallization process in GaN. Higher value of Peierls force in the homoepitaxial InN film, initiating plastic deformation, plays a major role for the maximum observed value of hardness than those for heteroepitaxial InN films.

ACHIEVEMENT

We provide a first atomistic model of recrystallization and defect dynamics in GaN using combined nanoindentation and Raman scattering studies. The present study implies important clues for reducing residual stress in a epi-GaN. A first quantitative study is performed to correlate mechanical properties with crystal orientation of InN with far reach implications of predicting mechanical properties for thin films. Part of this work is also appeared in 'Banyan Express@NCKU' publication as the editor's choice <http://research.ncku.edu.tw/re/reck/e/20081107/home.html>.

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1. S. Dhara, C. R. Das, H. C. Hsu, Baldev Raj, A. K. Bhaduri, S. K. Albert, L. C. Chen, K. H. Chen and Ayan Ray, "Recrystallization of epitaxial GaN under indentation", Appl. Phys. Lett. 92 (2008) 143114.
2. C. R. Das, S. Dhara, H. C. Hsu, L. C. Chen, Y. R. Jeng, A. K. Bhaduri, Baldev Raj, K. H. Chen, S. K. Albert, "Mechanism of recrystallization process in epitaxial GaN under dynamic stress field : Atomistic origin of planar defect formation", J. Raman Spectroscopy 40 (12) (2009) 1881.
3. S. Kataria, T. W. Liu, C. L. Hsiao, S. Dhara, L. C. Chen, K. H. Chen, S. Dash and A. K. Tyagi, "Growth Orientation Dependent Hardness for Epitaxial Wurtzite InN Films", Journal of Nanoscience and Nanotechnology, 10 (2010) 5170.

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Studies on Single Crystal Structure at Low Temperature using Laue Camera

EXECUTIVE SUMMARY

Facility for structure characterization of single crystals both at room temperature and low temperature has been set up in XS and CGS, CMPD, MSG. Laue patterns can be obtained right from 300 K down to 90 K using the Laue camera and liquid nitrogen cryojet cooler. Laue patterns can be obtained both in transmitted and back reflected geometry. The Laue camera makes use of imaging plate as area detector for recording the Laue pattern. The imaging plate scanner has been indigenously designed, developed and the data acquisition software also has been developed in-house. This IP based low temperature Laue diffraction facility is unique in India.

OUTLINE

The Laue camera along with low temperature crystal cooler is a unique facility for the study of single crystals as a function of temperature to track phase transition and lattice parameter variation. The symmetry changes associated with phase transformations can be easily studied from the two dimensional Laue patterns. Using the Laue camera and the low temperature CRYOJET cooler (Oxford Instruments, UK), Laue patterns of single crystals can be obtained from room temperature to 90K in steps of 1K, using a jet of cool nitrogen gas to produce the low temperature, along with a shroud of room temperature nitrogen gas sprayed on the single crystal, to prevent ice formation on the sample. A cryocan, housing the heater and transfer tube is used to pump the cold nitrogen gas. The sample to be cooled is placed on a goniometer located within 5mm from the tip of the cryojet. The temperature at the sample position has been independently calibrated using a Cr/Al thermocouple and the temperature stability found to be within ± 1 K. The Laue patterns were recorded using an Imaging plate as area detector as it drastically reduces the data acquisition time and also permits convenient pattern analysis and interpretation.

Low temperature Laue patterns were obtained on a single crystal of BaFe_2As_2 , which crystallizes in tetragonal structure (Space Group: $I4/mmm$ from room temperature to 90K. Laue pattern, recorded at room temperature clearly exhibits a four fold symmetry, corresponding to the (001) zone axis. The transmitted Laue pattern at 300, 120, 100 and 95 K are shown in Figure 1. In the temperature range 300 to 140 K, Laue patterns did not show any change in their symmetry pattern, indicating no structural transformation in this temperature range. The patterns recorded from 120K to 90 K, however did show perceptible changes in the transmitted patterns indicating a possible structural transformation over and above the lattice contraction.

As an extension, Laue studies on thin films were also undertaken. To overcome the problem of recording diffraction patterns from substrate, Laue pattern of the thin films were recorded at a grazing incidence of 1.5° , in addition to normal incidence mode. Figure 2 (top plane) shows both the transmitted and back reflected Laue patterns at the normal incidence. Bottom plane shows the corresponding patterns at an grazing incidence of 1.5° wherein thin film and substrate contributions could be clearly distinguished. The grazing incident transmitted Laue pattern has been analyzed using the freeware "Orient Express" programme. Using this programme, Laue diffraction pattern of the substrate was simulated to match the substrate pattern. After suitably subtracting the substrate contribution, thin film Laue pattern can be extracted and analyzed.

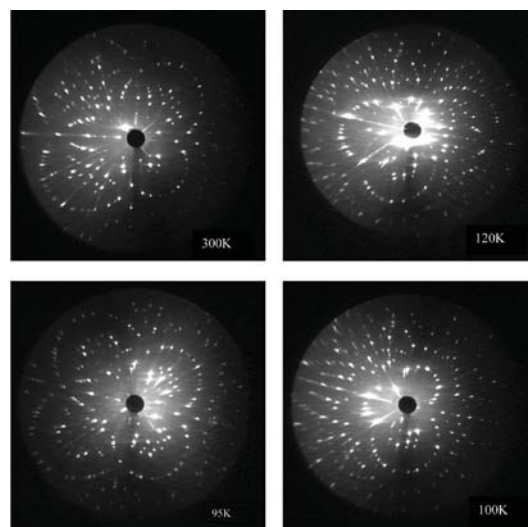


Fig.1: Transmitted Laue diffraction pattern of BaFe_2As_2 single crystal at various temperature

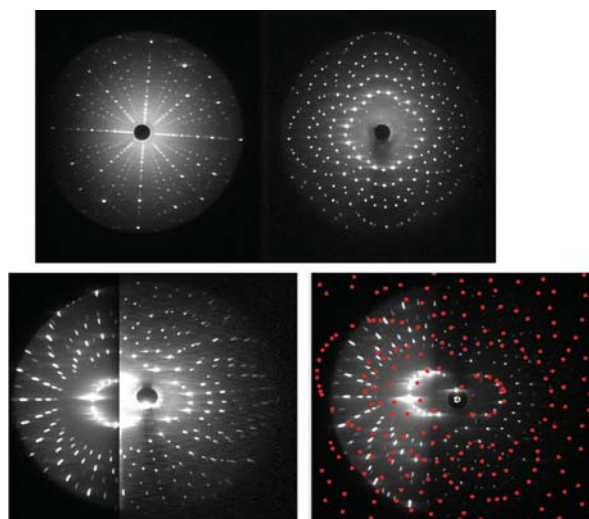


Fig. 2: Normal incidence (top) and grazing incidence (bottom) transmitted Laue pattern of $\text{FeSe}_{0.5}\text{Te}_{0.5}$ on LaAlO_3 substrate and with a simulated pattern shown in red. The bottom left pattern has been selectively enhanced to bring out the weak Laue spots

ADDITIONAL INFORMATION

Laue X-ray diffraction is a method for structural characterization of the crystals and is the first step in assessing the crystal quality. In this method, a beam of white radiation, the continuous spectrum from an X-ray tube, falls on a fixed single crystal. The Bragg angle is fixed for every set of planes in the crystal, and each set selects and diffracts that particular wavelength which satisfies Bragg's law. The Laue pattern is a direct map of the reciprocal space of the crystal. A single Laue diffraction spot contains reflection from a set of parallel planes with different d/n , where d is the interplanar distance and n an integer.

GENERAL DESCRIPTION OF LAUE CAMERA AND IMAGING PLATE

The Laue camera has two circular cassettes in which the imaging plate is loaded for recording the Laue pattern. The crystal is placed in between the cassettes and the X-ray beam is made to focus on the crystal passing through the centre of the cassettes with the help of a collimator (Figure 3). An X-ray tube with desired target and without any filter is used for this purpose. The imaging plate used is a photosensitive device, made up of fine powder of crystalline BaFBr:Eu^{2+} mixed with an organic binder deposited on to a flexible polymeric sheet. Upon absorption of ionizing radiation electron hole pairs are produced in the imaging plate and are trapped in the immediate vicinity where the ionizing radiation falls thus forming a latent image. The latent image is stable until read with a laser. On irradiating with an laser the trapped electron hole pair recombine and emit blue light of Eu^{2+} . The generation of electron hole pair involves excitation of electron from valence band to conduction band and since the band gap of BaFBr:Eu^{2+} is of the order of 8 eV no thermal generation of defect pair is possible at room temperature. Therefore the imaging plate can be exposed to X-ray over long time period without acquiring thermal background. Imaging plate has more advantages than conventional X-ray detectors, they are ultra high sensitive, have wider dynamic range, superior linearity, higher spatial resolution, permit digital data conversion and are reusable.

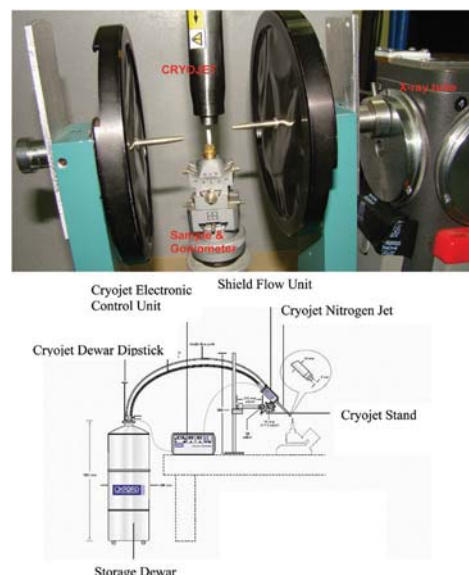


Fig. 3: Low Temperature Laue setup showing Laue camera, goniometer and cryojet and the schematic of the setup

IMAGING PLATE SCANNER

The imaging plate (IP) reader has been designed and developed indigenously. The imaging plate scanner is designed to scan an imaging plate of active area 250 X 200 millimeter. In the IP scanner we have used a focusable diode laser of 10 mW power and 635 nanometer wavelength. The focused spot size of the laser is 100 μm and is positioned on the imaging plate using a mirror inclined at 45° to the laser. When the laser light falls on the imaging plate photo stimulated luminescence (PSL) emission occurs, the emitted light is collected by a PMT. The optical assembly allows only the 390 nanometer light and filters the 635 nanometer light. The PSL intensity is converted in to digital data and image is constructed using the data. In the conventional Laue camera photographic film is used as an area detector. Here we have replaced this photographic film by an imaging plate. The exposed imaging plate is scanned by an imaging plate reader and gives the image in the digital form. Hence analysis of the diffraction pattern becomes easy.

ACHIEVEMENT

Laue diffraction characterization of single crystal is very important in determining orientation, symmetry, defects like twinning etc. Low temperature Laue patterns can be used to find phase transformation in single crystal as a function of temperature. Physical properties of the crystal are dependent on the crystallographic orientation thus determining the crystal orientation by and far determine the use of the particular crystal for a specific purpose. The Laue camera using the Imaging plate for data acquisition is a unique facility and the imaging plate scanner, used to read the imaging plate and the necessary data acquisition software for this has been developed in house and its worthiness has been proved. Using this low temperature Laue patterns for BaFe_2As_2 has been recorded up till 90K.

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Electron-Phonon Interaction in ZnO Nanocrystals

EXECUTIVE SUMMARY

Electron-phonon (e-p) interaction is the key parameter in determining the transport properties for device applications of nanostructures. Because of the confinement of charge carriers and the phonons in the nanocrystals, the electron-phonon interaction is modified. Furthermore, this could also be different for phonons of different symmetries. In order to obtain the electron-phonon interaction in the nanocrystals, Raman spectroscopic investigations were carried out on ZnO nanocrystals for various photon energies. Intensities of E_1 -LO and E_2 modes exhibit large changes as the excitation energy varied from 2.41 to 3.815 eV, signifying substantially large contribution of Frohlich interaction to the Raman polarizability as compared to deformation potential close to the resonance. Relative strengths of these two mechanisms is estimated for the first time in nanocrystals and compared with those in the bulk.

OUTLINE

ZnO nanocrystalline powder was synthesized at room temperature by precipitation in ethanol and characterized by X-ray diffraction (XRD) in a (STOE) diffractometer. Raman scattering measurement was carried out using a Spex 14018 double monochromator equipped with a Ar^+ laser and photomultiplier tube (Hamamatsu R943-02). Near-resonant Raman scattering measurements were performed using 325 nm line of He-Cd laser as the excitation and analysed using a double-subtractive triple-monochromator (Jovin-Yvon T64000), equipped with liquid nitrogen cooled CCD detector for spectra recording. The XRD pattern (Figure 1) of as-synthesized samples agrees with the wurtzite structure. The peak broadening suggests its nanocrystalline nature. The average particle sizes calculated, using the Debye-Scherrer formula, were ~ 7.7 nm.

Figure 2 shows Raman spectra of ZnO nanoparticle, excited using 514.5, 488 and 457.9 nm lines of a Ar^+ laser and 325 nm line of He-Cd laser. The spectra exhibit E_2 and E_1 -LO modes at 438 and 582 cm^{-1} respectively. When excited under near-resonant condition (325 nm), E_1 -LO mode exhibits overtone spectra (inset Figure 2) up to 4-LO modes. The enhancement due to resonance can be explained based on a third-order perturbation calculation for the intensity for Raman scattering. When the incident photon energy varies across an electronic transition, vanishing of energy denominators are expected to result in in- and out-resonances. Resonance Raman scattering in bulk ZnO is well documented, while in nanocrystals it is of current interest.

Figure 3 shows the intensity ratio $I(E_2)/I(E_1\text{-LO})$ as a function of excitation energy. The intensity ratio was analyzed to obtain insight about the e-p interaction. The intensity ratio represents the ratio of the matrix elements corresponding to the scattering mechanisms, i.e.

$$\frac{H_{ep}^{E_2}}{H_{ep}^{E_1\text{-LO}}} = \frac{DP_2}{DP_1 + FI}$$

where $H_{ep}^{E_2}$ and $H_{ep}^{E_1\text{-LO}}$ are the matrix elements for the e-p interaction for E_2 and E_1 -LO phonons respectively. DP and FI represent the deformation potential and the Frohlich mechanisms of the e-p interaction respectively. The subscripts 1 and 2 on DP imply E_1 -LO and E_2 phonons respectively.

It is seen from Figure 3 that the ratio decreases rapidly as a function of incident photon energy. The reported data for other sizes and for the bulk ZnO are also included in the figure for the sake of comparison. Note that the ratio increases as particle size reduces. The present results thus suggest that electron-phonon interaction is enhanced via Frohlich interaction as excitation energy approaches the resonance. Furthermore, a comparison of the ratio of E_2 and E_1 -LO intensities with that of bulk suggests that the electron-phonon interaction for E_1 -LO phonon decreases more rapidly than E_2 phonon with a reduction in size.

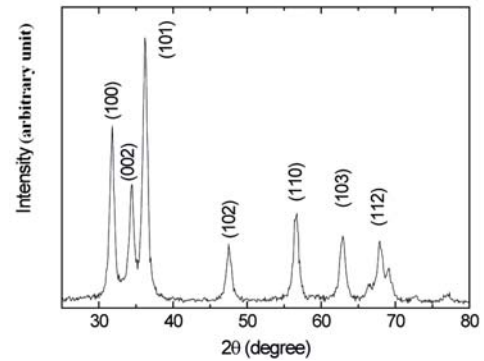


Fig. 1: X-ray diffraction pattern for 7.7 nm ZnO nanoparticles

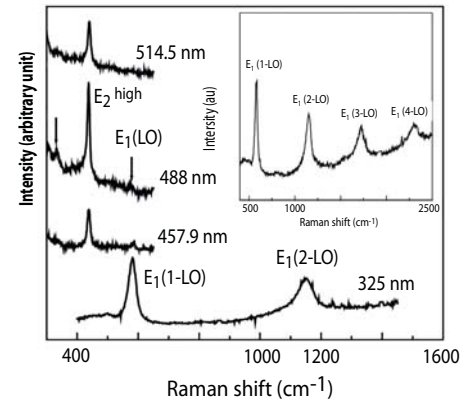


Fig. 2: Raman spectra of 7.7 nm ZnO nanoparticles using different excitation wavelength. The inset shows the overtone spectra for 325 nm excitation

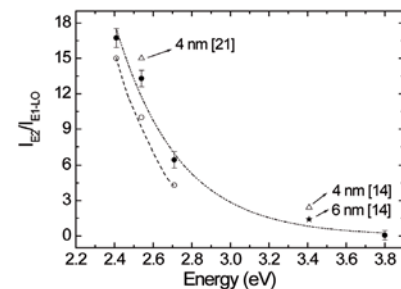


Fig. 3: Intensity ratio as a function of incident photon energy for 7.7 nm ZnO nanocrystals. For comparison, the data for a single crystal is shown as open symbols. Dot-dash curve through the present data and dashed curve through the open circles are guides to the eye

ADDITIONAL INFORMATION ABOUT ZINC OXIDE

Zinc oxide is a large gap semiconductor that belongs to hexagonal wurtzite structure with two formula units per primitive cell. The band-gap of a nanocrystal is blue-shifted as compared to the bulk value of 3.35 eV. The blue shift depends inversely on the diameter of the nanocrystal. Zone-center optical phonons predicted by group theory are $A_1 + 2E_2 + E_1$. In the wurtzite structure, the polar A_1 and E_1 modes split into longitudinal optic and transverse optic components with different frequencies due to macroscopic electric fields associated with the longitudinal optic phonons.

GENERAL DESCRIPTION

There is considerable current interest in zinc oxide nanostructures due to their potential applications in the areas such as photo-detectors, blue and ultraviolet lasers, optoelectronic devices, and radiation hard material. The controlled synthesis of ZnO nanostructures and a complete understanding of the physical properties are the key issues for the development of ZnO-based devices. In this context the electron-phonon interaction directly influences the carrier mobility and consequently determines the transport characteristics of an optoelectronic device. The electron-phonon (e-p) interaction is also responsible for the appearances of overtones of the longitudinal optic (LO) phonons in the resonance Raman spectra. In ZnO the LO phonon can have E_1 and A_1 symmetries; however, A_1 mode exhibits negligible intensity. In view of this often the ratio of the intensity of the first overtone of the E_1 -LO phonon to that of the fundamental is considered as a quantity that is proportional to the magnitude of the e-p interaction. This intensity ratio is found to be smaller in nanoparticles as compared to the bulk. However, the intensity of the Raman scattering from the LO phonons stems from the contributions from two processes: Frohlich interaction and the deformation potential. On the other hand, the Raman intensity of the non-polar phonon such as that of E_2 symmetry arises only from the deformation potential. Thus a study of the relative intensities of the E_1 -LO and E_2 phonons gives an opportunity to evaluate the relative strengths of the two processes. Furthermore, how the e-p interaction gets modified in nanostructures is also of interest.

BRIEF DESCRIPTION

ZnO nanocrystals of about 8 nm diameter were synthesized using precipitation in non-aqueous medium. Raman intensities of E_2 and E_1 -LO phonons were analyzed to obtain insight about electron-phonon interaction in nanocrystalline ZnO. The present results suggest that electron-phonon interaction is enhanced via Frohlich interaction as excitation energy approaches the resonance value. The electron-phonon interactions for phonons of both the symmetries, i.e. E_2 and E_1 -LO, reduce with decreasing particle size. However, a comparison of the ratio of E_2 and E_1 -LO intensities with that of bulk suggests that the electron-phonon interaction for E_1 -LO phonon decreases more rapidly than E_2 phonon with a reduction in size.

ACHIEVEMENT

This is first report of strengths of electron-phonon interaction for phonons of different symmetries in zinc oxide nanocrystals as a function of excitation photon energy. The dependence of the e-p interactions on the crystallite size is also reported.

PUBLICATIONS

S. Sahoo, V. Sivasubramanian, S. Dhara and A. K. Arora, "Excitation energy dependence of electron-phonon interaction in ZnO nanoparticles", Solid State Communication, 147 (2008) 271.

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Atomic Scale Study of Pressure Induced Amorphization in Zirconium Tungstate

EXECUTIVE SUMMARY

Zirconium tungstate is the most important material among the frame work structures which undergo negative thermal expansion (NTE) and pressure induced amorphization (PIA). Zirconium tungstate has a polyhedral network of ZrO_6 and WO_4 , which play an important role for these properties. Local structures of ZrO_6 have been studied in pressure amorphized zirconium tungstate using perturbed angular correlation (PAC), a hyperfine interaction technique. Existence of highly distorted ZrO_6 octahedra which remain stable up to 800 K in amorphous zirconium tungstate and their implications on PIA has been studied.

OUTLINE

Zirconium tungstate ZrW_2O_8 is a cubic compound (Figure 1) consisting of a three dimensional network of corner linked ZrO_6 octahedra and WO_4 tetrahedra, with the latter having one non-bridging W-O bond. This system exhibits negative thermal expansion (NTE) isotropically over the temperature range 4 to 1000 K. Large transverse vibration of the oxygen atom in the middle of the W-O-Zr linkage, which requires corresponding rotations of the polyhedra, is the primary origin of NTE due to unconstrained feature of the structure. This polyhedral structure undergoes PIA. As ZrO_6 is less rigid than more ionic WO_4 , we have carried out $^{181}\text{Hf}/^{181}\text{Ta}$ based Perturbed Angular Correlation (PAC) spectroscopy, a hyperfine technique, to probe local structure at ZrO_6 .

Using PAC Electric Field Gradient (EFG) tensors at the site(s) of occupancy of probe atoms are deduced. Principal component of EFG tensor is proportional to the quadrupole frequency (ν_Q) and inversely proportional to Zr-O bond length. Asymmetry parameter (ratio of relative change of other two components of EFG and the principal component) is sensitive to orientation distortion if any associated with ZrO_6 . PAC measurements were carried out on ZrW_2O_8 quenched from a pressure of 5 GPa, using BaF_2 based three detector, twin fast-slow coincidence spectrometer setup at MSD, at room temperature subsequent to each step of isochronal annealing, with a step time of 30 min in pressure amorphized sample. CCR is used for low temperature measurements.

Time dependent anisotropy spectra $R(t)$ and their Fourier transform $P(\omega)$ obtained at 250 K in crystalline sample is shown in top panel of Figure 2. Analysis of the spectra shows that there are four distinct Zr sites experiencing distinct quadrupole parameters such as quadrupole frequency, asymmetry parameters. The fraction which is predominant at 250 K is interpreted to be due to probe atoms occupying regular ZrO_6 . Earlier studies have shown that the fraction of probe atoms associated with regular octahedra decreases at the cost of other fractions at increasing temperatures, highlighting the role of contracted and distorted octahedra for NTE.

TDPAC spectra and their Fourier transforms for amorphized sample are shown in Figure 2. The spectra is non-vanishing though dampened implying the degree of non-uniqueness in the local structures of the octahedra due to distribution in bond lengths and bond angles. The short range ordering can thus be explained in terms of random network of structural units. The spectra could be fitted with six components for annealing treatments below 800 K. A high value of asymmetry parameter 0.4-0.7 of each of these fractions imply an appreciable distortion of these distinct ZrO_6 octahedra as occupied by the probe atoms. Variation of quadrupole frequencies with isochronal annealing temperature is shown in Figure 3. It is seen that below 875 K, apart from ν_{Q6} which exhibit a small change, all other quadrupole frequencies remain constant. Subsequent to annealing at 875 K ν_{Q1} and ν_{Q2} exhibit an increase while ν_{Q3} , ν_{Q4} and ν_{Q5} decrease. Beyond 875 K the values of ν_{Q1} , ν_{Q2} , ν_{Q3} and ν_{Q4} tend towards those of the untreated crystalline sample thus marking the onset temperature of the amorphous to crystalline transition in pressure amorphized ZrW_2O_8 .

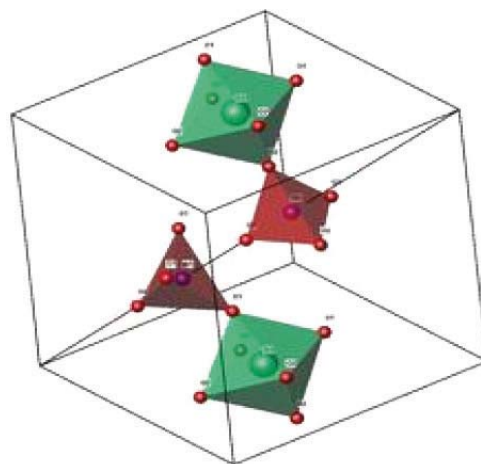


Fig. 1: Crystal structure of Zirconium Tungstate

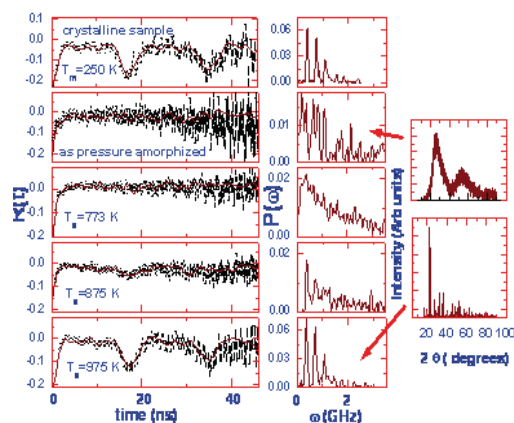


Fig. 2: Representative TDPAC spectra and their Fourier transforms obtained in crystalline and amorphous zirconium tungstate. T_m refer to temperatures of measurement in crystalline sample and annealing while T_a refers to isochronal annealing temperature of amorphized sample. Shown on the right are the XRD spectra of amorphized and recrystallised sample

NEGATIVE THERMAL EXPANSION AND PRESSURE INDUCED AMORPHIZATION IN ZIRCONIUM TUNGSTATE

Unusual property of contraction of volume on heating (Negative thermal expansion) is seen in some oxides, where the contraction is small and anisotropic and occurs only over a small temperature range. But Zirconium tungstate exhibits isotropic negative thermal expansion over a large temperature interval 4 to 1000 K. This article is about understanding negative thermal expansion in ZrW_2O_8 by studying the variation of quadrupole parameters associated at Zr sites of ZrO_6 using Perturbed Angular Correlation (PAC) spectroscopy.

GENERAL INFORMATION

Complete restoration of quadrupole parameters to those of untreated crystalline sample is seen subsequent to annealing at 975 K (Figure 2 and 3) commensurate with the bulk amorphous to crystalline transition as deduced by XRD measurements. Invariance of quadrupole frequencies below 800 K points to the fact that the terminal oxygen of WO_4 has got bonded with the other tetrahedra leading to frozen in configurations of ZrO_6 up to 800 K as rotational and/or translational motion of polyhedra are coupled. An overall increase in the dampening of the spectra and its Fourier transform subsequent to annealing at 775 K implies the tendency for relaxation of the frozen configurations at this temperature.

There are no additional low frequency components appearing in the pressure amorphized sample that are not present in equilibrium measurements carried out in crystalline ZrW_2O_8 at different temperatures. This observation indicates that there may not be any increase in the coordination number of Zr. Restoration of quadrupole parameters as that of the untreated crystalline sample for annealing beyond 950 K imply that there might not be any breaking of bonds due to amorphization.

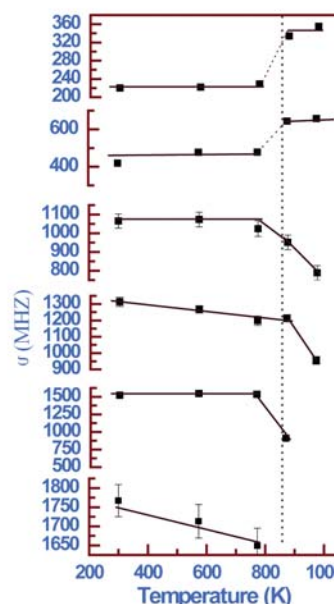


Fig. 3: Variation of quadrupole frequencies (associated with different fractions in amorphized sample) with annealing temperature. Dotted vertical line shows the temperature beyond which the values of quadrupole frequencies tend to become close to those of untreated crystalline sample

BRIEF DESCRIPTION

This atomic scale study in ZrW_2O_8 shows the existence of highly distorted octahedra in the pressure amorphized material. Invariance of quadrupole parameters in the amorphized sample also implies that the structures of these distorted octahedra remain stable up to 800 K. This is understood to be caused by the locking of polyhedra due to bonding of terminal oxygen of WO_4 with other tetrahedra. The present study also suggests that there may not be an increase in the coordination number of Zr atoms in pressure amorphized sample. The onset of amorphous to crystalline transition occurs around 870 K while the restoration of quadrupole parameters to those of the untreated sample is observed subsequent to annealing at 975 K.

ACHIEVEMENT

Evolution of local structures of ZrO_6 octahedra in pressure amorphized zirconium tungstate sample has been studied with isochronal annealing treatments. Observed configurations of ZrO_6 were seen to be stable up to 800 K which is understood to be due to bonding of terminal oxygen of WO_4 with other tetrahedra.

PUBLICATIONS

R. Govindaraj, C. S. Sundar and A. K. Arora, "Studies of local structure at Zr sites in pressure amorphized zirconium tungstate", J. Phys. Condens. Matter 21 (2009) 235402.

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Phase Stability in Nanostructured Alumina / Zirconia Multilayer Thin Films

EXECUTIVE SUMMARY

Phase stability of nanostructured materials can be significantly different from that of the same materials in bulk form. ZrO_2 is a technologically important material and is required in the tetragonal form for most high temperature applications. Apart from crystallite size control, a constraining effect of alumina layers in an alumina / zirconia multilayer system stabilises the tetragonal phase at a thickness greater than critical thickness. However, at high-temperatures inter-mixing occurs that results in an amorphous phase. Conventional techniques are inadequate to detect such changes at the nanometer scale; high-resolution TEM techniques have been successful in detecting and characterising these phase transformations. Present work deals with the study of phase stability in alumina / zirconia multilayers prepared by pulsed laser ablation.

OUTLINE

The nanostructured alumina/zirconia multilayered system is of interest for the development of functionally graded thermal barrier coating having high wear and oxidation resistance. In this article we demonstrate the combined effect of alumina addition and its nanoscale layer thickness on the stability of tetragonal zirconia down to room temperature, necessary for high temperature applications.

Alumina/zirconia multilayers were synthesised by pulsed laser deposition (PLD) (parameters listed in Table 1). Alternate nanostructured layers of Al_2O_3 and ZrO_2 were deposited on Si (100) single-crystal substrates at room temperature. One such sample was in situ heat-treated in a high-temperature X-ray diffractometer up to 1473 K under argon at a pressure of 2×10^{-5} mbar. HRTEM studies were carried out on cross-sectional TEM (XTEM) specimens of the as-deposited and heat treated multilayer samples in a JEOL 2000 EX II (T) transmission electron microscope operated at 200 kV.

The present studies show that the as-deposited multilayers are well-defined, of uniform thickness over large distances, with flat interfaces between the two phases (Figure 1(a)). X-ray diffraction studies indicated that the ZrO_2 layers were monoclinic while no peaks corresponding to Al_2O_3 were detected. Selected area electron diffraction (SAD) patterns recorded from the multilayers confirmed the presence of a mixture of large amount of monoclinic phase and a small amount of tetragonal phase in the Al_2O_3 layers, while the Al_2O_3 layers were seen to be amorphous. Figure 1(b) shows a phase-contrast lattice image from a region of the XTEM sample of as-deposited multilayers. Both tetragonal and amorphous regions have been identified in the central, darker region, showing lattice contrast, while the lighter regions on either side are amorphous alumina layers. On annealing the multilayer at 1473 K, it was seen that the ZrO_2 layer is no longer continuous and there are regions where it is amorphised (Figure 2). A majority of the diffraction rings in the SAD patterns could be indexed uniquely to tetragonal ZrO_2 while a small fraction was monoclinic. Distinction between alumina and zirconia was less clear in the regions where the zirconia has amorphised. Power Spectra from regions marked on the lattice image are shown alongside in Figure 2. Indexing such power spectra allows phase identification from regions as small as 3 – 5 nm.

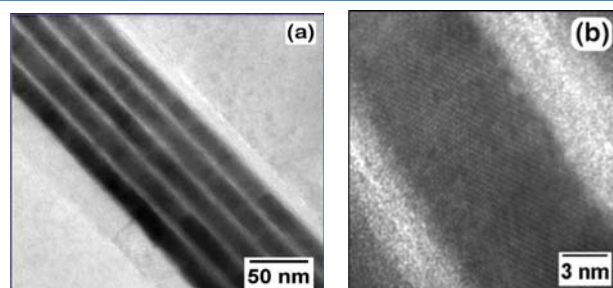


Fig.1: (a) Low magnification bright-field image showing multilayers of Al_2O_3 and ZrO_2 . Dark bands correspond to ZrO_2 while bright bands in between are Al_2O_3 layers, (b) Lattice image of a typical region of the as-deposited multilayers. The Al_2O_3 layers are amorphous while crystalline regions have been identified as monoclinic ZrO_2 layers

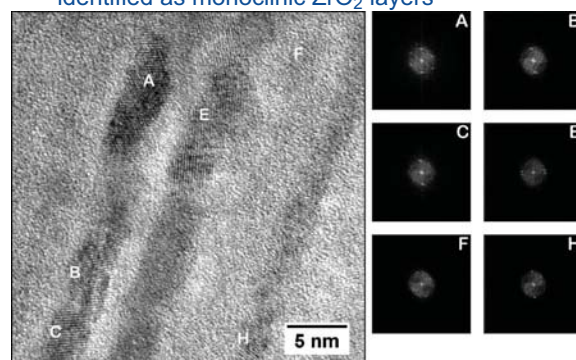


Fig. 2: High-resolution lattice image of Al_2O_3 - ZrO_2 multilayers. Lattice planes of ZrO_2 alone are imaged since the Al_2O_3 layers are amorphous. The layer boundaries are not distinct. Power Spectra from regions labelled A – H on the HRTEM image, shown alongside were used for phase identification from regions that are 3 – 5 nm wide

Table 1: Pulsed Laser Deposition Parameters

Laser	KrF Excimer
Wavelength	248 nm
Energy	300 mJ/pulse
Pulse rate	10 Hz
Targets	ZrO_2 , Al_2O_3
Substrate	Si (100)
Temperature	500 °C
pO_2	3×10^{-5} mbar

ADDITIONAL INFORMATION

Synthesis of ZrO_2 / Al_2O_3 multilayers: ZrO_2 and Al_2O_3 targets were synthesized by powder compaction and sintering to form pellets of ~25 mm diameter and ~3 mm thickness. Cleaned Si (100) substrates of dimension 10mm x 10mm x 0.5mm (N-type phosphorous doped with a resistivity of 20-30 Ω -cm) were mounted onto a sample holder using silver paste. PLD experiments were performed with a KrF excimer laser under the conditions listed in Table 1. Multilayers consisting of ZrO_2 and Al_2O_3 layers of thickness 15 nm and 5 nm respectively were deposited. Totally, eighty layers were deposited. PLD exhibits several advantages for the processing of Al_2O_3 / ZrO_2 thin films.

GENERAL INFORMATION

ZrO_2 is a technologically important material with applications ranging from thermal barrier or wear resistant coatings to use as radioactive waste immobilisation matrix. Pure ZrO_2 exhibits three structural modifications of which the tetragonal (t- ZrO_2) phase that is stable at 1443–2643 K is important for most technological applications. The tetragonal to monoclinic transformation at around 1443 K is accompanied by a significant volume change, which in constrained systems such as thin-film multilayers and coatings can result in considerable residual stress leading to damage of the coating. Hence it is necessary to stabilise the tetragonal phase of zirconia down to room temperature. Two approaches can be adopted for this purpose. Doping zirconia with calcia or alumina or by controlling the individual crystallite size to nanometre range can stabilise the high temperature tetragonal zirconia phase to lower temperatures. In the case of zirconia, it has been reported that controlling the crystallite size to below ~ 6 nm results in the stabilisation of the tetragonal phase down to room temperature.

BRIEF DESCRIPTION

This study on characterisation of high temperature annealed multilayer of zirconia/ alumina using HRTEM has shown the amorphous nature of alumina layers and crystalline regions of the zirconia layers which are almost completely transformed to the tetragonal structure with very few regions of monoclinic zirconia. While in most part the multilayer structures are still evident, the zirconia layers are seen to have become discontinuous at high temperatures, with regions of an amorphous phase separating regions of crystalline zirconia.

ACHIEVEMENT

ZrO_2 is a technologically important material with applications ranging from thermal barrier or wear resistant coatings to use as radioactive waste immobilisation matrix. Pure ZrO_2 exhibits three structural modifications of which the tetragonal (t- ZrO_2) phase that (stable at 1443–2643 K) is important for most technological applications. The tetragonal to monoclinic transformation at around 1443 K is accompanied by a significant volume change, which in constrained systems such as thin-film multilayers and coatings can result in considerable residual stress leading to damage of the coating. Hence it is necessary to stabilise the tetragonal phase of zirconia down to room temperature. Two approaches can be adopted for this purpose. Doping zirconia with calcia or alumina or by controlling the individual crystallite size to nanometre range can stabilise the high temperature tetragonal zirconia phase to lower temperatures. In the case of zirconia, it has been reported that controlling the crystallite size to below ~ 6 nm results in the stabilisation of the tetragonal phase down to room temperature.

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2. P.Kuppusami, G. Balakrishnan, S. Murugesan, E. Mohandas and D. Sastikumar, "Phase Evolution and Thermal Stability of Zirconia /Alumina Multilayer Thin films Prepared by Pulsed Laser Deposition" , National Conference on Emerging Areas in Thin Film Science and Technology, Focal Theme : MEMS and Nanotechnology, P S G College of Technology, 2009.

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Extinction of Light in a Disordered Medium

EXECUTIVE SUMMARY

We observe magnetic field induced extinction of light in Mie regime using a magnetic nanofluid containing Fe_3O_4 nanoparticles of 6.7 nanometer. At a critical applied magnetic field (H_{C2}), the transmitted light intensity becomes extinct where the H_{C2} follows a power law decay with volume fraction of nanoparticles indicating a structural transition. The light transmission was reversible with the magnetic field. We discuss various possible mechanisms responsible for the extinction of light under external magnetic field. The experimental finding may have significant application in tunable light controlling devices.

OUTLINE

Scattering and diffraction of light in magnetic suspensions, emulsions and mixtures of magnetic and nonmagnetic scatterers have been a topic of interest both theoretically and experimentally. Though there have been a few studies on scattering of light in magnetic scatterers, in the size limit of $a \gg \lambda$, scattering in the limit $a \ll \lambda$ (where a is the scatter size and λ is the wavelength of light) is still an unexplored area, probably due to the practical limitations in tailoring stable and monodispersed suspensions. Similarly, the exact mechanism responsible for the intense scattering or extinction of light in magnetic scatterers remains unclear and controversial. This was the main motivation to undertake the study. We investigate the effect of applied magnetic field on transmitted light intensity in a magnetically disordered medium, where the suspended particle size $a \ll \lambda$ (Mie regime).

We used a stable colloidal suspension of magnetite (Fe_3O_4) nanoparticles of average diameter 6.7nm, coated with oleic acid and dispersed in kerosene. The organic layer thickness around the particles is about 1.5nm. The suspensions used in our experiments had excellent long term stability with no agglomerations even after prolonged application of very strong magnetic fields. We measure both the forward and backward scattered light intensity as a function of applied magnetic field.

Figure 1 shows the normalized transmitted intensity as a function of external field for different magnetite volume fractions. The diffraction pattern at different magnetic field strength is also shown in Figure 1. The intensity was the ratio of the transmitted intensity through the sample with and without magnetic field. At lower concentrations, the transmitted light intensity remains constant upto a critical magnetic field (H_{C1} , shown by the dotted arrow) and above which the intensity starts to decrease drastically. At another critical magnetic field called (H_{C2} , shown by the solid arrow), the transmitted light intensity becomes a minimum. Above H_{C2} , the transmitted light intensity increases slowly.

Figure 2 shows the variation of the critical fields H_{C1} and H_{C2} with volume fraction of the nanoparticles. Both H_{C1} and H_{C2} follow power law decay with volume fraction ($H_C \propto \Phi^{-x}$) where the exponents are 0.423 and 0.283 respectively. The scaling analysis also predicts such power law dependence where exponents can vary between 0.25 and 0.75 for structural transitions under magnetic field. Also, with increasing concentrations, the slope of the transmitted intensity curve (between H_{C1} and H_{C2}) increases. At higher particle concentrations above 0.0176 vol., the transmitted light intensity increases slightly and then decreases with increasing in magnetic field. The experiments with light beam circularly polarized, linearly polarized with parallel and perpendicular to the field direction also gave similar results.

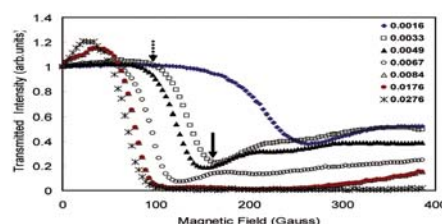


Fig. 1: The forward scattered light intensity as a function of applied magnetic field in magnetite based ferrofluid at different volume fractions. The average particle diameter was 6.7nm. The diffraction patterns at different magnetic field strength are also shown

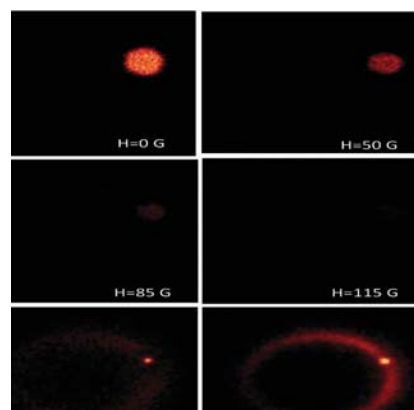


Fig. 2: The forward scattered patterns from ferrofluids of 0.0067 volume fraction at various magnetic fields (a) 0, (b) 50 (c) 115 (d) 140 and (e) 200 Gauss. The transmitted light intensity starts to decrease above 50G and is fully extinct at 115G, above which the forward scattered light shows a

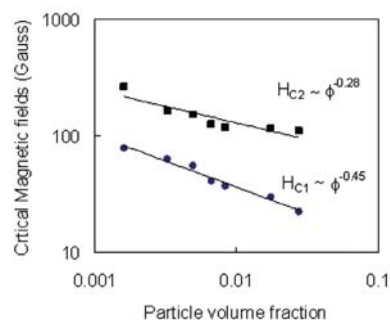


Fig.3: The critical magnetic fields (H_{C1} and H_{C2}) as a function of magnetite volume fraction. The critical fields H_{C1} and H_{C2} follow power law decay with volume fraction ($H_C \propto \Phi^{-x}$) where the exponents are 0.423 and 0.283 respectively, without and with magnetic field

ADDITIONAL INFORMATION

The ferrofluid sample is taken in a quartz cuvette and kept inside a solenoid, where magnetic field is varied by changing the current passing through its coil using a DC programmable power supply. The direction of magnetic field was parallel to the wave propagation. An amplitude and frequency stabilized polarized He-Ne laser (Spectra-physics) of wavelength 632.8 nanometer with an output power 1 mW is used as a light source. The light intensity is measured by using a photomultiplier tube (PMT). The output of the photomultiplier tube is fed to readout through a current amplifier with variable gain. The analog output from the readout is connected to a 12 bit analog to digital converter (ADC) that is interfaced with a computer.

GENERAL INFORMATION MEDICAL INFRARED THERMAL IMAGING

Magnetically polarizable fluids are easy to manipulate with applied magnetic field that makes them ideal candidates for many practical applications such as optical switches, modulators, filters, gratings etc. Scattering of light from disordered and turbid media can lead several fascinating phenomena. On increasing the amount of scattering beyond a critical value, the material undergoes a transition into a localized state as a result of interference of all the scattered waves. The weak localization of electromagnetic wave leads to a coherent enhancement of light in the backward direction, which has been experimentally observed in several systems such as dense latex microspheres, highly concentrated aqueous suspension of polystyrene particles, submicron sized SiO₂ beads in air, semiconductor microcavity etc. The first observation of strong modification of speckle patterns generated by diffusive transport of laser light through turbid granular samples under a strong magnetic field, that was modeled as random walk of field induced polarization changes, triggered further research. Also, optical limiting nature of colloidal suspension of particles under non-linear scattering due to photoinduced refractive index matching and magneto-optical correlation length in turbid media have been probed. Christiansen showed that a dispersion of powder in a liquid can act as an optical band pass filter when the dispersion curves of both media coincide at one wavelength.

BRIEF DESCRIPTION

What is the origin of the ring like patterns formed above the HC₁? The scattered wave from a long cylinder (length $\gg$ radius) forms conic sections on a screen placed perpendicular to the incident beam. The formation of the ring clearly establishes the formation of such rod like structures along the direction of propagation of the light wave above Hc₂, which is the coupling constant. The diffused ring at lower magnetic fields indicates the distorted columns due to weak magnetic dipolar attraction. As the magnetic field increases, both the average chain length and the chain number increases. The Rayleigh-Gans scattering intensity for such cylindrical scatterers should increase with the square of column length. At higher fields, the ring becomes sharper indicating the formation of smooth surfaced columns as evident from the hexagonal symmetry.

ACHIEVEMENT

Our results confirm the existence of magnetically tunable extinction of light by a dilute suspensions (~3 vol. %) of disordered magnetic media, in the size limit $a \ll \lambda$. The plausible reason for the extinction of light is the occurrence of dipole resonances within the scatters and the subsequent building up of standing waves inside the scatters that delay the light propagation. Our finding may have significant application in tunable light controlling devices.

PUBLICATIONS

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3. J.M.Laskar, J. Philip, and Baldev Raj, "Experimental evidence for reversible zippering of chains in magnetic nanofluids under external magnetic fields", Phys. Rev. E 80 (2009) 041401.
4. J. M. Laskar, J. Philip and Baldev Raj, "Experimental investigation of magnetic-field-induced aggregation kinetics in nonaqueous ferrofluids", Phys. Rev. E, 82 (2010) 021402.

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Phase Transitions in Thermo-responsive Nanogels

EXECUTIVE SUMMARY

The phase behavior of poly-N-isopropylacrylamide (PNIPAM) nanoparticles of 273 nanometer dispersed in aqueous medium is investigated as a function of temperature using static/dynamic light scattering techniques. We observed a series of phase transitions in PNIPAM nanogel dispersions of varying particle concentration as a function of temperature. A very dilute dispersion of these nanogel particles exhibited a sudden collapse in particle size at 32.4°C, which is identified as the volume phase transition (VPT), whereas relatively concentrated dispersion showed fluid-fluid transition at 31.5°C. Dispersions with high particle concentration exhibited crystalline and glass like ordering. The crystalline state is found to melt into a liquid like order (fluid) at 26.2°C. Upon further increasing the temperature a fluid-fluid transition occurred at 30.5°C. The system of PNIPAM nanogel is unique in that, across all these transitions the particle number density remains the same, whereas the volume fraction changes.

OUTLINE

Aqueous dispersions of thermo-responsive PNIPAM nanogels exhibit rich phase behaviour upon varying the temperature, T . However a systematic investigation on the phase behavior of these nanogel dispersions is not carried out. Towards understanding this phenomenon we have synthesized aqueous dispersion of thermo-responsive PNIPAM nanogel particles by free radical polymerization technique. The synthesized nanogel dispersion is purified by dialysis and characterized for its size and temperature dependence of particle size using dynamic light scattering (DLS) and the phase transitions are probed using static light scattering (SLS) technique. We have prepared four samples, (Figure 1. labeled S1, S2, S3 and S4) varying in particle concentration, np. SLS and DLS measurements were carried using a dynamic light scattering set-up (Malvern – UK, 4700 model) consisting of a goniometer and multi tau photon correlator and a mixed ion laser (Ar+ Kr) operating at 514.5nm wavelength.

Volume Phase Transition

Sample S1 with $np = 4.36 \times 10^{11} \text{ cm}^{-3}$ (Figure 1) is used to determine the particle size and temperature dependence on particle size. At $T = 25^\circ\text{C}$ the hydrodynamic diameter of the nanogel particles is found to be 273nm. Figure 2 shows the dependence of particle size as a function of temperature. At 32.4°C the nanogel particles are found to suddenly collapse, this is identified as the VPT (arrow in Figure 2). This transition is found to be reversible upon lowering the temperature.

Fluid-Fluid Transition

Static light scattering measurements are carried out on sample S2 with $np = 4.36 \times 10^{12} \text{ cm}^{-3}$ (Figure 1). This sample even after repeated annealing did not exhibit iridescence. The time averaged scattered intensity $I_s(q)$ measured as a function of scattered wave vector, q showed a peak (Figure 3) indicating short-range spatial correlations among the particles in sample S2. The absence of iridescence and the existence of well defined peak suggest that the sample has liquid-like structural order. Since the particle size varies with T in PNIPAM gel dispersions we investigated the effect of T in sample S2. It can be seen from Figure 3 that peak height decreases without a shift in peak position as T is increased. Further the peak disappears beyond 32°C. This implies a gas-like disorder in sample S2 at this temperature. The liquid-like to gas-like transition in sample S2, is identified by monitoring the peak intensity I_{max} as function of T (inset in Figure 3). Interestingly, we observed only a change in slope but not a sudden jump at around 31.5°C. This suggests that across the transition there is no variation in np. Hence we identify this transition as fluid (liquid-like) to fluid (gas-like) transition. The absence of change in np across this transition suggests that interparticle interaction is repulsive.

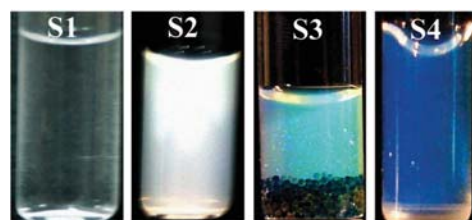


Fig. 1 : Photographs of samples S1, S2, S3 and S4 with varying particle concentration. Sample S3 shows iridescence (crystalline) for visible light and mixed bed of ion exchange resins can be seen at the bottom of the cell. Sample S4 is bluish in color without iridescence

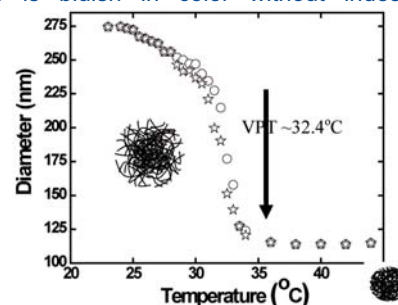


Fig.2: Hydrodynamic diameter d as function of temperature T in increasing and decreasing cycles measured in sample S1. The arrow indicates the VPT temperature. Schematic representation of PNIPAM nanogel particles below and above VPT are also shown

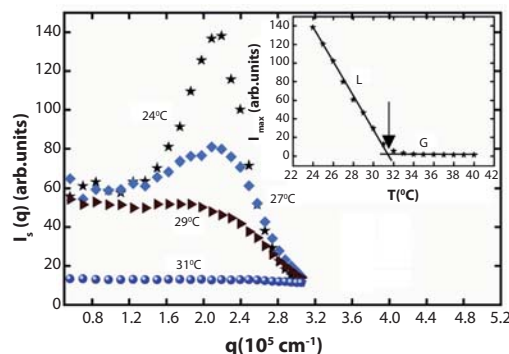


Fig.3: Scattered intensity $I_s(q)$ as a function of q for sample S2 at different temperatures. Inset shows peak intensity $I_{\text{max}}(q)$ as a function of T for sample S2. Arrow indicates the fluid (liquid-like (L)) to fluid (gas-like (G)) transition temperature ($\sim 31.5^\circ\text{C}$) in sample S2

CRYSTALLINE ORDER, MELTING TRANSITION AND GLASS-LIKE DISORDER

Iridescence due to Bragg diffraction of visible light and also a Bragg spot (inset of Fig. 4) for the laser light was observed in sample S3 with $n_p = 8.71 \times 10^{13} \text{ cm}^{-3}$ (Figure 1). SLS measurements on S4 revealed a sharp Bragg peak at $q = 2.84 \times 10^5 \text{ cm}^{-1}$. These observations suggest that sample S3 has crystalline order. The melting transition of the PNIPAM gel crystals is identified by monitoring the Bragg peak intensity I_{max} as function of temperature (Figure 4). The Bragg peak intensity disappeared at 26.2°C . Beyond this temperature the sample is not iridescent and $I_s(q)$ measured as function q showed a broad peak implying the liquid-like order in the sample. Thus we identified 26.2°C to be the melting temperature of PNIPAM nanogel crystals. Moreover the peak position remained the same across the melting transition, which suggests no change in n_p across this transition. Upon increasing the temperature beyond the melting point of PNIPAM nanogel crystals, the peak intensity decreases and showed a change in slope (Figure 4) at 30.5°C . Beyond 30.5°C , the structural ordering in the suspension is found to be gas-like similar to that observed in sample S2. Thus the change in slope observed at 30.5°C is due to the occurrence of fluid to fluid transition, as there is no change in particle concentration.

Sample S4 with $n_p = 10.12 \times 10^{13} \text{ cm}^{-3}$ (Figure 1) had a bluish hue and does not exhibit iridescence under white light illumination. Moreover, the sample did not flow when the cell is tilted upside down. SLS measurements on S4 at 22°C showed a broad peak at $q = 3.14 \times 10^5 \text{ cm}^{-1}$. Further the ensemble averaged electric-field correlation function, $f(q,t)$ as a function of time, t is observed to be non-decaying (Figure 5). These observations suggest that the structural ordering in S4 is glass-like.

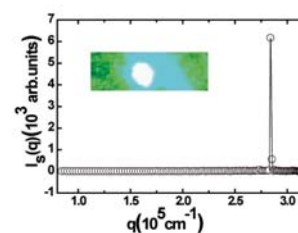


Fig. 4: Bragg peak intensity $I_{\text{max}}(q)$ as a function of T for sample S3. The inset shows C, L, and G represent the temperature region where sample S3 exhibited crystalline, liquid-like and gas-like disorder, respectively. The inset shows the photograph of the Bragg spot (arrow) from crystallites in sample S3

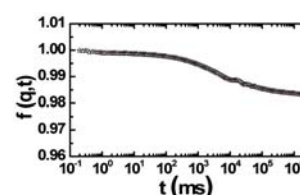


Fig. 5 : $f(q,t)$ versus t for sample S4 measured at $T = 25^\circ\text{C}$

INTERPARTICLE INTERACTIONS

At low temperatures (below VPT) the PNIPAM particles are highly water swollen. Hence the dielectric constant of the particle is almost same as that of water. This results in van der Waals attraction between microgel particles being negligible at low temperatures. So one expects PNIPAM particles to behave as hard-spheres for diameter, d up to VPT. If this is the case, the dispersion should have exhibited crystallization in the volume fraction, ϕ range ~ 0.5 to 0.74 . However the volume fraction of the crystallized sample S3 is found to be 0.93 at 25°C and 0.85 close to the melting. Such high values of ϕ suggest that PNIPAM particles behave as soft spheres. Hence, we conclude that the interparticle interaction between PNIPAM particles is soft-sphere repulsive below VPT and is responsible for the phase transitions observed in samples S2 and S3.

BRIEF DESCRIPTION

Monodisperse nanoparticles serve as superatoms and exhibit structural ordering analogous to that observed in atomic/molecular systems. A stable dispersion of nanoparticles can thus serve as an ideal model system to study universal phenomena like crystallization, glass transition, etc. In charged and hard sphere nanoparticle dispersions the particle size is fixed and temperature is not a controllable parameter to investigate the phase behaviour. We have shown that PNIPAM particles exhibit liquid-like order below a certain particle concentration and undergo fluid-to-fluid transition at 31.5°C without a change in the particle number density. At higher particle concentrations, these particles freeze into a crystalline or glassy state at room temperature. The crystalline order is found to melt into a liquid-like order at 26.2°C . A second transition (fluid-fluid transition) at 30.5°C is also observed. Across all these transitions there is no change in particle number density. Though the phase behavior of PNIPAM nanogel dispersions closely resembles that of hard-sphere colloidal system, the volume fraction at which the crystallization appears is much higher. These observations suggest that below VPT the interparticle interaction are soft-sphere repulsive in nature and is responsible for the observed phase behavior.

ACHIEVEMENT

This is the first report of a fluid-fluid transition in the PNIPAM nanogel system without undergoing a change in particle number density. PNIPAM nanogel crystals are found to melt into a liquid-like order at 26.2°C . We also identified the PNIPAM system to be soft sphere repulsive based on the occurrence of melting of PNIPAM crystals at high volume fraction compared to that of hard sphere system.

PUBLICATIONS

J. Brijitta, B.V.R Tata and T. Kaliyappan, "Phase behaviour of poly(N-isopropylacrylamide) nanogel dispersions: Temperature dependent particle size and interactions", Journal of Nanoscience and Nanotechnology 9 (2009) 5323.

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A Novel Approach for Measurement of Void Fraction in Primary Coolant System of Pressurised Water Reactor

EXECUTIVE SUMMARY

A unique approach for real time monitoring of void fraction in a binary phase steam-water system using a simple conductometric technique has been developed. A pulsating conductance sensor was designed and a few laboratory measurement campaigns for response of voids in water with variable air fraction in the form of small size dynamic bubbles were successfully conducted. In order to apply this technique for void fraction measurement in Pressurized Water Reactors an experimental campaign was successfully conducted in the steam-water simulation facility at Jadavpur University, Kolkatta. Encouraging results obtained from this simulated test facility gave enough confidence for its large scale deployment in Pressurised Water Reactors.

OUTLINE

As a collaborative project with the Reactor Safety Division (RSD) of BARC and Jadavpur University, Kolkatta, we in Innovative Instrumentation Section worked towards the development of a simple technique for measurement of void fraction in steam – water mixed system using our laboratory made pulsating sensor. The major aim of this project is to evolve a simple and reliable technique for real time monitoring of void fraction in Pressurised Water Reactors which is presently considered as one of the important requirements in the context of reactor safety.

A large number of experimental campaigns at room temperature were conducted in our laboratory at IGCAR, Kalpakkam by designing appropriate pulsating conductance sensors with associated instrumentation. In these experiments a vertical type conductance probe with a pair of stainless steel electrodes was mounted in a specially designed Perspex vessel containing distilled water in it. Arrangement was made to create air bubbles in the water by passing Ar gas from a cylinder through a sintered glass disc placed in the bottom of the vessel. Steady value of conductance in water medium and gradual decrease of conductance in the mixed heterogeneous phase (water and gas mixture) was observed. Quick response of conductance while voids were created by passing argon through water was realized. By increasing the flow rate of argon it was also observed that conductance of the medium decreased further. In order to meet the requirement for reactor application at high temperature and high pressure a special type probe fabricated by reactor safety division as per our design was also tested in our laboratory which showed similar performance. The experimental setup is shown in Figure 1.

Further simulation experiments on void fraction in steam – water system pertaining to reactor condition were conducted in the ‘Simulated Facility’(SF) of Mechanical Engineering Dept., Jadavpur University, Kolkata. A schematic diagram of the steam – water two phase simulation facility at Jadavpur University is shown in Figure 2. The simulated facility consists of a storage tank, heat exchanger, buffer chamber, the test section housed with the probe and sump. A series of nitrogen gas cylinders were used for pressurization of the entire system.

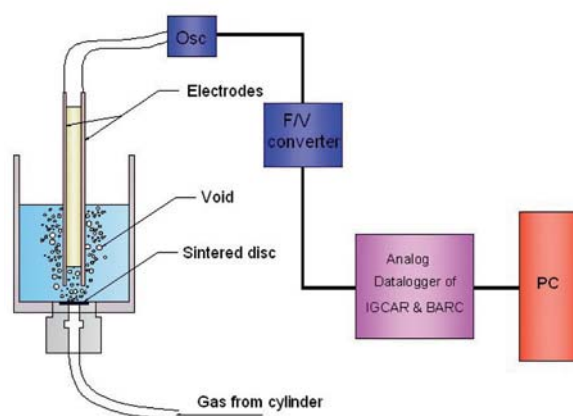


Fig.1: Experimental set-up

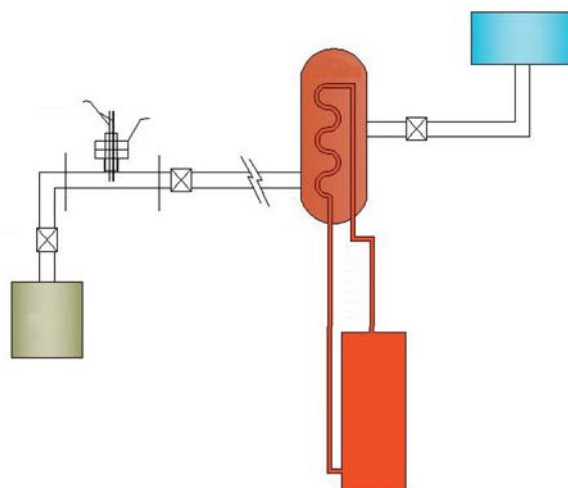


Fig.2: Schematic diagram of the Simulation Facility at Jadavpur University

ADDITIONAL INFORMATION

Experimental runs conducted in the SF are broadly divided into two parts (i) Cold run (at room temperature, 90 bar pressure) (ii) Hot run (at 2500°C, 90 bar pressure). In both cases experiments were conducted in (i) limited blowdown condition i.e. allowing a part of water from buffer chamber passing through the test section for blow down by opening the outlet valve (ii) total blowdown condition i.e. allowing all water passing through the test section for blow down suddenly by opening inlet and outlet valves. The real time shift in conductance in each run was recorded in personal computer. The screen capture view of two blowdown experiments during a hot run are shown in Figure 3. In both cases, the steady value of conductance in homogeneous water medium before the onset of void and sudden decrease in conductance to attain another steady value in heterogeneous phase where voids were created, were measured by the probe with excellent precision. Figure 3 shows a graphical view of data collected from two limited blowdown and two total blowdown experiments. It shows rapid response of probe towards shift in conductance and high precision in measurement. In this measurement campaign our primary attention was focused to examine the probe for its quick response and reliability in such type of work. Only the conductance shifts due to the presence of voids are shown in all experiments. In order to present the data in terms of void fraction for engineering application the measured conductance can be converted in terms of void fraction using appropriate calibration technique. The coefficients thus generated can be loaded in our system software to get direct display of void fraction.

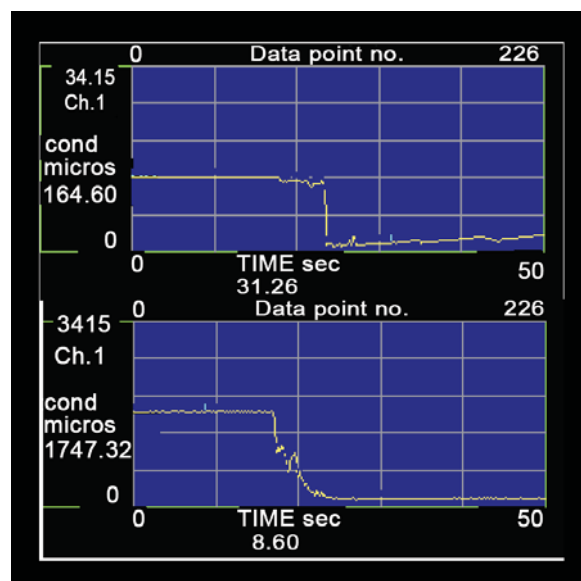


Fig. 3: Screen capture view of blowdown experiments during hot run

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

When conductance probe is fully dipped in water or in any aqueous solution it shows a fixed maximum value of conductance which is purely governed by the nature of liquid and the geometry of the electrodes. In this single phase liquid medium if some air or gas is introduced to create bubbles or voids near the electrodes zone, there will be some reduction in conductance. The bubbles sticking to the electrodes offer resistance in conduction. With increase in void in the aqueous solution the conductance decreases further. When the electrodes are fully withdrawn from the liquid phase conductance becomes zero which will be considered as 100% void and when the electrodes are fully placed in liquid phase without containing any void it shows maximum conductance value. These two extreme values can be used to calibrate the probe for quick response and determination of void fraction. This simple conductance approach is realized due to evolution of a high performing conductance meter in IGCAR.

ACHIEVEMENTS

This conductance principle was deployed to capture the liquid level oscillations in the $\frac{1}{4}$ simulation facility for PFBR.

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Diffusional and Displacive Phase Transformations in 9Cr based Reduced Activation Ferritic Martensitic Steel

EXECUTIVE SUMMARY

The knowledge of phase transformation characteristics of Reduced Activation Ferritic - Martensitic (RAFM) steels is of paramount interest in developing these steels for fusion reactors. The design of low or reduced activation steels aims at replacing the highly radioactive transition elements like Nb, Mo, Cu, Ni, Al, N etc., with their less active counterparts like Ta, W, Mn etc. In order to understand the phase stability in this system, the nature of stable and metastable phase fields that emerge upon cooling from high temperature γ - (fcc) phase is investigated using calorimetry and metallography techniques. It is found that the transformation mode is extremely sensitive to the cooling rate, holding temperature and time of stay in the γ -phase. Both diffusive nucleation growth and displacive martensitic modes of relieving the γ -phase instability are exhibited depending on actual thermal history.

OUTLINE

A study of phase stability and phase transformation characteristics, as a function of temperature, composition and processing variables is important for the design of nuclear reactor materials, wherein the performance of a material are tested against one of the most challenging operating conditions. A fusion reactor material should have adequate thermal, chemical and mechanical stability under normal operating conditions which include high temperature, moderate stress levels and high level of neutron irradiation, in addition to possessing good thermal conductivity, minimum thermal expansion, and minimal induced activation. The 9Cr-based ferritic steel with controlled composition, especially residual elements such as P, B, Sb, Sn etc. besides chromium contain W, Ta, V, C with minimal nitrogen. This article highlights the studies related to basic thermodynamic and kinetics of phase transformations encountered in reduced activation 9Cr ferritic -martensitic (RAFM) steel.

The 'as received' alloy in the normalized and tempered condition was investigated using the Differential Scanning Calorimetry (DSC) and Isothermal Drop Calorimetry. In the former technique, a sample of known mass with homogeneous starting microstructure is heated in an inert atmosphere to temperatures above 1823 K at various controlled heating rates. Various on-heating phase transitions are recorded by DSC as distinct peaks in a heat flow versus time plot (Figure 1). This figure shows all the expected phase changes, namely α' (martensite) + carbides $\rightarrow$ γ -austenite + carbides $\rightarrow$ γ -austenite $\rightarrow$ δ -ferrite $\rightarrow$ Liquid. This phase evolution is also attested with distinct discontinuities in the enthalpy - temperature data, corresponding to the onset of various phase changes on heating as shown in Figure 2. This latent heat of phase transformation is an important thermodynamic quantity in modelling the phase stability of RAFM steels.

An observation is the influence of heating rate on the kinetics of austenite phase formation. It is found that faster the rate the longer it takes to complete the austenite formation reaction. In other words, the ferrite + austenite two phase region, can be stabilized to higher temperatures by using faster heating rates. This has bearing on the industrial solution treatment step that is usually carried out above 1273 K for this steel. The adoption of slower heating rates on the other hand facilitates the dissolution of carbides as the alloy spends more time in the dissolution temperature domain.

Another observation is the role of cooling rate in influencing the decomposition mode. While very slow cooling aided in maintaining full thermodynamic equilibrium as it allowed enough time for the diffusion of substitutional solutes and C atoms to occur, faster rates promoted segregation and also the realization of metastable thermodynamic states. Thus, for cooling rates above 3-5 K/ min. for this steel, a threshold known as the critical cooling rate, hard martensite is obtained. This suggests that RAFM steel is air - hardenable. The hardenability monitored by measuring hardness variation with cooling rate (Figure 3) clearly suggests the role of cooling rate in processing of the RAFM steel.

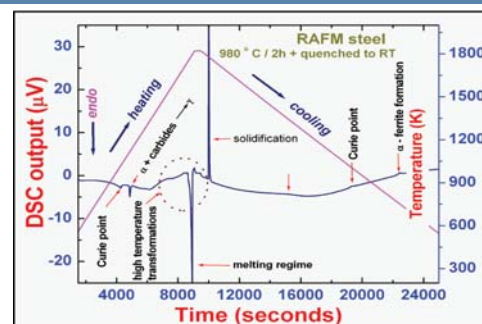


Fig.1: A typical DSC profile obtained for RAFM steel during heating and cooling

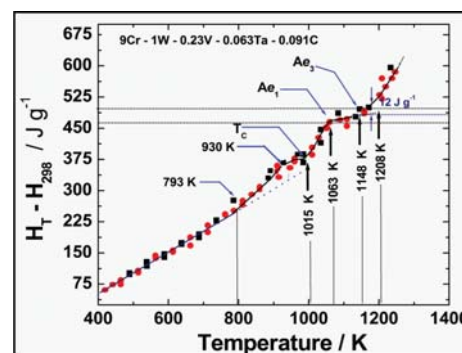


Fig. 2: Enthalpy increment versus temperature variation. A highly non linear variation is observed in the region of phase transformation

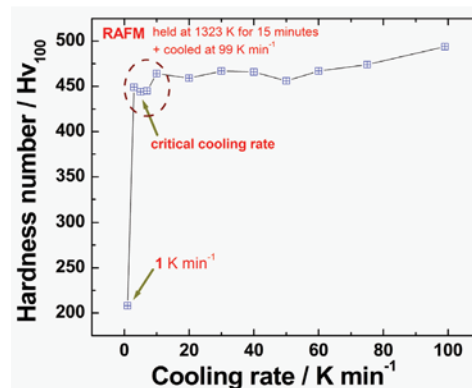


Fig. 3: The variation of martensite hardness with cooling rate for RAFM steel cooled from 1323 K after 15 minutes hold

ADDITIONAL INFORMATION ABOUT THE STEEL

Ferritic- martensitic steels of the 9Cr variety are replacing the presently used austenitic steels for core components in fast reactors due to their excellent resistance to neutron irradiation induced void swelling. This property together with their adequate creep resistance until 773K and resistance to helium embrittlement makes them attractive materials for the test blanket module for fusion reactors. The resistance to induced radioactivity is achieved by elimination of elements like Cu, Co, N and replacement of Mo by W and Nb by Ta, which impart similar properties to the steel, which has resulted in the Reduced Activation Ferritic Martensitic steel. Although the 9Cr steels have inherently a lower tendency for embrittlement as compared to higher Cr ferritics, a strict control on chemistry with respect to trace elements such as P, Sn, Sb, Bi is maintained by use of pure raw materials and improved melting methods.

GENERAL EXPLANATION

The 9Cr RAFM steel is used in the normalized and tempered condition. It consists of a microstructure of tempered martensite with carbides of two kinds: one of the type $M_{23}C_6$ with $M = Fe, Cr, W$ and $C = Carbon$. the other carbide is of MX type with $M = Ta, V$ and $C = C, N$. Of these two principal carbides, the dissolution of $M_{23}C_6$ can be realized at temperatures fairly close to A_{c3} , the austenite completion temperature. The dissolution rate is decided by the effective time provided for this purpose. On the other hand, the MX carbide is very stable to dissolution and requires temperature higher than 1373 K to completely dissolve. Thus, the extent of austenite homogenization, grain growth etc. are decided by the carbide dissolution kinetics. Adopting a higher temperature of homogenization such as 1323 K although enables complete carbide dissolution, provided enough time is ensured, also may lead to grain growth. The selection of heat treatment conditions is based on a balance between these two processes. The influence of these two factors on the microstructure is effectively reflected by corresponding hardness variation.

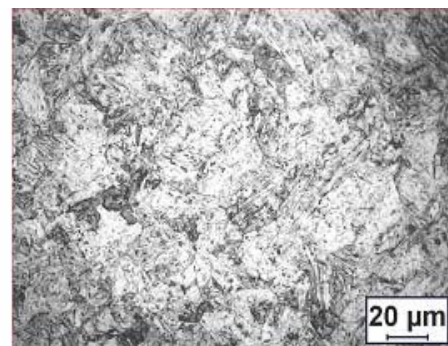


Fig. 4: Microstructure of RAFM steel in the normalised and tempered condition showing tempered martensite structure

BRIEF DESCRIPTION

The significance of such fundamental studies on the basic phase equilibria characteristics is that they throw considerable insight into factors that affect the practical stability of metallic alloys under typical service conditions. Actual in-pile or real reactor simulations are very complex to be realized in laboratory frames. There are many synergistically acting factors that govern the ultimate useful life span of a nuclear reactor material. But idealized laboratory studies like the one presented here attempt to isolate one or few of the key factors and explore their significance on the overall metallurgical stability. The results of these studies serve to advance the frontiers of our understanding on materials characteristics and their performance. A rationalized alloy design is therefore facilitated.

ACHIEVEMENT

An indigenous material development programme for the test blanket module of fusion reactor has been taken up for the first time. The complete assessment of phase boundaries and evaluation of temperature limits has been carried out in an indigenously produced 9Cr RAFM steel. This study has clearly revealed the various transformations that this steel undergoes and the role of alloy chemistry and process parameters in influencing the transformation temperatures. This data serves as a useful input to optimise the thermomechanical and heat treatment conditions for this steel during fabrication.

PUBLICATIONS

1. S. Raju, B. Jeya Ganesh, Arun Kumar Rai, R. Mythili, S. Saroja, E. Mohandas and M. Vijayalakshmi, K. B. S. Rao and Baldev Raj, "Measurement of Transformation Temperatures and Specific Heat Capacity of Tungsten added Reduced Activation Ferritic-Martensitic Steel", J. of Nuclear Materials, 389 (2009) 385.
2. S. Raju, B. Jeya Ganesh, Arun Kumar Rai, S. Saroja, E. Mohandas, M. Vijayalakshmi and Baldev Raj, "Drop Calorimetry Studies on Ta, W added 9Cr based reduced activation steel", Int. Journal of Thermophysics, 31 (2010) 399.

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Synthesis of Nano-crystalline Al_2O_3 and Yttria Stabilized Zirconia Coatings by Flame and Laser Pyrolysis

EXECUTIVE SUMMARY

Flame and Laser Pyrolysis are two important techniques for the synthesis of large surface area nano-structured coatings under open atmospheric conditions. In the flame mediated process the ultrasonically generated precursor aerosols are made to interact with flame and form a coating on the substrate held in or near the flame. In laser pyrolysis the precursor aerosols and the laser beam are coupled in to a quartz tube and directed on to the substrate to form a coating. Flame and Laser Pyrolysis facilities were indigenously designed and developed for synthesis of large surface area ceramic coatings for tribological and high temperature corrosion resistance applications. Alumina and yttria stabilized zirconia coatings were made on various substrates using the facility and effect of substrate temperature, precursor composition and concentration on coating structure, morphology and adhesion were studied.

OUTLINE

There is considerable interest in nanostructured alumina and Yttria Stabilized Zirconia coatings due to their potential applications in areas such as tribology, thermal barrier coatings, solid oxide fuel cells and sensors. A variety of synthesis techniques are available for their synthesis. However, simple, cost effective open atmosphere, techniques for large surface area coatings are very rare and have not attained commercial status. In this context flame and laser pyrolysis facilities were developed in-house.

In flame pyrolysis the ultrasonic or compressed air generated aerosols of 1 to 5 micron size are premixed with the fuel and coaxially injected into the annular burner and combusted. Inside the flame, the precursor undergo desolvation, rapid thermal vaporization and at the substrate surface decomposition reaction. Desired surface coverage is achieved by executing ultra-precise computer controlled movement of the substrate mounted on board the raster stage. Desired thickness is achieved by carrying out deposition in multiple passes. Laser pyrolysis is a variant of the flame pyrolysis in which the flame is replaced with a laser. Here the precursor aerosols are fed into a quartz tube through which a CW-Nd:YAG laser (1064) is coaxially coupled. Photo-thermal decomposition of the precursor by the laser beam at the substrate results in the formation of nano-crystalline coatings. Large surface area coatings are obtained by using a computerized raster stage. The primary requirement for laser pyrolysis is that the emission line of the laser overlaps with an absorption line of one or more precursors. If this requirement is not met one has to use additional photocatalysts which will absorb laser energy and transmit to the precursor by shocks. The strength of the technique is that the process can be controlled through systematic parameters such as beam cross section, absorber concentration and laser power.

Nano-structured Al_2O_3 coatings and YSZ coatings were synthesized as a function of flame temperature in the range 500 to 1000°C and also by laser pyrolysis. The coatings were characterized by X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM). The coating adhesion was determined using a scratch tester. The evolution of structure from amorphous to γ to θ to κ - Al_2O_3 with temperature in the range 500 to 1000°C is shown in Figure 1. The Al_2O_3 coatings were amorphous at all laser powers whereas the YSZ coatings were of cubic structure. The critical load for coating failure obtained from the scratch test is ~12N. Figure 2 shows the acoustic emission signal during scratch. The failure mode was continuous ductile perforation. The FESEM picture of the coating synthesized at 900°C is shown in Figure 3.

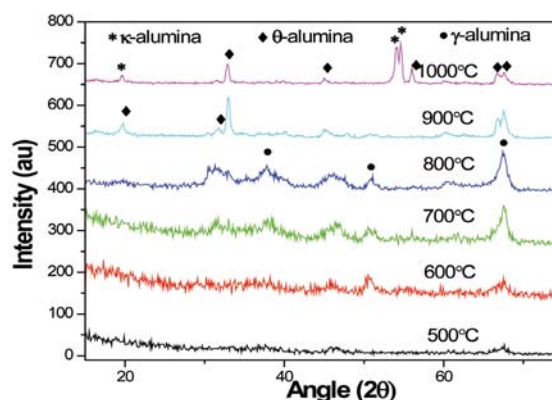


Fig. 1 : XRD pattern of Al_2O_3 coating at various temperatures

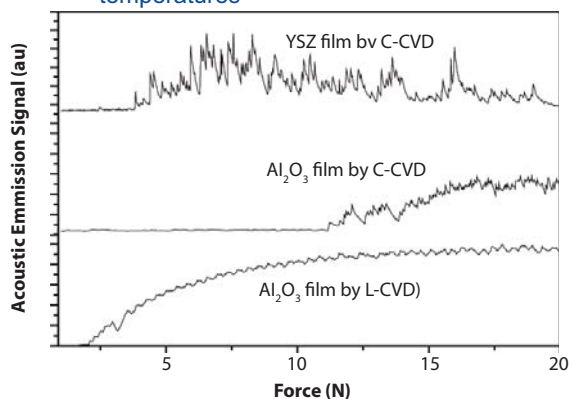


Fig. 2 : Acoustic emission spectra during scratch

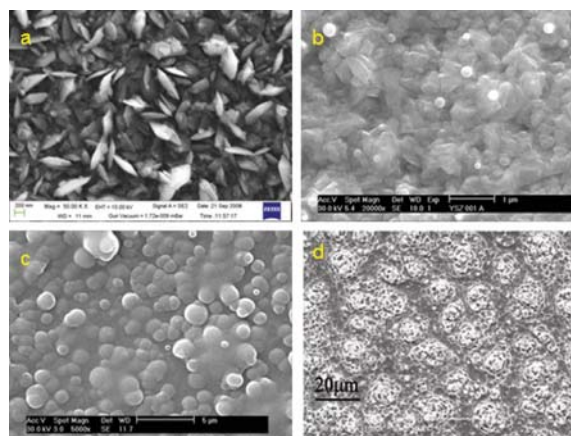


Fig. 3 : FESEM images a) Alumina and b) YSZ by flame pyrolysis c) alumina and d) YSZ by laser pyrolysis

ADDITIONAL INFORMATION

Flame and laser pyrolysis are open atmospheric coating techniques suitable for large surface area coatings. In the flame mediated process the flame forms the CVD reactor and the non-equilibrium rapid heating characteristics of the flame are used to derive nano-structured coatings. The criteria necessary to obtain adherent coatings using flame pyrolysis is that the precursor should completely vaporize in the flame and the vapors on reaching the substrate undergo reactive decomposition. Hence metal organics are generally chosen. Precursors like metal nitrates do not vaporize but decomposes in the flame giving rise to powdery deposits. In laser pyrolysis precursors like metal organics and nitrates were found to give adherent coatings. This is because in laser pyrolysis the chemical process involved is photo-thermal reaction. The coatings obtained in laser pyrolysis by using nitrates were rough compared to those obtained by using metal organics.

COMPARISON BETWEEN FLAME AND LASER PYROLYSIS

In flame pyrolysis a flow stabilized conical flame element having a parabolic temperature profile provides the medium for coating synthesis. Since the flame stability, dimension and temperature can not be fine tuned, microstructure tailoring and coating patterning are not feasible. In addition the requirement of an oxygen atmosphere for sustaining the flame results in substrate surface oxidation. The choice of substrate material is severely limited in this case. In contrast laser pyrolysis is a precise coating synthesis technique because the laser parameters like laser power, beam cross section and beam path can be precisely controlled. Since laser can be focused to a fine spot patterned and guided nanometric architecture is realizable. It has a higher degree of spatial control over particle nucleation and growth and therefore ability to tailor the microstructure. Substrate surface oxidation is not an issue since the process can be carried out under any ambient.

BRIEF DESCRIPTION

Nanostructured Al_2O_3 and yttria stabilized zirconia coatings were made on various substrates using flame and laser pyrolysis techniques. In flame pyrolysis evolution of structures from amorphous to γ to θ to κ - Al_2O_3 with temperature in the range 500 to 1000°C were observed. Laser pyrolysis gave amorphous alumina at all laser powers. Both laser and flame pyrolysis produced cubic yttria stabilized zirconia. The coating adhesion was determined using a scratch tester.

ACHIEVEMENT

Open atmosphere large surface area coating facilities were designed and developed indigenously. Using the facilities nanostructured Al_2O_3 and yttria stabilized zirconia coatings were made. Other ceramic oxide coatings can also be made using the facility by selecting the appropriate precursor. By a simple modification of the laser pyrolysis facility, in addition to oxides, carbide and nitride coatings can be synthesized.

PUBLICATIONS

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2. T. Mathews, B. P. Dhonge, R. Krishnan, S. Dash, A. K. Tyagi and Baldev Raj, "Open atmosphere laser assisted spray pyrolysis technique for deposition of Al_2O_3 and yttria stabilised zirconia nanostructured coatings", Surface Engineering (online available April 15, 2009).

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Synthesis of Novel Compounds by Laser Heated Diamond Anvil Cell Technique

EXECUTIVE SUMMARY

One of the foremost challenges in Materials Research is to design and synthesize new and exotic materials, hitherto unknown. The unique Laser Heated Diamond Anvil Cell (LHDAC) facility, established at Materials Science Group at IGCAR offers excellent opportunities in this pursuit. This facility, only one of its kind in India, can be used to investigate material properties, synthesize metastable compounds, in the ultra high P-T regime. Here, we summarize the recent achievements in the area of synthesis of novel compounds. Firstly, direct conversion of pyrolytic graphite to diamond-like carbon is reported. Secondly, high pressure synthesis of novel phase of Ruthenium Carbide is reported. This has been accomplished at a very low pressure of 10 GPa. Lastly, novel III-V compound BSb in bulk form has been successfully synthesized for the first time in the laboratory.

OUTLINE

Interest in synthesis of carbides, stems from the broader motivation in looking out for materials harder than diamond. It is observed that as we move along the 4d transition metal series, the bulk modulus B_0 of the elements increases up to Ruthenium and thereafter it decreases. It is also observed that B_0 of carbides and nitrides of transition metal is further enhanced compared to 4d metal. This enhancement in the bulk modulus of transition metal Carbides is attributed to the increase in the covalent nature of the bonding between the p and d orbital of C and transition metal. Most of these carbides are formed in rock salt structure with few notable exceptions like WC which has hexagonal structure. Ruthenium carbide has been synthesized earlier in hexagonal phase isomorphous with WC, with very high micro-hardness. However, it is suggested that synthesis of RuC should be carried out under pressure to prevent its dissociation. It would be interesting to study the structure adopted by RuC when synthesized under pressure and also its B_0 . As a prelude to these experiments first we have carried out laser heating of graphite alone. This is because in the synthesis experiments it is the graphite which couples with the laser rather than the transition metal.

Direct conversion of pyrolytic graphite to diamond-like carbon

The cell is taken to $P \sim 16.5$ GPa and $T \sim 2000$ K. Laser beam was rastered on the sample and the sample was thoroughly laser heated. The sample was retrieved from DAC for characterisation. The diamond Raman peak at 1331 cm^{-1} , was clearly riding on a typical DLC background as shown in Figure 1. The transition pressure observed here of 16.5 GPa is much lower than what is seen in shock experiments i.e 19.6 GPa.

Synthesis of novel transition metal carbide Ru-C

Ru and C in 1:10 atomic ratio is pelletized and loaded in the DAC. The sample is pressurized to 6.7 GPa and laser heated to a temperature of ~ 2000 K. High Pressure X-Ray Diffraction (HPXRD) patterns of the sample before and after laser heating are shown in the Figure 2. X-ray pattern before laser heating contains intense Bragg peaks of Ruthenium and Ar. Disappearance/reduction in intensity of Ru peaks associated with appearance of two new X-ray reflections indicates synthesis of ruthenium carbide. These two new reflections are observed in experiments carried out with NaCl as pressure transmitting medium. The cell was taken to further high pressures to study stability of the new phase formed and is also shown in Figure 2.

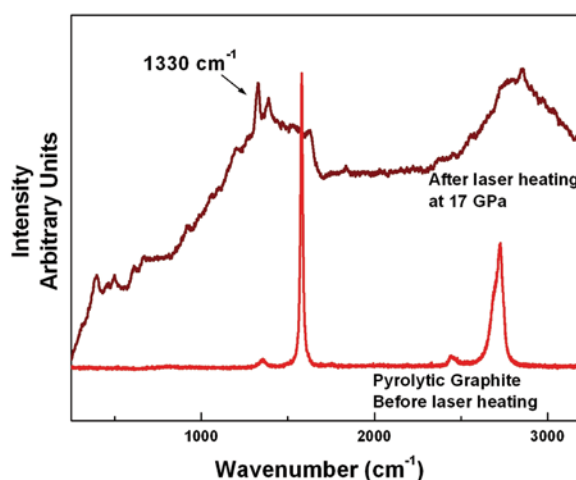


Fig.1: Bottom curve shows the typical DLC characteristics with a double hump due to bond stretching of all pairs of sp^2 atoms. Top curve shows clear second order Raman of diamond at 1331 cm^{-1}

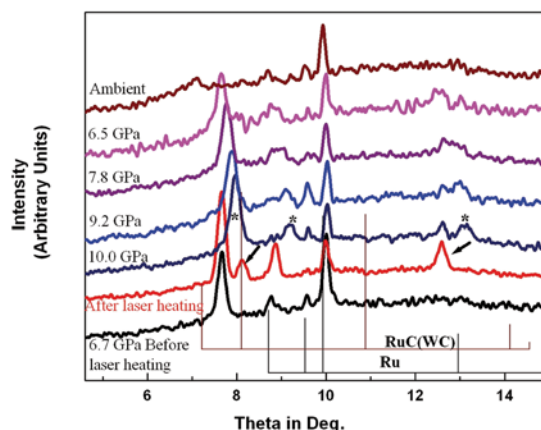


Fig.2: HPXRD patterns of Ru:C before and after laser heating under high pressure. Stick plots are the standard PDF patterns

ADDITIONAL INFORMATION

Why ultra high pressures and temperatures ?

Under extreme conditions of pressure and temperature, nature of bonding and electronic structure undergo changes leading to drastic increase in their chemical reactivities. This paves way for synthesis of novel phases which otherwise are not permitted by thermodynamics at ambient. Several novel and superhard materials have been synthesized by employing the LHDAC technique.

GENERAL DESCRIPTION

The observed HPXRD pattern at 6.5 GPa after laser heating is compared with the earlier reported synthesis of RuC in WC (hexagonal) structure. It is seen that apart from the peak at 8.10 the other prominent reflections for WC type RuC is not observed in our experiment.

Determination of the high pressure phase is a difficult task. This is especially so in high pressure synthesis experiments as the volume fraction of the new phase formed is very small as compared to starting material. In the present experiment we observe only two new reflections in the d range of 4.74 to 1.5 Å. Systematics in analogous compounds is used as guide to start the iterative search process. In general transition metal carbides adopt cubic structure. Least square analysis carried out with the possible cubic lattices yields very poor figure of merit.

High pressure synthesis of Re_2C is reported recently in hexagonal phase. Least square analysis carried out with these trial lattice parameters yields a good figure of merit. The lattice parameter obtained after carrying out the analysis is $a=3.20$ Å and $c=10.07$ Å. Further experiments are being carried out to confirm the synthesis and also to explore formation and phase stability as a function of pressure.

BRIEF DESCRIPTION

The interest in Boron compounds is due to their unusual properties due to small core size and absence of p electrons in Boron. Theoretical electronic structure calculations predict BSb to be an indirect band gap semiconductor similar to BP and BAs. B and Sb in 3:1 atom ratio is taken, this mixture is pelletized and a small piece of ~ 150 μm is loaded in DAC. Sample assembly done is similar to the one done in RuC experiment. The cell is taken to $P \sim 3$ GPa and $T \sim 1900$ K. Coupling of the laser to the sample was good and lot of convection currents were seen. Micro-Raman was carried out on the retrieved sample which shows a prominent peak at 151cm^{-1} , is shown in Figure 3. The same micro Raman feature is seen on all regions of the sample, implying that the compound has been formed in single phase and uniformly across the sample.

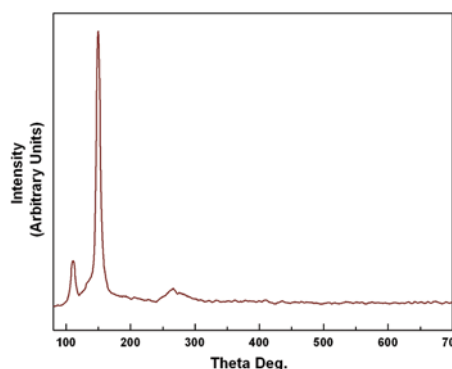


Fig. 3 : Sharp peak in micro-Raman at 151cm^{-1} which is due to B - Sb mode

ACHIEVEMENT

Ruthenium Carbide has been formed in hexagonal phase at moderate pressure of 7 GPa for the first time. In the process we observe direct conversion of pyrolytic graphite to diamond at a low pressure of 16.5 GPa. Also, we report for the first time bulk synthesis of BSb not reported earlier. These novel results highlight the emerging importance of laser heating technique coupled to diamond anvil cell high pressure experiments.

PUBLICATIONS

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Determination of Effective Activation Energy for Vacancy Migration in D9 Steel using Positron Annihilation Spectroscopy

EXECUTIVE SUMMARY

Migration and clustering of vacancies at elevated temperatures essentially determines the void-swelling behavior of structural materials used in fast reactors. Towards investigating the vacancy migration behavior, cold worked and proton irradiated Ti- modified D9 steel have been studied by positron annihilation spectroscopy. D9 samples have been irradiated with 3 MeV protons followed by isochronal annealing at various temperatures. The dramatic decrease in positron annihilation parameters viz., positron lifetime and Doppler S-parameter, around 500 K indicates the migration and annealing of vacancy defects. Isothermal annealing studies have also been carried out at the temperature wherein vacancies distinctly migrate. Assuming a singly activated process for defect annealing, the effective activation energy for vacancy migration is deduced to be 1.13 ± 0.08 eV. This is an important parameter for the determination of void-swelling behavior of D9 steel.

OUTLINE

Solution annealed and 20% cold worked D9 were irradiated at room temperature with 3 MeV protons at doses of 1×10^{15} , 1×10^{16} , 5×10^{16} and 1×10^{17} protons/cm² with a beam diameter of 10 mm. Isochronal annealing was performed in steps of 50 K ranging from 323 to 1323 K for a period of 30 minutes each. Temperature range for isothermal studies was so chosen wherein the sample recovers from the damage, viz 623 K, with a time scale ranging from 5 minutes to few hours in a vacuum of 1×10^{-6} mbar. Positron lifetime measurements were performed at room temperature on identical set of samples, in standard sample-source sandwich geometry. A fast-fast coincidence system coupled with a pair of BaF₂ detectors having a time resolution of 260 ps was used. To complement the lifetime results, Doppler broadening measurements were also carried out using a HPGe detector having 1.3 keV energy resolution. From these measurements, a defect-sensitive line shape S-parameter was deduced to monitor the annealing stages at different temperatures.

Figure 1 shows the variation of positron lifetime as a function of annealing temperature for cold worked (CW) and proton irradiated D9 samples. Two clear stages viz., (i) a noticeable reduction in positron lifetime during 300 K to 800 K, corresponding to vacancy migration and annealing (ii) a broad maximum in lifetime around 1100 K, corresponding to the nucleation and growth of TiC precipitates, are observed. Complementary Doppler S-parameter has also exhibited similar annealing stages. There is only a small increase in lifetime values during first annealing stage for proton irradiated sample as compared to CW sample, suggesting that proton irradiation induces only marginal increase in vacancy defect concentration as compared to the cold worked state. Further, for proton irradiated solution annealed sample, the second stage was not observed which is attributed to absence of TiC precipitation due to annealing of dislocations. Irradiation dose dependence of lifetime and S-parameter values has also been obtained. Having identified the temperature range of vacancy migration (Figure 1), isothermal positron lifetime studies were carried out at selected temperatures for various time durations on proton irradiated D9 steel.

Figure 2 shows the variation of positron lifetime corresponding to isothermal annealing at different time durations at 623 K (where maximum lifetime reduction is seen in comparison to initial state). Assuming that the vacancy migration is a single-activated process, the isochronal and isothermal results are analyzed to obtain the time duration at different temperatures to produce the same change in positron lifetime. This plot is shown in Figure 3, from which, the activation energy for vacancy migration is deduced to be 1.13 ± 0.08 eV. In general, a value around 1.0 eV has been reported for various stainless steels. The present value deduced for D9 steel is consistent with this general trend. To conclude, using positron annihilation spectroscopy we have been able to deduce the effective activation energy for vacancy migration in D9 steel.

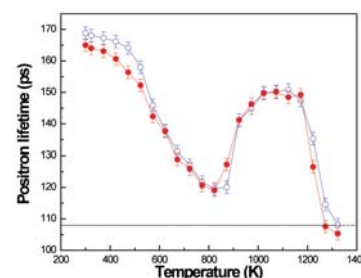


Fig. 1 : Variation of positron lifetime as a function of annealing temperature for proton irradiated (blue circles) and cold worked (red circles) D9 steel. Horizontal dashed line indicates reference lifetime value

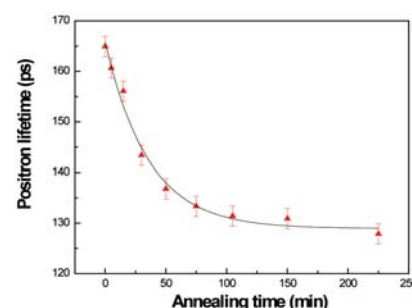


Fig. 2 : Variation of positron lifetime as a function of annealing time at a temperature of 623 K for proton irradiated D9 steel

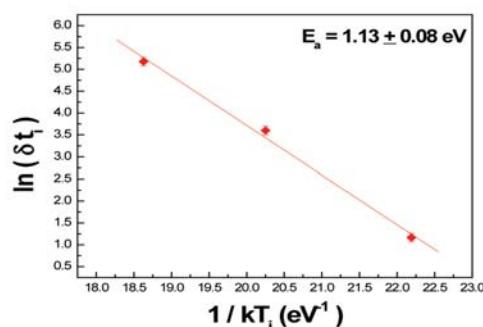


Fig. 3 : Determination of activation energy for vacancy migration

GENERAL DESCRIPTION

Fundamental understanding of the migration, clustering and annealing behavior of vacancy defects in D9 steel is essential, since D9 is the core component material in PFBR. The critical parameter for void-swelling behavior is the effective activation energy for vacancy migration, which is not reported for alloy D9 in literature. There are only two prominent experimental methods namely resistivity and positron annihilation to determine this. Hence, detailed positron annihilation spectroscopy studies.

BRIEF DESCRIPTION

D9 steel subjected to cold working and proton irradiation has been studied using positron lifetime and Doppler S-parameter studies. Isochronal annealing studies have established two annealing stages corresponding to vacancy migration and annealing as well as the nucleation and growth of TiC precipitates. Based on these, isothermal studies have been carried out at selected annealing temperatures. From these studies, the effective activation energy for vacancy migration has been deduced to be 1.13 ± 0.08 eV.

ACHIEVEMENT

One of the important parameters determining the void-swelling behavior of D9 steel is the effective activation energy for vacancy migration. From the present isochronal and isothermal positron annihilation experimental studies, this value has been deduced to be 1.13 ± 0.08 eV.

PUBLICATIONS

J. Arunkumar, S. Abhaya, R. Rajaraman, G. Amarendra, K. G. M. Nair, C. S. Sundar and Baldev Raj, "Defect recovery in proton irradiated Ti- modified stainless steel probed by positron annihilation", Journal of Nuclear Materials, 384 (2009) 245.

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Mechanical Behavior from Small Volumes of Material

Recent Advances in Experimental and Modeling studies

EXECUTIVE SUMMARY

Small specimen test technology has reached a significant level of maturity and acceptance with the development of an array of techniques to extract the mechanical properties such as tensile, fracture and creep from relatively small volumes of the material. The ab-initio research work at IGCAR in this area include development of high precision loading equipments and test fixtures, finite element (FE) simulation and analysis, standardization of experimental procedures and validating the finite element results, and development of correlations with mechanical properties derived from conventional test methods. The techniques such as shear punch and ball-indentation have been exploited for evaluating the properties of irradiated SS316, weld joints, aged and creep exposed Mod 9Cr-1Mo steel.

OUTLINE

The development of small specimen testing technology at IGCAR, India, has been an integral part of material irradiation programme of various candidate alloys chosen for fast reactor core structures and post-irradiation characterisation of their mechanical properties. The techniques that have been extensively studied are shear punch, small punch and ball-indentation tests.

The shear punch (ShP) test involving blanking or shearing of disc specimen (approx 8 millimeter diameter and 0.5 millimeter thick) with a flat cylindrical punch was modeled using finite elements to study the specimen deformation up to yielding. The finite element results indicated a large influence of the punch compliance on the elastic portion of load-displacement plot. By suitably modifying the experimental setup, the fixture compliances were eliminated and the elastic loading lines of experimental curve matched well with the finite element generated curve (Figure 1). By analyzing the tensile and shear punch tests data of alloys like carbon steel, 2.25Cr-1Mo, modified 9Cr-1Mo steels, SS316, the tensile-shear correlations for yield and maximum strength were rationalized using universal correlations without involving material specific empirical constants.

The ball-indentation (BI) technique based on repeated loading-unloading cycles of indentation on a material using a spherical indenter has been standardized using in-house developed servo hydraulic test equipment. Figure 2 shows the finite element model of the spherical indentation and the comparison of finite element generated load-depth plot with the experimental data. A better overlap of the loading curves was obtained only after including the indenter holder in the model which simulated closely the experimental conditions. The evaluation of stress-strain values from the indentation load-depth data rely on accurate determination of contact diameter which in turn is influenced by the amount of pile-up or sink-in of material around the indentation edge. Using finite element analysis, the pile-up effects were visualized and correction factors for pile-up were verified.

The small punch (SP) test involving the bulging of a disc specimen using a spherical punch produces a load-displacement curve different from that of shear punch test. The small punch test has been modeled using finite element method and the effects of initial clamping force on the specimen, friction between specimen-ball and specimen-die interfaces on the load-displacement curve have been examined. The extent of overlap of the finite element generated curve with experimental curve was dependent on the range of the stress-strain data as input to the finite element model. These observations will provide useful insight during the implementation of inverse finite element methodology for estimating the constitutive stress-strain curve from the small punch load-displacement curve. Combining shear punch technique with electron microscopy and swelling measurements, the irradiation behaviour of SS316 duct of FBTR has been systematically studied using small specimens. The ball-indentation technique has been applied in a novel way to investigate the progression of the creep damage in mod 9Cr-1Mo. By terminating the uniaxial creep tests at various predetermined strains and performing spherical indentation tests on the unstressed head and stressed gage portions, the extent of softening of the steel due to thermal and creep exposures was determined (Figure 3).

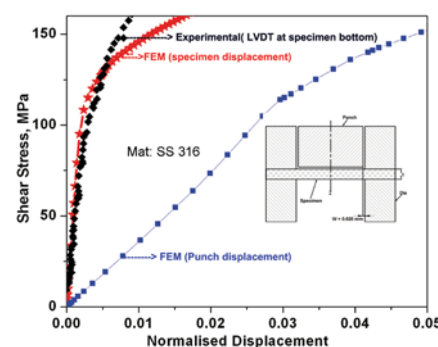


Fig.1: Modified shear punch experimental set up and the resultant curve compared with FE generated plot

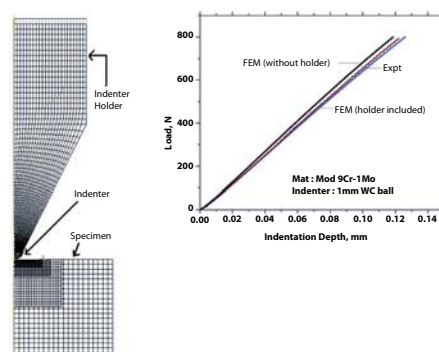


Fig. 2 : FE model of spherical indentation and the resultant L-d plot superimposed on the experimental data

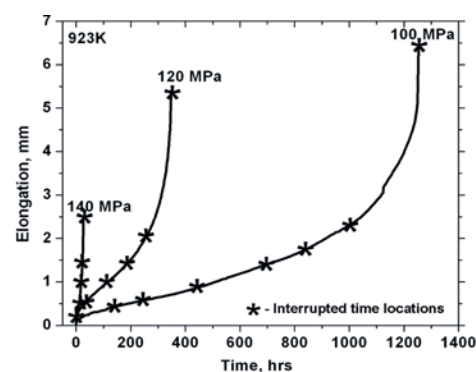


Fig.3: Conventional creep tests on Mod 9Cr-1Mo steels at 923K at various levels

ADDITIONAL INFORMATION

The use of small specimens for mechanical testing actually originated in nuclear industry caused by space limitations in nuclear reactors for irradiation material testing with the added advantage of reduced radiation dose from smaller volumes during post-irradiation testing. As a spin off, the small specimen testing also finds applications in many other areas where conventional mechanical test techniques are practically difficult due to limited availability of material volume like in failure analysis, study of weld joints, coatings and in alloy development etc. The concept of small specimens for evaluating mechanical properties is also gaining popularity for residual life/structural integrity assessment of ageing power plant components due to advantages of sampling small volume of material non-destructively directly from the operating component without affecting its structural integrity.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The small specimen test techniques can be broadly classified into the following categories: (a) Tests, which are based on miniaturization or scaling down of conventional specimen size (like miniature tensile, fatigue, impact and fracture toughness tests) (b) Tests that are based on novel techniques using disk sized specimens of approximately 0.5 millimeter thickness (like disk bend tests, shear punch / small punch tests) (c) Techniques based on ball-indentation (BI) testing.

The punch and ball-indentation tests do not have equivalent conventional counterparts and require extensive validation and benchmarking before being effectively put to use. The methodology adopted at IGCAR is to first carry out experiments which involve performing both conventional and small specimen tests on various heat treated conditions of widely ranging microstructures and mechanical properties prepared from the alloy of interest. The data from the small specimen test technique is analyzed, compared and correlated with the corresponding conventional test properties. Unlike a tensile test, the conversion of the load-displacement response to stress-strain data is not directly possible due to the complex stress states and inhomogeneous strain distribution in the specimen during deformation. Hence, finite element modelling, simulating the loading and boundary conditions becomes necessary for analysing the experimental load-displacement response and to obtain the stress-strain properties. From the numerical simulation, the stress states and strain distribution in the specimen during deformation can be well understood.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Shear Punch testing is basically a blanking operation and the principal mode of deformation is shear occurring in the narrow clearance zone between the specimen and lower die surface. The relations between the shear and the uniaxial tensile is linear in nature.

The Small Punch test or the buldge test involves cupping of a disk specimen with a spherical punch. The principal mode of deformation is biaxial and the load-displacement data can be analyzed for both tensile and fracture toughness properties. The small punch test is also useful for transition temperature determination similar to the Charpy Impact test.

Indentation tests using spherical indenters are unique because the geometry of indentation changes with increasing penetration, corresponding to unique stress-strain value for a particular depth. As a result, spherical indentation under triaxial compression loading is a unique method for determining the complete stress-strain curve of metals. Compared with the conventional hardness tests, this technique offers both strength and ductility parameters from analysis of the measured indentation load-depth curve.

ACHIEVEMENTS

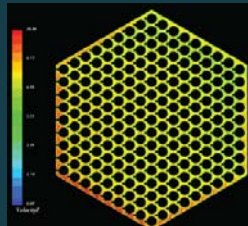
The various small specimen test techniques have been effectively put to use for quantifying the properties of irradiated materials, weld-joints and service aged materials. Integrating the experiments with FEM simulations has been valuable for understanding the deformation behaviour of smaller specimens and for extracting practically useful information. The established test facilities and the insights gained from FEM will serve as a foundation for ongoing and planned experimental irradiation programs on advanced FBR structural materials. The expertise will also be useful for predicting the in-service integrity of actual plant structures.

PUBLICATIONS

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ENGINEERING

Development of a Computer Code for Analysis of Partitioning Cycle of PUREX Process

EXECUTIVE SUMMARY

A computer code PUSEP (Plutonium Uranium Solvent Extraction Program) has been developed for the analysis of partitioning cycle of PUREX process involving in-situ electrochemical reduction of uranium and plutonium. The development of model equations involves countercurrent multi-component extraction coupled with chemical and electrochemical reactions. The model is specific to mixer-settler contactors and is restricted to mass transfer equilibrium. Computer code provides transient behaviour of mixer-settler contactor from the start-up conditions to the steady-state conditions. The code has been validated using experimental data on in-situ uranium reduction in electrolytic mixer-settler and the data available in literature. The calculated results are consistent with the experimental results.

OUTLINE

In the PUREX process of nuclear fuel reprocessing uranium and plutonium are co-extracted from aqueous nitrate solution into organic (TBP) phase as U(VI) and Pu(IV) nitrates to achieve separation from fission products. Further, it is necessary to separate uranium and plutonium from each other and also purify them separately for the end use. Stripping of plutonium from metal rich organic phase is accomplished by selectively reducing the plutonium to poorly extractable trivalent species [Pu (III)] by means of a reducing agent (i.e., by ferrous sulphamate, hydroxylamine nitrate or uranus nitrate) or by in-situ electrochemical reduction. The extent to which separation of plutonium from uranium is achieved depends upon the effectiveness of the reduction reaction. Externally generated uranous [U(IV)] is generally used as reducing agent for U/Pu partitioning in the contemporary salt free reprocessing flowsheet. But, the addition of this external uranium increases the separation load as large excess uranous (more than 6-10 times of stoichiometry) is required for effectively separation, it becomes unwieldy for the FBR fuels due to high content of Pu (about 25-30% Pu).

A computer program (PUSEP) has been developed to model the various steps in the electrochemical separation. The basis of model equations development involves countercurrent multi-component extraction with coupled chemical and electrochemical redox reactions. The model is specific to mixer-settler contactors and is restricted to mass transfer equilibrium. It is assumed that complete mixing is assumed in mixer compartment of mixer-settler unit and mass transfer due to extraction occurs only in mixer compartment and the components are always at equilibrium between the phases. It is also assumed that stage residence times are sufficiently long so that diffusional resistances can be ignored. The rate of electrochemical reactions as well as the chemical reductions is accounted in the equations. The developed code generates transient concentration profiles from the initial conditions to steady-state conditions. Though calculation of steady-state profiles via transient behavior consumes a considerable amount of computer time, it provides important information about the variation of solute concentration with time in the contactor bank which cannot be obtained otherwise from steady-state solutions. Transient calculations can also provide the information about unsafe accumulation of solute if any. Simulation of almost all the published experimental results concerning to electrolytic partitioning of Pu/U were carried out in order to test the validity of the model equations and associated computer program. Two typical examples to compare the experimental and literature data are described in Figure 1 & 2. It can be seen from Figure 1 that reasonably stable profile is reached in 9 hours of run. The experimental concentration is in mg/l, whereas very low values are predicted by the program. This deviation in the low Pu concentration is probably due to slow strip rate of Pu-HDBP complex as well as uncertainty in the estimation of distribution coefficient in low concentration region. Another experiment (carried out at IGCAR) with uranium reduction in a 20 stage mixer settler with 14 extraction stages and 6 scrub stages, has been modeled. Uranium is more difficult to reduce electrolytically and the concentration profile prediction is very reasonable (Figure 2).

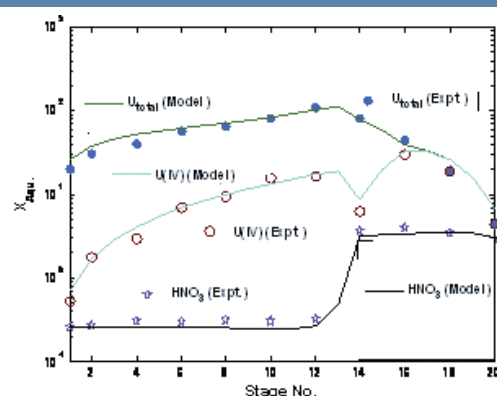


Fig. 1: Comparison of calculated and experimental data on uranium reduction in a 20-stage electrolytic mixer settler (details given elsewhere). Aqueous feed is at 14th stage and the concentrations are: U: 67.4 g/L and HNO₃: 4.35 M. The organic scrub is 30% TBP in NPH. The strip solution concentrations are: HNO₃: 0.26 M and N₂H₅⁺: 0.35 M. The respective flow rates are: 6mL/min, 6mL/min and 4mL/min respectively for feed, scrub and strip

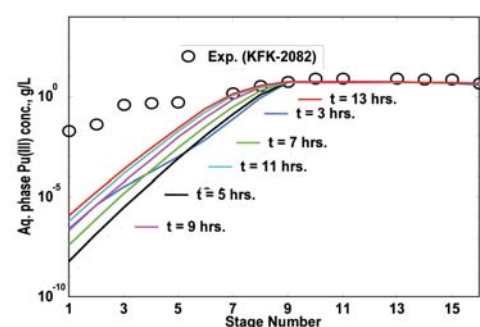


Fig. 2: Comparison of experimental and calculated profile of Pu(III) in 16-stage 1B contactor. Experimental data [KFK-2082] is based on 20% TBP in n-dodecane. The organic feed stream is at 10th stage and the concentrations are: U: 49.6 g/L and Pu: 2.33 g/L. The organic scrub is 20% TBP in n-dodecane. The strip solution concentrations are: HNO₃: 0.4M and N₂H₅⁺: 0.26M. The respective flow rates are 0.75, 0.156 and 0.12 L/hr respectively for feed, organic scrub and strip

ADDITIONAL INFORMATION ABOUT SX CODES: WHAT IS THE OBJECTIVE OF SOLVENT EXTRACTION CODES?

PUREX solvent extraction flowsheet has been greatly facilitated by the development of computer codes to do the tedious stage-by-stage equilibrium calculations involving multi solutes. It is a valuable tool for simulating various conditions and to arrive at optimum flowsheet conditions. In majority of the cases it helps in cutting down costly and hazardous (from the nuclear safety point of view) experimental runs required. Some of the important computer codes developed in the past for simulating PUREX solvent extraction systems are SEPHIS and its modifications, PUBG, PUMA, SOLVEX, etc., all of them designed for mixer-settler contactors. Except for PUMA, all these codes provide transient response of a contactor from some initial conditions to the steady-state conditions. Each of these codes is addressed to a specific case.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Electrochemical In-situ Reduction Process

The plutonium is chemically and electrochemically reduced to lower valence state and is back-extracted into the aqueous phase. Within an electro-reduction mixer-settler Pu(IV) reduction results from the following three reactions:

- i. Electrochemical Pu(IV) reduction,
- ii. Indirect Pu(IV) reduction by U(IV) [electrochemically formed],
- iii. Reduction of Pu(IV) by hydrazine.

The major fraction of Pu(IV) reduction is caused by U(IV) formed electrochemically within the extractor. Hydrazine reduces Pu(IV) much more slowly than does U(IV). It is common to add hydrazine as a holding reductant to prevent Pu(III) re-oxidation by nitrous acid, which is produced during electrolysis as well during dissolution step.

Electro-reduction Extractors

Pulsed column and mixer-settlers are used for electrochemical separation of uranium and plutonium. But mixer-settler offers more flexibility in operating conditions in comparison with column with regard to phase separation and accessibility for sample taking. At IGCAR, an electrolytic mixer settler based on ejector mixer type has been developed which was used for the uranium reduction experiment. In this unit, the extraction section of the mixer-settler bank is equipped with the electrodes in each settler without diaphragms. The cathode is made of titanium and the anode made of platinum or platinized Ti and Ta. Due to selective placing of the anode in the settling chamber, electrolysis takes place preferentially in the aqueous phase of the settler compartment.

BASIS OF MATHEMATICAL MODEL

To simulate partitioning process, detailed models of the distribution equilibria for the species involved under the prevailing conditions of partitioning cycle, the mass transfer kinetics and the redox reactions are needed. The development of model equations involves countercurrent multi-component extraction coupled with chemical and electrochemical reactions. It is assumed that complete mixing is assumed in mixer compartment of mixer-settler unit and mass transfer due to extraction occurs only in mixer compartment and the components are always at equilibrium between the phases. It is also assumed that stage residence times are sufficiently long so that diffusional resistances can be ignored. Material balance equations based on an idealized model for mixer-settlers incorporating distribution coefficients of the species involved and redox reactions are solved numerically in order to obtain concentration profiles of the species.

ACHIEVEMENT

The computer code, PUSEP has been developed for the analysis of partitioning cycle of PUREX process. It provides transient behavior of mixer-settler contactor from the start-up conditions to the steady-state conditions. The unique feature of the developed code is that it is applicable to all stages of PUREX process extraction, stripping and partitioning cycle. It has been used for the simulation of stripping runs of CORAL facility.

PUBLICATIONS

N.K. Pandey, R. Sivasubramanian, M. Sampath, R. Srinivasan, and S.B. Koganti, "Development of Electrolytic Mixer-settler for the In-situ Reduction of Uranium and Plutonium", CHEMENT - 2009.

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Development of Ultrasonic Guided Wave Based Methodology for Inspection of Hexcan Sheath of Fuel Subassemblies of PFBR

EXECUTIVE SUMMARY

An ultrasonic guided wave based methodology has been developed for rapid inspection of the hexcan sheath. Using the immersion guided wave based inspection methodology, both inner surface (ID) and outer surface (OD) reference defects of the order of 6mm (L) $\times$ 200 μ m (W) $\times$ 100 μ m (D) could be detected reliably in the hexcan sheath.

OUTLINE

Fuel subassembly of Prototype Fast Breeder Reactor (PFBR) consists of a hexagonal wrapper tube (Hexcan) housing fuel pins. The hexcans of PFBR are 3600 mm long and made up of 3.2 mm thick cold worked Alloy D9 (Ti-modified 316 austenitic stainless steel). Structural integrity of the hexcan sheath is important as any failure of this during service may result in separation of fuel pins from the subassembly interfering with fuel subassembly unloading operation and/or failure of fuel pins leading to contamination of the coolant with fuel and radioactive fission products. Hence, stringent nondestructive quality control of hexcan sheath is essential. This demands nondestructive evaluation (NDE) techniques employed to be sensitive to detect defects of the order of 10 mm (L) $\times$ 200 μ m (W) $\times$ 100 μ m (D) in hexcan sheath face and corners.

Scanning of large area ($\sim 3600 \times 75 \text{ mm}^2 \times 6$ faces / subassembly) using conventional focused angle beam ultrasonic transducer is highly time consuming, hence The developed methodology is quite fast and requires only one (/two) longitudinal line scans using four (/two) transducers for detection of any longitudinal defect in one face of the hexcan sheath. Similarly, for detection of transverse defects, transverse line scans at 20 mm spacing are only required for complete coverage of the hexcan sheath. The developed methodology substantially reduces the total amount of scanning time required as compared to the conventional angle beam inspection with similar defect detection sensitivity. However, as the sensitivity of the ID and OD defects are different in guided wave based inspection, guided wave based methodology is proposed to be used only for detection of defect. If a defect indication is observed, sizing would be carried out using conventional angle beam inspection using a point focused ultrasonic immersion transducer.

A new methodology and corresponding software were developed in LabVIEW for generating collated B-scan images. Such a collated B-scan provides image of the whole tube by integrating various B-scans (line scans) acquired at regular intervals. The collated B-scan images from simulated ID and OD face defects is shown in Figures 1 & 2.

For detection of defects in the hexcan faces, angle of incidence ($34 \pm 2^\circ$) at water/ specimen interface was standardized. The A-scans corresponding to a ID defect and a defect-free location are shown in Figure 3. The signal corresponding to water/specimen interface is also marked in the B-scan. It can also be observed from Figures 3 (a) and (b) that the defect is more than 20 mm away from the point of entrance of ultrasonic wave in the specimen. If the defect is nearer to the point of entrance, higher amplitude of reflection is expected from the defect.

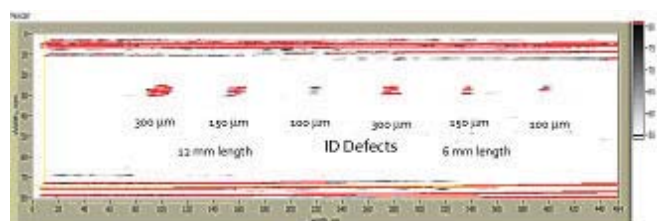


Fig. 1: Collated B-scan image of ID longitudinal defects (generated by processing four longitudinal B obtained at 20 mm intervals)



Fig. 2: Collated B-scan image of OD longitudinal defects (generated by processing four longitudinal B-scans obtained at 20 mm intervals)

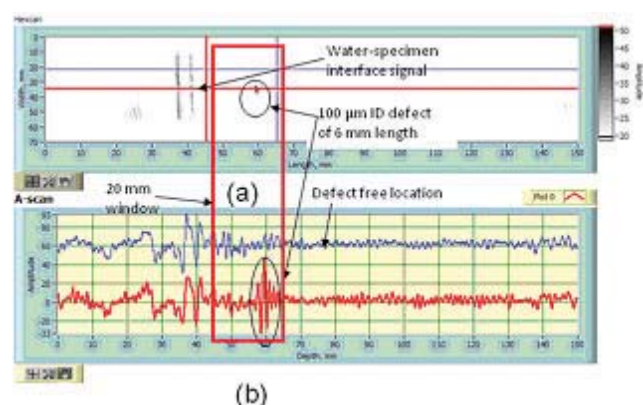


Fig. 3 : (a) B-scan corresponding to a ID defect of 6mm long, 100 μ m deep (smallest defect to be detected) (b) A-scan signal corresponding to defect (Fig. 3b) and defect free signal (window selected for generating a collated scan is also shown)

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Ultrasonic surface wave is found to contain its energy within a depth of about one wavelength and is able to travel along the surface of a component for a long distance with low attenuation. When the inspected component wall thickness is comparable to the wave length of the ultrasonic wave, the wave propagates along the full thickness and is guided by the two surfaces of the component. Such waves are called “Guided waves” and are gaining significant importance in inspection of thin walled components and complex geometries. The mode and sensitivity of such waves depend upon the frequency of the wave, thickness of the wall and angle of incidence of ultrasonic wave in the component.

ACHIEVEMENTS

Ultrasonic guided wave based methodology was successfully employed to characterise the artificial defects of various sizes in hexcan sheath successfully.

PUBLICATIONS

Development of ultrasonic guided wave based methodology for inspection of hexcan sheath of fuel subassemblies of PFBR, UMAS Report: IGC/NDED/UMAS/PFBR/UT/2008/12.

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Optimum Plant Layout for Twin Units of Future Sodium cooled Fast Reactors

EXECUTIVE SUMMARY

Beyond PFBR, it is planned to design five FBRs, each of 500 MWe capacity. Certain key features are to be introduced in these four reactors to achieve improved economy and enhanced safety. Among others, the optimum plant layout has a significant benefit on the reduction in the capital costs. Plant layout is optimized with a few constraints respecting safety and functional considerations. Twin reactor concept would be adopted for the future FBRs. The first two units viz. FBR-I & II would be constructed at Kalpakkam site. Further, various layouts of national and international reactors including certain advanced water reactors are studied thoroughly to arrive at the concept.

OUTLINE

The layout for the twin Fast Breeder Reactors at Kalpakkam is under optimisation with the prime objective of economy, safety, traditional design philosophies, O&M related issues, also taking into account comments of various committees and specialists groups. Further, various layouts of national and international reactors including certain advanced water reactors are studied thoroughly to arrive at the concept. Although there are many twin units available for water reactors, very few have been conceived for sodium cooled reactors. The area for the layout has already been earmarked near to PFBR. The size of selected site is 580 x 450 m, which is 580 in north-south direction and 450 in east west direction. The orientation of buildings is selected to be architecturally compatible with remaining facilities in the site. The reactors will have rectangular containment. The concept of Nuclear Island Connected Building (NICB) that has been adopted for PFBR will be retained. With these constraints, the layout has been optimised after through understanding of various building and their interface functions (Figure 1), from which broad guidelines have been derived, which has been taken as the reference for the present study, particularly to fix a few major dimensions of buildings.

The broad guidelines derived from literature review are: (i) Location of steam generator building (SGB) close to reactor containment building (RCB) to minimize the length of large diameter sodium piping from economic considerations; (ii) Location of Fuel building (FB) closer to RCB to facilitate efficient fuel handling operations; (iii) Location of Turbine building (TB) close to SGB to have minimum length of steam piping; (iv) Location of TB such that buildings housing safety and safety related equipment should be protected from low trajectory turbine missile; (v) Location of Control building (CB) such that cabling between CB & RCB is kept minimum; (vi) Location of Rad Waste Building (RWB) closer to RCB & FB to have minimum length of active piping; (vii) Location of Stack closer to RWB; (viii) Location of buildings linked by safety / safety related interconnected piping/fuel transfer/cabling preferably on a common raft; (ix) Provision of two separate diesel generator buildings (DGB) to ensure two independent trains of power supply free from common mode of failure; (x) Location of Switchyard closer to TB & transformer yard; (xi) Location of decontamination facilities preferably outside RCB to minimize use of water inside RCB and (xii) Sharing of certain facilities like FB, special handling facilities of component handling, decontamination facility, dismantling facility, transfer arm examination facility and sea water intake and outfall between twin units from economic considerations.

Based on the analysis of various options, an optimum layout has been arrived at (Figure 2). The salient features are: sharing of FB and RWB, CB, electrical building, switchyard, site assembly shops, two turbine buildings oriented in a manner that there is no concern of missile impact, provision of decontamination building in between the two RCBs (Figure 3), common to both units, provision of a traverser linking the decontamination building with RCBs through which the radioactive components are brought for decontamination and maintenance, provision of outer peripheral wall similar to PFBR for personnel movement and entry to various buildings. Normal personnel entry is through CB entrance and then to various buildings using peripheral corridor. NICB area of PFBR is 7705 sq.m. and 14017 sq.m. for FBR I & II. It is estimated that there will be an overall reduction of 20 % as compared to twice the layout area of PFBR.

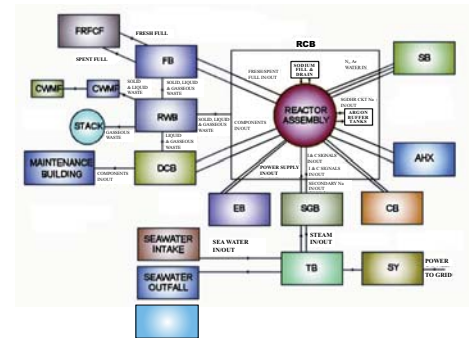


Fig. 1: Important buildings and the interface functions

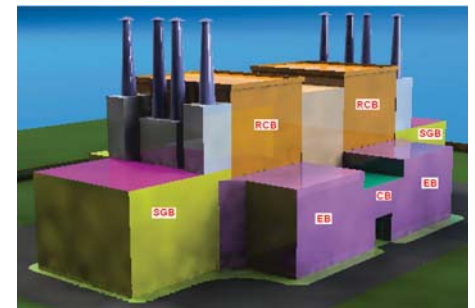


Fig. 2: 3-D view of optimum plant layout for twin units

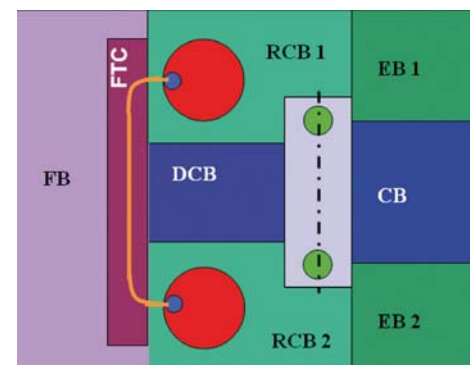


Fig. 3: Concept of common fuel buildings

ADDITIONAL INFORMATION

Engineering features of recommended layout have been identified and a systematic roadmap for R&D activities have been identified. Based on the detailed seismic analysis, it is recommended to isolate the reactor vault, by directly constructing from the base raft without inter-connecting it with the NICB, from the seismic loading considerations (Figure 4). This also provides some flexibility to design and analyze the reactor assembly components independently without linking to the finalization of design of rest of the civil structures. Therefore, additional cylindrical concrete wall is required around reactor vault to support the floors adjacent to the vault. One of the major revisions being studied is adopting anchored safety vessel concept. As per this, the safety vessel is removed. The steel liner is made to serve the functions of safety vessel. Accordingly, sodium compatible concrete embedded with thermal insulation is being conceived. With this, the inner diameter of vault can be reduced significantly (~ 700 mm).

Use of high strength (M60) for reduction of structural wall thickness, high density (3.6 / cc) for the reduction shielding thickness and self compacting concrete for reduction in the congestion of beam column junctions is to be considered. Further, optimisation of structural wall thicknesses for various buildings through static and seismic analyses, integration of components and piping layouts with the plant layout to ensure the compactness as well as the availability of sufficient space for inspection, maintenance and replacement and 3D visualization of plant layouts including construction sequences (Figure 5) are some of the immediate tasks to be completed for finalizing the layout.

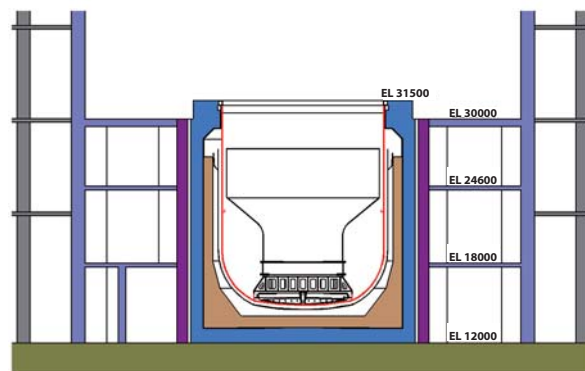


Fig. 4 : Concept of isolated reactor vault in NICB

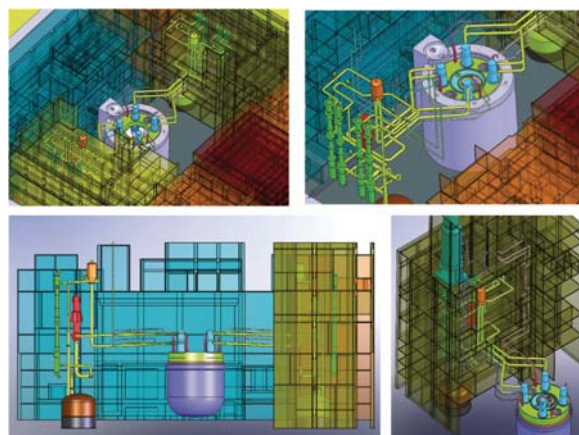


Fig. 5: Computer simulation of coupled civil and mechanical systems

ACHIEVEMENTS

The optimum plant layout can bring down the capital cost of the FBR project to the extent of 10%. This will also have high impact to the international future FBR developments.

Further inquiries:

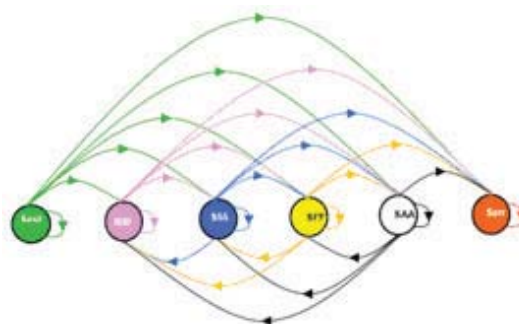
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The reliability of Programmable Logic Devices (PLD) especially when they are used to build safety-critical systems is very important and it is necessary to validate the failure rate value by subjecting the device to a quantitative Accelerated Life Test (ALT). During ALT, the devices are subjected to one or more combinations of the stress parameters that include environmental parameters like temperature. According to the I & C safety norms, to enhance the reliability, PLDs are de-rated and used in the implemented designs. Considering the testing environment and interface circuitry, a testable stress design is proposed instead of the existing available designs of the PLD. The same is achieved to an extent using Synthetic Benchmark Circuit (SBC) approach.

Temperature is the preferred primary stress parameter for electronic devices. It is due to the fact that for every 10°C rise in temperature, the leakage current doubles. For ALT, along with temperature, the stress design is proposed as a secondary stress parameter. This design should be inline like a benchmark circuit to the targeted PLD. Since the design is exclusive for test purpose, the specifications for the testable stress design is determined by the targeted device parameters, test conditions and test environment conditions. To start with a commercial vendor's Field programmable Gate Array (FPGA) device is selected for this purpose and a set of stress specification is defined. According to them, a testable stress design is generated. A design is called a stress design of a device when it is able to meet each of these parameters. The parameters include maximum FPGA logic utilization, FPGA I/O utilization, inter-connection circuitry, power consumption and operating frequency. In addition, the design should be highly testable to find out whether the device has failed or not. ALT testing constraints dictate that the amount of additional logic circuitry, like the circuitry for input pattern generation, circuitry for checking outputs etc, need to be incorporated into the FPGA itself, because, adding additional devices for these functions pose problems during diagnosis of a failure as it will be impossible to determine the cause of failure (whether it was due to failure of the FPGA or the failure of the supporting devices).

Table 1: Results of stress design

Tool	Comb Modules.		Seq. Modules		Total		RAM		I/O	
	Out of 1184	%	Out of 1230	%	Out of 2414	%	Out of 2560 bits	%	Out of 176	%
Synthesizer	1691	143	1109	1109	2800	116	2048	80	172	97
Place and Route	856	72	1109	90	1965	81	2048	80	172	97



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GENERAL EXPLANATION RELATED TO DESCRIPTION

Today's CAD tools and PLD architectures under development are for tomorrow's designs. The requirement and complexity of future designs will be definitely exponential compared to the present. So it is vital to venture the possible validation techniques for evolving tools and device architectures, thereby certifying their suitability to face the challenges of the future. Even validating the existing tools and used PLD architectures is of utmost importance for high integrity systems. Therefore, SBC helps in a large way in this effort. A circuit can be characterized with respect to a circuit space. A circuit space is a multi-dimensional space formed by the set of characteristic parameters that include size, topographical and functional parameters. As the name suggests, size parameters express the size of a circuit in terms of number of nodes, number of nets, number of flip-flops, number of primary inputs and primary outputs. Topological parameter represents the interconnection structure of the circuit with fanin distribution and netdegree distribution. In a circuit, the number of blocks to which a net is connected is called the net-degree of a net. In a circuit, the statistics about the nets with the same net-degree is denoted by the net-degree distribution. A design's complexity is determined by the interconnection complexity of the circuit. Finally, functional parameters are defined based on the desired functionality of the user/tool. For example, to evaluate a test generation tool one may need a benchmark with many undetectable single-stuck faults.

ACHIEVEMENTS

The SBC generation and the associated experimental results imply that generation of SBCs that meet all the criteria for stress testing is not easy. After many permutations, a benchmark testable stress design is achieved with good interconnection complexity. The benchmark for the FPGA device is designed using HDL. Even the same building blocks can also be utilized to test any programmable logic devices in future for similar test.

PUBLICATIONS

L. Srivani, V. Kamakoti and S. Ilango Sambasivan, "Constructing Synthetic Benchmark Circuits To Stress Test FPGAs" in Proceedings of 13th IEEE VLSI design and Test Symposium (2009).

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Critical Analysis of Prototype Fast Breeder Reactor Designs for Sixty year Plant Life

EXECUTIVE SUMMARY

Higher design life is one of the major contributing factors in cost reduction of FBR. As a follow-up to PFBR with 40 y design life (75 % load factor), it is planned to construct four 500 MWe commercial fast breeder reactors (CFBRs), having 85 % load factor and 60 y design life. Under higher operating temperature over a prolonged period in association with temperature variations following thermal transients, the hot pool components are subjected to creep, fatigue and creep-fatigue interactions which are the main life limiting factors. Apart from these, a few special types of failure modes, viz. thermal stripping and thermal ratchetting occur on components when they are subjected to temperature fluctuations caused by thermal hydraulic phenomena. Based on a critical review of PFBR design and the conservative loads considered vis a vis realistic loads, it is established that 60 y of plant life can be achieved comfortably, with adequate margin.

OUTLINE

Major Issues Related to Plant Life of SFRs

Deduction from PFBR R&D and international experience reveals that material degradation issues do not influence design life of FBR. Structural mechanics aspects:

- 1) Creep-fatigue damage
- 2) High cycle strain controlled fatigue damage
- 3) Thermal ratchetting
- 4) Loss of wall thickness in intermediate heat exchanger (IHx) and steam generator (SG) tubes control the design life.

The various failure modes in hot pool components are depicted in Figure 1.

High cycle strain controlled fatigue damage

Sodium free level fluctuations, oscillation of stratified sodium layers and thermal stripping are the special problems which cause strain controlled high cycle fatigue (HCF) damage. Sodium free level produces cyclic variations of temperature and stress on the adjoining metal wall surface. The presence of high cycle fatigue cycles at the location where LCF and creep damage are significant, is the most damaging scenario. Permissible thermal stripping limits (ΔT) are derived as a function for two cases:

- (1) There is no thermal attenuation on the surface wall temperature ($h - a$) and
- (2) There is thermal attenuation depending upon the frequency. The effect of frequency-dependent thermal attenuation on the structural wall surface is quantified and found to be significant. The maximum range of temperature fluctuations can be as high as 70 K where the accumulated creep-fatigue damage is insignificant (<0.1) and 45 K is acceptable where the creep-fatigue is very significant (0.9), as depicted in Figure 2.

Thermal Ratchetting

The sodium free levels vary slowly following changes in hot and cold pool temperatures. The level variations cause large temperature and stress variations. The affected components are main vessel (MV), inner vessel (IV) and thermal baffles, of which MV is critical. The maximum axial temperature gradient ($\Delta T/\Delta X$) along the main vessel is about 1200 K/m at about 850 K during safety grade decay heat removal condition (SGDHR). High stress and temperature conditions prevail for sufficiently longer time (~ 1 d) to introduce significant creep deformations. The stress variations cause thermal ratchetting (progressive growth of strain) enhanced by creep deformation. Studies show that the vessel undergoes shakedown after ~ 20 y. So strain accumulation after 60 y is same. Maximum deflection of (~ 15 millimeter) due to ratchetting is less than the MV thickness (25 millimeter), hence there is no risk of buckling. Maximum accumulated hoop strain after 60 y is ~ 0.3 % which is lower than the acceptable value of 0.5 % for welds.

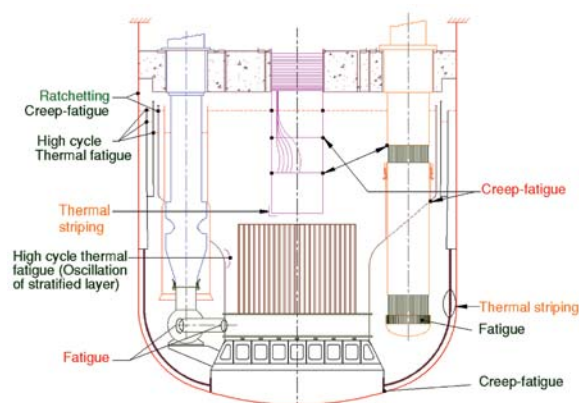
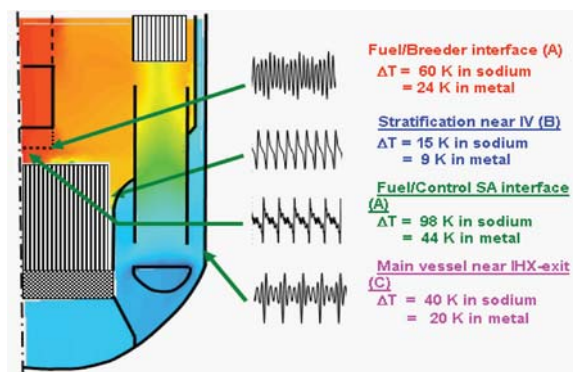
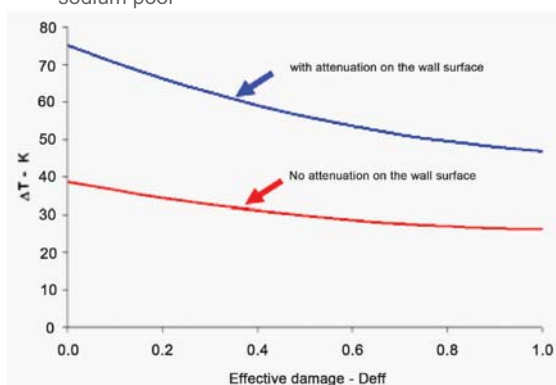


Fig. 1: Failure modes in hot pool of a FBR



Thermal stripping zones and temperature fluctuations in sodium pool



Permissible thermal stripping limits for SFR structures, made of SS 316LN

Fig. 2: Thermal stripping zones and temperature fluctuations in sodium pool

GENERAL EXPLANATION RELATED TO DESCRIPTION

Creep fatigue damage: The creep-fatigue damage is assessed for the high temperature components, viz. MV, control plug (CP), inner vessel (IV) and IHX as well as steam generator (SG) shell-skirt support, as per RCC-MR,2002 (Figure 3). For the MV and inner vessel the stresses and strains are computed using viscoplastic analysis, since it is not possible to respect the creep fatigue damage limits through elastic analysis route for these components. For the viscoplastic analysis, the Chaboche model is employed. For other components viz. control plug, IHX and steam generator shell-skirt support, elastic analysis route is followed. For the control plug and inner vessel, the thermal transients following reactor scram are considered. For the IHX and steam generator, the shutdown-normal operation-shutdown conditions constitute load cycles. The main vessel is subjected to cyclic thermal stresses due to free level variations under DHR conditions. The summary of computed creep fatigue damage assessment of hot pool components for 60 y of design life is presented in Table 1. Permissible life of IHX can be 85 y as per codes.

Loss of wall thickness in IHX and SG tubes: Tubes in IHX are straight with 0.8 millimeter nominal wall thickness and 19 millimeter OD. After subtracting the corrosion allowance 'C' (0.0035 millimeter/y) of 0.14 millimeter for 40 y and 0.21 millimeter for 60 y and tolerance on the wall thickness of 0.08 millimeter, the net thickness available is 0.58 millimeter at the end of 40 y and 0.51 millimeter at the end of 60 y. Further fretting wear rate 'W' of 0.01 mm/y is considered at the belt location. IHX tube with thickness of 0.8 millimeter with fretting wear rate 0.01 millimeter / a can go up to 41.8 y (~ 55 y with 75 % LF), for 60 y of design life 1.045 millimeter tube thickness would be required. Hence, minimum thickness required for design life of 60y for IHX is ~ 1.05 millimeter. In view of pessimistic value of fretting wear rate, it is judged that IHX could have 60 y of design life with tube thickness 0.8 millimeter. Bend tubes of 17.4 millimeter OD and 2.4 millimeter nominal wall thickness would be used for steam generator of CFBR. As the steam outlet side has higher temperature as compared to water inlet side, steam outlet side would govern the minimum thickness requirement. The adequacy of tube thickness is checked based on corrosion, reduction due to edge preparation and weld thinning and fretting wear at support location. For 60 y design life steam generator tube thickness should be ~ 2.452 millimeter from fretting wear consideration at support location (higher by 0.052 millimeter from steam generator tube thickness value of 2.4 millimeter).

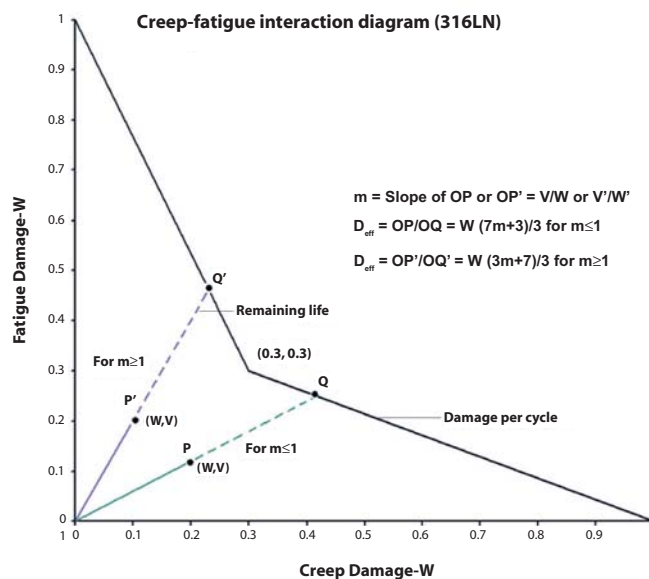


Fig. 3 : Bilinear curve to determine effective damage

Table 1: Creep fatigue damage of hot pool components for 60 y life

Component	Load cycle/annum	Hold time/cycle (h)	Creep Damage (Dc)	Fatigue Damage (Df)	D _{eff}
Main Vessel	4 SGDHR	24	0.030	0.0105	0.054
Inner Vessel	19 Scrams	350	0.075	0.0225	0.128
Control Plug	19 Scrams	350	0.540	0.0075	0.558
IHX	22 Shutdowns	305	0.675	0.0045	0.686
SG	22 Shutdowns	305	0.450	0.0450	0.555

ACHIEVEMENTS

This work is of high relevance for the future CFBR, wherein the primary focus is economy. Based on PFBR design studies and reassessment of conservatism in-built in the design, it is established that 60 y plant life for CFBR can be comfortably achieved with adequate safety margins.

PUBLICATIONS

Plant Design Life for CFBR, PFBR/30000/DN/1099/R-A.

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Simulation of Safe Operational Regimes of Primary Sodium Pump for Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

One of the important safety requirements is the demonstration of smooth operation of primary sodium pump (PSP) to ensure adequate core flow during all the operating conditions, including seismic events. The kind of investigations carried out, are the investigation of forward and backward whirling instability of pump and seizure of pump under seismic forces. Towards this, the dynamic characteristics of hydrostatic bearing (HSB) and gyroscopic effects under all the operating conditions are numerically simulated. The stability analysis indicates that primary sodium pump can enter into unstable regime once speed is greater than 1.5 times rated rotation per minute. Further, the radial displacement of shaft at the hydrostatic bearing location is found to be $250\text{ }\mu\text{m}$ which is less than the nominal radial clearance of $400\text{ }\mu\text{m}$ that ensures that there is no risk of impact of shaft on the journal of hydrostatic bearing during safe shutdown earthquake.

OUTLINE

In PFBR, there are two primary sodium pumps (Figure 1) delivering sodium flow to the core, which are mechanical pumps with long shafts (12 m). The pump's impeller is immersed in sodium on one side and is thrust bearing on the other side. The thrust bearing at the top sees the normal atmosphere, while the bottom portion (impeller side) sees sodium pool. For good vibration stability, it is required to use a bearing immersed in sodium. Since oil is not permissible within sodium pool due to safety reasons, hydrostatic bearing which uses sodium itself as a fluid is generally adopted in the design. The life of reactor and in turn the bearing should be to the extent of 40-60 years. Since the pump has to operate at variable speeds in the range of 200-590 rpm, it is important to ensure that there is no instability and seizure at any operating speed.

Resonance occurs when the natural frequency of the pump matches with the seismic excitation. Whirling may occur when the natural frequency matches with the speed of pump. A situation of great concern is the coincidence of pump natural frequency ($\sim 10\text{ Hz}$), nominal speed ($\sim 10\text{ Hz}$) and predominant frequency of seismic excitations ($\sim 10\text{ Hz}$), which may damage the pump. So the dynamic stability of the pump should be investigated critically while designing them. This investigation for dynamic instability has concluded that, even with the perfect coincidence of resonance and whirling, the pump rotates under stable condition, due to the inherent damping mechanisms and insufficient time available for the system to attain instability. Further, investigation is required to ensure that there is no seizure in case of enhanced dynamic displacements under the influence of seismic induced forces. For a comprehensive investigation on these failure modes, a dedicated software has been developed and the PFBR pump has been analysed for vibration, instabilities and seizure (Figures 2 and 3). The results are highlighted below.

Vibration Analysis

The fluid mechanics analysis quantifies the leakage flow through HSB ($81\text{ m}^3/\text{h}$ and $16.2\text{ m}^3/\text{h}$ for 590 and 120 rpm respectively) and minimum and maximum pressures in pocket (42 and 65 m of sodium head at 590 rpm). The load carrying capacity was derived as a function of shaft eccentricity and at the rated rpm, the maximum possible load bearing capacity when eccentricity is very close to the radial clearance is $\sim 45\text{ t}$. The vibration analysis of PSP subjected to unbalanced forces has been carried out by integrating the governing fluid mechanics equations for HSB to derive stiffness coefficients at every instant and gyroscopic effect. Analysis indicates that the maximum displacement of shaft at 590 rpm is less than $60\text{ }\mu\text{m}$ under unbalanced condition, which reduces to $14\text{ }\mu\text{m}$ under balanced condition. The resonant speed has been obtained by dynamic response analysis, as 700 rpm which has a margin of 1.2 against the nominal speed. The maximum amplitude of shaft is limited to $160\text{ }\mu\text{m}$, when the shaft runs even at resonance speed. At 15% speed, the vibration levels are insignificant. The dynamic forces on the roof slab and primary pipe header are found to be less than 3t which are judged to be small.

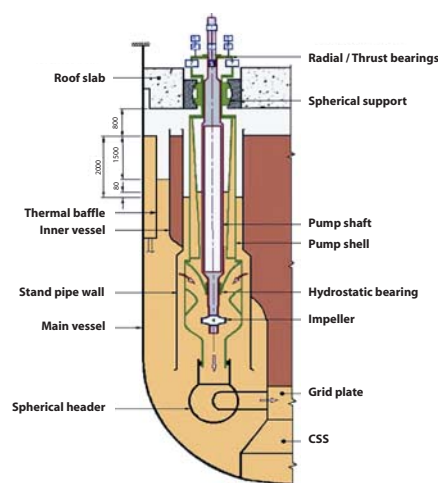


Fig. 1: Schematic of primary pump

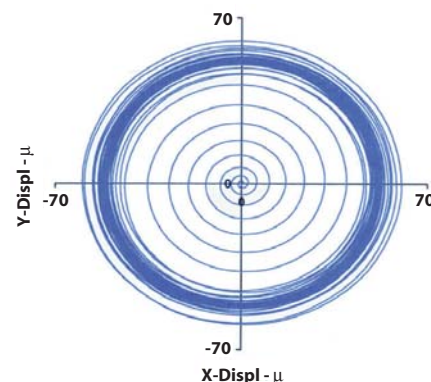


Fig. 2: Orbit of CG of shaft at HSB location

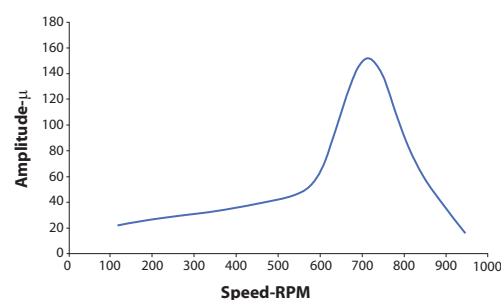


Fig. 3: Pump shaft resonance

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Dynamic Stability Analysis

Developed for predicting the dynamic behaviour and stability of the pump shaft with HSB. The investigations on the forward and backward whirling instability has been investigated and found that PSP can enter into unstable regime once speed is greater than 1.5 times rated rpm (Figure 4 & 5).

Pump Seizure Analysis under SSE

The seismic analysis of primary pump has been carried out by time history. The stiffness coefficients of hydrostatic bearing are computed as function of eccentricity, speed and angular orientation of shaft. The associated fluid mechanics equations are formulated and solved integrating with the governing structural dynamics equations. The important outcome of the analysis are: (i) the maximum stress intensity values at the support shell and shaft are found to be insignificant (<20 MPa) and (ii) the imposed displacements on flexible coupling (~ 1 mm peak) are insignificant. The eccentricity of $60\text{ }\mu\text{m}$ experienced by the shaft during normal operation increases to $250\text{ }\mu\text{m}$ under Safe Shutdown Earthquake (SSE). Hence, there is no problem of seizure since the eccentricity experienced by the shaft is $< 400\text{ }\mu\text{m}$. At speeds lower than 200 rpm , the eccentricity of shaft is greater than radial clearance and hence the shaft can impact on the shell under SSE. Under Operation Basis Earthquake (OBE) up to 20% speed ($\sim 120\text{ rpm}$), there is no fear of impact. Peak impact force experienced by the shaft & shell at HSB at 235 rpm under SSE is $<3t$, which is judged to be insignificant and may not cause the pump to seize. This needs to be confirmed by experiments. Transient forces on the primary pipe spherical header and roof slab have been extracted for the subsequent check on structural integrity. The maximum force on the pump spherical header at the nominal speed and roof slab under SSE, is found to be less than $20t$, which are judged to be insignificant particularly for the roof slab which is relatively rigid structure. In summary, the performance of PSP during seismic events is found to be satisfactory.

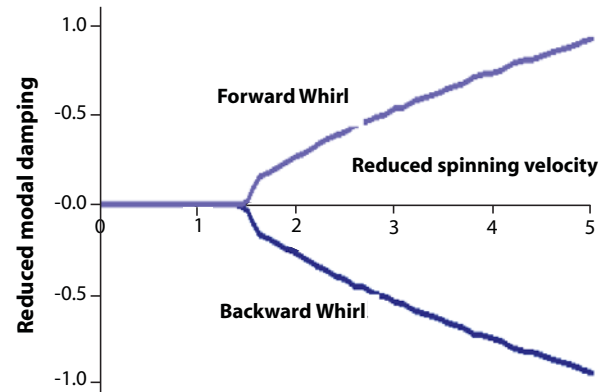


Fig. 4 : Stability chart for primary sodium pump

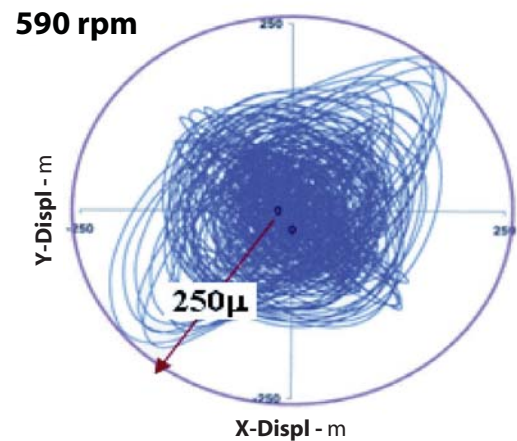


Fig. 5 : Orbit plot of PSP at HSB

ACHIEVEMENTS

Investigation of pump for vibration, background and forward instability and seizure at hydrostatic bearing location under seismic forces calls for dedicated computer code, which has been developed and applied to the investigation of PFBR pump. This raises the confidence of safe operation of sodium pump. The code can be used for the investigation of future reactor pumps.

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Simulation of Fast Transient Fluid Structure Interaction in Prototype Fast Breeder Reactor for Safety Analysis

EXECUTIVE SUMMARY

For the analysis of mechanical consequences of Core Disruptive Accident (CDA), i.e., vessel strains, sodium impact and sodium ejection to reactor containment building (RCB) in a pool type Sodium Cooled Fast Breeder Reactor, a sophisticated computer code called FUSTIN has been developed. Among many challenges such as, large distortion in the fluids and structures and fluid structure interaction effects, the simulation of complicated core bubble expansion / contraction around the reactor internals cannot be simulated by the state of art system viz., Arbitrary Lagrangean Eulerian (ALE). To solve this, a special finite element based fluid element viz., multi-phase component fluid element has been developed and implemented in the FUSTIN code. Using this code, the transient responses of PFBR main vessel are determined, with good understanding on the complicated fluid motions, sequence of loadings and effects of introduction of internals on the fluid structure interaction effects.

OUTLINE

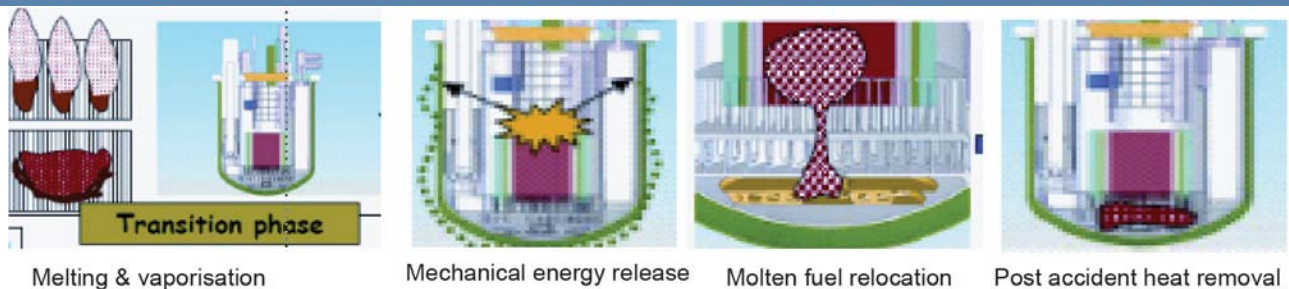


Fig. 1: Mechanical and thermal hydraulic consequences of CDA

The scenario depicting the consequences of CDA is illustrated in Figure 1. The Unprotected Loss of Flow Accident (ULOF) leads to melting of almost whole core, with simultaneous generation of vapour phase consisting of fuel, coolant and structural materials. In the pre-disassembly phase, the heat generated due to power excursion could melt the core, to the extent of attaining a saturated liquid state. Subsequent heating generates saturated liquid/vapor phases at high temperature (~ 5000 K) and pressure, which has potential to do mechanical work (Figure 1a). The mechanical energy release is due to expansion of vapor phase from initial pressure to the ambient pressure. During this rapid expansion of core bubble in the liquid sodium pool, the pressure/shock waves are generated, which deform the shell structures surrounding the core (Figure 1b). Subsequent to the mechanical work, the vapor phase condenses due to the cooling effect of sodium pool, which is at 855 K and molten fuel starts moving downwards melting the support structures, such as grid plate and core support structure (Figure 1c). Ultimately, the liquid settles down on the core catcher structure and subsequently debris collected on the core catcher will be cooled by natural convection (Figure 1d). The scenario brings out clearly the complicated distortions, undergone by the core bubble, particularly with the presence of above core structures.

Precise numerical simulation of the core bubble expansion amidst the structures immersed in the sodium pool is not successful even with state-of-the-art computer code such as FUSTIN. The main difficulty is in the inadequacy of existing rezone algorithm, which itself is advanced. To overcome this difficulty, a 'homogeneous fluid element concept' has been developed, with which the existing rezone algorithm can yield desired results, and implemented in FUSTIN code. The homogenized fluid element can be only liquid or only gas or homogenized mixture of liquid and gaseous phases (Figure 2). In this concept, the identification of exact liquid and gas interface is naturally lost. However, because of the large difference in the density values of liquid and gaseous phases, the graphical display of isovalue density contours indicates clearly the state of the interface within a narrow band. The main issue is derivation of appropriate equation of state for such multi-phase element which has been solved successfully and implemented in FUSTIN code.

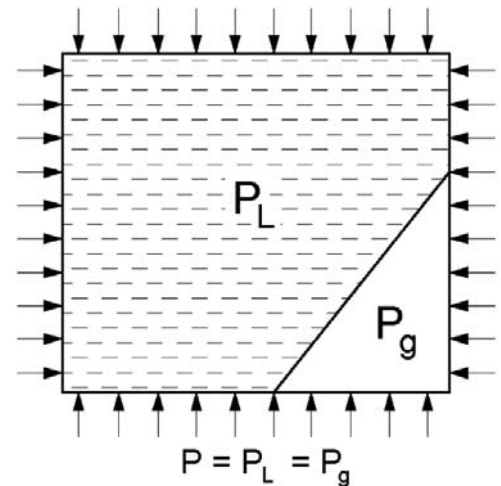


Fig. 2: Two phase fluid element

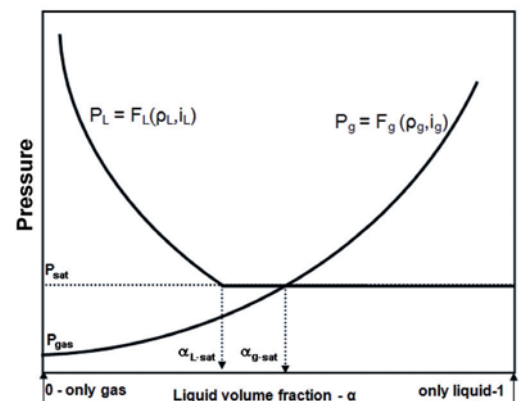


Fig. 3: Two phase fluid element

GENERAL DESCRIPTION

The two phases are assumed to be immiscible and each phase keeps its own behavior law (equation of state) unchanged within the element. The equation of state is derived for typical element, from the known quantities, viz. net element mass M , internal energy I , volume V , liquid mass fraction is β , internal energy fraction γ . The liquid volume fraction α is adjusted so that pressures in the liquid and gaseous phases are equal, which is expressed in the following equations which involve the equations of state for liquid (F_L) and gas (F_g):

$$P = F_L(\rho_L, i_L) = F_g(\rho_g, i_g)$$

where $\rho_L = \beta.M / \alpha_{sol}V$, $\rho_g = (1 - \beta).M / (1 - \alpha_{sol})V$, $i_L = i_g = I/M$. The only unknown α_{sol} is solved by using Newton-Raphson method, which calls for upper and lower bound values of α_{sol} . There exists a possibility of vapourisation of liquid phase that poses challenge in the solution algorithms. In order to take in to account the vapourisation effects, various possible scenarios are considered carefully. The possible situations that lead to net pressure of the element higher than the saturated vapour pressure (P_{sat}), so that there is no vapourisation of liquid phase have been identified. There can be a situation at which the volume of the liquid at which $\alpha_{g-sat} < \alpha_{L-sat}$ and hence, there is no solution for α (Figure 3). Under this situation, the liquid vapourises. Since the entire element contains two gases, the element pressure (P) is sum of gas pressure (P_g) and vapour pressure (P_{sat}).

Employing the new fluid element, the mechanical consequences of CDA are depicted accurately with the FUSTIN code. Figure 4 shows the overall sequence of loading up to 200 ms at an interval of 50 millisecond, beyond which the results are stabilized. The important sequence of events observed from the analysis are: upward bowing of core cover plate of control plug and bulging of inner vessel (Figure 4ii), downward displacement of main vessel bottom portion (Figure 4 ii), core bubble crossing the control plug (Figure 4iii), sodium slug impact on the top shield (Figure 4iv), reflection of pressure waves from top shield and downward bowing of core cover plate (Figure 4.v) and downward displacements of sodium free levels (Figure 4v). High local straining of main vessel due to load transmitted at the triple point and the sodium impact restricted to a small zone on the top shield around the control plug and no impact of sodium slug within the control plug on the top shield can be seen in the Figure 4. With the kind of rezone algorithm employed and efficient multi phase element employed, it is possible depict clearly the complicated motions of core bubble around the control plug and sequential immersion of inner vessel in the sodium pools, while preserving the regularities of meshes as smooth as possible till the end of analysis.

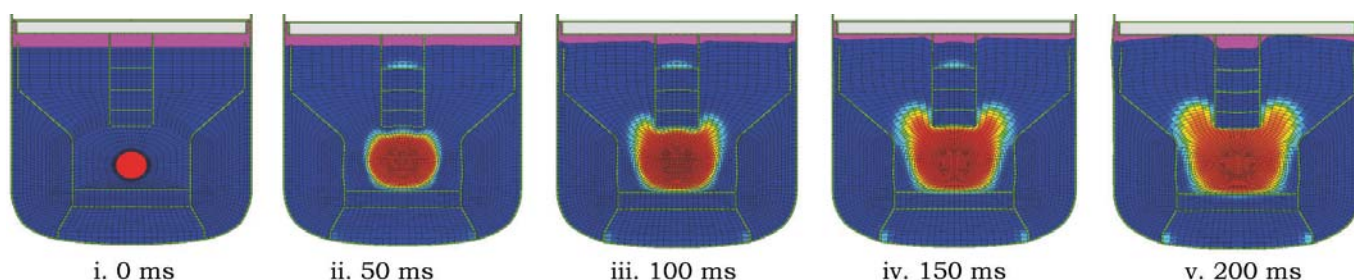


Fig. 4: Core bubble behaviour as depicted by the analyses

BRIEF DESCRIPTION ON IMPACT TO FBR PROGRAMME

Advanced CFD computations coupled with fast transient fluid structure interaction effects enhance the confidence on the safety of PFBR under severe accident conditions. Further, this is truly an international effort.

PUBLICATION

P. Chellapandi. S.C.Chetal and Baldev Raj, "Structural Integrity Assessment of Reactor Assembly Components of A Pool Type Sodium Fast Reactor under Core Disruptive Accident", Paper sent to J. Nuclear Technology (2009).

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Design of an Array Receiver for defect Detection by Finite Element Modeling of Remote Field Eddy Current Technique

EXECUTIVE SUMMARY

Three-dimensional finite element (FE) modeling of remote field eddy current (RFEC) technique has been developed to detect localized defects in ferromagnetic steam generator (SG) tubes of Prototype Fast Breeder Reactor (PFBR). The excitation frequency, inter-coil spacing and the design of localized receiver coil have been optimized using this model. Amplitude and phase of remote field eddy current signals of radial and axial receiver coils have been predicted for a through hole and two flat bottom holes (FBH). The detection sensitivity of the receiver coils has been compared and the model has been validated for a few cases.

OUTLINE

Remote field eddy current technique has been chosen for in-service inspection (ISI) of steam generator tubes of PFBR. The Remote field eddy current technique is a potential technique to detect uniform wall thinning due to corrosion in steam generator tubes. This low-frequency eddy current technique uses separate exciter and receiver coils and detects internal and external corrosion wall loss with equal sensitivity. Use of conventional circumferential receiver coil based remote field eddy current probe, results in poor detection sensitivity for localized defects. In this context, use of localized receiver coils (Figure 1) is advantageous. Design and development of localized or array type remote field eddy current sensor is a challenge and requires a thorough understanding of the Remote field eddy current phenomenon. This involves accurate determination of direct, transition and remote field regions in the tube at different frequencies and will be useful for optimizing the test frequency and design of localized receiver coils such as axial receiver coil and radial receiver coil. Finite element model based optimization and analysis has been performed in collaboration with Michigan State University, USA to avoid time consuming and tedious experimentation.

Detailed analysis of the model predicted amplitude and phase of the induced voltage calculated for a finite width bobbin coil in the case of a tube without defect at frequencies of 1100, 1300 Hz, 1500, 1700 and 1900 Hz, revealed that the optimum frequency is 1500 Hz and intercoil spacing of the remote field eddy current sensor is 45 millimeter. This optimized parameters has been used for predicting the remote field eddy current amplitude and phase signals due to a 1.5 millimeter diameter through hole, 60% and 20% flat bottom holes. The remote field eddy current signals for these defects have been predicted for two different types of array receiver coils, viz., radial and axial type as shown in Figure 2. The remote field eddy current peak phase difference relative to the baseline for through hole, 60% FBH and 20% FBH using axial and radial coils are given in Table I. It is clear from Table that the detection sensitivity of radial coil is better than the axial coil. The enhanced detection sensitivity of radial coil is due to larger radial leakage fields entering from the tube wall due to the presence of localized defects.

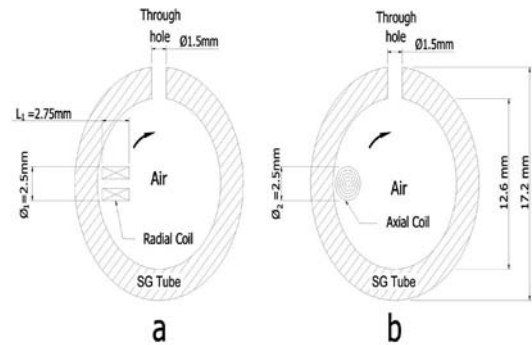


Fig. 1: Cross sectional view of a) radial receiver coil and b) axial receiver coil in SG tube having a through hole of 1.5 mm diameter

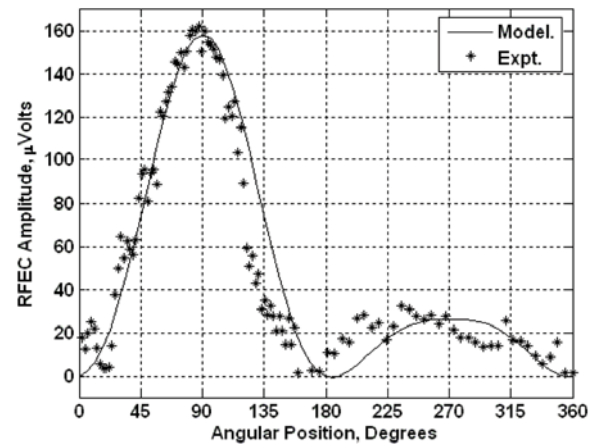


Fig. 2: Validation of model predicted amplitude for a 1.5 mm diameter through hole in SG tube at 1500 Hz

Table I: Peak phase values of RFEC signals for axial and radial type array receiver coils

Defect type	Phase difference, degrees	
	Axial Array	Radial Array
Through hole	10	136
60% FBH	1.7	43
20% FBH	0.25	7

ADDITIONAL INFORMATION

Detailed analysis of the model predicted amplitude and phase of the induced voltage calculated for a finite width bobbin coil in the case of a tube without defect at frequencies of 1100, 1300, 1500, 1700 and 1900Hz, revealed that the optimum frequency is 1500 Hz and intercoil spacing of the remote field eddy current sensor is 45 millimeter. This optimized parameters has been used for predicting the remote field eddy current amplitude and phase signals due to a 1.5 millimeter diameter through hole, 60% and 20% flat bottom holes. The remote field eddy current signals for these defects have been predicted for two different types of array receiver coils, viz., radial and axial type as shown in Figure 1. The remote field eddy current peak phase difference relative to the baseline for through hole, 60% FBH and 20% FBH using axial and radial coils are given in Table I. It is clear from Table that the detection sensitivity of radial coil is better than the axial coil. The enhanced detection sensitivity of radial coil is due to larger radial leakage fields entering from the tube wall due to the presence of localized defects.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The governing partial differential equation of remote field eddy current phenomenon for quasi-static time harmonic case can be derived from the following Maxwell's equations as

$$\Delta^2 A = j \omega \mu \sigma A + \sigma \mu \nabla V - \mu J$$

Analytical solution to above equation is difficult due to multiple interfaces and boundaries, especially in the presence of defects. These can be readily handled by finite element methods and they are versatile and computational efficient. This equation has been solved to investigate the remote field eddy current phenomenon in steam generator tubes. The remote field eddy current model geometry consists of a steam generator tube and excitation coil surrounded by air region. The outer diameter of exciter coil is 6.2 millimeter. The modeled region is discretized with hexahedral elements. The mesh is optimized after systematic trials that give due emphasis to solution accuracy and computational time. The optimized mesh consists of 671744 hexahedral elements and 697125 nodes. Figure 3a and 3b shows the meshing in vertical and horizontal cutaway sections of the modeled region. The model was based on eight noded hexahedral elements with linear shape functions. Quasi Minimal Residue (QMR) iterative solver is used to solve the global matrix to obtain the electric scalar potential (V) and magnetic vector potential (Ax, Ay, Az) at the nodal points of hexahedral mesh. A preconditioning technique based on incomplete LU decomposition scheme, was used for approximating the solution matrix (prior to QMR) to minimize the computational time and resources. A high performance cluster computational facility was used for solving the global matrix.

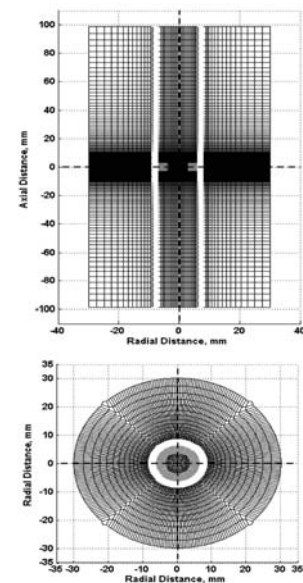


Fig. 3: a) Vertical and b) horizontal cutaway sections of finite element mesh generated to model the RFEC phenomenon

ACHIEVEMENT

Three-dimensional FE modeling of remote field eddy current technique has been developed and successfully used to design an optimal array receiver for detection of localized defects in steam generator tubes. Optimum frequency and inter-coil spacing are found to be 1500 Hz and 45 millimeter. Studies reveal that the radial array receiver coil is ideal for detection of localized defects as compared to axial array receiver coil. When radial coil is used, a through hole produced unique two-peak amplitude signal and this behavior is attributed to formation of conical field inside the tube due to strong perturbation of magnetic flux inside the tube wall. This two-peak behavior is promising for reliable detection of pin-hole type defects and other leak paths in the tubes.

PUBLICATIONS

S. Thirunavukkarasu, B.P.C. Rao, S. Mahadevan, T. Jayakumar, Baldev Raj, Z. Zeng, L. Udpa and S. S. Udpa, "Finite element modeling for detection of localized defects using remote field eddy current technique", Journal of Research in Non destructive Evaluation, 20 (2009) 245.

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Mono-Articulated Links Manipulator for Remote In-Service Inspection

EXECUTIVE SUMMARY

Reprocessing of spent fuel is a crucial stage in Indian Nuclear Energy Programme since it facilitates recovery of fissile fuel. Reprocessing of spent fuel involves operations in highly disordered and radioactive environment. Reliable performance and safe operation of fuel reprocessing plants is essential for sustained throughputs. Despite successful commissioning of a reprocessing plant, periodic in-service inspection (ISI) and monitoring of critical plant components is necessary in order to ensure that service-induced defects and abnormalities are detected and remedied beforehand. During in-service inspection and in many other instances, limited access, radiation and acidic environment prevailing in a reprocessing plant obviate direct human intervention necessitating remote tools and gadgets. Remote visual inspection of process tanks below the radioactive containment box was carried out successfully in COMpact facility for Reprocessing of Advanced fuels in Lead cells (CORAL) at Kalpakkam.

OUTLINE

One of the in-service inspection (ISI) tasks identified in CORAL is the inspection of the space below the containment box where tanks and vessels containing highly active process solution are located. Inaccessibility and hostile environment had necessitated remote intervention by deploying a remote gadget for inspection. As part of ISI of CORAL, a remote inspection device was developed for inspecting the space below the containment box including the process tanks and successfully demonstrated in the plant. Visual examination is the NDE technique adapted for the inspection of the process tanks located below the radioactive containment box in the CORAL reprocessing plant.

The stainless steel containment box is shielded by lead bricks all around the box and the process tanks and vessels with highly active process solution are kept below the containment box. The gap between the containment box and the lead brick wall is nominally 100 mm and decreases wherever the window protrudes from the lead wall towards the containment box. The difficulty comes in the form of restricted access, lack of ISI-specific penetrations available in the lead cell, narrow space between the lead cell and the containment box and piping work between the lead cell and containment box.

A thorough tube opening of mini master slave manipulator for deploying the inspection device which will pass through the gap between the lead wall and the containment box has been designed. This manipulator will be able to manipulate the camera below the containment box. The narrow gap between the lead cell and containment box precluded the possibility of deploying a single rigid arm, 2 metres long with the end-effector. A manually actuated multi-link manipulator consisting of a series of small links having a length of 60 mm has been conceived with the end-effector having two degrees of freedom, namely pan and tilt. A CCD-based camera device has been selected as the end-effector for carrying out the inspection. The pan and tilt motions facilitate the camera to achieve maximum coverage of the space below the containment box during the inspection. The configuration of the manipulator is such that it is rigid in one direction and flexible in complementary direction in the same plane making it steady during the actuation of the pan and tilt motions. The bottom-most part of the links carried the array of white LEDs for lighting the inspection environment.

A manually actuated flexible manipulator has been developed for the visual inspection of equipment and tanks below containment box. The device has been fabricated and tested for its basic functions in a mock-up cell. Subsequent to extensive trials, the device was used in the CORAL plant to assess the condition of the space below the containment box. Figure 1 shows the device being tested in the mock-up cell and Figure 2 shows few snapshots of the space below the containment box. Further inspection campaigns from different ports are being planned.

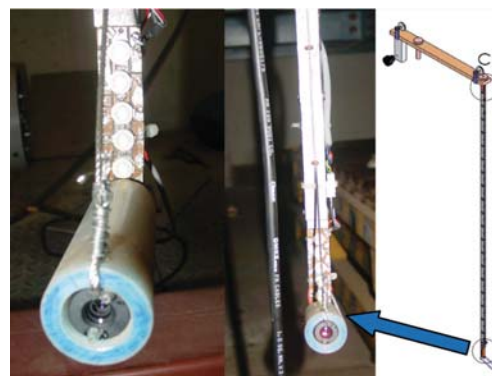


Fig.1: Photographs and the CAD Model of the Remote device for the ISI of the space below containment box in CORAL



Fig. 2 : Snapshots of few equipment and vessels below the containment box obtained during the inspection campaign from the video streamline

ADDITIONAL INFORMATION

In addition to the above described device, an electrically actuated-servo controlled snake-like robot is being developed to undertake inspection in such inaccessible locations. Snake-like robots are serial manipulators with links connected in series providing flexibility and redundancy to avoid obstacles, pass through tortuous path and manipulate the end-effectors dexterously.

GENERAL EXPLANATION RELATED TO DESCRIPTION

Main processes in reprocessing of spent fuels takes place in a radioactive containment box made of stainless steel and the consequent process solutions are stored in tanks located below the containment box, shielded all around by lead bricks or RCC walls. The space below the containment box where the process tanks are located is highly inaccessible and hostile. The process tanks contain highly radioactive and corrosive dissolver solution and other process fluids. Failure of the tank will cause leakage of highly radioactive liquid into the space below the containment box, which is not acceptable. Hence, periodic inspection of this space, and its contents, is essential to assess the integrity. The lead cell with the containment box is shown in the CAD model in the Figure 3.

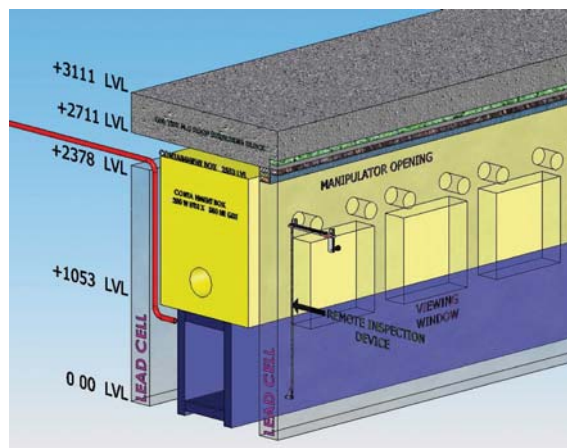


Fig. 3 : CAD Model of the CORAL cell showing the containment box and the region below the box with the Remote Inspection Device

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

As part of the program for comprehensive in-service inspection of critical components of the reprocessing plants, the space below the containment box of the CORAL is required to be inspected periodically using NDE techniques to assess its healthiness and integrity. Visual examination is the NDE technique envisaged using a camera and remote device.

ACHIEVEMENTS

A challenging task has been successfully completed for remote visual inspection of process tanks and other equipment, below the containment box as part of in-service inspection. Such comprehensive in-service inspection campaigns in reprocessing plants, especially for process tanks, are not reported elsewhere and it is a significant step forward in our in-service inspection programme. The development of various gadgets for the in-service inspection and its validation using a mock-up facility and successful implementation in the field has given immense confidence for undertaking such tasks in other areas of reprocessing plant. The feedback gained from the experience of this campaign will be put to use while designing such systems for the demonstration as well as plant-level reprocessing facilities. Such an experience is also useful in providing various plant features and equipment layouts at the design inception stage for future reprocessing plants.

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Grain Boundary Engineering in Ferritic Steels

EXECUTIVE SUMMARY

Embrittlement phenomena in ferritic steels, manifested as increase in ductile-to-brittle transition temperature (DBTT) due to exposure to high temperature or irradiation, poses a major challenge in developing them as a material for core components in fast reactors. High energy boundaries are more susceptible to segregation of impurities and precipitation of brittle phases. Grain boundary engineering is a possible method to increase the amount of low energy boundaries and thereby mitigate the grain boundary embrittlement problem. Modeling studies demonstrated the synergistic effect of small grain size and amount of crack resistant or low energy boundaries in reducing the propensity for initiation of intergranular crack. A two-step normalisation treatment was designed to refine the prior-austenite grain size in 9Cr-1Mo steel which reduced the ductile-to-brittle transition temperature.

OUTLINE

Ferritic steels, owing to their excellent void swelling resistance against irradiation and good high temperature mechanical properties, are being considered as a wrapper material in fuel subassembly of future fast reactors. However, ferritic steels exhibit Ductile-to-Brittle transition phenomena at low temperature. Temper/irradiation embrittlement can also cause increase in DBTT and/or lowering of upper shelf fracture energy, on long-term exposure to radiation and high temperature (400-600°C) environment. Assessment and control of embrittlement problem, so as to retain adequate ductility/toughness over all handling temperatures, after target fuel burn-up, is thus essential for ensuring the successful use of ferritic steels as fast reactor core component materials.

Low-angle and Coincidence Site Lattice (CSL) grain boundaries have low surface energy, and are more resistant to segregation, precipitation phenomena and less susceptible to intergranular failure, compared to 'random' grain boundaries. The 'Grain Boundary Engineering' approach aims to increase the fraction of low energy/'special' boundaries in 9Cr-1Mo ferritic steel by thermomechanical treatment.

An assemblage of grains containing both 'random' and 'special' boundaries and the intergranular crack connectivity along susceptible (random) boundaries was modeled in a simulated hexagonal grain structure, for various amounts of crack resistant (special) grain boundaries, distributed randomly using Monte Carlo method. Brittle failure of materials is primarily dictated by crack initiation stages and there exists a specific critical crack length, beyond which material is bound to fracture. The statistical probability of interconnected random boundaries exceeding this 'critical crack length' is an index indicating the susceptibility to intergranular fracture. This was evaluated (based on 1000 Monte Carlo cycles in a 100 x 100 hexagonal matrix) for two types of grain sizes, and the result is given in Figure 1; inset illustrates the hexagonal grain lattice, a specific instance of distribution of crack susceptible and crack resistant boundaries and crack network along susceptible boundaries from top. Reduction in grain size leads to lower failure probability for a given fraction of susceptible boundaries. For example, if the crack resistant boundaries are 50%, and if the critical crack length for failure is 100 μm, then the probability of intergranular failure is seen to reduce from 0.97 in a coarse (25 μm) grained lattice to 0.20 in a fine (12 μm) grained lattice.

A two-step normalisation treatment (1050°C/2 hours/Water quenched + 960°C/2 hours/Air cool) was designed to refine the prior-austenite grain size from 26 μm to 12 μm. Subsequent tempering gave a uniform tempered martensitic microstructure similar to conventional N&T treatment, but with improved Ductile-to-brittle transition characteristics; DBTT reduced by 15°C and upper-shelf-energy improved by 50 Joules (Figure 2). Several types of thermomechanical treatments were carried out and grain boundary character distribution was evaluated using SEM-EBSD technique. The amount of Coincidence Site Lattice increased from 35.4% (N&T) to a maximum of 42.9% for '20% cold rolled + Annealing' treatment (Table 1).

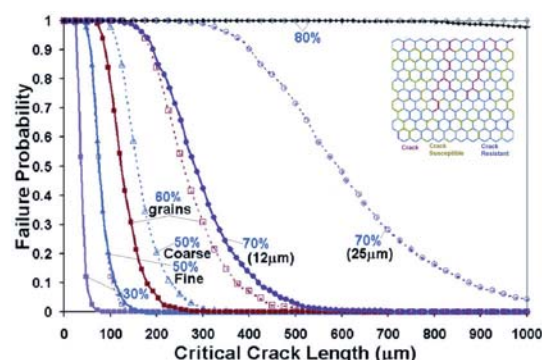


Fig. 1 : Calculation of probability for initiation of intergranular crack in a hexagonal grain lattice

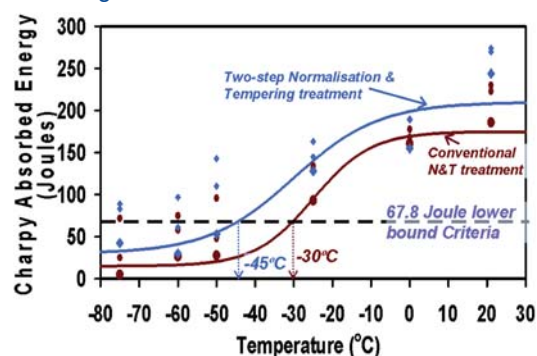


Fig. 2: Ductile-to-brittle transformation of 9Cr-1Mo steel, subjected to different heat treatments to alter grain size

Table 1: Grain boundary characteristics after thermo mechanical treatment

TM Treatment		Low Angle %	CSL %
Hea treatment	Deformation%		
N & T	5	8.0	19.1
	10	5.9	25.2
	15	4.9	38.9
	20	5.6	27.5
Annealing	5	7.2	25.7
	10	3.8	38.9
	15	5.0	39.7
	20	5.5	42.9

ADDITIONAL INFORMATION ON GRAIN BOUNDARIES IN POLYCRYSTALLINE MATERIALS

Grain boundaries play an important role in determining the properties of metallic materials. They hinder the movement of dislocations leading to dislocation pile-ups, and thereby strengthening the material. The yield strength is indirectly proportional to the square-root of average grain size (Hall-Petch relation), and smaller grain size gives higher strength, ductility and toughness in metals, and also reduces ductile-to-brittle transition temperature in steels. Periodic arrangement of atoms is disturbed at the grain boundary (especially at random boundaries) and thus exhibit free surface energy, which promotes impurity segregation and secondary phase precipitation.

GENERAL INFORMATION ABOUT EVALUATION OF GRAIN BOUNDARY NATURE USING EBSD

Grain boundary is an interface separating two grains having the same crystal structure (for single-phase material) but different crystallographic orientation. In SEM-EBSD (Scanning Electron Microscope-Electron Back Scattered Diffraction) technique, a fine high energy (~30 keV) electron beam is incident on the specimen. Back scattered electrons are emanated, which in-turn undergo diffraction by the crystalline phase at the surface (~100 nanometer) to produce pseudo-Kikuchi diffraction patterns, collected using the Phosphor screen/CCD detector. By analyzing the diffraction pattern, the type of crystal lattice and the orientation of the unit cell with respect to the specimen axes can be precisely calculated (within 1°) by an automated procedure. An areal EBSD scan is carried out on the region of interest, and a typical crystal orientation map result is shown in Figure 3; colors represent the type of crystallographic plane (legend key) that is aligned with the specimen surface. Grain orientation changes discretely across a grain boundary, and a low angle boundary exhibits a small change in orientation and a small disturbance in periodic arrangement of atoms. In a Coincidence Site Lattice (CSL) boundary, certain fraction of lattice points is common to both the crystals, and they occur for a specific misorientation between the adjoining grains. By calculating the misorientation (represented using rotation axis R -misorientation angle ω) between neighboring pixel data, the grain boundary ($\omega > 10^\circ$) as well as its nature (low-angle/ Coincident Site Lattice/Random) can be evaluated.

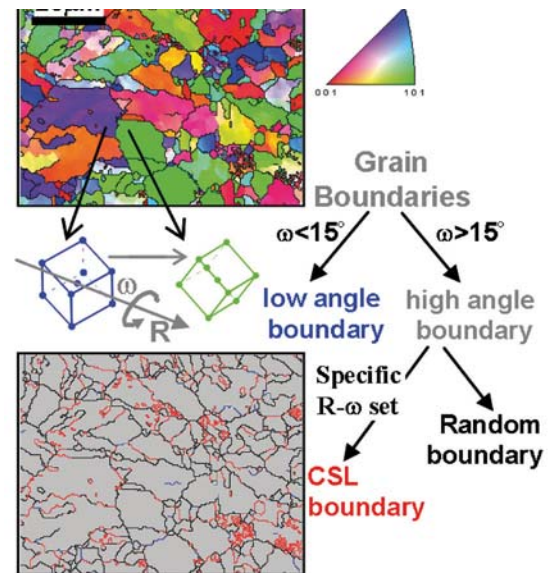


Fig.3: Evaluation of crystal orientation and grain boundary type using SEM-EBSD technique

BRIEF DESCRIPTION ON MODELING OF INTERGRANULAR CRACK CONNECTIVITY

The propagation of a crack requires connectivity of the 'random'/crack-susceptible boundaries or in other words, a network, which has been modeled assuming a regular hexagonal grain lattice. There exists a certain critical amount of 'special' boundaries known as 'percolation threshold', below which the network is thoroughly percolating leading to imminent failure (23% for this case). For 'special' boundaries greater than the threshold, there exists a certain finite probability for crack initiation depending on the actual distribution which has been statistically evaluated based on large number of Monte Carlo runs.

ACHIEVEMENT

Modelling studies demonstrated quantitatively the synergistic effect of amount of 'crack resistant' grain boundaries and finer grain size in reducing the propensity for initiation of intergranular crack. Two-step normalisation treatment was developed in 9Cr-1Mo steel to reduce the prior austenite grain size by one-half, which reduced the ductile-to-brittle transition temperature by 15°C. Using thermomechanical treatments, improvement in the amount of CSL boundaries up to 43%, as compared to 35% in N&T condition was achieved.

PUBLICATIONS

1. T. Karthikeyan, "Grain Boundary Engineering – A concept to overcome embrittlement problem in ferritic steels", Annals of the Indian National Academy of Engineering, 6 (2009)189.
2. Baldev Raj, S. Saroja, K. Laha, T. Karthikeyan, M. Vijayalakshmi and K.Bhanu Sankara Rao, "Methods to overcome embrittlement problem in 9Cr-1Mo ferritic steel", Journal of Materials Science, 44 (2009) 2239.
3. T. Karthikeyan, V. Thomas Paul, S.K. Mishra, S. Saroja, M. Vijayalakshmi and I. Samajdar, "Effect of Thermomechanical Treatment on Grain Boundary Distribution in 9Cr-1Mo Ferritic Steel", Metallurgical and Materials Transactions. 40 (2009) 2030.

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Innovative Computational Fluid Dynamic Studies for Argon Macro bubble formation in Sodium near Pump Shaft

EXECUTIVE SUMMARY

Primary sodium pump (PSP) shaft is partially immersed in sodium and partially exposed to argon gas. Due to large rotational velocity of the shaft (590 rotation per minute), formation of a vortex around the shaft is possible with the associated risk of gas entrainment. This has been investigated in detail by Computational Fluid Dynamic (CFD) studies employing a novel Volume of Fluid method. Detailed parametric studies have been carried out to understand the effects of hydrostatic bearing (HSB) flow, pump vessel ribs and ECFM tubes. It is found that maximum depth of vortex around the shaft is 0.4 meter which is much shorter than the submergence depth of hydrostatic bearing, viz. 2.88 meter. Further assessment of gas entrainment risk indicates that there is no possibility of macro-sized argon bubbles (> 2 millimeter diameter) entraining into sodium. This study has been executed as a collaborative research with Tridiagonal Solutions Pvt. Ltd, Pune.

OUTLINE

Radioactive primary sodium is circulated through the core by two primary sodium pumps operating in parallel. The nominal speed of the pump is 590 rotations per minute and the pump shaft is partially immersed in sodium and partially exposed to argon cover gas (Figure 1). The bottom end of the shaft is supported by a hydrostatic bearing. The shaft is housed inside a pump vessel, in the cold pool. The outer diameter of the shaft is 630 millimeter, which leads to a linear velocity of about 20 m/s. Due to the rotation of the shaft in a confined zone having sodium-argon interface, formation of a vortex around the shaft is possible. About 50% of the sodium flowing through the hydrostatic bearing enters the pump vessel. The interaction of vortex and the hydrostatic bearing flow could be a source of argon gas entrainment in cold pool.

Traditionally, gas entrainment phenomenon is studied only by an experimental approach. But in the present study, a computational fluid dynamic method is employed. For tracking the argon-sodium interface, volume of fluid approach (VOF) is used where liquid surface properties and wetting behavior is accounted in the model. Vortex interaction with hydrostatic bearing flow for various power levels of the reactor has been studied.

The predicted average free surface swirl velocity at full load is 3.07 m/s and at part load it is 1.56 m/s. It is observed that the vortex depth is unaffected by the amount of liquid sodium in the pump vessel and depends on the shaft speed. To reduce the computational time, a single phase approach has been developed for further studies related to liquid surface deformation. The free surface deformation and stream line distribution predicted by 2-D simulations are depicted in Figure 2.

It is found that the bearing flow is dominant up to outlet holes located above upper ribs, i.e., up to 0.7 meter above the hydrostatic bearing. Also the average swirl velocity on the interface without bearing flow for full load is 3.09 m/s and that with bearing flow it is 3.05 m/s and for part load without bearing flow it is 2.39 m/s, whereas with bearing flow it is 2.30 m/s.

3-D single phase model is used for studying influence of internals (ribs, gusset plates and ECFM tubes) and results show that vortex depth decreases in presence of internals. The vortex depth is (Figure 3):

- Without internals: 1.06 meter
- With ribs, and gusset plates: 0.668 meter
- With tubes: 0.51 meter.

Simulation results show that vortex depth is mainly governed by average tangential velocities in the bulk and internals reduce the average bulk tangential velocities considerably and hence the vortex depth. But, tangential velocities near the vicinity of the shaft are unaffected by internals. Comparing the axial velocity at the vortex tip against the bubble rise velocity, for range of bubble sizes, it is established that no macro-sized bubbles (> 2 millimeter) can entrain in the sodium.

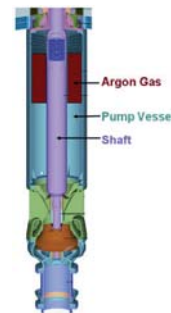


Fig. 1: Pump shaft partially immersed sodium

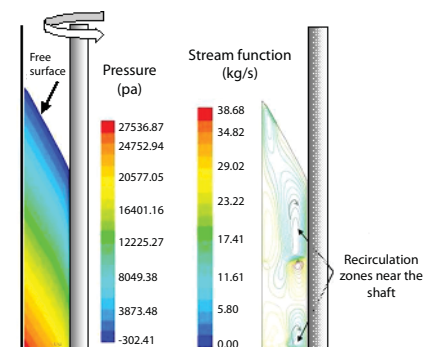


Fig. 2: Free surface profile and stream function contours demonstrating bearing flow does not percolate upto free surface

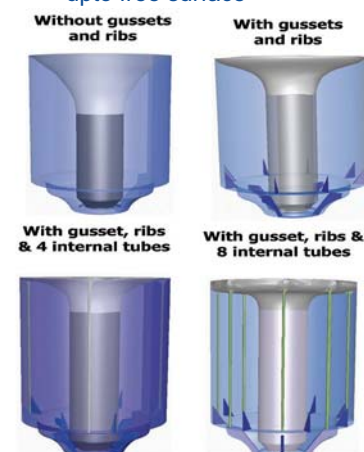


Fig. 3: Free surface profiles depicting the effects of ribs and ECFM / TC passage tubes (3-D study)

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

In this study detailed computational fluid dynamic model for rotating shaft immersed in sodium pool along with associated hardware components have been developed. For interface interaction with the shaft surface, volume of fluid approach is used where liquid surface properties and wetting behavior are accounted. For rotating shaft, multiple reference frames (MRF) approach is used for accounting shaft surface rotations. High Reynolds number version of standard k- ϵ model with wall function treatment has been used to account for turbulence effects. Based on the vertical velocity at the vortex tip and argon gas rise velocity, the risk of gas entrainment is assessed.

ACHIEVEMENT

This work is first of its kind in any nuclear industry. Prediction of sodium-argon interface by computational fluid dynamic simulations employing volume of fluid method and assessment of associated gas entrainment risk based on the predicted vortex tip velocity and gas rise velocity are innovative applications of computational fluid dynamic. This study has established that there is no risk of macro-sized argon bubbles entraining into sodium due to the rotation of pump shafts.

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Wavelet Transform Based Processing of Eddy Current Signals from Bend Regions of Steam Generator Tubes

EXECUTIVE SUMMARY

Remote field eddy current (RFEC) signals from expansion bend regions of ferromagnetic steam generator (SG) tubes of Prototype Fast Breeder Reactor (PFBR) are expected to be influenced by noise from disturbing variables such as exciter-receiver coil misalignment, bending stresses, probe wobble and magnetic permeability variations. Wavelet transform based signal processing has been developed for noise reduction and enhancement signals. This has been successfully applied to process RFEC signals from bend regions of SG tubes.

OUTLINE

Remote field eddy current (RFEC) technique is proposed for in-service inspection of modified 9Cr-1Mo SG tubes of PFBR as part of structural integrity assessment. During periodic in-service inspection of the SG tubes, detection of defects in straight as well as bend regions of the tubes is essential. Detection of defects in bend regions is influenced by disturbing noise such as changes in electromagnetic coupling due to exciter-receiver coil misalignment, presence of bending stresses, magnetic permeability variations and probe wobble. Hence, reliable detection of defects in the bend regions of SG tubes using RFEC technique is challenging. Studies have been performed on SG tubes having grooves of 10% and 20% wall thickness (WT) and fuel tubes having artificial notches and flat bottom holes of different size.

RFEC signals from four SG tubes with wall thickness grooves in the expansion bends have been used for application of the wavelet transform technique. Figure 1 shows the location of the grooves namely straight-to-bend transition region and bend-to-bend transition region. In one tube (T1), no artificial defect has been introduced. Continuous wavelet transform (CWT) method has been applied for reduction of noise from disturbing variables for detection of defects in bend regions.

Systematic studies based on the signal to noise ratio (SNR) revealed that the Bior2.8 wavelet is optimum for elimination of disturbing noise and enhancement of signals due to defects in the bend regions of SG tubes (Table I). Figure 2 shows the as-received (raw) RFEC signals of tube without defect (T1) and tubes having 10% WT (T2), 20% WT (T3) grooves and corresponding wavelet transform processed signals are shown in Figure 3. When the noise amplitude of tube T1 is used as threshold, shown as dotted line in Figure 3, both 10% and 20% WT grooves have been detected unambiguously.

The disturbing noise in RFEC response due to misalignment of coils, bending stresses and probe wobble is expected to be of low-frequency and extended in nature, thus essentially contribute to shift in the baseline. The RFEC response due to permeability variations is expected to be of higher frequency and distributed. On the contrary, the RFEC response for wall loss defects would be highly localized and expected to be of intermediate frequencies, depending on the type and size of the defects. Thus, processing of RFEC signals based on band-pass filtering seems attractive for noise reduction. Also, RFEC technique produces double-peak or two-Gaussian signals for defects, one when the exciter coil moves past the defect and the other when the receiver coil moves past the defect. This aspect can be conveniently used to design a template for cross-correlation and also to choose a mother wavelet for signal enhancement enabling reliable detection of defects in the presence of disturbing noise.

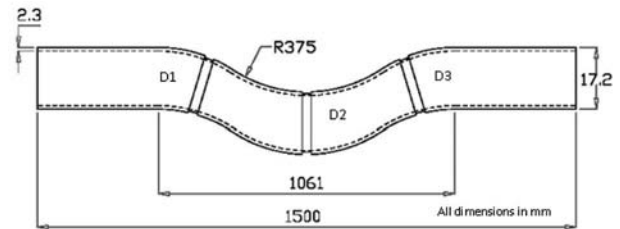


Fig. 1: Schematic of SG tube with grooves D1, D2 and D3 at three characteristic locations in bend region

Table I: Performance of different wavelets for detection of 20% WT grooves in SG tube

S. No.	Wavelet Type	Maximum SNR, dB	Optimal Scale
1	Gaus2	6.50	7
2	Mexh	6.48	5
3	Bior2.8	7.23	19
4	DB5	6.70	15

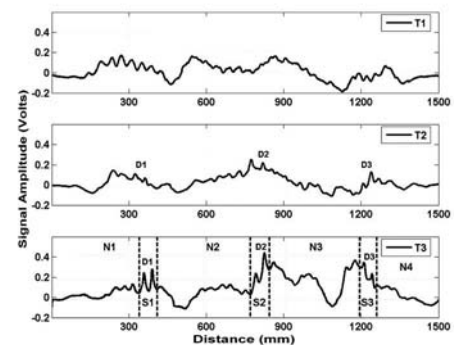


Fig. 2: As-received RFEC signals from bend region of tube T1 (defect-free) and T2 (10% WT grooves) and T3 (20% WT grooves)

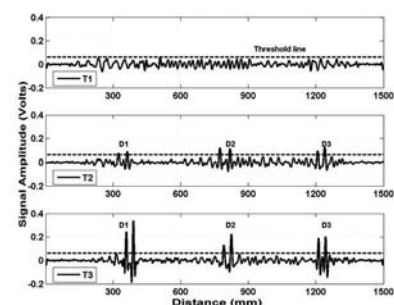


Fig. 3: Wavelet transform processed RFEC signals of tubes T1 (defect-free), T2 (10% WT grooves) and T3 (20% WT grooves) using Bior2.8 wavelet

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Wavelet transform is a signal processing technique that allows a signal of interest to be examined in both time and frequency domains simultaneously. The continuous wavelet transform of a time domain signal $x(t)$ is defined as

$$X_w(s,b) = 1/\sqrt{s} \int_{-\infty}^{\infty} x(t)\psi^*(t-b/s) dt$$

where $\psi(t)$ is the mother wavelet which is a continuous function in both time and frequency domains, b is the translation factor that depends on time, and s is the scale factor which is a function of frequency. Daughter wavelets are derived from the mother wavelet by varying the scale factor. Thus, the frequency bandwidths of daughter wavelets are different from each other. The wavelet coefficients $X_w(s,b)$ are obtained by cross-correlation of $x(t)$ with different daughter wavelets. The time frequency localization property of the wavelets enables one to use them as band-pass filters at the bandwidth of the wavelet.

ACHIEVEMENT

A 10-fold reduction in the disturbing noise from the bend regions and a 7 dB improvement in the SNR have been observed after wavelet transforms processing of remote field eddy current signals and has resulted in reliable detection of 10% WT grooves anywhere in the bend regions of steam generator tubes. Besides, the wavelet transform technique has preserved the double-peak behaviour of the remote field eddy current signals.

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New Method of Temperature Compensation for Mutual Inductance type Continuous Level Probes in Sodium Systems of Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

Mutual inductance (MI) type continuous level probes are used in primary and secondary sodium systems of PFBR for monitoring sodium level in sodium tanks and vessels. The probe output varies with level and temperature of sodium for given excitation frequency. A new method to compensate the variation of probe output due to temperature was implemented and effect of temperature on output of level probe is reduced to $\pm 1.5\%$.

OUTLINE

While using a Mutual Inductance type continuous level probe, (MILP) the effect of frequency is minimized by choosing optimum operating frequency. The optimum operating frequency is the frequency for which the effect of temperature on secondary output is minimum. The effect of temperature on secondary output is eliminated by connecting suitable external shunt resistor across the terminals of the secondary winding. By this new method the error in accuracy of measurement has been brought down to 1.5%. Figure 1 shows the secondary output of a MI level probe (with optimum frequency of excitation) at different temperatures without application of new temperature compensation method.

Presently temperature compensation is achieved by measuring the primary voltage variation, which is the measure of average temperature of the probe. Based on this primary voltage, temperature compensation is applied to open circuited secondary voltage in the temperature range of 200°C-550°C. This compensation is given through electronics. This existing electronic compensation is partial because variation in secondary O/P from zero level to full level is different for different temperatures which lead to an error of $\pm 3\%$ in measurement.

A new method for temperature compensation by adding a resistor across the secondary coil was tried out on 9600 mm MI type continuous level probe through sodium testing experimentally. Test Vessel-1 of LCTR was used for sodium testing of 9600 mm MI level probe. A standard pipe of size 1½" SCH10 was used as pocket for the continuous probe for easy insertion and withdrawal. A 45 mm thick new top flange for TV-1 was fabricated for calibration of the longest MILP. To avoid vibration of long pockets during sodium flow, a supporting ring with 4 tie rods surrounding the pocket was welded to the TV-1 top flange. This 9600 mm probe is the longest level probe calibrated in Fast Reactor Technology Group.

The value of external resistance was calculated using simulation and it was fine tuned using open circuit experimental data. This value of resistance was connected across probe secondary. The experiment was repeated and secondary output of the probe was recorded at different temperatures from 200°C-550°C. Figure 2 shows the secondary output after adding a resistor for temperature compensation. It can be seen that the effect of temperature in secondary output is eliminated.

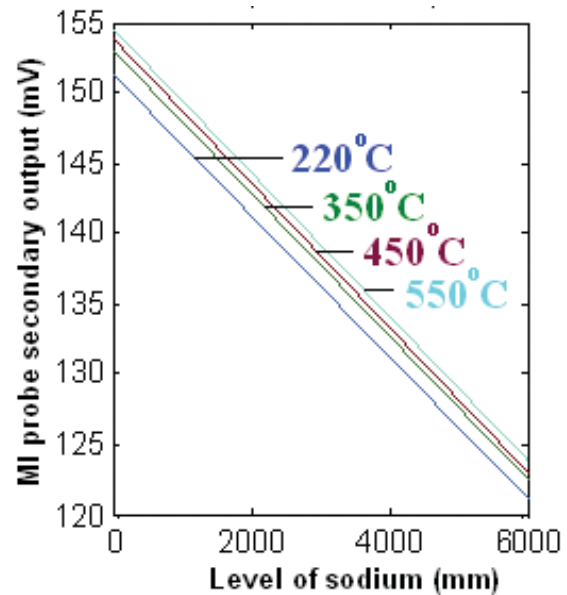


Fig. 1: Secondary output without temperature compensation

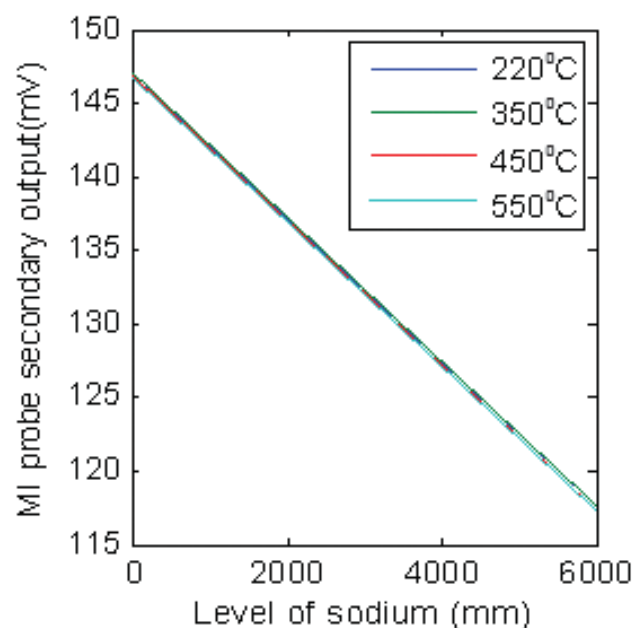


Fig. 2: Secondary output with temperature compensation

ADDITIONAL INFORMATION

Effect of sodium temperature on the level probe secondary output: As the temperature of sodium is varied, resistance of sodium around the level probe also varies. This variation in resistance of sodium causes variation in eddy current produced in sodium, resulting in variation in secondary voltage. Hence it is seen that the secondary voltage of the probe is a function of sodium level as well as temperature.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND OF NEW METHOD OF TEMPERATURE COMPENSATION

In this method of compensation a suitable external resistance, R_{ext} , is connected across the secondary. The increase in the induced secondary voltage due to temperature rise is compensated by the voltage drop in internal resistance of the coil since the secondary coil is now closed by external resistance. Now, the voltage across the secondary terminal is only due to sodium level variation. Figure 3 shows the arrangement required for compensation where E is the induced voltage, R_i is the internal resistance of the secondary winding, R_{ext} is the external resistance required for temperature compensation, i is the loop current and E' is the terminal voltage which is a function of sodium level and is independent of temperature.

The external resistance required for temperature compensation of 9600 millimeter probe was determined to be 1530 Ω .

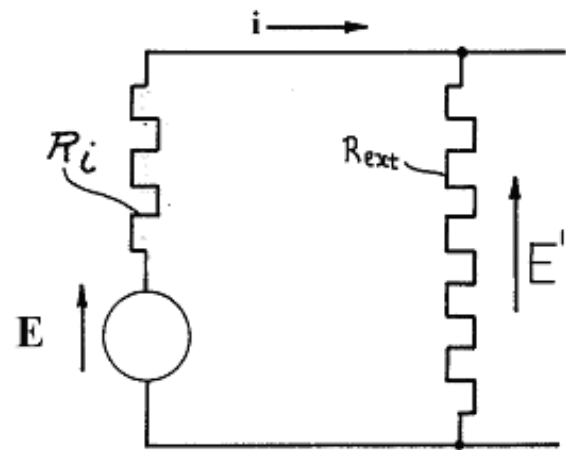


Fig. 3: Arrangement for Temperature Compensation

ACHIEVEMENT

The new method of temperature compensation was validated experimentally for 9600 millimeter long mutual inductance type continuous level probe. The method of calculating external resistance with minimum sodium data has been established. Using this new method, the temperature effect on secondary voltage was found to reduce without change in sensitivity of the probe. This new method of adding an external resistor for temperature compensation simplified the electronics by eliminating the temperature correction circuitry and reducing the error due to temperature variation to $\pm 1.5\%$.

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Assessment of Dynamic Instability in Once Through Steam Generator

EXECUTIVE SUMMARY

Hydrodynamic flow instability in Steam Generators (SGs) is one of the important problems in the design and operation of Liquid Metal Fast Breeder Reactors (LMFBRs). Water flow in once through steam generators is susceptible to instability due to the close coupling between the thermal and hydraulic processes. Sustained flow oscillations due to instability are undesirable since they result in flow mal-distribution among the tubes, thermal stress, mechanical vibrations and system control problems. Instability experiment in Steam Generator Test Facility (SGTF) was carried out to establish the stable and unstable operating regimes of the steam generator and to study the effect of various operating parameters on the stability of steam generator and also for validating the analytical code developed.

OUTLINE

Steam Generator Test Facility is setup to test the model of Once Through Steam Generators (OTSG) of PFBR, which is similar to that of PFBR SG in all aspects such as operating condition, material of construction and structural layout etc. It is a counter current shell and tube type heat exchanger in which sodium flowing in the shell side exchanges heat with the water/steam flowing in the tubes to produce high pressure superheated steam. Instability experiment was carried out in SGTF SG at 20% power level. By decreasing the SG outlet pressure, instability regime was approached. Fluctuations in the steam outlet temperature and in the module inlet flow were monitored continuously to detect the onset of instability phenomenon.

When instability region is approached, fluctuation levels in individual tube feed water flow and the outlet steam temperature increase with small sinusoidal oscillations superimposed. During the stable operation of SG at 1.1 MWt power (20% of nominal power), the fluctuation in the steam temperature measured from the thermocouples installed in various SG tube outlets is around 2-30°C (Figure 1). During operation in the unstable region, the mean outlet temperature in all the tubes was found to be decreased and large temperature fluctuations of about 18-240°C were observed in all the tubes (Figure 2). Fluctuations were observed in the output of the DPTs connected across the orifice in SG tubes during the instability phenomenon. It is also observed that during the instability phenomenon, the SG outlet pressure showed oscillation similar to that observed in the temperature and DPT outputs. A similar trend is also observed in the feed water inlet flow to SG during instability.

Water flow oscillation in tubes manifests as oscillations in the steam temperature at the tube outlet/pressure fluctuations. However, it is difficult to instrument individual tubes in SG for such measurement in an operating plant. Figure 3 shows the tube layout of SGTF-SG with instrumented tubes. If the flow oscillations in the tube manifest itself in the overall module flow, then fluctuation in the overall flow/flow noise could be utilized for on-line stability measurements. The result of the experiment has indicated the feasibility of an instrumentation scheme for on-line measurement of dynamic instability in once through Steam Generator in an operating plant.

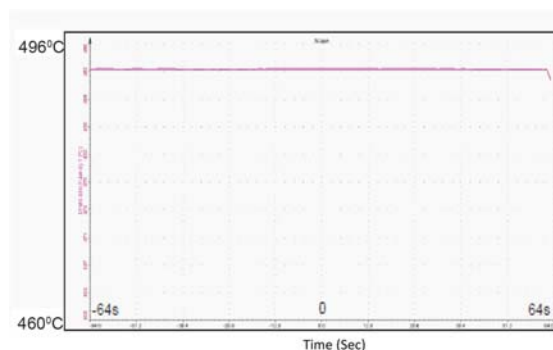


Fig. 1: Temperature Fluctuation during Stable Condition

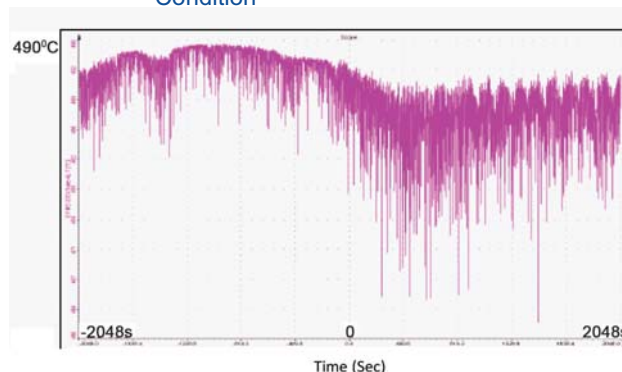


Fig. 2: Temperature Fluctuation during Unstable Condition

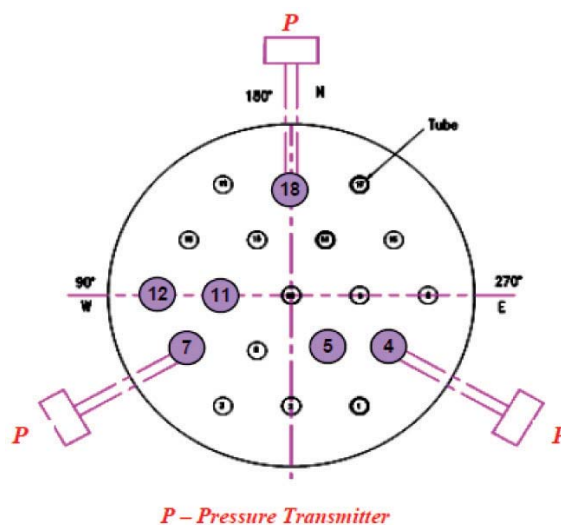


Fig. 3: Instrumented tubes in SGTF-SG

ADDITIONAL INFORMATION ABOUT FLOW INSTABILITIES

Flow instabilities have been classified as static instabilities and dynamic instabilities. A flow is said to be subjected to a static instability if, when the flow condition changes by a small step from the original steady-state one, another steady state is not possible in the vicinity of the original state. A static instability can lead to operation with a different steady state flow. Static instability results in different steady state water flows in different tubes and steam generator modules. Measurement of steam temperatures at the outlet of tubes and steam generator modules will indicate whether static instability has set in. Many types of dynamic instabilities can occur in a steam generator tube, but the cause of the main type of instability important for the design of steam generators is the propagation of density waves. Density wave oscillation causes very large fluctuation in the steam outlet temperature. For example, when water flow is reduced, or sodium flow is increased, vapour generation rate and pressure drop change. Time delays between these processes can, under certain operating conditions cause sustained oscillations, leading to the onset of dynamic instabilities. Dynamic instability has been experimentally studied by many investigators and a coarse correlation has been found indicating that the period of density wave oscillation is approx. 1-2 times the transit time of fluid in a tube. However, it is also reported that the period of density wave oscillation could be even $\frac{1}{2}$ or even a small fraction of the transit time.

GENERAL EXPLANATION RELATED TO DESCRIPTION

SG is one of the most critical components of a fast reactor plant that determines plant availability. It is therefore, necessary to predict the conditions, under which instability occurs so that the system may be designed to operate always under stable conditions. Dynamic instability (density wave oscillation DWO) occurs because of the phase mismatch between the primary perturbation (water flow) and the response to this perturbation (pressure drop). As many tubes are operating under essentially constant pressure heads, this mismatch can lead to sustained oscillations. Dynamic instability manifests itself in the form of self sustaining feed water flow oscillations in the individual tubes. These oscillations in the feed water flow results in oscillations in tube wall temperature, which could cause the rapid failure of tubes due to thermal stress cycling depending on the frequency and amplitude of oscillations. Experiments were carried out in SGTF to study the dynamic instability phenomenon and to establish the stable and unstable operating regimes of SG.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

In a steam generator of once through design, connecting parallel steam generating tubes linking inlet and outlet headers, there is a possibility of dynamic flow instability under certain thermal hydraulic conditions. Many types of dynamic instabilities can occur in a steam generator tube, which can be ascribed to a number of different causes, viz. propagation of pressure or density waves, variation of flow patterns and thermodynamic non-equilibrium between the phases in the superheated steam region. However, the cause of the main type of instability important for the design of steam generators is the propagation of density waves. This type of low frequency instability is referred to in literature as parallel-channel, density wave, time delay or mass flow-void feedback instability. Dynamic instability (density wave oscillation) occurs because of the phase mismatch between the primary perturbation (water flow) and the response to this perturbation (pressure drop). As many tubes are operating under essentially constant pressure heads, this mismatch can lead to sustained oscillations.

ACHIEVEMENTS

Dynamic instability measurement was carried out in SGTF steam generator at 20% power level. Very large fluctuation in steam temperature has been observed during instability. The experiment has indicated the feasibility of an instrumentation scheme for on-line measurement of dynamic instability in an operating plant. Based on these experimental inputs, development of an on-line instability measurement system for SGTF is in progress.

PUBLICATIONS

V. Prakash, M. Thirumalai, P. Murugesan, V. Vinod, V. A. Sureshkumar, I. B. Noushad, K. K. Rajan, P. Kalyanasundaram and G. Vaidyanathan; Orlando, Florida, USA; "Assessment of Dynamic Instability In Once Through Steam Generator" Paper No: 48253, ICONE 16, 3 (2008) 315.

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Establishing Thermal Striping Limits for Sodium Fast Reactors

EXECUTIVE SUMMARY

Less pessimistic thermal striping limits have been established for future sodium cooled fast reactors (SFR) based on fracture mechanics-based method employing 'σ-d approach'. Random temperature fluctuations and frequency-dependent thermal attenuation on the structural wall were considered. The limits are derived at various potential locations in FBRs which are also subjected to creep-fatigue damage due to major cycles caused by startup, shutdown, power failures pump trips, etc. Pool hydraulic computations carried out to identify and quantify the thermal striping zones confirmed that the proposed limits can be respected with good margins for FBRs. This is an important work to confirm the sixty years design life for future SFRs planned by the Department

OUTLINE

While sodium has several distinct advantages from the point of view of safety, there are certain critical structural mechanics issues, especially with the austenitic stainless steels, commonly used structural materials in view of its excellent compatibility with sodium and high strength at elevated temperatures. The high thermal fatigue damage caused by a special phenomenon called 'thermal striping' is one among them. Thermal striping is a complex thermal hydraulics phenomenon, which generates random fast temperature fluctuations, originating from the incomplete mixing of hot and cold jets of fluid, sodium in the present context, in the vicinity of adjoining structural wall surface (Figure 1). The range of frequency of oscillations under thermal striping is reported as 1-10 Hz. Thermal striping occurs at a few locations in the hot and cold sodium pools in the reactor assembly, predominantly on the core cover plate of control plug and at mixing 'Tee' junctions in the secondary sodium pipelines. There are a few reported failures, in the form of extensive cracking due to thermal striping in the operating reactors at: secondary sodium pump vessel in Phenix, Tee junction of an auxiliary pipe of the secondary circuit in SPX, control rod guide tube in PFR and primary cold trap in BN 600. In view of these reported failures, the international structural mechanics specialists have been critically investigating thermal striping phenomenon both experimentally and theoretically. Investigations focus both on thermal hydraulics and structural mechanics aspects.

Internationally, pessimistic values are reported as low as 40 K, in view of lack of robust thermal hydraulics and structural mechanics investigations. This has been resolved in the present studies. A general methodology has been developed based on fracture mechanics concepts for establishing allowable ΔT and recommendations of thermal striping limits at a few potential locations in the PFBR reactor assembly, as predicted by detailed computational fluid dynamic computations (Figure 2). The methodology can be applied to other potential locations in the sodium circuits (Figure 2). The novel features of the investigation is the fundamental derivation of an equivalent damage (D_{eff}) to represent the creep, fatigue and creep-fatigue damage interactions and derivation of equivalent crack depth corresponding to D_{eff} and idealization of temperature fluctuations through power spectral density, computation of temperature distributions in the plate taking into account beneficial effects of attenuation of high frequency components and associated stress and stress intensity distributions. The methodology has been applied to derive thermal striping limits for PFBR, as a function of accumulated creep-fatigue damage and assessment of these limits with the operating experiences. The effect of frequency-dependent thermal attenuation on the structural wall surface is found to be significant (Figure 3).

Thermal limits are derived at various potential locations in FBRs which are also subjected to creep-fatigue damage due to major cycles caused by startup, shutdown, power failures, pump trips, etc. The maximum range of temperature fluctuations can be as high as 70 K where the accumulated creep-fatigue damage is insignificant (0.1) and 45 K where the creep-fatigue is significant (0.9).

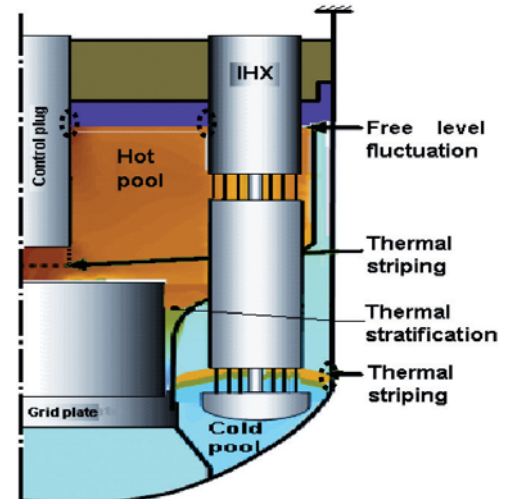


Fig . 1: Thermal striping locations in FBR

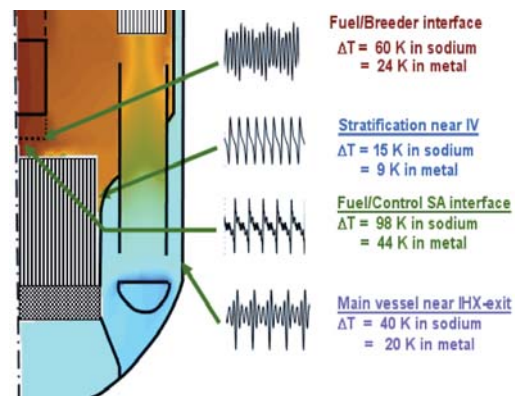


Fig. 2: CFD predictions of thermal striping Locations in PFBR

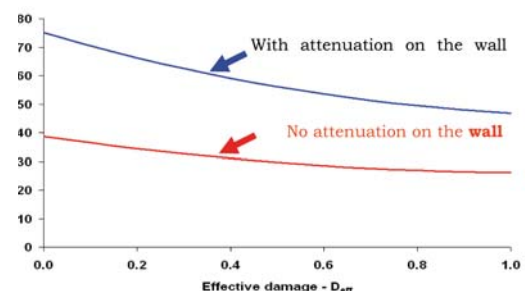


Fig. 3: Effect of temperature attenuation on striping limits

GENERAL DESCRIPTION

These limits are found to be consistent with the broad limits extrapolated from the failure experiences of international FBRs and sodium facilities. Pool hydraulic computations carried out to identify and quantify the thermal striping zones confirmed that the proposed limits can be respected with good margins for future FBRs which are designed for longer design life (sixty years) and higher load factor (85 %).

In order to derive a typical spectrum of temperature fluctuation, a setup simulating thermal striping phenomenon on the core cover plate of PFBR using water model has been commissioned and data generated was used for validating the thermal hydraulics input (Figure 4).

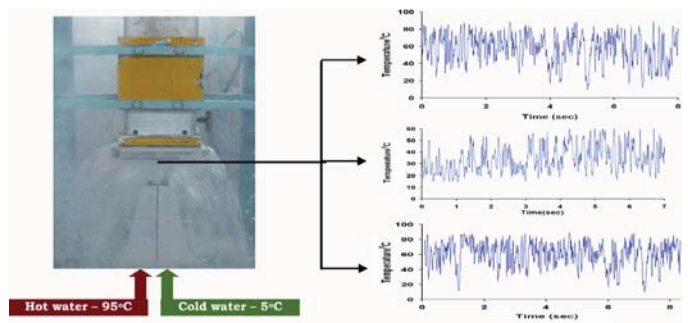


Fig. 4: Water test facility to simulate thermal striping

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The idealized problem has been formulated and governing differential equations towards solving for temperature, stress and stress intensities were derived and exact solutions were obtained by applying frequency response spectrum method. Since it is the analytical solution, the effects of various governing parameters could be studied efficiently, in terms accuracy and speed.

ACHIEVEMENTS

The outcome of this study is based on a comprehensive research in the areas of fracture mechanics, computational thermal hydraulics and bench mark experiments, taking into account of international failure experiences. The outcome of this study is very essential for design of future Indian FBR with 60 years life without changing the basic structural material. It is an international benchmark, providing guidelines for thermal striping design of future FBR of the world.

PUBLICATIONS

1. P. Chellapandi, S. C. Chetal and Baldev Raj, 'Investigation of Structural Mechanics Failure Modes in FBR', Pressure Vessels and Piping : Codes, Standards, Design and Analysis, Narosa Publishing House, New Delhi ,1 (2009) 89.
2. P. Chellapandi, S. C. Chetal and Baldev Raj, 'Thermal Striping Limits for Components of Sodium Cooled Fast Spectrum Reactors', International Journal of Nuclear Engineering and Design, 239 (2009) 2754.

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Ab initio Computational Fluid Dynamic Simulations of Flow and Temperature Distributions in 217 Pin Fuel Subassembly

EXECUTIVE SUMMARY

PFBR fuel subassembly (SA) has 217 fuel pins with thin spacer wires helically wound over each pin. Computational Fluid Dynamic (CFD) simulation of complete fuel subassembly is highly demanding in terms of computational time and memory. Rotational symmetry exhibited by velocity and pressure distributions, under forced convection regime, has been judiciously utilized to minimize the computational time and construct flow and pressure distribution for the entire fuel pin bundle. The temperature distribution has been estimated considering axial as well as pin-to-pin variations in heat generation rate. To reduce the number of mesh, a customized structured 3-D mesh generator has been developed. Computational fluid dynamic calculations have been performed for typical fuel and blanket subassembly in all the flow zones and hotspot factors have been estimated.

OUTLINE

Traditionally, sub-channel approach is followed for the thermal hydraulic analysis of the fuel pin bundle. This approach is used in the codes like SUPER-ENERGY, wherein porous body formulation is adopted and the exchange of mass, momentum and energy among the various sub-channels is defined explicitly based on limited experiments performed on fuel pin bundles. This approach provides only a global picture of the thermal hydraulics within FSA without much detail on pin-to-pin circumferential variations. Contrary to this formulation, computational fluid dynamics (CFD) based approach has more maneuverability to capture intricate details of the flow physics, which are very difficult to measure in a tightly packed pin bundle. In this approach, complex thermal hydraulic characteristics of the FSA are predicted by numerically solving the conservation equations of mass, momentum and energy.

The mesh requirement for a complete simulation of entire pin bundle is highly demanding in terms of memory and computational time. Hence, a structured CFD mesh generator for an optimum mesh has been developed. A part of the mesh for FSA is depicted in Figure 1. To reduce the computational time further, the rotational periodicity in the velocity and pressure distributions has been judiciously utilized. In this novel approach, detailed velocity and pressure distributions are estimated only for $1/6^{\text{th}}$ length of the spacer wire pitch. Then the flow and pressure fields for the entire length of the fuel pins are established from the simulation results corresponding to $1/6^{\text{th}}$ pitch. This flow field is utilized in the energy equation to determine the temperature distribution of sodium in the entire pin assembly, taking into consideration the axial variation of the heat generation rate.

The axial velocity and temperature distributions at the exit of the active zone are depicted in Figures 2 and 3. The velocity is highly non-uniform in the wall sub-channels, varying from 10.5 m/s to 8 m/s, and is maximum in the sub-channels housing the spacer wire, despite a larger hydraulic resistance. Since the spacer wire revolves around the pin, the location of the wall sub-channels with maximum velocity also revolves around the inner periphery of the hexcan. It is evident from the temperature contours that the mixing induced by the spacer wires is insufficient. The temperature difference at the top of the active zone is over 100 K between the central and wall sub-channels, with wall sub-channels being colder due to flow by-pass. This difference reduces in the top axial blanket region to 60 K. The value of non-dimensional peak clad temperature (ξ) defined as,

$$\xi = (T_{\text{Peak-Clad}} - T_{\text{Na-Bulk}}) / (T_{\text{Avg-Clad}} - T_{\text{Na-Bulk}})$$

where $T_{\text{Peak-Clad}}$ is the peak clad temperature at any axial location,

$T_{\text{Na-Bulk}}$ is the bulk sodium temperature at that level and $T_{\text{Avg-Clad}}$ the average clad temperature, is found to vary from 2 to 5. Another hallmark of this collaborative project is the development of integrated models for CFD analysis of entire FSA starting from orifices at the foot to top of the pin bundle considering pin-to-pin variations in the power generation. A customized mesh generator has been developed for generating tetrahedral mesh for complex honey-comb orifice plates. The pressure drops predicted by the CFD code has been found to compare within 20% of the experimental data.

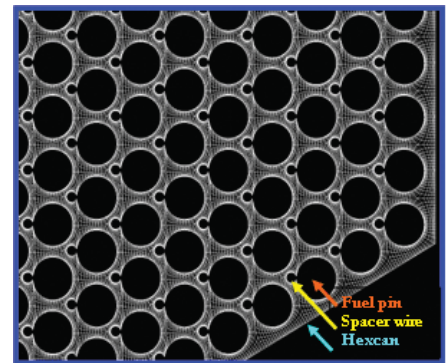


Fig. 1: Structured finite volume mesh for CFD simulation of fuel subassembly

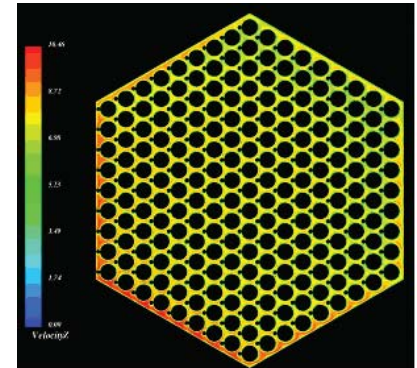


Fig. 2: Cross sectional axial velocity distribution in a cross section of the central FSA (m/s)

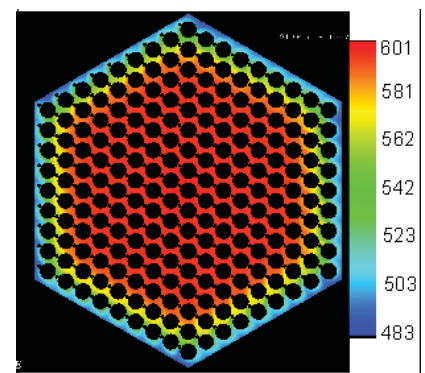


Fig. 3: Sodium temperature distribution at the top of active zone in central fuel subassembly (°C)

GENERAL DESCRIPTION

In PFBR, fuel subassembly (FSA) is a 217 pin bundle generating a huge heat flux of $\sim 1.5 \text{ MW/m}^2$. The spacing between the fuel pins is maintained by helically wound spacer wires over each pin. The spacer wires also induce cross stream flows that promote mixing of sodium among the various sub-channels of the FSA. There are significant flow and temperature variations around the fuel pins with local hot spots beneath the spacer wires. The life of the fuel pin is a strong function of the peak temperature seen by the clad, which in turn is affected by the sodium flow distribution. In conventional thermal hydraulic analysis of the FSA, the peak value of clad temperature is estimated by the use of hotspot factors. This approach leads to a conservative estimate of the temperature. Unfortunately, it is impossible to measure this temperature in any operating reactor. Realistic estimate of this temperature requires detailed knowledge of flow and temperature distributions within the fuel pin bundle. The fuel pin assembly forms a tight triangular lattice with helically wound spacer wires, forming over 514 sub-channels with free mixing of sodium from one sub-channel to the other. The hydraulic diameter of the sub-channel is very small ($\sim 3.5 \text{ mm}$) and it offers little scope for small scale experiments. Experimental determination of local flow and heat transfer characteristics is very difficult due to the miniature nature of the sub-channels demanding non-intrusive-type of instruments. Since the Prandtl number of sodium is very small (0.005), water cannot simulate the heat transfer behavior of sodium. Hence, experiments need to be conducted only in sodium. Due to the violent exothermic reaction of sodium with air/moisture, compounded with opaque nature of sodium that offers no possibility of flow visualization, sodium experiments are very difficult to perform and a modeling approach is adopted

ADDITIONAL INFORMATION

The computational fluid dynamic simulation is based on finite volume method, employing structured mesh for the fuel pin bundle region and unstructured mesh for the orifice region. The 3-D studies have been carried out using the computer code CXDExpert, developed by Zues Numerix, IIT-Bombay. Turbulence has been modeled using the standard k- ϵ model. Multi-grid method has been employed to promote convergence of the solution scheme. The computational fluid dynamic mesh consists of over 72 lakh volumes for each pitch distance.

ACHIEVEMENTS

This work is an international benchmark wherein computational fluid dynamic simulation of the entire fuel subassembly having 217 pins along with their helical wire wrap, is achieved to predict detailed flow and temperature distributions and to derive hotspot factors. By employing a novel concept of rotational symmetry, the huge computational effort has been reduced to minimum. No such study has been reported for any other reactors. This is vastly different from the traditional approach of sub-channel analysis. A structured mesh generator and computational fluid dynamic solver have been developed for special application to FBR subassembly thermal hydraulics. The predictions of the code have been validated against IGCAR / international experiments.

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Comprehensive Fluid Hydraulic Analysis of Reactor Vault Temperatures under Loss of Cooling

EXECUTIVE SUMMARY

Structural integrity of reactor vault is important to be ensured under all normal operating and event conditions of the plant as this being a very important structure supporting the main vessel and safety vessel of the reactor. An extreme scenario of loss of reactor vault cooling happening during a station blackout event has been analysed to demonstrate that the structural temperature limits are respected. In view of the modifications in the originally proposed arrangement of cooling pipes due to construction difficulties, additional cooling arrangement for the safety vessel support region has been worked out through detailed computational studies. Apart from this, a compact arrangement for this additional circuit has also been proposed in a novel manner. This work demonstrated safety of the plant design and could overcome construction difficulties without diluting the design requirements.

OUTLINE

The reactor vault (RV), a concrete structure supporting the main vessel and the safety vessel gets heated due to the heat emanating from the outer surface of the safety vessel insulation (Figure 1). The RV consists of the inner wall and the outer wall which are separated by expanded polystyrene insulation. The inner wall supports the safety vessel while the outer wall supports the main vessel. From design considerations the temperature limits on the reactor vault are (i) 65°C during steady state operation, (ii) 90°C during safety grade decay heat removal (SGDHR) condition and (iii) 177°C during a station blackout condition. The vault is lined by 6 mm thick carbon steel on the inner side and is cooled by water flowing through square pipes embedded in the concrete and welded continuously to the liner. Complete loss of cooling to vault is considered as a Category 3 event. Evolution of vault temperature during this event has been analyzed and the safety action required has been finalized.

The thermal hydraulic analysis has been carried out in two parts: (i) Global analysis for the bottom shield, lower lateral shield and upper lateral shield, and (ii) Local analysis for the safety vessel support embedment (SVSE). The computer code HEATING5 has been used for the global analysis, and commercial CFD code for the latter.

Two extreme scenarios of loss of vault cooling have been considered. They are: (i) controlled shut down of the reactor within a short time after the occurrence of the event and (ii) station blackout extending for 14 h. In the former, the main vessel temperature does not exceed 420°C, which is brought down gradually to 200°C by controlled cooling of the primary sodium system. The maximum temperature of the liner is only 70°C after 18 h. After this instant, the peak temperature reduces as the heat inflow into the vault inner surface reduces while heat diffusion in the concrete continues at a faster rate. Thus, the peak temperature on the inner surface of the vault for most part of the vault surface is only 70°C, which is acceptable.

In the second scenario, the main vessel temperature increases from nominal value to a maximum value of 575°C during the transient as the cooling rate is very slow, due to the low decay heat removal capability of the SGDHR system. The evolution vault temperature during a station black out is depicted in Figure 2. The thermal response of the RV is sluggish due to its large thermal inertia. Even 14 h after the loss of cooling, the liner temperature is only 76°C. As in the earlier transient, the thermal effect is limited very close to the liner.

SVSE region during station blackout has also been analysed in detail. The peak temperature reached is only 122°C, which is far below the allowable value of 177°C. It is therefore established that the event of loss of cooling to vault is easily manageable in the case of PFBR due to relatively large thermal inertia of the concrete structure. The operator action recommended for this event is controlled shut down of the plant, in about ½ h after confirmation of the event.

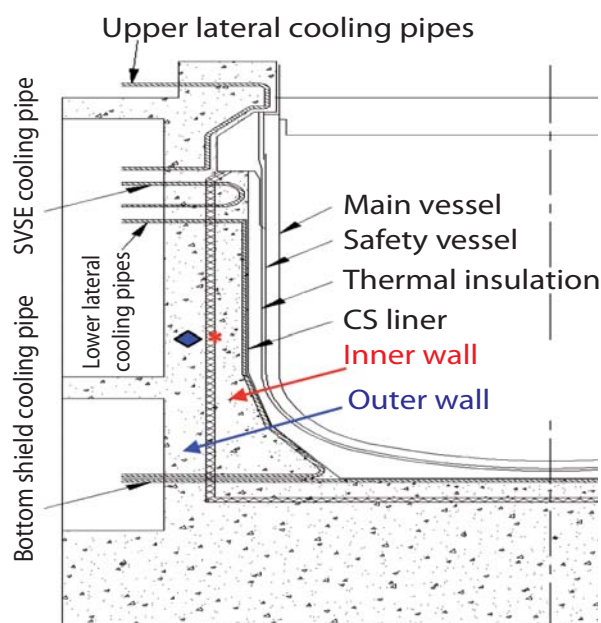


Fig. 1: Reactor vault

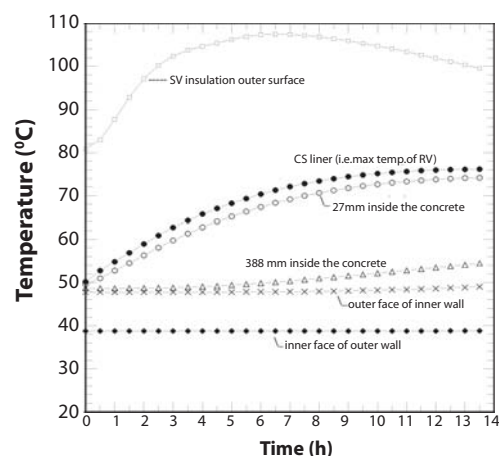


Fig. 2: Temperature evolution in reactor vault during station blackout

ADDITIONAL INFORMATION ABOUT EFFECT OF LONGER OUT-OF-PILE DELAY

The original arrangement of cooling pipes proposed for reactor vault is a square pipe layout with miter bends welded to the liner all along up to the support location of the safety vessel to the reactor vault. However, from construction considerations, this was modified leaving the support location un-cooled. Detailed studies were performed to arrive at an additional cooling loop for the support location giving due consideration to all the layout constraints. Because of the limitation in the number of headers that can be accommodated in the layout, special design measures to ensure uniformity of coolant flow among various circuits were also adopted.

GENERAL EXPLANATION ON ANALYSIS OF SVSE TO DEVICE A SPECIAL COOLING ARRANGEMENT

The re-routing of original proposed layout of cooling pipes necessitated the provision for a separate cooling circuit for the SVSE location maintain its temperature within permissible levels under all operating conditions of the reactor. A three dimensional thermal hydraulic analysis of reactor vault has been carried out to arrive at an acceptable layout for the circuit with the consideration of other geometrical and construction constraints. Temperature of distribution of RV predicted in the support location under normal operation conditions of the reactor with only one loop of the cooling system in operation is shown in Figure 3.

Because of space limitations, the additional cooling circuit had to be designed with compact layout. It was also desirable to have only minimum headers for the distribution of coolant flow among various cooling pipes. Several configurations such as converging and diverging headers, headers with supply connections at the ends, headers with supply connections at the middle, orificed pipes etc. have been investigated. Finally, a configuration of headers with a single header catering to forty four cooling pipes, supply connection to the headers at its mid location and cooling pipes provided with special orifices has been selected which meets all the requirements.

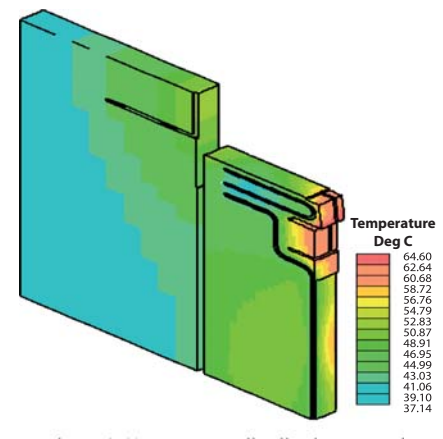


Fig.3: Temperature distribution around safety vessel support embedment

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Thermal hydraulic analysis of reactor vault involves multi mode conjugate heat transfer study considering all the three modes of heat transfer. The model considers conduction in walls, radiation heat exchange among various surfaces of structures through the transparent nitrogen gas and natural convection of nitrogen gas in the annuli between the main vessel, safety vessel and the reactor vault, simultaneously. The solution for the coupled 3-D transient thermal and hydraulic problem has been obtained using commercial CFD code STAR-CD and HEATING5. The contact resistance between cooling pipe and concrete has been obtained through an innovative experimental procedure combined with theoretical calculations.

ACHIEVEMENT

This work is of high relevance to the project. By a comprehensive thermal hydraulic study, solutions have been provided to the site to ease construction by re-routing the cooling coils respecting space limitations. It is also demonstrated that the concrete temperature limits are respected even under a station blackout condition. Also, there is no need for providing emergency power for the vault cooling system. Benchmark contact resistance data between concrete and pipe has been obtained through an innovative approach of combining experimental and theoretical studies.

PUBLICATIONS

1. "Reactor Vault Temperature in Upper Lateral Shield Region:Effect of Long Radius Bend In Cooling Pipes", PFBR/21100/DN/1026/RA.
2. "Assessment of Reactor Vault Temperatures Under Loss of Cooling", PFBR/21100/DN/1007/R-A.
3. "Hydraulic Investigation of Flow Distribution in SVSE Cooling System", PFBR/21100/DN/1025/R-A.
4. "Temperature Distribution in Safety Vessel Support Embedment", PFBR/21100/DN/1028/R-A.

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Modelling of Inter-Wrapper Flow in Core Thermal Hydraulics from first Principles

EXECUTIVE SUMMARY

For improving the reliability of decay heat removal system of a reactor, it is necessary to take advantage of heat removed by the inter-wrapper flow. Modeling Inter-wrapper flow is complex and calls for high fidelity analysis. Porous body models can be used to overcome this problem for which it is necessary to have a good understanding of the characteristics of inter-wrapper flow pressure drop and heat transfer. In literature, correlations for pressure drop and heat transfer are not available for such complex flow paths. Hence, these relations have been developed from first principles and correlations are proposed for pressure drop and heat transfer coefficients as a function of Reynolds number. These correlations are being used in the global analysis of decay heat removal system.

OUTLINE

Unlike fossil fuel power plants, nuclear reactors generate heat (decay heat) even after reactor shutdown. An important task in the successful thermal hydraulic design of a reactor is the demonstration of safe and reliable Decay Heat Removal (DHR) even under the worst possible conditions like complete power failure. In PFBR, heat generated in the fuel and blanket subassemblies (SA) of the core is removed by forced circulation of sodium (coolant) through the SA using Primary Sodium Pumps (PSP) (Figure 1). The PSP are driven by off-site power and backed by diesel generators. A highly reliable Class I power supply (powered by batteries) is also provided to run the PSP for 4 h at 20 % speed, which is sufficient to remove decay heat. The highly reliable DHR system is designed such that even under an unlikely event of complete power failure, the decay heat from the SA is removed keeping all the temperatures within design safety limits.

During loss of forced flow through the core, natural convection currents are set up inside and outside of the SA i.e., in the gaps between SA called Inter-Wrapper Flow. The former is supported by the buoyancy forces generated by the heat transfer from primary sodium to secondary sodium in the IHX and the heat transfer in core, while the latter is due to the complex thermal hydraulic interaction amongst the inter-wrapper space, hot pool and DHX. Heat removed by the IWF is significant during natural circulation of coolant through the core and has large potential to reduce SA peak temperatures during onset of natural convection.

The IWF gaps are narrow (~ 3.4 mm) compared to length (~ 3 m) and width of the SA (131.3 mm). A large number of SA (1758), stacked vertically in a large pool of sodium (12 m diameter) makes the flow path complex and three dimensional. A transient analysis with complete 3-D model for the IWF integrated with hot and cold pools calls for a high fidelity analysis involving billions of computational grids. One method to circumvent it is to use porous body model for the core linking it with hot pool. In porous medium formulation, a relatively coarser mesh can be used with appropriate surface permeability and volumetric porosity. The pressure drop encountered by the fluid is specified as additional resistance in the momentum equations and the heat transfer from the solid is specified as additional heat source in the energy equation. In literature, correlations for pressure drop and heat transfer are not available for such complex flow paths. These relations have to be developed from first principles. Towards this, detailed multi-dimensional computational fluid dynamics investigations have been carried out (Figure 1). The variation of friction factor and heat transfer coefficient as a function of Reynolds number has been predicted and the correlations proposed for both axial and cross flow over hexagonal subassemblies. (Figures 2 & 3).

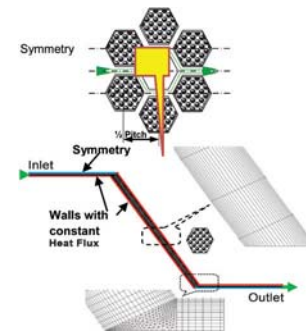


Fig.1: CFD mesh for inter-wrapper flow

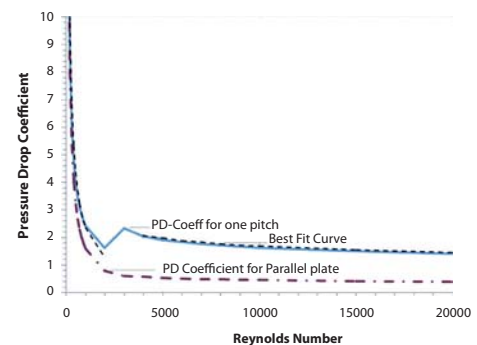


Fig.2: Variation of pressure drop coefficient as a function of Re. Correlations proposed for cross flow pressure drop (Figure 2) $C_f = 1034 Re^{-0.88}$ $200 < Re < 2000$ $C_f = 12.82 Re^{-0.22}$ $3000 < Re < 20,000$ Where $C_f = \Delta p / (\rho V^2)$

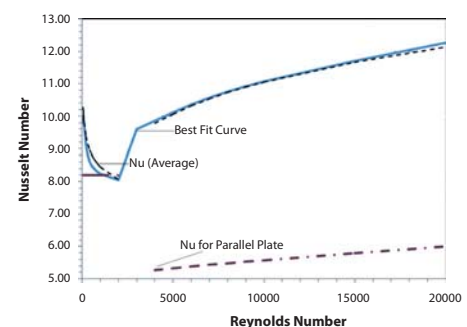


Fig. 3: Variation of Nusselt number as a function of Re. Correlations proposed for heat transfer $Nu = 13.23 Re^{-0.065}$ $200 < Re < 2000$ $Nu = 3.217 Re^{0.134}$ $3000 < Re < 20,000$

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The geometry has been modeled with unstructured grids. In laminar flow regime, the laminar model is used. In turbulent flow regime, the turbulence is modeled using Low Reynolds number model. This eliminates the uncertainties in using wall functions and estimates the pressure drop and heat transfer from first principles. In order to model the periodic behavior of the cross flow, cyclic boundary conditions are imposed at the inlet and the outlet.

PUBLICATIONS

U. Partha Sarathy ,T. Sundararajan,C. Balaji and K. Velusamy, “Characterization of Sodium Cross Flow Over Hexagonal Subassemblies’, 20th National and 9th International ISHMT-ASME Heat and Mass Transfer Conference at Mumbai, India, 2010.

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Indigenously evolved Source Subassembly Design for Fast Reactors

EXECUTIVE SUMMARY

Neutron detectors are housed in control plug to monitor the reactor. In order to monitor the reactor during shutdown condition and to facilitate the reactor start-up after fuel handling, three counts per second (cps) count rate is required at the detector location. This warrants use of neutron source subassembly (SA). Neutron source subassembly is designed meeting the challenging neutronics, structural and thermal hydraulics requirements. It employs a standard hexcan with 61 pins of Antimony oxide (Gamma emitter) for a length of 500 millimeter surrounded by a hexagonal Beryllium block (Neutron emitter by gamma-neutron reaction) of 20 millimeter thick. The thermal striping probability due to low heat generation by source material is overcome by addition of fuel pin bundle to the required volume, which is a novel approach. Three source subassemblies are loaded symmetrically in the first row of blanket subassembly locations in the core. This integrated design is first of its kind.

OUTLINE

The location and number of source Sb_2O_3 -Be (γ -n) type source SA is very important as they would displace some fuel/blanket SA in the core. Fuel SA displacement would result in penalty on the total power and hence an optimum position is arrived at. Three source SAs are located 120° apart symmetrically in the blanket region (8th ring) next to core-II fuel SAs in the PFBR core. The life of source SA is 4 cycles of operation and they remain in storage location for 3 cycles for decay and cooling. The number of source SA required is arrived at from the requirement of minimum shutdown count rate of 3 cps at the detector location after two months of shutdown. The variation of count rate vs shutdown period for different number of source SAs is shown in Figure 1. It is inferred that with two source SAs, count rate of 3.2 cps is available after two months of shutdown, but during replacement of source SA, the source SA in the reactor is not sufficient to provide the necessary count rates. Hence three numbers of source SAs are considered for PFBR.

The source SA consists of 61 pins of Sb_2O_3 surrounded by a hexagonal beryllium block of 500 mm length. The source volume in the pin starts exactly at the active core bottom level and extends up to the middle of the core. Since the heat generation in source SA is very low as compared to the adjacent fuel SA, to avoid thermal striping in the control plug, short length fuel pins (217 nos.) are incorporated to increase heat generation in source SA which is a novel feature. Fuel pins in which the fuel column (MOX fuel with 27.7% PuO_2) has a length of 275 mm are supported above the source pins. The external features, head and axial shielding of source SA are identical to that of blanket SA. The design features of source SA are shown in Figure 2.

The required source (Sb_2O_3) volume per SA (455 cm^3) is achieved by selecting suitable pin diameter, length & number of pins in a SA. The constraints being:

(a) Low melting point (655°C) of Sb_2O_3 as well as thermal conductivity. This restricts the pin diameter to be small since the centerline temperature of Sb_2O_3 at 115% peak power under hotspot conditions shall not exceed its melting point; (b) Restricted source length between the bottom and middle levels of the active core region (500 mm max.) with a minimum length of 250 mm from active core bottom to avoid the direct contribution of source neutrons to detector; (c) The beryllium surrounding Sb_2O_3 should have minimum thickness of 20 mm on all sides to produce enough source neutrons, hence less number of pins; (d) Coolant flow in the source SA should be such that the temperature difference between the fuel SA and the source SA at the exit is less than 100°C from thermal striping considerations and low melting point of Sb_2O_3 . Hence, the requirement of source material volume, to accommodate within the standard SA geometry meeting the temperature limits is challenging and the design incorporates appropriate engineering solutions to meet the above challenges.

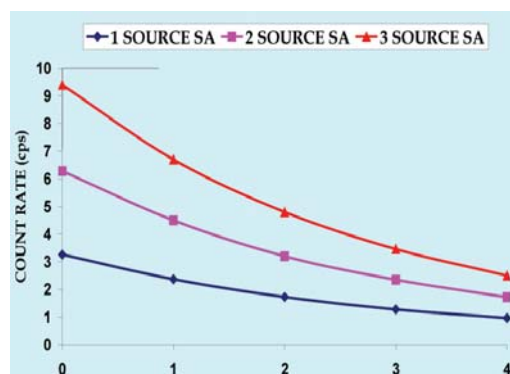


Fig. 1: Count Rate Vs Shutdown Period

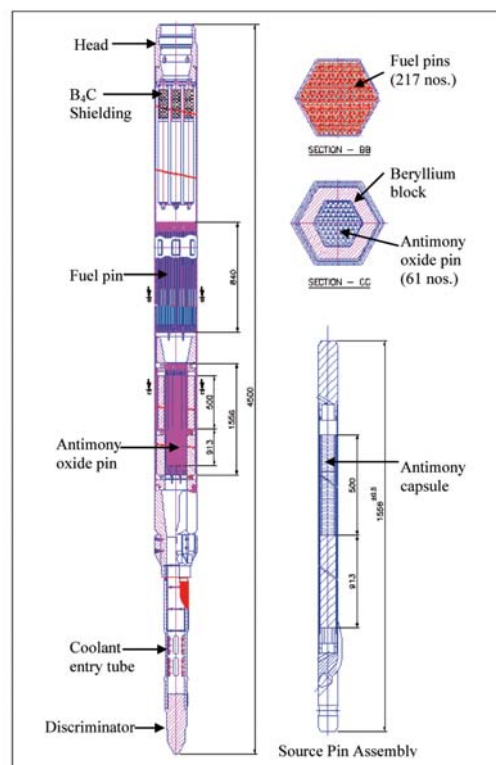


Fig. 2: Source Subassembly

ADDITIONAL INFORMATION ABOUT EXTERNAL NEUTRON SOURCE

In thermal reactors (PHWRs), external neutron source is not required, since the (γ, n) and $(n, 2n)$ reactions of D_2O produce sufficient neutrons for detection during shutdown period whereas in fast reactors, external neutron source is a must. It is to be noted that the source volume requirement and the heat generation is very high in PFBR as compared to FBTR and hence more challenging. In FBTR, irradiated source is introduced in the reactor whereas in PFBR, the source subassembly is loaded without any irradiation and its source strength is zero initially. The source subassembly gets activated insitu in the reactor. This is to avoid handling of antimony oxide which is a Gamma emitter and hence highly radioactive. However, for initial start-up of reactor, in-core detectors are used to monitor the flux level. In-core detectors are removed from the reactor after the initial cycle (cycle-I). The added advantage of having external neutron source is that it helps in reduction of counting times and hence provides faster approach to criticality.

GENERAL EXPLANATION RELATED TO DESCRIPTION

Source Pin Assembly

Cast form of Sb_2O_3 is selected since it gives high density and thermal conductivity as compared to powder/sintered form. To have defect free casting of Sb_2O_3 pellets, an optimum length of 50 millimeter is chosen and is cast inside a SS 316 M clad of outer surface 5.1 millimeter and inner surface 4.36 millimeter. This capsule is seal welded with end plugs on both ends. Ten numbers of such capsules are stacked one over another in an outer clad tube (pin) of outer surface 6.6 millimeter and inner surface 5.7 millimeter and will be capped at both ends with another set of end plugs. Sodium is selected as the conducting medium between the capsules and the clad to respect the melting point of Sb_2O_3 . The source pins are arranged in triangular pitch forming a hexagonal array. Coolant flow of 12 kg/s is required to respect the temperature limits.

Beryllium block assembly

Beryllium is toxic and hence is housed in a helium filled leak tight container. Beryllium block assembly consists of beryllium block covered by outer and inner hexagonal sheaths welded with end plates at top and bottom. The swelling and thermo-mechanical analysis (Figure 3) of beryllium block assembly were carried out and the dimensions are arrived at.

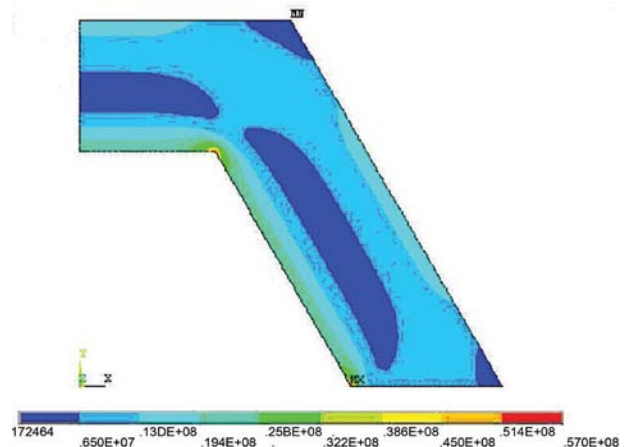


Fig. 3: Thermal Stress (Von-Mises) Distribution In Beryllium Block

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The reactor physics studies decide the number of source subassembly required and the location of source subassembly in the core. Inter subassembly heat transfer analysis between the source and adjacent fuel subassemblies has been done to find out any significant change in the exit sodium temperature of fuel subassembly and it is observed that there is no concern on this aspect. The thermo-mechanical analysis of beryllium block involves modeling of Be block in FEM software to find the temperature drop in Be block and hence the thermal stress pattern in it which indicates that there is no concern over the integrity of Be block.

ACHIEVEMENTS

Source subassembly design for fast reactors is a very challenging task as it involves reactor physics, heat transfer, thermal-hydraulic and mechanical design aspects. Source subassembly design has been indigenously evolved incorporating suitable features addressing the design challenges and satisfying the design criteria. The thermal stripping probability due to low heat generation by source material is overcome by addition of fuel pin bundle to the required volume, which is a novel approach. The integrated design is first of its kind.

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Development of Novel Gas-Gap Type Irradiation Capsule for Irradiation of Specimens at Temperatures higher than bulk Sodium for Fast Breeder Test Reactor

EXECUTIVE SUMMARY

Fuel cladding and wrapper tubes are important core structural components of Fast Reactor. Due to bombardment of high energy neutrons and high temperature prevailing in the fast reactor, the core structural materials undergo severe changes in mechanical and physical properties. Irradiation capsules are useful to irradiate specimens of the structural materials under desired experimental conditions in a test reactor for subsequent post-irradiation examination of the exposed specimens in the radioactive hot cells. A gas-gap irradiation capsule has been designed and developed to irradiate structural material specimens at a higher temperature (upto 650°C) than the temperature of bulk sodium coolant (380-540°C) in Fast Breeder Test Reactor (FBTR). The temperature range of 400-650°C is the main operating temperature range of interest for fast reactor core structural components.

OUTLINE

Irradiation performance testing of fast reactor fuel cladding and wrapper materials can be carried out in FBTR using irradiation capsules. Irradiation capsule is a long tubular fabricated component in which the specimens are kept for irradiation. The irradiation capsule can be inserted in the central bore of fuel, blanket and reflector special subassemblies of FBTR which can in turn be loaded in FBTR core for irradiation of the specimens.

A new irradiation capsule has been designed and fabricated to irradiate structural material specimens at a temperature higher than that of the sodium coolant flowing in the reactor. This capsule has been developed for irradiation of pre-pressurised capsules, tensile, swelling and other specimens in FBTR at 400-650°C to meet the temperature range of interest of fast reactor core components. Pressurised capsules are made of cladding tubes and sealed with inert gas such as argon at high pressure to develop biaxial stress conditions in the tube wall during their exposure. Tensile/swelling specimens are required to determine the change in the mechanical properties/volume due to radiation exposure.

There are several subcapsules coaxially located in the gas-gap irradiation capsule where the specimens are kept in static sodium inside the sub capsules with gas-gap between subcapsules and irradiation capsule. Figure 1 shows the sketch of an irradiation capsule containing five subcapsules. The gap between each subcapsule and the irradiation capsule is filled with helium and argon gas mixture of a distinct composition. The specimen temperature will depend on the neutron and gamma heating rate at the location of irradiation. The width of the gas-gap and the gas composition in the gas-gap is adjusted to attain a desired temperature of irradiation in the specimens. The annular spaces around different subcapsules are not inter-connected due to which, the specimen irradiation temperatures of 400, 450, 500, 550 and 600 °C (for instance) can be achieved in the five subcapsules during irradiation. The specimen temperatures can be chosen by varying the gas width and/or gas composition.

A mock up irradiation capsule containing five subcapsules has been fabricated (Figure 1). Sodium filling in the sub capsules was carried out at Radiochemistry Laboratory in an argon atmosphere glove box and an innovative sealing method was developed to seal the sodium-filling path in a leak tight manner. The subcapsules were machined in such a way that they are located coaxially within irradiation capsule one over the other and have a separate uniform gas insulation layer around each of the subcapsules. X-radiography examination was carried out on the mock up irradiation capsule to verify the internal configuration of the capsule. This type of capsule will be used in FBTR for generation of irradiation performance data on fuel cladding and wrapper materials of interest at higher irradiation temperatures.

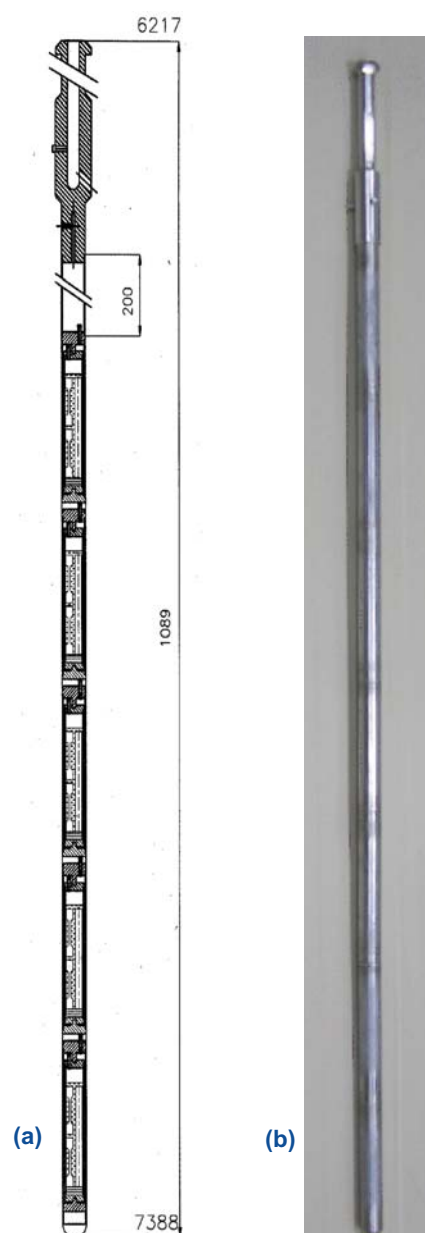


Fig.1(a): Sketch of non instrumented gas gap capsule
(b): Photograph of mock-up gas-gap capsule fabricated

ADDITIONAL INFORMATION

Irradiation temperature is an important parameter that affects the mechanism of defect formation under irradiation and the macroscopic effect of radiation on materials. The defects introduced by irradiation can vary from single vacancies and interstitials to voids and bubbles. At temperatures between 0.1 and $0.3 T_m$, where T_m is melting point in K, the interstitials are mobile and they combine with vacancies and get annihilated; interstitials also agglomerate to form clusters of interstitials. Macroscopically, between 0.1 and $0.3 T_m$, low temperature embrittlement and increase in ductile brittle transition temperature occur in ferritic steels. At temperatures between 0.3 and $0.5 T_m$, concentration of vacancies produced by irradiation far exceeds thermal equilibrium concentration and they coalesce to form voids and get stabilized by helium formed by irradiation. This leads to increase in volume known as 'void swelling' in austenitic stainless steel in this temperature range.

GENERAL EXPLANATION RELATED TO DESCRIPTION

Any material specimen under irradiation will generate heat due to attenuation of neutron and gamma rays on it. This will lead to rise in temperature of the specimen over sodium temperature flowing around the irradiation capsule. However, the desired temperature of irradiation may still be higher. Gas-gap capsule will be useful in achieving the desired higher irradiation temperature in the specimens. The thermal conductivities of helium and argon differ approximately by eight times and by mixing the appropriate composition, higher temperature is achieved in the specimens. The design of gas-gap capsule requires the data on sodium coolant temperature and neutron and gamma heating rates both along length of irradiation capsule. The width of the gas-gap and the gas composition to be filled in the gap will then be decided based on the desired higher irradiation temperature. Gas-chromatography equipment was used to fill the mixture with exact composition of the gas. The closure of the filling path of sodium in the subcapsule, and the gas mixture in the gas-gap in a leak tight way posed challenges and methods have been developed to carry out this task. Figure 3 shows the details of a subcapsule.

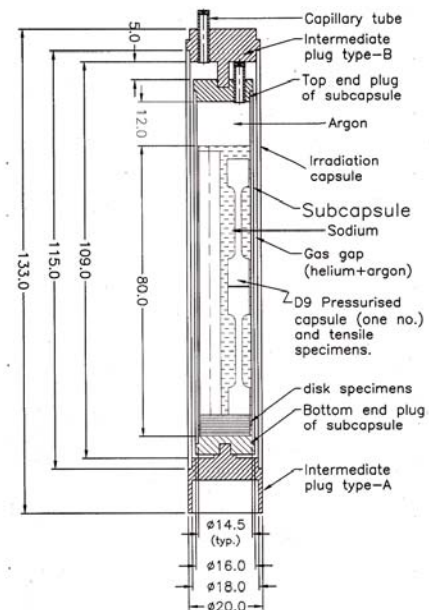


Fig. 3: Details of a subcapsule

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The neutron flux varies in a cosine shape along the length of irradiation capsule and nuclear heating rate also varies in a similar manner. Temperature of flowing sodium along the irradiation capsule varies linearly. The gap-width and the gas composition have been arrived at by considering both the above variations and the intended temperature of specimens in the subcapsule. Heat transfer due to conduction and radiation has been considered in the gas-gap. The variation of thermal conductivity of helium, argon, stainless steel and sodium with temperature have also been taken into account. An iterative procedure has been used.

ACHIEVEMENTS

The development of gas-gap irradiation capsule is a significant achievement which enables to carry out irradiation at the temperature of interest to fast reactor program. Using this irradiation capsule, any irradiation temperature higher than bulk sodium temperature can be achieved. The methodology to seal the gas filling path and the sodium filling path in a leak tight way has been developed at IGCAR. Sealed capsules have been tested for integrity by exposing them to temperatures upto 650°C and their integrity demonstrated.

PUBLICATIONS

S. Murugan, V. Karthik, K.A. Gopal, N.G. Muralidharan, S. Venugopal, K.V. Kasiviswanathan, P.V. Kumar and Baldev Raj, "Irradiation Testing of Structural Materials in Fast Breeder Test Reactor", in IAEA Technical Meet (TM – 34779) on Research Reactor Application for Materials Under High Neutron Fluence, IAEA Headquarters, Vienna, Austria 2008.

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Core Temperature Monitoring – 3 Dimensional Computational Fluid Dynamics Studies

EXECUTIVE SUMMARY

Core monitoring thermocouples (TC) continuously check the adequacy of subassembly (SA) cooling. They are located in control plug, at ~100 millimeter above the subassembly top. Accurate measurement of subassembly sodium outlet temperature is essential for timely safety actions. Due to fabrication and erection tolerances, the position of thermowells change. Owing to irradiation as well as thermal effects, the subassembly bow outwards. Further, cold sodium issuing out of control subassembly may dilute the thermocouple readings of neighboring subassembly. All these aspects have been investigated by a comprehensive 3-D CFD study, wherein flow and temperature distributions below the core cover plate have been predicted for fresh and irradiated core configurations. Based on this study, it has been established that the maximum dilution in fuel subassembly is 2.5 K while that in blanket subassembly is 8 K. Further, the control subassembly flow is seen not to influence the neighboring thermocouple readings.

OUTLINE

Totally 210 SA (180 fuel and 30 blanket SA) have monitoring thermocouples (TC), enclosed in thermowells. The central subassembly outlet temperature is monitored using four fast acting bare TC held by central canal plug. Core monitoring thermocouples have several important safety functions. At the bottom of the control plug (CP), there are two horizontal plates and a vertical shell. Immediately after exiting vertically from the SA, which is positioned directly under the CP, hot sodium faces the first horizontal porous plate placed 475 mm above SA top. This is the lattice plate (LP), with a porosity of 60%. Subsequently, it enters a cylindrical cavity enclosed at the top and bottom by the core cover plate (CCP) and LP respectively. The CCP which is a solid plate is located at a distance of 1260mm from the SA top. The lateral wall of the cavity, joining the CCP and LP, is a porous skirt with a porosity of 10%, which is provided to control thermal stratification in the hot pool. The thermowells holding the TC are supported on CCP and guided by LP.

The above system leads to a series of changes in the direction of flow coming out of SA. Thus, the flow which is vertical when coming out of the SA develops a horizontal component after it exits the cylindrical cavity, either below the porous skirt or through the porous skirt itself, with the former mode dominating. These directional changes in velocity of hot sodium emanating from various SA are non uniform and depend on the relative positions of the SA themselves. Thus, this whole setup leads to a phenomenon called 'flow flowering'. This occurrence in turn gives birth to the problem investigated i.e., spatial dilution of SA outlet flow leading to temperature masking at TC positions. Due to fabrication and erection tolerances, the position of thermowells change. Also, the SA bow outwards due to irradiation and differential thermal expansion. For accurate temperature measurement, the tips of TC should be as close to the SA top as possible. To improve accuracy, a radial shift of 20 mm from SA centerline is provided for majority of the TC. But, this shift could not be provided to 36 TC close to absorber rod drive mechanism shrouds and neutron detectors. Clear understanding of all these effects on temperature dilution is very essential to validate the trip threshold.

Towards this, 3-D flow and temperature distributions below the core cover plate have been predicted for fresh and irradiated (100 MWd/kg) cores (Figure 1). All the monitored SA and absorber rod shrouds have been physically modeled and the following inferences have been drawn (a) The cold sodium from control SA does not influence the TC readings of the neighboring SA, (b) The maximum dilution in fuel SA readings is 2.5 K (Figure 2) and that in blanket SA readings is 8 K, which is acceptable (c) The present position of core monitoring TC meets all the thermal hydraulic requirements.

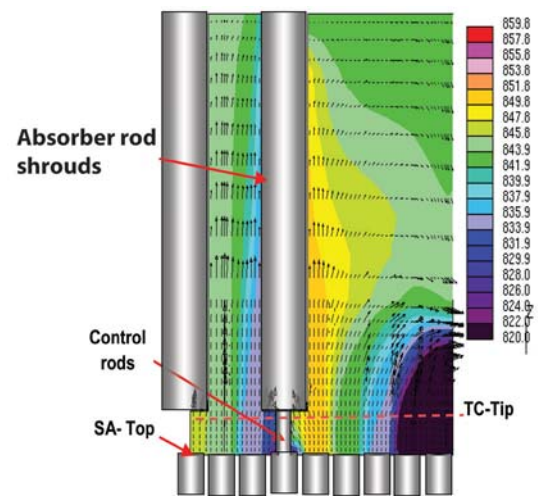


Fig. 1: Sodium flow and temperature (K) distributions below CCP in a vertical plane through control SA

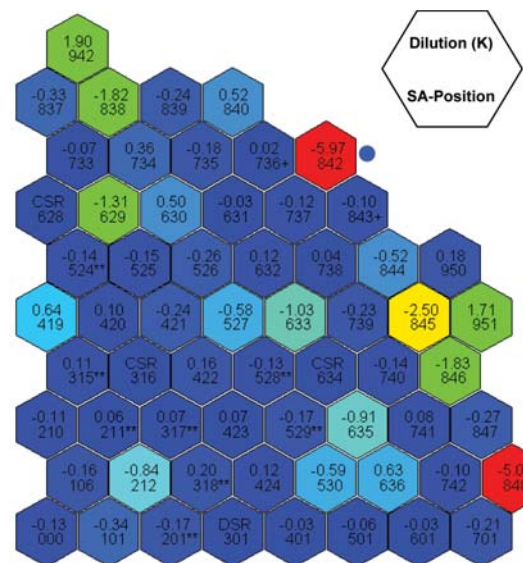


Fig. 2: Dilution in TC readings (K) (fresh core with maximum outward movement of TC)

ADDITIONAL INFORMATION: MODELS APPLIED

Commercial CFD codes have been used for the above studies, wherein the 3-D form of conservation equations of mass, momentum and energy are numerically solved by control volume based discretisation method. In this study the SIMPLE algorithm is used to resolve pressure-velocity coupling. The effect of turbulence is taken care of by the standard high Reynolds number K- ϵ model, while the buoyancy effects are considered through the Boussinesq approximation.

ACHIEVEMENTS

This work is of high relevance to the centre, wherein the acceptability of present position of core monitoring thermocouples, in irradiated core, has been demonstrated by a comprehensive 3-Dimensional computational fluid dynamic simulation. Due attentions have been paid to modeling individual sodium jets, thermal / irradiation bowing of subassemblies, manufacturing / erection tolerances of thermowells and dilution effects of sodium issuing out of control subassemblies.

PUBLICATIONS

3-D Thermal Hydraulic Investigations on subassembly Outlet Temperature Dilution: Effect of CSRDM Shrouds and Bowing of subassembly”, PFBR/31410/DN/1081/R-A.

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Innovative Design of Structures for handling of Over Dimensioned Components of Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

Large and thin shell components of PFBR are manufactured with very tight tolerances. Hence, good care is essential to avoid errors and damage to the parts during erection. Also, the functionalities and specification of these components are different from conventional components requiring specialized erection techniques. The safety and main vessels are to be placed in reactor vault pit, having narrow gap between the vessel and vault. Since the components are thin shell structures, special attachments and handling fixture are designed for erection. A comprehensive structural analysis of the components has been carried out considering all the loads occurring during handling. The safety vessel (diameter 13.5 meter and 16 meter height) has already been erected successfully on the inner reactor vault within a gap of 70 millimeter. Erection of further components is planned and mockups are performed wherever required.

OUTLINE

The main/safety vessels and other structures are unique to PFBR. The functional and quality requirements are also different from conventional industries. These vessels are large diameters thin shell structures and are to be placed in narrow gaps. Erection of components and construction of reactor building have to be carried out simultaneously. So, erection and construction methodologies are planned in advance. Narrow gaps and limited space call for tight tolerances in construction. Also, erection procedure for the components demands new attachments and equipment.

Normally, spiders are attached to the thin shell structures to retain their shape within tolerable deformation. It is planned to erect the components with minimum attachments, so as to reduce the risk of damage to the components. Special attachments and handling spiders are designed to handle the vessels. The vessels are handled without attaching stiffening spiders.

Erection methodologies for main vessel and safety vessel have been finalized after a detailed structural integrity analysis of the handling spiders. The safety vessel (SV) is first erected inside the inner reactor vault (IRV). Subsequently, the main vessel (MV) assembly with core support structure (CSS) and core catcher (CC) is lowered in the safety vessel. During this erection, four lugs are attached to core support structure. The main vessel will be lifted through four ropes attached to these lugs. Ropes will be connected to a spider at the top and subsequently to the crane hook. Then the spider will be supported on the top of the outer reactor vault (ORV). The main vessel will be kept hanging.

While hanging from the outer reactor vault, a box type ring will be attached to the main vessel top portion with 126 vertical stiffeners. The main vessel will then be lifted through the box type ring with 126 rods attached to it. The rods will pass through the sleeves present in outer reactor vault. The load of the main vessel will then be transferred to the rods and the spider will be removed, to facilitate erection of grid plate, thermal baffle, main vessel cooling pipes, primary pipes and inner vessel in the main vessel.

Finally, roof slab will be brought to reactor vault and fixed on the outer reactor vault. The main vessel along with the internals will be lifted further to bring in position to the roof slab and welded to it. To carry out this operation, the lifting spider of the main vessel will again be brought back on the top of outer reactor vault. The spider will be placed on twenty four jacks. The main vessel will be attached to the spider through four ropes attached to the lugs of the core support structure. Main vessel will be lifted by the jacks and welded to the roof slab. Subsequently, the attachments will be removed. The erection sequence is depicted in Figure 1.

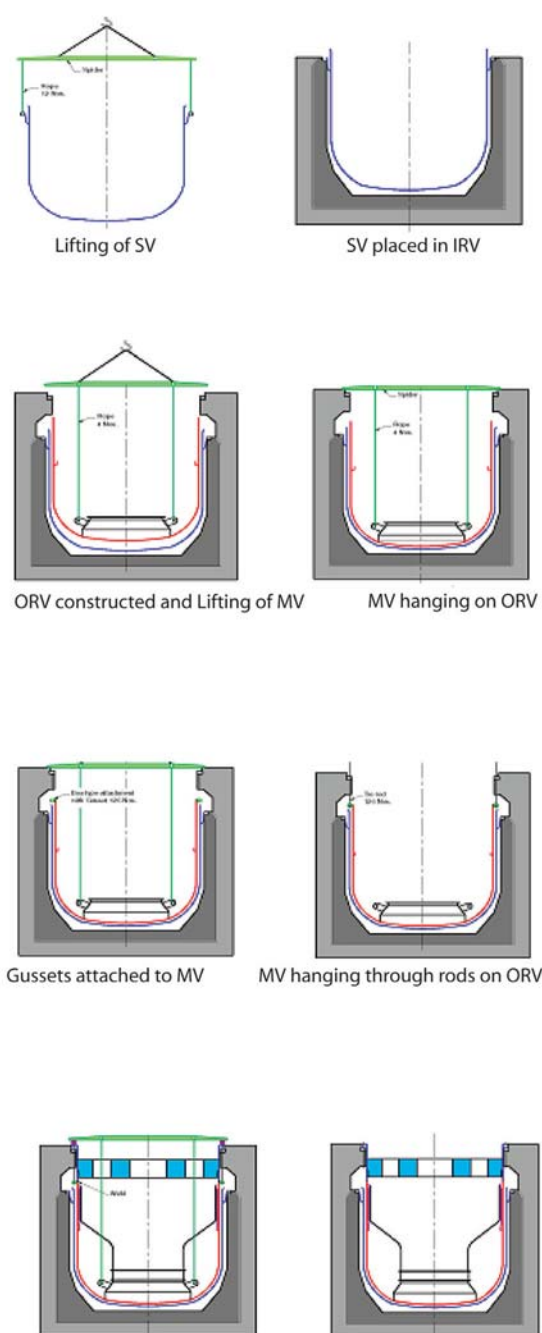


Fig. 1: Erection sequence of reactor assembly

GENERAL EXPLANATION RELATED TO DESCRIPTION

Analysis for Safety Vessel

Inner diameter of safety vessel is 13.5 meter and its weight is 155 tonnes. The safety vessel support is a box type flange with upper and lower plates connected with stiffeners. The lugs for lifting safety vessel are designed such that the load of the safety vessel gets transferred with both top and bottom plate of the flange to avoid bending of the plates (Figure 2). A lifting spider is designed to facilitate vertical lifting of safety vessel, i.e., the lifting lugs are connected to spider with vertical ropes. The analysis has demonstrated that six lugs are adequate for lifting safety vessel. The stresses on different parts are limited within elastic limit (yield stress). However, considering possible uncertainties in load distribution in actual site condition, twelve supports were recommended. The radial deflections at the conical support to safety vessel junction are also found to be small (<0.4 millimeter), which are insignificant. Hence, there is no requirement of putting spider at that location during erection. The lifting spider is made of twelve radial beams which are interconnected by tangential beams. The spider is also analysed for the erection loads and the design is optimized based on rigidity and ease of manufacturing.

Analysis for Main Vessel

Main vessel is 12.9 meter diameter and 25 millimeter thick vessel with dished end and without any penetration and attachments. It is subjected to axial load of 2200 tonnes throughout its life. It is intended to have axial attachment on main vessel for handling to avoid any risk of circumferential deflection. While main vessel is hanging during erection of its internal components, the supporting attachments are subjected to about 500 tonnes load. Hence, to hold main vessel, 126 vertical stiffeners are attached to it. Two shells are attached to the gussets to have torsion rigidity. The shells are connected with a base plate which will be used for attaching lifting rods. While holding in position, main vessel bends inwards at the top causing compressive hoop stress. To reduce hoop stress and avoid risk of buckling, a top plate is attached to main vessel with stitch weld. A thin plate with stitch weld is attached at bottom of stiffener to reduce local stresses. The height of the stiffeners and shells are optimized based on site condition and stresses in different parts of the attachment are determined. The attachment is analysed and the stresses are limited within the elastic limits (Figure 3). The main vessel is also checked for buckling risk.

The lifting spider of MV is made of four cross beams connected by square beams. A circular channel made by two plates connected by a web plate is attached to the above cross beams through which twenty four box type radial arms are attached (Figure 3). The arms are joined to the channel with 150 millimeter diameter pin, so as to facilitate minor adjustments for jack positions. The spider will be used for lifting and placing the main vessel inside the safety vessel and for lifting the main vessel with internals to be welded to roof slab. The spider is analysed for all the loads during the above conditions and the stresses are limited within elastic limits.

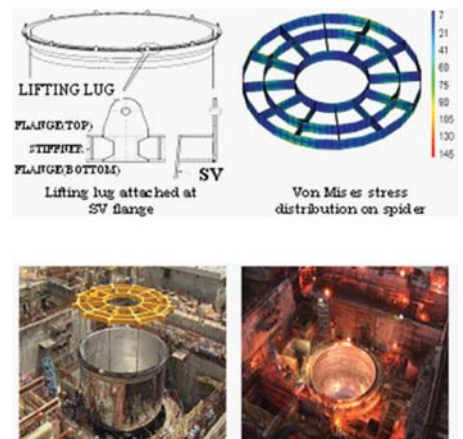


Fig. 2: Stress analysis of safety vessel erection spider and successful erection of safety vessel

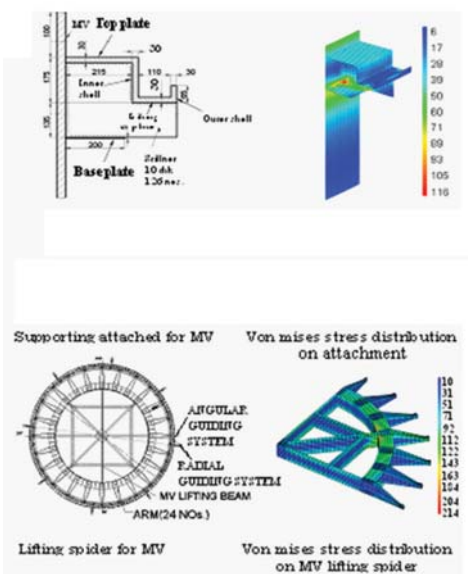


Fig. 3: Stress analysis of main vessel erection spider and attachment and successful erection of safety vessel

ACHIEVEMENTS

In the absence of established guidelines and experiences, this is an international benchmark, wherein original and innovative ideas have been implemented to arrive at an optimum and robust handling systems for thin shells of large diameters, viz., main vessel and safety vessel, with narrow gaps between the vessels and reactor vault. The erection sequence has been backed up by comprehensive structural analysis, mock-up tests and detailed safety reviews. The robustness of the design and erection procedures has been validated by successful erection of safety vessel.

PUBLICATIONS

B. C. Sati, V. Balasubramanian, P. Chellapandi, and S. C. Chetal. "Structural Analysis towards Erection of Prototype Fast Breeder Reactor Components." SMiRT-20, 2009.

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Innovative Design and Development of Steam Generator for Future Fast Breeder Reactors

EXECUTIVE SUMMARY

The concept of Steam Generator (SG) with 30 meter long tubes has been conceived for future Fast Breeder Reactors (FBR) as one of the measures to improve economy and enhance safety of the plant. Steam generator transfers heat from secondary sodium circuit to steam-water system to produce High pressure and high temperature steam. The tube sheet and tube bundle of steam generator are designed as per French code for fast reactors, namely, RCC-MR Class 1 except for water/steam dished head, which is designed as per ASME Sec VIII Division 1 with due modification for a design life. The rest of steam generator is designed as per RCC-MR Class 2. Robust design is chosen towards ensuring reliable tube to tube sheet joint. Based on the experience with design, development and manufacture of steam generator for PFBR, an elaborate and detailed review of number of steam generator modules per loop for future FBR together with design optimization of each module has been undertaken. Also, a technology development exercise for indigenous manufacture of 30 meter long seamless tubes has been initiated.

OUTLINE

Based on design, development and manufacturing experience with SG for PFBR, integral once through SG with modified 9Cr 1Mo as principle material of construction is chosen for future FBR (Figure 1). Owing to the constraints in tube manufacture, the PFBR SG was designed with a tube length of 23m. However, for future FBR a longer length of tubes is preferred in order to reduce number of tube to tube sheet joints for minimizing possibility of sodium-water reaction through out the design life. The tube length as a function of heat transfer area, related to number of tube to tube joints, and joint failure rate are presented in Figures 2 & 3 respectively. It is seen that a tube length of 34 m is desirable as it meets all the design limits yielding lower joint failure rates / annum, thereby increasing the availability of SG module. However, due to constraints, indigenous manufacture of tubes upto 30 m is only possible at present and hence, the same is chosen.

The option of raised spigot tube to tube sheet square butt joint (Figure 1) by internal bore welding torch, followed by post weld heat treatment and 100% radiography with micro focal rod anode X-ray machine is retained. Further, the design incorporates exclusive thermal expansion bend for the tubes at cold end to accommodate the differential thermal expansion between the shell & tube bundle and between tubes. Above innovative concepts ensure relatively high reliable SG as compared with designs of BN600, PFR etc, which also have satisfactory operating experience.

Detailed optimization studies, taking into consideration overall effect of capital cost, outage cost and construction schedule, have indicated that for 500 MWe plant with 40% thermal efficiency, a design with 3 modules / loop each having 475 tubes/module (ID / OD: 12.6 / 17.4 mm) are optimal leading to 35% lesser joints thereby, improving the safety and economy. The tube dimensions are arrived after accounting for the allowances for corrosion, chemical cleaning and fretting wear over a design life of 60 years. The above configuration reduces the number of modules from 8+1 for PFBR to 6 x 2 + 1 for the twin unit design of future FBR. The strategy of sparing one module for the twin unit plant is to reduce the overall cost of spares.

Also for future SG, tube-sheet and dished end thickness will be nearly same as PFBR SG for which the manufacturing experience already exists. Further, their number is reduced with the reduction in number of modules. The SG is supported through a conical support at its Centre of Gravity and guide supports are provided at either of the tube-sheet ends acting as seismic movement arrestors. Also as compared to PFBR, the bend location in SG for future FBR is located at around 1.5 m nearer to bottom tube sheet leading to corresponding reduction in height of the outer shell.

A comprehensive technology development exercise is already underway to address all the technical issues relating to 30 m long tubes and demonstrate the indigenous manufacturing capability well ahead of start of manufacture of SG for future FBR. Simultaneously, indigenous development of 34 m long tubes is being explored.

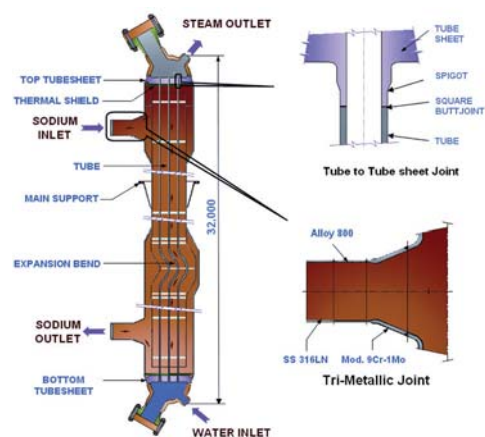


Fig. 1: Steam generator

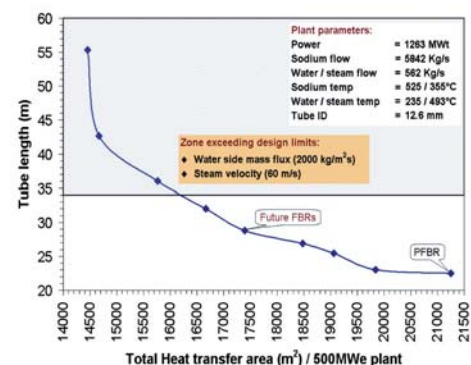


Fig. 2: Variation of tube length as a function of total heat transfer area

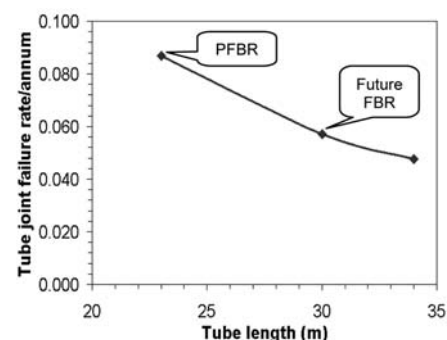


Fig. 3: Variation of tube joint failure rate as a function of tube length

ADDITIONAL INFORMATION

Also for future SG, tube-sheet and dished end thickness will be nearly same as PFBR SG for which the manufacturing experience already exists. Further, their number is reduced with the reduction in number of modules (Figure 4). The SG is supported through a conical support at its CG and guide supports are provided at either of the tube-sheet ends acting as seismic movement arrestors. Also as compared to PFBR, the bend location in SG for future FBR is located at around 1.5 m nearer to bottom tube sheet leading to corresponding reduction in height of the outer shell.

A comprehensive technology development exercise is already underway to address all the technical issues relating to 30 m long tubes and demonstrate the indigenous manufacturing capability well ahead of start of manufacture of SG for future FBR. Simultaneously, indigenous development of 34 m long tubes is being explored. Further, it is judged that issues relating to transportation and erection on account of increase in tube length of SG are resolvable.

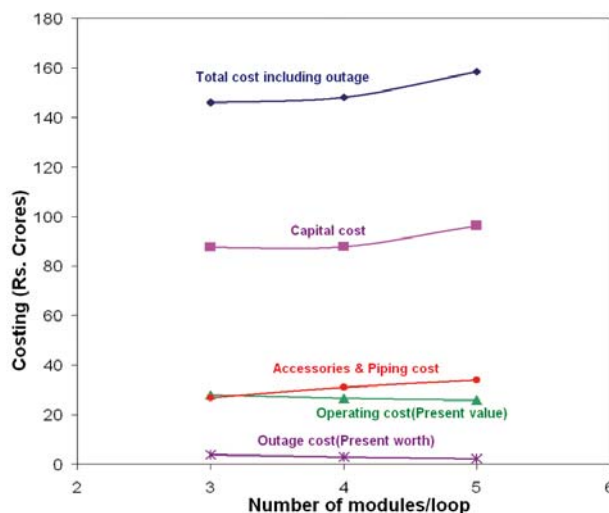


Fig. 4: Variation of cost as a function of number of modules

GENERAL EXPLANATION RELATED TO DESCRIPTION

In steam generator, the hot sodium enters the shell side through a single inlet nozzle, flows upward in annular region wherein flow distribution arrangement is located to evenly distribute the flow around the tubes in the bundle. The cold sodium exits through the bottom outlet plenum to the outlet nozzle. Feed water enters the tubes from the bottom tube sheet through orifices, flows upward in counter current direction to the down coming sodium. Superheated steam exits the steam generator through the nozzle at top dished end. A tri-metallic joint between nozzle and secondary sodium piping with Alloy eight hundred intermediate pipe spool is provided at the nozzle-piping junction to take care of differences in thermal expansions between SS 316LN and Mod 9Cr – 1Mo. Detailed technology development programme preceded the manufacture of steam generator for PFBR (Figure 5) in order to sensitise the Indian industries with respect to complexities in manufacture of steam generator and also develop indigenous manufacturing capability.



Fig. 5 : PFBR Steam generator under manufacture

ACHIEVEMENTS

The concept of steam generator with 30 meter long tube has a saving of 20% on materials and expected to result in similar savings in manufacturing and accessories costs. This will contribute to the economy of future FBR. Also, the three modules / loop concept will result in associated reduction in the manufacturing schedule, which is very important from the overall economy of the plant. Further, the reduction in number of tube to tube-sheet joints on account of increased length of tubes enhances the overall availability of the plant and contribute to improved safety.

PUBLICATIONS

1. Tube size optimisation studies for CFBR Steam Generator : CFBR/33130/DN/1002/R-A, 2009 (Internal report)
2. Tube wall thickness calculation for CFBR SG: CFBR/33130/DN/1003/R-A, 2009 (Internal report)
3. Optimum number of modules for twin unit concept : CFBR/33130/DN/1005/R-A, 2009 (Internal report)
4. Process design and part load operation studies for CFBR SG : CFBR/33130/DN/1004/R-A, 2009 (Internal report)

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Real Time Computer (RTC) systems are extensively used in Instrumentation and Control of Safety Class 1 (SC1), Safety Class 2 (SC2) and Non-Nuclear Safety (NNS) systems of PFBR. Software used in these systems should have highest level of quality. Quality of the software product can be assured by applying appropriate and systematic techniques throughout the software life cycle. Hence software development methodology for real time computer systems was standardized at Real Time Systems Division, EIG.

In PFBR, RTC systems located at various local control centers, do the scanning, processing, generation of physical outputs and send the data to process computer of Distributed Digital Control System (DDCS) for storage and display purposes. Each RTC system consists of VME bus based 68020 CPU card, analog I/O cards and digital I/O cards. For RTC systems software development, waterfall model was selected, because of its simplicity, ease of Verification & Validation and tracking & control on deliverables across lifecycle. Waterfall model represents the sequence of activities needed to develop a new piece of software product as a linear series of steps beginning with the definition of user requirements. At each stage an intermediate deliverable is produced that feeds into the next stage. Typical representation of the waterfall model is shown in Figure 1. The output from each stage is subjected to various verification and testing activities to ensure that it conforms to the requirements defined in the previous stage. Different phases of software development process are listed below. AERB safety guide SG/D-25 was followed in all phases of software development. Computer Aided Software Engineering (CASE) tools were also extensively used.

System Architectural Design (SAD): SAD documents were prepared as per IEEE standard 1471. These documents were prepared only for the system whose architecture is complex and stored in Telelogic DOORS CASE tool.

Software Design Description (SDD): SDD documents were prepared as per IEEE standard 1016. These documents follow the SRS. Context diagram, data flow diagram (Figure 2), state transition diagram, state event matrix, structure chart and flow chart were prepared using Software through Pictures/Structured Environment (Stp/SE) tool. SDD documents were stored in Telelogic DOORS CASE tool. Traceability between SRS and SDD was established.

Software Testing: Latent defects present in the software are exposed during software testing. LDRA tool was used to generate software metrics and software quality aspects like maintainability, testability and analyzability.

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graph TD
    SR[System Requirements] --> SAD[System Architectural Design]
    SAD --> SWR[Software Requirements]
    SWR --> SD[Software Design]
    SD --> IUT[Implementation & unit testing]
    IUT --> IST[Integration & System Testing]
    IST --> OM[Operation & Maintenance]
    OM --> SR
    OM --> SAD
    OM --> SWR
    OM --> SD
    OM --> IUT
    OM --> IST
  
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ADDITIONAL INFORMATION

What is Real Time Computer System?

A real time computer system is a computer system in which the correctness of the system behavior not only depends on the logical results of the computation, but also on the time at which these results are produced. The real time computer system is a special-purpose computer system designed to perform a dedicated function. Unlike a general-purpose computer, such as a personal computer, these systems perform one or a few pre-defined tasks, usually with very specific requirements and often include task-specific hardware.

In PFBR, Real Time Computer systems, Computer Based Systems (CBS) and Embedded Systems are all used interchangeably to refer the same.

GENERAL EXPLANATION RELATED TO DESCRIPTION

Software metrics are used to identify critical problem areas. Metrics are static measurements (i.e. obtained without executing the program). They provide indications related to the analyzed program complexity, readability, maintainability and its reliability. Some of the metrics and their importance with respect to the quality factors of the software are described below :

Cyclomatic Complexity Metric ($v(G)$): (McCabe number)

Cyclomatic Complexity ($v(G)$) is a measure of the complexity of a module's decision structure. It is the number of linearly independent paths and therefore, the minimum number of paths that should be tested. A popular use of the McCabe metric is for testing.

Fan-in and Fan-out:

The Fan-in and Fan-out metrics are used in estimating the complexity of maintaining the software. Fan-out indicates the number of functions, a function calls.

Halstead metrics:

Halstead metrics is based on the fact that the complexity of a program is related to the number of operators and operands in the program. Operators are syntactic elements such as +, -, <, > and operands are quantities that receive the operators namely variables and constants. Halstead metrics calculates the Program Volume, Difficulty and Programming Effort.

Kiviat Diagram:

Kiviat diagram is used to highlight quality metrics which is shown in Figure 3. Each metrics displayed on an axis with all axis having a common virtual interpretation. The achieved value of the metric is plotted on its axis together with user definable upper and lower bounds. If a metric exceeds upper or lower bound, the same is displayed using shaded red colour. Based on this diagram, necessary changes were carried out on software design / coding so as to bring the value of the metrics within the boundaries.

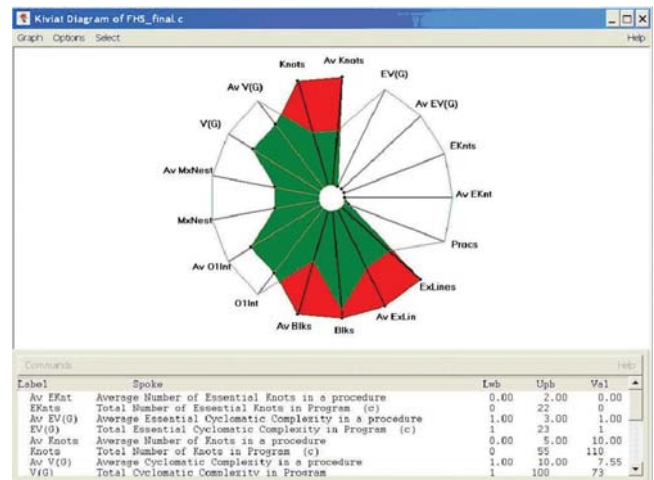


Fig. 3 : Kiviat Graph generated from LDRA tool

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Software engineering is the practice of using selected process techniques to improve the quality of a software development effort. This is based on the assumption that a methodical approach to software development results in fewer defects and, therefore, ultimately provides shorter delivery times and better value. The documented collection of policies, processes and procedures used by a development team or organization to practice software engineering is called its software development methodology or system development life cycle.

ACHIEVEMENTS

Software quality was improved by following software development methodology, which was verified by generating reports like software design review, code review and quality review using LDRA tool. Feedback from internal V&V committee also enabled us to generate quality software, which will be deployed at PFBR.

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Gas Flow Measurements through Concrete

EXECUTIVE SUMMARY

Numerous studies have been carried out in the field of fluid transport through concrete, widely used in civil engineering discipline. These studies throw light on the physical, chemical and mechanical properties of the concrete. When pressure acts as an influencing parameter in the transport process, Darcy's law allows the prediction of fluid flow rate across a porous medium under differential pressure ΔP . The measured volumetric flow rate across any porous media can be used to calculate the permeability, which is the ability of a medium to allow liquid or gas flow. Permeability changes considerably with aging and degradation of the medium. It also depends on the presence and nature of cracks. Hence permeability is an indirect but the most important measure of worthiness of concrete vis-à-vis its properties. An experimental setup was designed and installed at Radiological Safety Division (RSD) and permeability of reactor grade concrete was studied from the standpoint of transport of radioactive gaseous species.

OUTLINE

Permeability of a porous medium controls the fluid flow across the medium. The linear flow, through the porous medium, of an incompressible fluid with viscosity η , can be calculated using Darcy's law.

$$Q = \frac{KA\Delta P}{\eta L} \quad (1)$$

Gas can be treated as a compressible fluid with low viscosity. As the gas is compressible, at these low pressures the gas flow through porous medium differs from that of liquid flow. i.e. instead of following a linear relation with difference in pressure, the flow rate follows a difference in square of input and output pressure. Eqn.1 can be represented for this case as given below.

$$Q(m^3s^{-1}) = \frac{K A (P_i^2 - P_o^2)}{2 P L \eta} \quad (2)$$

where K is permeability of the concrete (m^2), Q = flow rate (m^3s^{-1}), P_o = outlet pressure (Pascal), P_i = input pressure (Pascal), L = length of medium (m), A = cross section area (m^2) and η = Dynamic viscosity of air at room temperature (Pa.s).

The experimental set-up consisted of two MS chambers of dimension 60 x 60 x 60 cm, viz., source and collection chamber connected with a concrete block of 20 cm length in between (Figure 1). Two ΔP pressure transmitters of 'Rosemount' make were used to measure pressure in the range 0-500 mbar. One of them was connected between source and collection chamber to measure the difference in pressure, while another is used to continuously monitor the pressure of the collection chamber. Additionally, a pressure gauge was fitted to the source chamber, with a higher range of 0 – 2 kg/cm² and least count of 0.25 kg/cm² to measure pressures above 500 mbar. A K-type thermocouple measures the temperature inside the collection chamber. The output of the two pressure transmitters and thermocouple were given to an 'HP Agilent' make data logger, for processing the logged data at the end of each experiment. During the experiment, the source chamber was pressurized to desired level using a pump and the change in pressure at the collection chamber was monitored continuously. Using the pressure-volume relation and time taken, flow rate is calculated (Figure 2). By applying the flow rate values in equation (2), the permeability value of the concrete can be calculated (Figure 3). The calculated permeability values were in the range of reported ones. For the given samples, the average permeability value for air was 8.89E-16 m² and 1.18E-15 m² for nitrogen.



Fig. 1 : Experimental set-up

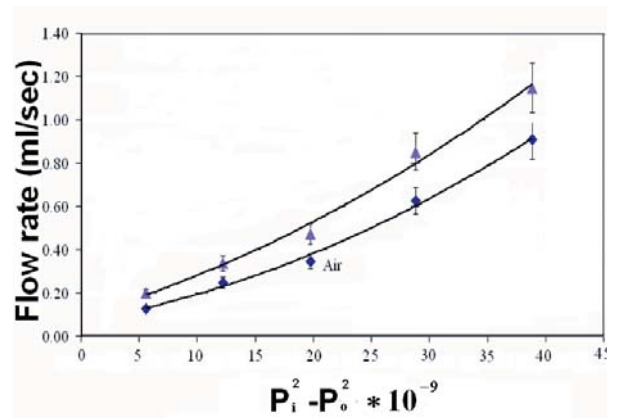


Fig. 2 : Variation in flow rate with respect to $P_i^2 - P_o^2$

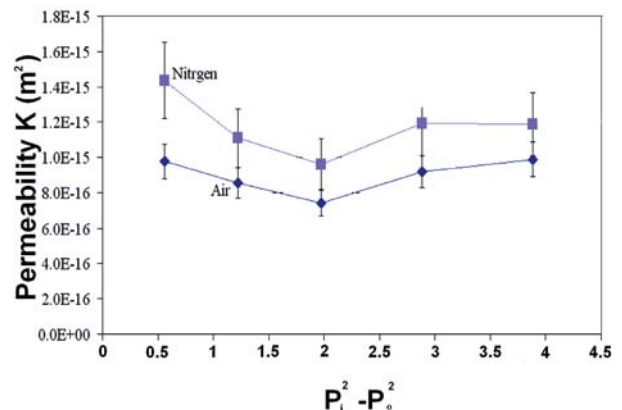


Fig. 3 : Variation in permeability with respect to $P_i^2 - P_o^2$

ADDITIONAL INFORMATION

During a Core Disruptive Accident (CDA) event, the core melts down and the fission product radioactivity gets released to the Reactor Containment Building (RCB). The released radioactivity exists in all physical forms (gaseous, volatile and particulate) and gets distributed inside the reactor containment building volume. Thus whole reactor containment building becomes a volume source and gives external exposure to the plant personnel and public. During this event both the supply and the exhaust for the reactor containment building is stopped for isolating the reactor containment building so as to prevent the release of radioactivity to the environment via stack. The raise in reactor containment building temperature due to core disruptive accident raises the reactor containment building pressure. Since reactor containment building is a concrete structure and concrete being a porous medium it allows the Fission Product Noble Gas to leak from the reactor containment building with increased pressure. The results obtained in the present experimental study can very well be extended to determine the inherent leak rate of reactor containment building during a design basis accident event. Using the estimated leak rate, ground release of fission product noble gas and the dose that would be received by the occupational workers as well as the public in such a scenario can be estimated.

ACHIEVEMENTS

Generally, to estimate the permeability, chloride ion test is being used, which takes minimum of 6 hours and upto a maximum of few days; present experimental setup provides another active method to determine the same. The main advantage of using this technique is that permeability can be calculated within a short duration (two hours) and experiment can be repeated many times without affecting the condition of concrete which is not possible in chloride ion test which requires a fresh sample for each test. Further reduction in experimental time can be achieved by the use of bubble flow meters.

PUBLICATIONS

A. Dhanasekaran , K.C.Ajoy ,B. Danalakshmi ,S. Balasundar and R. Santhanam, "Gas flow measurements through 200 millimeter concrete under different pressure gradient", Report no: IGC/SG/RSD/RSS/92604/EP/3002/REV - A 3002/A

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Grain Boundary Microstructural Modification for achieving very High Resistance to Sensitization in Austenitic Stainless Steel

EXECUTIVE SUMMARY

The main objective of this investigation is to enhance the sensitization resistance of austenitic stainless steel wrought products during its manufacturing stage by modifying the initial grain boundary nature by specific heat treatment without involving mechanical treatment so that a very high resistance to sensitization is achieved in the material. By performing a specified heat treatment in the temperature range of 1100-1250°C for durations of thirty minutes to two hours the Effective Grain Boundary Energy (EGBE) is altered to such an extent that chromium carbide precipitation and concomitant chromium depletion are delayed resulting in an increase in the sensitization resistance.

OUTLINE

Conventional austenitic stainless steels have a low resistance to sensitization. Fabrication process which involves welding or stress relieving heat treatments or long period of service exposure cause sensitization in these steels leading to intergranular corrosion/ intergranular stress corrosion cracking in corrosive environment. Control of sensitization by lowering of Carbon content or addition of carbon stabilizing elements (Titanium, Niobium) involves a higher cost on the material. The low carbon variety also has a lower mechanical strength. This has been overcome by introducing N in the low C varieties, but it again involves a higher cost. The solution annealing of sensitized components followed by fast cooling introduces harmful residual stresses which leads to intergranular stress corrosion cracking in corrosive environment during service. Modifying the initial microstructure of the austenitic stainless steel through a special heat treatment as invented by us, would develop a very high resistance to sensitization which would be of great benefit to stainless steel users. The present invention consists of giving a specific heat treatment to austenitic stainless steel wrought or fabricated product after its conventional mill-annealing treatment. The specific heat treatment consists of heating the mill-annealed product to a higher temperature in the range of 1100-1250°C for a duration of thirty minutes to two hours followed by fast cooling. The process results in modification of grain boundary nature wherein a very high resistance to sensitization is achieved. By adopting this process a low sensitization resistant material can be converted to a very high resistant material. AISI type 304 SS (C-0.044%), 316SS (C-0.054%) and 316LN SS (C-0.03%,N-0.086%) in the mill-annealed condition were subjected to the specific heat treatment. Both as-received as well as special heat treated specimens of all these grades were exposed to the so called “nose temperature” where time required for sensitization is minimum, for time periods $> t_{min}$. The degree of sensitization was measured by double loop EPR technique. For 304SS and 316SS, the sensitization resistance can be enhanced by about three times and for 316LN SS by about ten times. Therefore the specific heat treatment is applicable to the three different varieties of austenitic stainless steels which are commonly used in industries. The achievement of high resistance to sensitization by the specific heat treatment can be generalized for different varieties of austenitic stainless steels also.

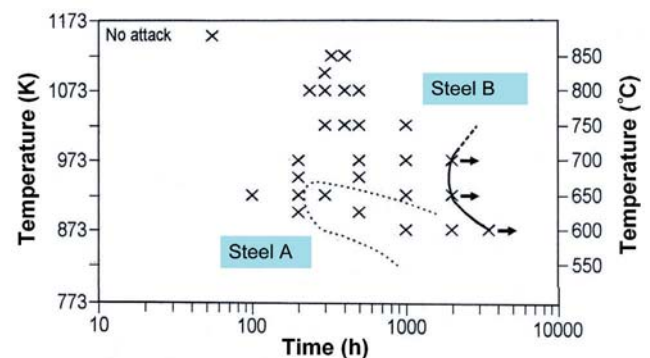


Fig. 1: Time-Temperature-Sensitization diagram for AISI 316LN SS (Grade A & Grade B)

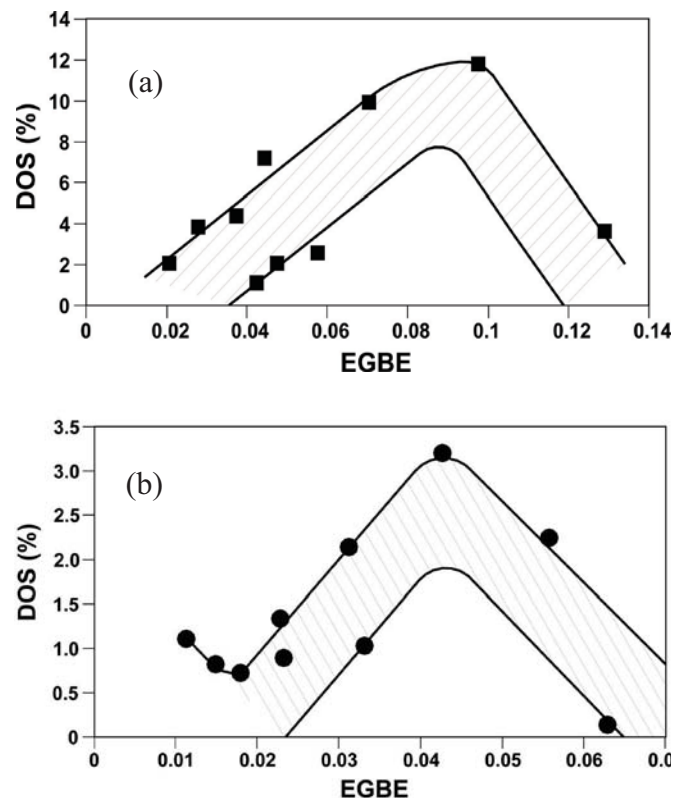


Fig. 2: Degree of Sensitization as a function of EGBE (Effective Grain Boundary Energy - as in equation (1)) in (a) Grade A and (b) Grade B

ADDITIONAL INFORMATION

Two heats of 316LN SS (Grade A and Grade B) having chemical composition within specification showed marked difference in sensitization kinetics as shown in Figure 1 ($t_{\min}$ was two hundred hours for steel A and two thousand hours for steel B). When the effective grain boundary energy was altered by various heat treatments, both the Grades showed low degree of sensitization at extremely low as well as high effective grain boundary energy values as shown in Figures 2a and 2b. However the special heat treatment resulted in very low Degree Of Sensitization in Steel A comparable to that of Steel B.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Electron Back Scattered Diffraction (EBSD) studies measurements were made using a Fei Quanta 200 HV scanning electron microscope with TSL-OIM system. For each specimen, at least an area of 3 X 3 millimeter (a minimum of five thousand grains) was scanned and several such measurements were made to arrive at comfortable statistics. Subsequent analysis of the electron back scattered diffraction data brought out different parameters of the grain boundary, viz., grain size, coincident site lattice fractions and the respective deviations from ideal Σ , coherent twin boundary fraction and random boundary connectivity. To club different types of coincident site lattice boundaries and the grain size, effective grain boundary energy was calculated as follows:

$$\text{EGBE} = \left[\left(\sum F_i \gamma_i \left(\frac{\Delta \theta_i}{\theta_{\text{Maximum}}} \right) \right) \left(\frac{4}{d} \right) \right] \text{-----(1)}$$

where F_i and, γ_i are respectively the fraction and energy of a coincident site lattice boundary of type i and d is the grain size. $\Delta \theta_i$ is the average deviation from perfect coincident site lattice and θ_{Maximum} is the maximum deviation allowed by Brandon's criteria. F_i and θ_i were estimated for different types of coincident site lattice boundaries directly from electron back scattered diffraction scans, while γ_i was approximated as,

$$\gamma_{\Sigma} = (1 - 1/\sqrt{\Sigma})$$

γ_{Random} where γ_{Σ} is the energy of a coincident site lattice boundary of notation Σ and γ_{Random} is the energy of a random boundary.

316LN SS (A and B) were subjected to different thermal treatments to alter the grain boundary nature significantly. Degree of sensitization was evaluated using DL EPR studies and grain boundary nature was evaluated by electron back scattered diffraction studies in terms of:

- Grainsize
- Coincident site lattice (CSL) fraction (fraction $\Sigma 1$ - $\Sigma 29$)
- Fraction of low angle boundaries (fraction $\Sigma 1$)
- Deviation from ideal $\Sigma 1$
- Fraction of $\Sigma 3$
- Deviation from ideal $\Sigma 3$
- Fraction of coherent twin boundaries
- Random boundary connectivity

The special heat treatment reported in this investigation resulted in favorable effective grain boundary energy leading to very low DOS and hence very high resistance to sensitization.

ACHIEVEMENT

This process does not involve any thermo-mechanical treatment on the wrought or fabricated products. The process consists of a specific heat treatment which can convert a sensitization - prone material to a high resistant material. During the manufacturing operation of stainless steel plates and tubes, the specific heat treatment consisting of heating the mill-annealed product to a higher temperature in the range of 1100-1250°C for a duration of thirty minutes to two hours followed by fast cooling can be adopted after its conventional mill – annealing treatment so that high resistance to sensitization is obtained in the material.

PUBLICATIONS

1. R.K. Dayal, N. Parvathavarthini, Baldev Raj, S. Mulki and I. Samajdar; Patent: Development of a Very High Resistance to Sensitization In Austenitic Stainless Steel Through Special Heat Treatment Resulting in Grain Boundary Microstructural Modification, Indian patent application no.: 893/MUM/2008 and European (Germany, France and Sweden) Patent Application no.: EP 08 159613.2 (2008).
2. N. Parvathavarthini, S. Mulki, R.K. Dayal, I. Samajdar, K.V.Mani and Baldev Raj; Sensitization control in AISI 316L(N) austenitic stainless steel: Defining the role of the nature of grain boundary, Corrosion Science 51 (2009) 2144.

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Cryogenic Gas Loading in Mao-Bell Type Diamond Anvil Cell

EXECUTIVE SUMMARY

Diamond anvil cells (DACs) are used for investigating materials under high pressure (~ few megabar) and high temperature (~ 5000° C) conditions. To provide a hydrostatic and inert atmosphere around the sample at such extreme conditions, inert gases like He, Ar and N are loaded in the high pressure chamber of the diamond anvil cell. For loading argon fluid at cryogenic temperatures in a Mao-Bell type diamond anvil cell, a simple system has been developed. It is done in a two-step process in which the piston-cylinder assembly alone is submerged in the cryogenic chamber for trapping the liquefied inert gas. Liquid nitrogen is used for condensing the argon gas. This system is now being efficiently used for loading liquid argon in the diamond anvil cell for high pressure – high temperature experiments. The success rate of trapping liquefied argon in the sample chamber is about 75 %. Several novel experiments, such as direct conversion of pyrolytic graphite to diamond, synthesis of RuC, BSB etc. under high pressure-high temperature conditions have been performed by using laser heated diamond anvil cell facility.

OUTLINE

Diamond anvil cells (DACs) are used for investigating materials under high pressure (~ few megabar) and high temperature (~ 5000° C). Inert gases like He, Ar and N are loaded in the high pressure chamber of the DAC to provide a hydrostatic and inert atmosphere around the sample at such extreme conditions. The cryogenic technique of loading gases in a DAC requires either the entire DAC or part of it to be submerged in the liquefied gas. However, it is not convenient for the Mao-Bell type DAC to be submerged in the liquefied gas due to their large size and thermal mass. In this article we present our novel design of a simple device to load liquid argon in the Mao-Bell type of DAC. In this design, only piston-cylinder assembly of the DAC is partly submerged in the liquefied argon (boiling temperature 87.3 K) which is cooled by liquid nitrogen (boiling temperature 77 K). The piston-cylinder assembly is locked in a two step process and then transferred in to the pressure cell holder for further pressurization.

Description and Operation:

The apparatus consists of a cylindrical chamber with a lid and a two-step locking device, all made of copper for good thermal conductivity and dimensional stability. The entire apparatus is kept inside a small thermocole box as shown in Figure 1. The copper vessel is provided with a lid, which has gas inlet, outlet, and a viewing port. Liquid nitrogen is poured inside the thermocole box and argon gas is let inside the copper vessel for liquefaction. Figures 2&3 shows the two step locking device for trapping and sealing of liquefied gas in the sample chamber of the piston-cylinder assembly. At the outset, the sample is loaded inside the SS gasket hole of dia 250 μ m and depth 60 μ m. Also, beside the sample a ruby chip of dimension of 5- 10 μ m is placed for pressure calibration. The piston is then locked to its bottom copper plate. The cylinder is then carefully inserted along with the top copper plate and the two distance adjustment screws. The distance adjustment screws are then tightened to maintain the optimum gap between the two anvils. The entire piston-cylinder assembly along with the copper plates is anchored at the bottom of the copper vessel by two dowel pins. The main vessel is then purged with argon gas in order to remove any residual air. Liquid nitrogen is poured into the outer thermocole box enclosing the copper vessel to liquefy the argon. Once the level is reached, the distance adjustment screws are loosened so that the two anvils come in contact with trapping liquid argon inside the sample chamber. The auxiliary locking screws are then tightened, thereby generating slight pressure on the sample assembly. The piston-cylinder assembly along with the locking plates is then taken out and defrosted. Two locking screws (with Belleville washers) are then tightened to retain the sample pressure. Once the piston-cylinder assembly is clamped together with the locking screws, the two auxiliary locking screws and the clamping plates are released and the piston-cylinder assembly is inserted in to the pressure cell holder for further increasing the pressure to carry out High pressure - High Temperature (HP-HT) experiments.

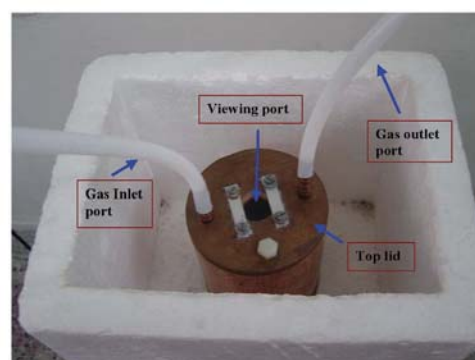


Fig.1: The copper vessel containing the piston cylinder and the locking device in a thermocole container

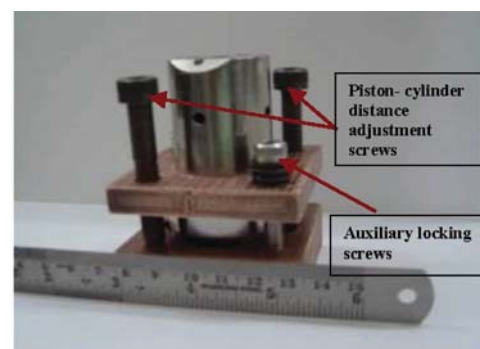


Fig. 2: Piston-cylinder assembly with the locking mechanism showing the first stage

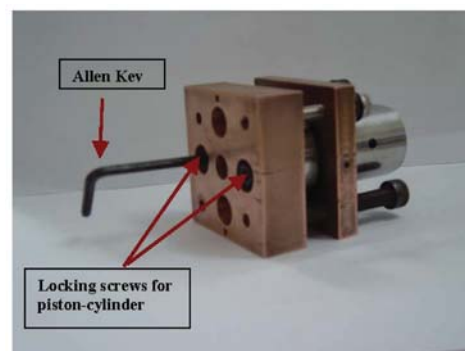


Fig. 3: Piston-cylinder assembly with the locking mechanism showing the second stage of two step locking

ADDITIONAL INFORMATION ABOUT DIAMOND ANVIL CELL (DAC)

Diamond Anvil Cells (DACs) are being extensively used for investigating materials under HP- HT conditions. Synthesis of novel and super hard materials under extreme pressure and temperature are also carried out using Laser Heated Diamond Anvil Cells (LHDAC). The compressive load on the diamond anvil cell is converted into hydrostatic pressure on the sample by using appropriate pressure transmitting media eg; methanol-ethanol mixture, silicone oil and rare gases depending on the nature of the sample under investigation. For LHDAC, the pressure transmitting media have to satisfy more stringent conditions. It has to be a thermal insulator, transparent to IR laser beam used for heating pressurized sample and also be chemically inert. Rare gases as pressure transmitting medium satisfy these conditions to provide purely hydrostatic environment until they freeze and are also stable over a wide range of pressure. There are two ways to load gases in a DAC, one is to use cryogenic technique wherein liquefied gas is loaded (Cryogenic Gas-loading Technique) and the other method is to use a gas compressor to densify the gases at room temperature (High Pressure Gas-loading Technique) before loading in the high pressure chamber. Although both methods have their own advantages and disadvantages, cryogenic technique is very simple and inexpensive.

The DAC consists of two identical gem quality diamonds shaped in the form of anvils. One of them is mounted on to a piston and the other on to a cylinder of the DAC and is aligned in such a way that when the piston-cylinder is assembled together, the two anvil face each other perfectly. A pre-indented metal gasket, with a 200 μm (dia) X 75 μm (depth) hole to act as sample chamber, is spaced between the two culets (the flat polished tip of the diamond is called the "culet"). Very high pressure is generated on the sample when the piston is moved against the cylinder using an appropriate arrangement.

GENERAL INFORMATION

The performance of the system has been checked by carrying out a few dummy runs with ruby chips loaded in the sample chamber. The success rate of trapping the argon liquid in the sample chamber was ~75%. Figure 4 shows the X-ray diffraction pattern of argon trapped inside the sample chamber at ~ 17 GPa. Another HP-HT experiment for directly converting graphite into diamond in the inert argon pressure transmitting medium using the LHDAC facility was also carried out. In this experiment, we loaded a piece of pyrolytic graphite in the argon pressure transmitting medium and pressurized it to ~16.5 GPa. It was then laser heated to a temperature of ~2000 K by focusing the high power CO₂ laser beam onto the sample. The recovered sample was characterized by micro-Raman. Clear indication of the direct conversion of pyrolytic graphite into diamond and diamond like carbon could be seen. This test experiment clearly established the utility and successful performance of cryogenically loaded argon as pressure transmitting medium by this novel device. Subsequently, several novel compounds such as RuC and BSb were synthesized successfully under HP-HT conditions using Ar as the pressure transmitting medium.

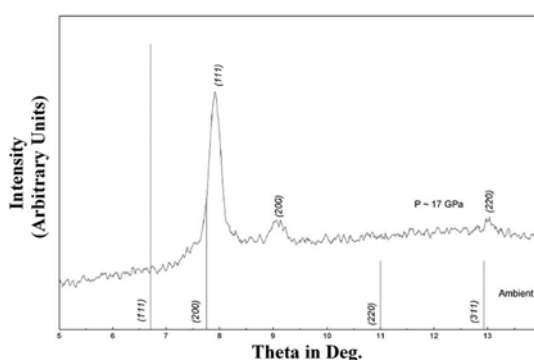


Fig. 4: X-ray diffraction pattern of solidified argon at 17 GPa in the DAC, the stick plot is at ambient

ACHIEVEMENT

A novel device has been designed to load argon cryogenically in a Mao-Bell type diamond anvil cell. This has helped in the successful synthesis of novel material like diamond, diamond like carbon, RuC and BSb under HP-HT conditions.

PUBLICATIONS

M. Sekar, N. R. Sanjay Kumar, P. Ch. Sahu, N. V. Chandra Shekar and N. Subramanian, Cryogenic gas loading in a Mao-Bell type diamond anvil cell for high pressure - high temperature investigations, Review of Scientific Instruments, 79 (2008) 076103.

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Ferroboron as an alternative In-Core Shield Material in Fast Breeder Reactors

EXECUTIVE SUMMARY

Shields around core and blankets form a major part of reactor assembly in fast reactors. The spectrum of neutrons leaking from the blankets is hard with negligible thermal component and has anisotropic angular distribution. Boron carbide and stainless steel have been the main choice shield materials for fast reactors. A study of the use of ferroboron as a new alternative shield material was made choosing the reference case as Prototype Fast Breeder Reactor (PFBR) which is a pool type FBR. It was demonstrated through 2-D transport calculations, that this relatively cheap material as shield is capable of satisfying the radiological safety criteria equally well and hence a prospective material for use in fast reactors. An experimental study conducted on ferroboron in KAMINI, has shown its good neutron attenuating properties.

OUTLINE

Fast reactor fuel and blankets are surrounded by large quantity of shields. They occupy nearly 60-70 % of the core volume. The leaking neutrons from core and blankets have energies peaking around 10-100 keV. The calculations span an energy range of 0.025 eV to 14 MeV and attenuation through more than forty mean free paths of shield thickness. As a consequence, PFBR has more than 1200 shield subassemblies (SA) even though the number of fuel subassembly is much lower at 181. The outer shield beyond fuel storage locations consist of six SS rings of shield SAs (609 numbers) and three rows of B_4C subassemblies (417 numbers). The shield design is dictated not only by radiological safety criteria but also limits on radiation damage to components and structures. Intermediate Heat Exchangers (IHXs) are immersed in the reactor vessel itself and serve the purpose of heat transfer to a secondary coolant loop. Sodium is the coolant in a large majority of fast reactors though Pb-Bi, Na-K and He have been considered. The advantage of Na is its excellent heat transfer properties, low density and high boiling point. However, primary Na in vessel and also secondary sodium passing through intermediate heat exchangers become radioactive. Without adequate shielding, this activation can cause unacceptably large dose in the steam generator Building. Thus fast reactors are characterized by large in-vessel shields which is also one of the parameters determining the reactor vessel size.

Stainless steel and B_4C have been the main choice shield materials for PFBR. This study is on the effectiveness of shields, if absorber material boron is spread throughout shields albeit with lower atom densities rather than concentrating in the last rows as in PFBR (Figure 1). Ferro-boron has been considered as such a potential material. It has about 15-18 wt % boron and is much cheaper than B_4C . Two dimensional transport calculations were carried out with DORT code with one hundred and seventy five group cross sections. The secondary sodium activity computed in the ferroboron case is lower than that of the reference case. The reasons are clear from the Figure 2. In the case of ferroboron shields, the sodium capture rates are about twice that of PFBR in intermediate heat exchanger region in the core mid plane. But this is only a small fraction of the peak sodium capture rate. In the position in intermediate heat exchanger region showing maximum value of flux, sodium capture rate in the ferro-boron case is less than that of the reference case. The reason is that top parts of shield subassemblies in between core subassemblies and the intermediate heat exchanger peak position have significant boron atom densities. The calculations also show that by replacing the shield assemblies with ferroboron, the reduction in neutron counts at detector location are only marginal.

Ferro-boron shields thus satisfy the radiological safety criteria and neutron detection requirements. Its effectiveness is due to the fact that Fe and B are spread throughout shields.

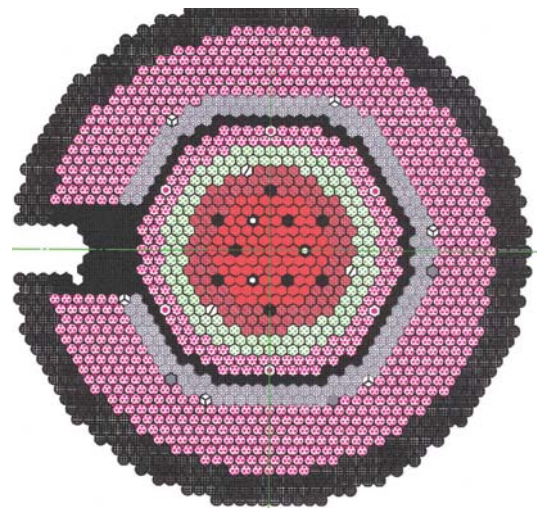


Fig. 1: PFBR Core and Shield Plan. There are 609 SS shield (6 rows) and 417 B_4C shield (3 rows) assemblies

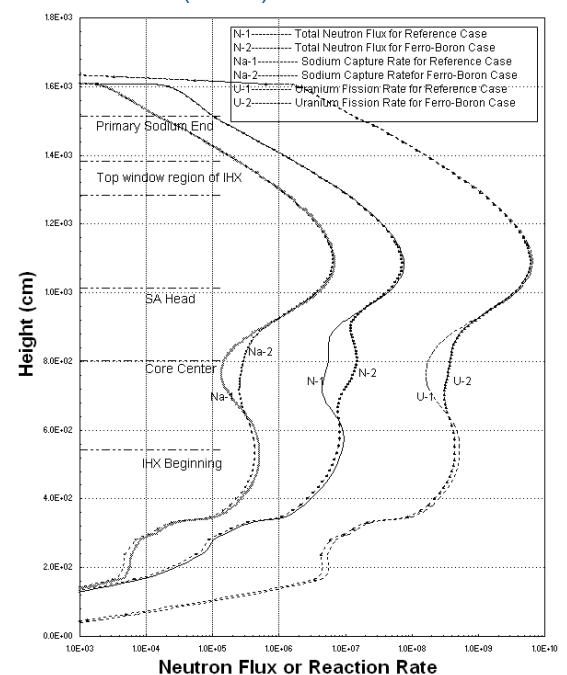


Fig. 2: Total neutron flux ($n/cm^2/s$) and reaction rates ($10^{-24} Bq/atom$) of $^{23}Na(n,\gamma)^{24}Na$ and $^{235}U(n,f)$ along IHX for PFBR (reference case) and ferroboron case

ADDITIONAL INFORMATION ABOUT EXPERIMENTS BEING PERFORMED ON FERROBORON

Potential of ferroboron as shield material was tested in neutron attenuation experiments in KAMINI reactor. The measured attenuation behaviour is shown in Figures 3 and 4. Metallographic characterization of ferroboron powder has shown that boron is in the form of iron borides. Preliminary hot-sodium experiment has shown that ferro boron does not react with liquid sodium upto 650° C. High temperature clad-ferroboron interaction tests with varying time durations and estimation of clad penetration depth at 700 °C are in progress.

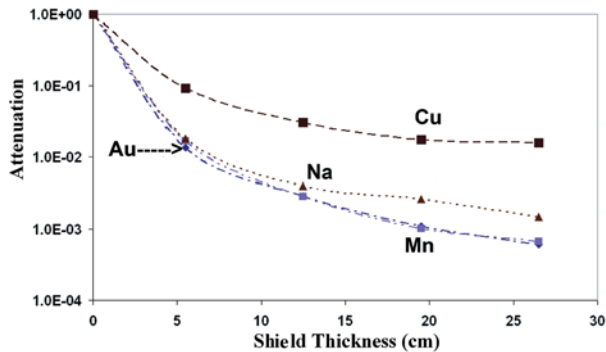


Fig. 3: Thermal and epithermal neutron flux attenuation represented by capture rates of Au, Mn, Na and Cu in ferroboron granules in KAMINI reactor

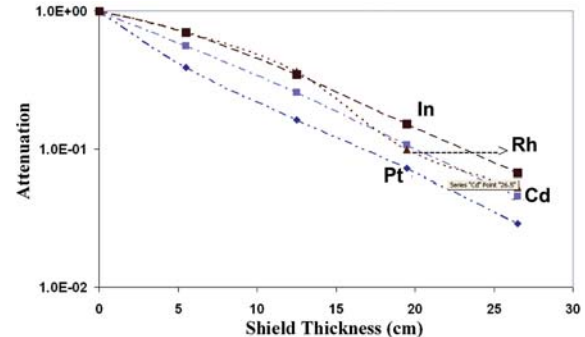


Fig. 4: Fast neutron flux attenuation represented by measured inelastic reaction rates of Pt, Cd, Rh and In in ferroboron granules in KAMINI reactor

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Typical composition of commercially available ferroboron is 15-18 wt % boron, 0.3 % carbon and rest is iron. Chemically, much of boron is present as borides of iron. The material is used as a master alloy in steel industry as an additive for boron. Density is around 6.7g/ cm³. The material is available in the form of lumps, granules and powder. Typically, the bulk density of ferroboron is around 4 g/ cm³. This density was used in these calculations. In PFBR shield assemblies, boron carbide is used in powder form and packed 304L tubes of ID 110.0 millimeter and OD 114.3 millimeter. Double containment is provided by an outer 304L tube of ID 120.0 millimeter and OD 130.0 millimeter. In the calculations, the same configuration with boron carbide powder replaced with ferroboron, was used. The inner SS shield assemblies are also replaced by these ferroboron assemblies. The use of ferro-boron brings down the cost of shield substantially.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The work is based on neutron 2- dimensional transport theoretical calculations in R-Z geometry. The discrete ordinates approximation used is S_8 and order of anisotropy used for cross sections is P_3 . For bulk shielding calculations this approximation is known to work well. The IGC-S3 cross sections in one hundred and seventy five groups are basically derived from ENDF/B-VI. The radial geometry extends up to reactor vault. Axial geometry extends up to top shield in the top direction and under vessel detector block in the bottom direction. The radial (571) and axial (718) meshes are chosen to achieve point flux convergence of 10^{-4} .

ACHIEVEMENT

Boron carbide and stainless steel have so far been the main choice materials for shields in fast reactors. For the first time it is shown that ferroboron, as in-core shield material in pool type fast breeder reactors is capable of satisfying the radiological safety criteria equally well. Use of this material results in substantial saving in shield material cost. In the FBRs planned to be built in Kalpakkam, the extent of saving can be up to 20 %.

PUBLICATIONS

1. D. Sunil Kumar, R. S. Keshavamurthy, P. Mohanakrishnan and S. C. Chetal , “A feasibility study of ferro-boron as In-Core Shield Material in Fast Breeder Reactors”, Nuclear Engineering and Design, 240 (2010) 2972.
2. D.Venkata Subramanian, Rajeev Ranjan Prasad, A.Haridas, and RS.Keshavamurthy, “Measurements of Neutron attenuation in Ferro- boron granules”, KAMINI/O1115/KS/6/R-0 (2009).

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Calibration of Eddy Current Flow Meter for Core Flow Measurement

EXECUTIVE SUMMARY

Eddy current flowmeter (ECFM) is used for measuring core flow (Q). This signal is used in combination with power (P) to trip reactor as P/Q . Eddy current flowmeter signal protects reactor against primary pipe rupture, transient over power, primary pump trip/seizure and class-IV power failure events. Hence, knowledge of uncertainty in eddy current flowmeter reading is essential for safety assessment. Towards this, two possible routes for calibration of eddy current flowmeter viz., (i) direct calibration by water and sodium tests in conjunction with pump characteristics and (ii) thermal balance method from steam-water system have been identified. It is seen that the uncertainty in the former is 4 % while that in the latter is 2 %. Acceptability of these uncertainty values has been demonstrated by plant dynamic studies. Suitability of eddy current flowmeter tapping point has been demonstrated by 3-D CFD studies.

OUTLINE

Sodium flow through core is monitored at the primary sodium pump using ECFM. The requirements of ECFM are in getting an indication of flow reduction through the core in case of primary pipe rupture event and in measuring the core flow. ECFM is located in a bypass line from pump discharge to suction, as depicted in Figure 1. This location is chosen from the point of view of easy assembly without calling for any additional structure for it. Detailed CFD analysis has been carried out to estimate the pressure head developed at the ECFM tapping location. The CFD model comprises of 180° sector of the cold pool, IHX-outlet, PSP suction bell, inlet guide vanes, rotating impeller, diffuser, discharge pipe spherical header with internal baffles and primary pipes. The pressure distribution in the pump assembly predicted by the CFD investigations is depicted in Figure 2, for full flow conditions of the pump. It is clear that the pressure at the tapping location is ~ 5.5 bars, which is very close to the total head developed by the pump (6.2 bars). Similar studies were carried out for various speeds of the pump and it was found that the pressure at the tapping location bears a constant ratio to the total head developed by the pump and this ratio is very close to unity demonstrating that the present location for ECFM tapping is acceptable. Subsequently, experimental hydraulic studies were carried out on 1:1 model to establish the head drop across ECFM and flow through ECFM. Studies were carried out in sodium to establish the voltage output of the ECFM for various flow rates and temperatures. Using the hydraulic studies and sodium studies, relation between the head drop across ECFM and ECFM signal was established.

Head drop across ECFM is the partial head developed by the pump. The actual pump head is higher than the head drop across the ECFM. It is planned to confirm this value during prototype pump performance testing in water, at various speeds. After installation of the pump in the reactor, the pump will be run at a given speed and the ECFM signal is measured. For this ECFM signal, the head across the ECFM (partial head) is determined. From the partial head the pump flow is estimated based on pump characteristics. Through these direct tests, the estimated accuracy is $\pm 4.46\%$.

Subsequently during plant operation, the flowmeter will be calibrated against the reactor thermal balance at steam water circuit. Thermal power across steam generator is computed. By adding heat losses in secondary and primary circuits, power generated in the reactor is computed. From this thermal power flow through the core is estimated. Through thermal balance, the flowmeter can be calibrated to an accuracy of $\pm 2\%$. Detailed plant dynamic studies have been carried out for various enveloping design basis events and the acceptable inaccuracy is found to be 5 %. This is higher the maximum possible uncertainty demonstrating the acceptability of calibration procedure.

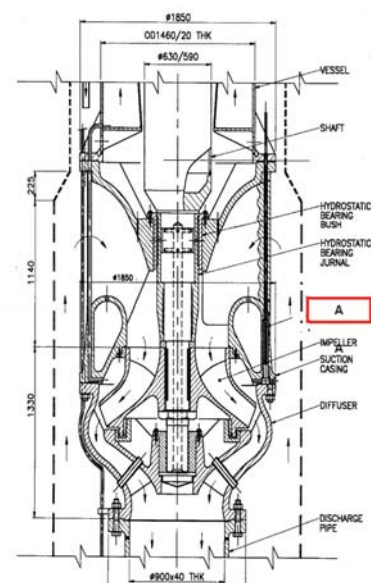


Fig.1: Location of ECFM in primary sodium pump indicated as A

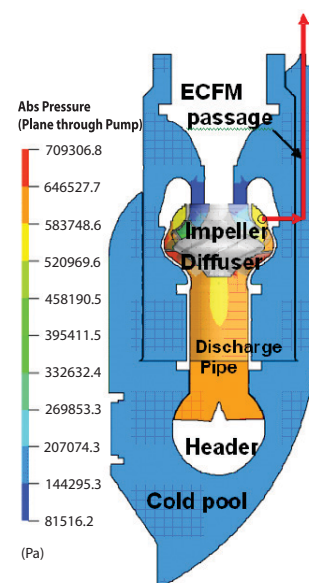


Fig.2: Pressure distribution across PSP and Pressureat ECFM tapping is close total head developed by PSP

ADDITIONAL INFORMATION ABOUT EDDY CURRENT FLOW METER

Eddy current flowmeter consists of one primary coil and two secondary coils. Primary coil is at the middle. Secondary coils are wound symmetrically on either side of the primary coil on the same core. Primary coil is excited with a constant current (200 mA) source at a constant frequency (~400 Hz). Due to magnetic flux generated by the primary coil, secondary coils will have induced voltages, due to transformer action. When this flow meter is inserted in flowing sodium, magnetic field interacts with sodium and produces eddy current which influences signal output. When the flow is established across the flow meter, voltage induced in the upstream secondary coil is less than downstream secondary coil. The difference between two secondary coil voltages is proportional to the sodium velocity.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Eddy current flowmeter probe assembly consists of flow sensor and guide tube assembly. Each probe consists of two sensors. The guide tube assembly consists of inner guide tube and outer guide tube. Inner guide tube passes through the outer guide tube provided in the pump flange and enters the hole in the pump suction casing. Inner guide tube is supported at the bottom on a support tube. This inner guide tube is surrounded by Fe-Ni tube that is part of the pump. High pressure sodium enters the Fe-Ni tube through an opening of 30 millimeter dia, and flows through the annular space between inner guide tube and Fe-Ni tube and exits through the holes provided at the top of the suction web to the pump suction. Eddy current flowmeter probe is inserted in the inner guide tube. This probe is replaceable. In each primary sodium pump two such assemblies are provided. Hydraulic studies were carried out on 1:1 model to establish the head drop and flow across eddy current flowmeter and different temperature. Studies were carried out in sodium to establish the voltage output of the eddy current flowmeter for various flow rates.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

3-Dimensional CFD studies to determine pressure development in primary sodium pump has been carried out using the CFX code, wherein an integrated model of the cold pool along with the components of the pump are modeled in detail. The finite volume mesh comprised of over 30 lakhs control volumes for accurate prediction of the pressure and velocity features. The CFD part of the study has been carried out as a collaborative research with Anna-University, Chennai. Design basis events have been analysed using the in-house developed plant dynamics code, DYANA-P.

ACHIEVEMENT

This work is of high relevance to the project, wherein (i) the practical routes for calibration of eddy current flow meter which monitors core flow, (ii) uncertainty in each route, (iii) acceptability of the uncertainty from plant safety point of view and (iv) acceptability of tapping location for the flow meter in pump, have been established by detailed 3-D CFD studies and transient plant dynamics investigations, to satisfaction of safety committees.

PUBLICATIONS

1. "Eddy Current Flow Meter for Core Flow Measurement", PFBR/63210/DN/1007/R-A.
2. "Primary Sodium Pump Flow Estimation from Eddy Current Flow Meter (ECFM) output", FRTG/99140/DN/3011.

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X-ray Radiography Analysis of Irradiated Boron Carbide Control Rods of Fast Breeder Test Reactor

EXECUTIVE SUMMARY

X-ray radiography examination of the irradiated boron carbide control rods of Fast Breeder Test Reactor was carried out to evaluate the condition of the boron carbide pellets encapsulated in a stainless steel cylinder. The primary objective is to measure the diameter of the boron carbide pellets and the outer diameter of the control rod from the radiography images. Among nine pellets stacked, pellet to pellet gap and pellet chipping indications on the first and second pellets are clearly seen. Radiography images show pellets three, four, five and nine are cracked radially at the center. Pellets six to nine show multiple branched cracks. The bottom pellets are cracked in the axial direction. For pellets one-six from the spring region, the measured value of diameter was about 40 millimeter as against the nominal value of 38 ± 0.1 millimeter (about 5% radial swelling). For the pellets seven-nine, the radial swelling was about 12%.

OUTLINE

Six Boron Carbide control rods are in use inside the FBTR for start up, power control and shut down of the reactor. The control rod drive mechanisms are constantly monitored and on-line measurements of frictional force and rod drop time are being monitored. Two control rods are sufficient to shutdown the reactor.

A dose level of 8 Gy/hr was recorded at one meter distance in the as received condition. To minimize gamma fogging, during X-radiography inspection, the bottom end of the control rod was trimmed by diamond wheel cutting. The radiation dose level is reduced considerably and the measured value was 1R/hr at 1 meter distance. A temporary radiographic inspection facility was set-up behind hot-cell No.4 to facilitate radiographic inspection of irradiated control rod outside the hot cell, in the isolation area. The irradiated boron carbide control rod is inserted from the hot cell into this aluminum tube which is shielded with 250 mm thick lead bricks on all sides. A 110mm wide aperture is provided in the lead brick below the aluminum tube for film positioning. An X-ray film carriage drive is accommodated into this aperture for high speed positioning and retrieval of X-ray films. Figure 1 shows the photograph of the X-ray machine positioned above the shielding blocks behind the hot-cell 4.

Figures 2 (a) and (b) show radiography images of the pellet region of the control rod. The pellets 1-9 from the spring region are numbered. From Figure 2 (a), it is observed that the pellets are cracked at regular intervals (almost at the centre of each pellet). The cracked location in the pellets is indicated by arrows. It is observed that in three of the pellets (3, 4 and 5) in Figure 2a are cracked radially at the centre of each pellets. However, at the bottom region pellets, the cracks are formed in the axial direction. Branched cracks are also observed in various pellets. The damages to the pellets of the control rod would have originated from the structural changes in B_4C pellets (such as lattice distortion, formation of helium bubbles, swelling and micro-cracking) due to recoiled atoms and the incident neutron flux. The radial cracks in the pellets could be due to a combined effect of mechanical and radial thermal stresses.



Fig. 1 : Photograph of the X-ray machine and shielding block positioned in the lower isolation area for radiography of the irradiated control rod

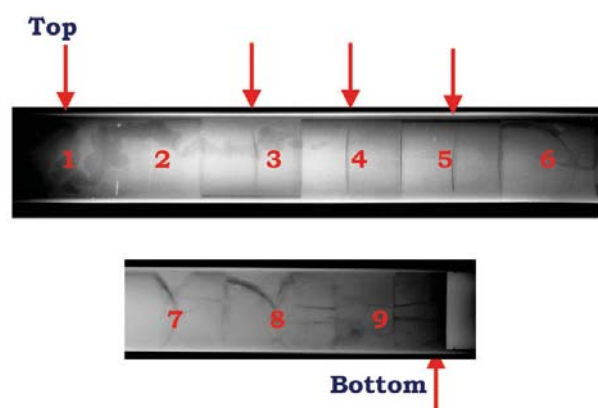


Fig.2: Digitized radiography image of the pellet region of boroncarbide control rod. Arrows indicate radial cracks

ADDITIONAL INFORMATION

X-ray films were digitized using Kodak Lumiscan LS85LF X-ray film scanner with fourteen bit gray level resolution. The diameter of the control rod is measured from these digitized radiography images using image processing tools. For this, a known physical dimension is chosen as reference for calibration of pixel size. From the above analysis, the diameter of the control rod was estimated as 45.17 ± 0.1 millimeter as against the nominal value of 45.1 millimeter. The diameter measurements were made at selective locations along the length of the rod. The diameter of the control rod was estimated as 44.93 ± 0.010 millimeter using dial gauge matches well the radiography data.

GENERAL EXPLANATION RELATED TO DESCRIPTION

In the present campaign, X-ray film radiography was carried out to ascertain the irradiation damage on the boron carbide pellets. X-ray radiography technique clearly shows the cracks, diametral swelling in the pellet, pellet to clad gap and control rod damages due to high neutron flux. Radiography was carried out with single wall single Image Technique technique on two orientations namely 0 and 90° apart. Based on the radiographic trials, it was decided to carry out radiography at two different voltages. We have chosen 160kV and medium grain Agfa D4 film for boron carbide pellet region (top region) and 250 kV for structural materials. To ascertain the level of gamma fogging from the foot region of the control rod, auto-radiography was carried out. During trials, it was found that beyond ten seconds of X-ray exposure in the foot region, the optical density on the film was measured to be above 4.0. To have a better control of gamma fogging and higher radiography sensitivity, fine grain and slow speed Kodak DR50 film was used for the foot region of the control rod.

ACHIEVEMENTS

Radiography investigations have been successfully carried out on irradiated boron carbide control rods of FBTR. By adopting suitable methodologies to eliminate gamma fogging, we have been able to obtain high quality radiography images of the pellets, spring, and bottom region of the control rod.

PUBLICATIONS

Report No. IGCAR/MMG/NDED/SMARTS/02/ 2009 (2009)

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Development of Polyetheretherketone (PEEK) Coating on the Rotors in the Motors of Centrifugal Extractors

EXECUTIVE SUMMARY

For the corrosion protection of rotors of motors in centrifugal extractors (CE) from nitric acid vapour, polyetheretherketone (PEEK) coating was chosen. It is a high performance engineering thermoplastic which has a radiation resistance of 10⁹ rads and it is resistant to a wide range of chemicals at elevated temperatures. The coated rotor, after curing was put in the motor and the extractor was run with nitric acid, which simulates the operating conditions of the reprocessing plant. After the run the rotor was removed from the centrifugal extractors and the performance of coating was evaluated. Motor load tests were conducted to assess the performance degradation due to increased gap between rotor and stator. The performance of the coated rotors when subjected to functional testing under simulated conditions was found to be satisfactory.

OUTLINE

Centrifugal extractors (CE) are extensively used in the reprocessing of the spent fuels of Fast Breeder Reactors. They are driven by electrical motors. The rotors and stators of the motors are made from stampings of silicon steel. These rotors and stators are continuously exposed to radiation and vapour of nitric acid in the plant, leading to corrosion. For the purpose of preventive maintenance of motors, corrosion protection coatings are essential on the rotors and polyetheretherketone, (PEEK) coating was identified, as it is a high performance engineering thermoplastic which has a radiation resistance of 10⁹ rads and it is resistant to a wide range of chemicals at elevated temperatures in harsh environments.

Liquid dispersion ferrous grade (PEEK) was coated on the rotors and the performance of (PEEK) as a protective film to resist corrosion had been evaluated by measuring the thickness and hardness of the coating as well as subjecting the rotors to real time testing.

Initially coating of PEEK was developed on one rotor. dispersion a water was coated on the rotor by spraying. It was then dried immediately at 110°C for 30 minutes and cured at 400°C for ten to fifteen minutes. The coating was characterized for thickness, hardness and electrical resistivity. The coating thickness was measured to be 80 to 90 microns. The hardness value determined was 80 to 84 Shore D. The electrical resistivity, surface resistivity was measured to be 10¹¹ to 10¹² ohms/sq. The performance of the coating was evaluated by running the rotor in the extractor with operating conditions.

The coated rotor was machined and 0.5 micron of material was removed to accommodate the coating, without changing the dimensions of the rotor. To test the performance of the coating in the plant, the coated rotor was put in the motor and the extractor was run with nitric acid which simulates the operating conditions of the plant. After the run the rotor was removed from the centrifugal extractors and the condition of coating was examined. There was no change in the thickness of PEEK coating after carrying out a run, in the motor. Hardness and surface resistivity values did not alter after exposure to acid vapour. As there was no peeling or swelling of the coated layer, it is confirmed that the PEEK coating on the surface of the rotor has enabled to give adequate protection to the motor of centrifugal extractors against corrosion.

Figure 1 shows the picture of rotor before coating, 1(b) rotor with coating before testing and 1(c) rotor with coating after testing. Motor load tests were conducted to assess the performance degradation due to increased gap between rotor and stator. Performance of the motor also depends on the air gap between rotor and stator. Figure 2 depicts the torque with current characteristics of the motor. From the motor load test it was found that the current drawn at the operating conditions has not increased significantly (Figure 2).

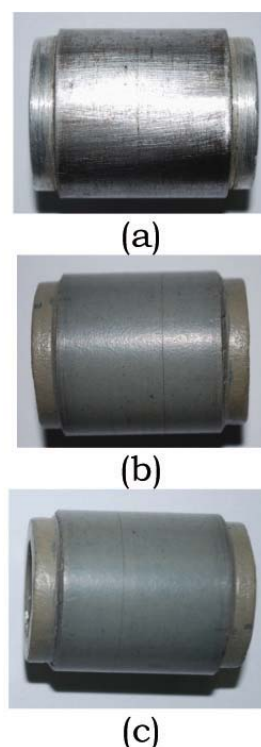


Fig. 1: Pictures of rotors (a) without coating, (b) with coating before testing and (c) with coating after testing

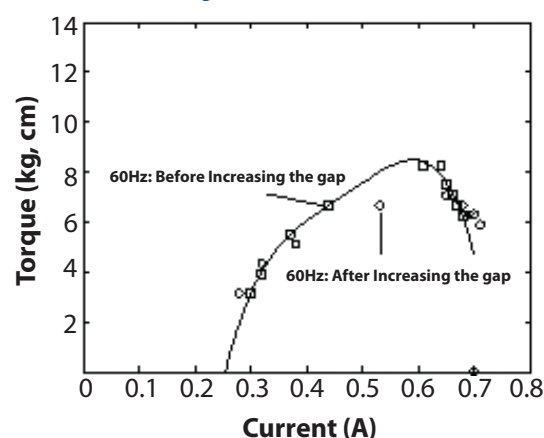


Fig. 2 : Torque-current characteristics of motor

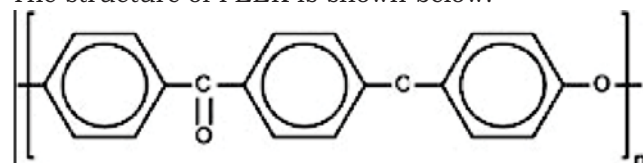
ADDITIONAL INFORMATION ABOUT POLYETHERETHERKETONE

Polyetheretherketone (PEEK) is a crystalline thermoplastic which makes it a chemically resistant product. (PEEK) can function in a wide pH range, from 60% sulfuric acid to 40% sodium hydroxide, and at high temperatures. Additional chemical resistance includes the tolerance to organic solvents, aqueous reagents, and long term performance in superheated water at 260°C. The heat distortion temperature is 315°C, with continuous service possible at 240°C. Good mechanical properties, very low smoke emission and self extinguishing characteristics bring PEEK into numerous demanding applications, for example as automotive engine parts, aerospace components, compressor valve parts and bearings.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The linear aromatic polyether ketone family consists of several variations on the theme of repeated monomers of ether and ketone. The first one produced in the laboratory in 1977 was the polymer polyether ether ketone B (now simply known as PEEK) and is claimed to be one of the first designer polymers.

The structure of PEEK is shown below:



From the above structure it is seen that the backbone consists of aromatic rings. This is responsible for high performance properties with respect to mechanical, thermal, chemical, etc., Hence, PEEK is termed as high performance engineering thermoplastic. Its radiation resistance was measured and it was found that up to 10^9 rads of radiation there is no significant change in mechanical properties. It is evident from Figure 3 that the elongation at break falls to 50% of its original value at 10^9 rads. So PEEK can be used up to 10^9 rads without any degradation in its properties.

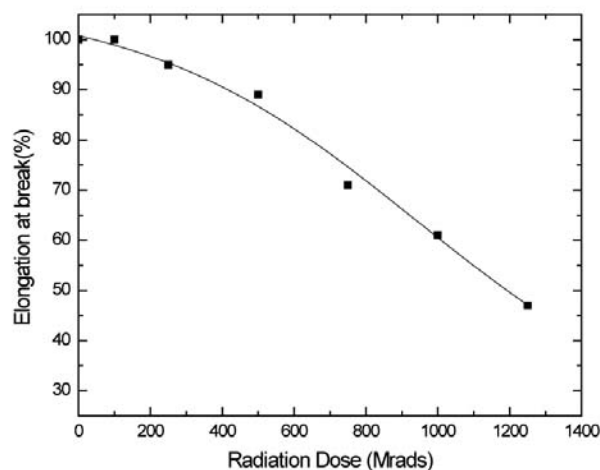


Fig. 3 :Elongation at break as a function of radiation dose

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

PEEK consists of aromatic rings in the backbone. This aromaticity is responsible for the chemical and radiation resistance of the polymer. When a polymer is irradiated lot of highly energetic free radicals are generated. In the presence of oxygen these radicals are responsible for chain scission in the polymer leading to degradation in mechanical properties. In PEEK these radicals are stabilized by aromatic ring and hence, PEEK is more stable to radiation.

ACHIEVEMENTS

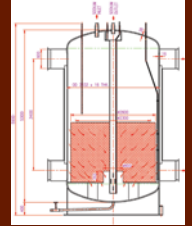
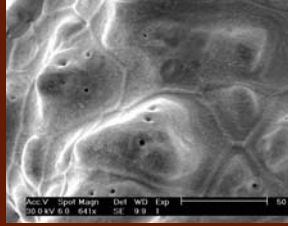
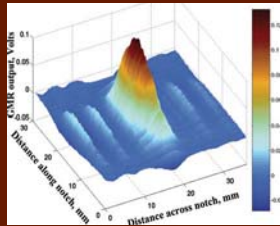
Polyetheretherketone, is a linear aromatic polymer which is inert to a wide range of chemicals and acids in addition to its tolerance to energetic radiation. It has optimum mechanical properties and was identified as the material for providing a protective coating to the motor components of centrifugal extractors, which are prone to corrosion due to the attack by nitric acid vapour.

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TECHNOLOGY

Reprocessing of High Burn-up Mixed Carbide Fuel: Closure of Fast Reactor Fuel Cycle

EXECUTIVE SUMMARY

Fuel with as high a burn-up as 155 GWd/t was taken up for reprocessing the first time anywhere in the world and two campaigns have been completed successfully in the CORAL facility with good recovery and purity of the recovered materials. The satisfactory operation of various equipment and systems enabled fine-tuning of design of process and equipment for future plants.

OUTLINE

PUREX process for the reprocessing of Plutonium rich fuel with a burnup of 155 GWd/t and cooling time of less than two years, was successfully demonstrated in CORAL plant (Figure 1). The reprocessing of this $(\text{Pu}_{0.7}\text{U}_{0.3})\text{C}$ mixed carbide fuel was a challenge from many fronts such as chopping, dissolution and solvent extraction. Based on the experience with the reprocessing of spent fuels of 25, 50 and 100 GWd/t burnup, the reprocessing of spent fuels with still higher burn-up and lower cooling times was taken up in CORAL facility. The fuel pins, though brittle due to high burn-up, could be chopped in single pin chopper without any problem. Inspection of the chopped pin pieces during the CORAL campaigns indicated that there was no significant crimping, which enabled good dissolution characteristics, demonstrated the use of single pin chopper for the slender pins of fast reactor fuels. This could be confirmed from the fact that the dissolution kinetics was almost same as that of the fuels with lower burn-ups. Dissolution was carried out in a thermosyphon dissolver which was operated under reflux conditions to destroy the carbide carbons which on dissolution can interfere with the solvent extraction process. The centrifugal contactors used for extraction due to the low residence time, enabled good recoveries and decontamination factors in spite of high radiation dose to the solvent. The recovery and decontamination performance is comparable to the campaigns with 100 GWd/t. Recoveries of more than 99.8% for plutonium and 99.9% for uranium could be achieved. The decontamination factors realised with respect to critical nuclides such as ^{106}Ru and ^{137}Cs are only marginally lower than 100 GWd/t fuel but within acceptable limits.

The product has been refabricated into fuel subassembly and loaded into FBTR. This marks the important land mark of closing the fuel cycle of FBTR after a burnup of 155 GWd/t. The operation and maintenance of various equipment in CORAL, enabled validation of the design, experience of these units, provided the unique opportunity for appropriate choice for their application and gave adequate confidence for scaling up for the future plants. Also, the design of master slave manipulators which are very vital in contamination-free operation especially when the tongs which aid in maintenance, has been scaled up to higher capacities for future plants, (Figure 2) based on the feedbacks.



Fig.1: CORAL Facility

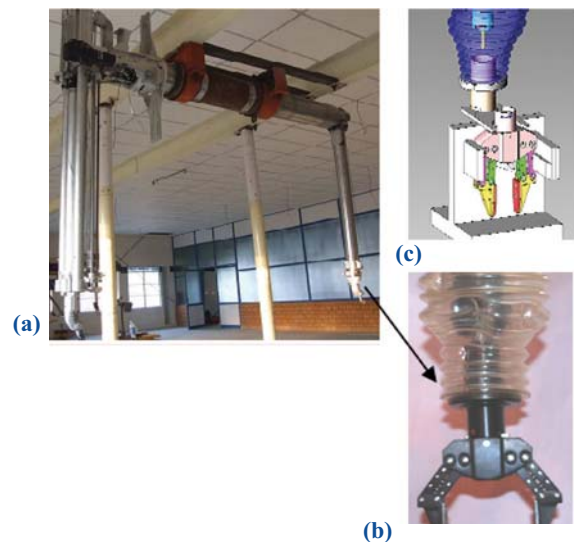


Fig. 2: α-tight Master slave extended reach manipulator with remotely removable tong

GENERAL DESCRIPTION

Reprocessing is the Chemical treatment of spent reactor fuel to separate uranium and plutonium into pure products from the fission products. During the reprocessing campaign of this high burnup fuel, another major issue was addressed in CORAL which is the demonstration of reduction of waste volumes of solvent. The solvent undergoes degradation inspite of using a short residence time contactor which holds the plutonium during the recycle. Hence in the reprocessing of fast reactor fuels with high burnup, recovery of plutonium from the lean organic stream before recycle is important. For this purpose, extensive laboratory studies have been carried out using various solvent wash processes (Figure 3) involving ammonium carbonate, hydrazine carbonate, sodium carbonate and $U^{4+}/0.1\text{ M } N_2H_4$. The results of the studies indicated that ammonium carbonate and hydrazine carbonate are the most promising candidates considering all the aspects of recovery and easiness of deployment in the hot cells. Process flow sheet based on these reagents have been developed and deployed in CORAL.

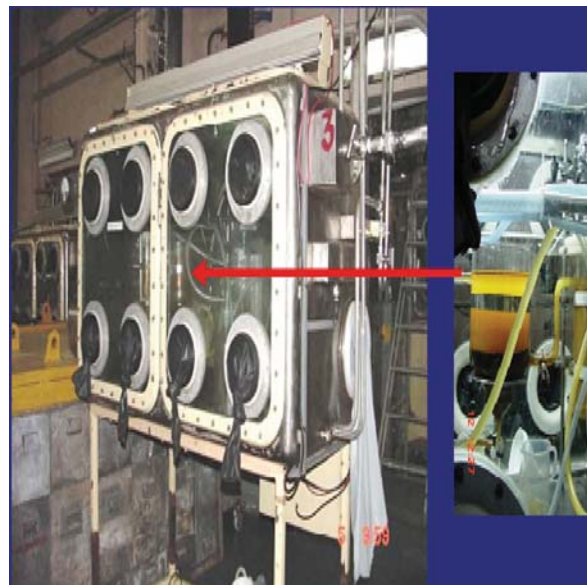


Fig. 3: Solvent wash set up in a glove box undergoing testing

ACHIEVEMENTS

Uranium and plutonium from the spent fast reactor fuel with a high burn-up of 155 GWd/t, were separated to the required purity and recovery levels. Reprocessing of this type of fuel with this high burn-up has been carried out successfully for the first time in the world. Several process development work have been successfully completed while carrying out this operation.

PUBLICATIONS

R.V. Subba Rao, M.Venkataraman, R. Natarajan and Baldev Raj, "Operating experience of Fast Reactor Spent Fuel Reprocessing facility, CORAL", Global 2009, Paris, France, 2009.

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Modifier Free Delivery of Ligands for Supercritical Fluid Extraction

EXECUTIVE SUMMARY

A Supercritical Fluid Extraction method has been developed for the controlled delivery of ligands including solid extractants using supercritical carbon dioxide (Sc-CO₂). The ligand delivery vessel with a restricted geometry, where its inlet and outlet are located beside each other, accomplished the task of the controlled delivery of ligands. The proposed method eliminates the usage of modifier solvent such as methanol for the delivery of various ligands during the supercritical fluid extraction of metal ions. Using neat Sc-CO₂, the delivery profiles for the various ligands such as octyl(phenyl)-N,N-diisobutylcarbomoylmethyl-phosphineoxide(CMPO), tri-n-butyl phosphate(TBP), tri-n-octyl phosphine oxide(TOPO), theonyltrifluoroacetone(TTA) and di(2-ethylhexyl) isobutyramide (D2EHIBA) were established. The technique was demonstrated for extraction of uranium and thorium nitrates.

OUTLINE

Supercritical fluid extraction (SFE) is more advantageous technique for the extraction of metal ions even though the initial applications were established for the extraction of natural products. The commonly used supercritical fluid, CO₂ has the advantages of being less expensive and possessing convenient critical temperature and critical pressure i.e. 32°C and 74 bar respectively. Since CO₂ is non-polar and can extract efficiently the various non-polar compounds, a modifier or co-solvent like methanol is added to make it effective for the extraction of polar compounds.

Direct extraction of metal ions using Sc-CO₂ is inefficient because of charge neutralisation requirement and the weak solute-solvent interactions. However when metal ions are complexed or chelated with ligands or chelating agents, they may become more soluble in Sc-CO₂. To achieve the extractability of metals, specific ligands are added to CO₂. Variety of ligands such as dithiocarbamates, beta diketones, organophosphorous compounds, amides and macrocyclic compounds e.g. crown ethers are used for SFE of various metal ions such as transition elements, lanthanides and actinides.

Different modes are established to introduce the ligands into Sc-CO₂ stream in a SFE technique. An on-line chelation method is carried out by introducing ligands directly into Sc-CO₂ and the modified Sc-CO₂ is passed through the sample containing metal ion. A separate pump is used to deliver the ligand and mix with CO₂, which is delivered from another pump. The stream is passed through the extraction vessel containing the sample. Such mode of delivering the ligands is possible only for the liquid reagents of low viscosity but difficult for viscous liquid extractants and the solid reagents, which can not be pumped directly and rather needs a co-solvent like methanol. On contrary, in the in-situ chelation method, it is performed by spiking a suitable ligand into the extraction vessel containing the sample matrix before subjecting it to SFE. In general, on-line chelation method is used for the introduction of ligands which are liquid in nature and easy to pump while in-situ chelation is used for the introduction of ligands which are either a solid or liquid.

Among the various delivery modes of ligands established so far for the extraction of metal ions, the delivery mode which has no modifier or co-solvent results in elimination of liquid secondary waste. It is also essential to minimize the phase perturbation by a modifier to safeguard the integrity of the supercritical fluid phase of carbon dioxide. The modifier free modes of delivering the ligands in the on-line chelation mode are mainly studied for ligands of liquid nature. However, such methods are not available for the delivery of highly viscous liquid and solid extractants like TTA, CMPO, TOPO etc, the commonly used extractants for the actinides. Hence studies were carried out to evolve out a method to deliver the ligands without using a liquid modifier like methanol (Figure 1&2).

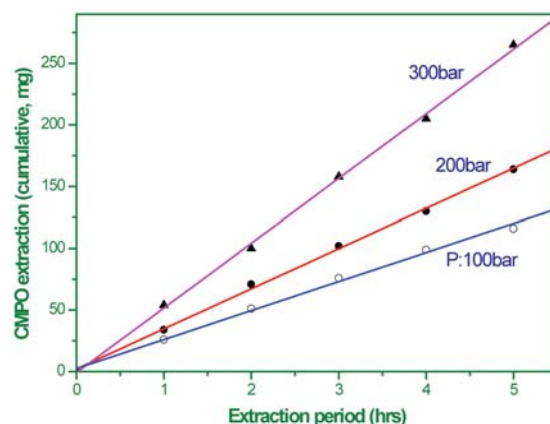


Fig. 1: Extraction of CMPO from 10 mL vessel (LDV-RG) as a function of pressure. CMPO initial amount: 600 mg. Sc-CO₂ flow rate: 3 mL/min; P: 100-300 bar and T: 40°C.

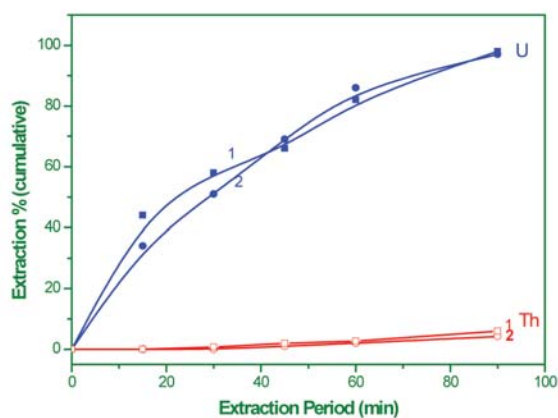


Fig. 2: Use of Modifier free technique for preferential extraction of uranyl nitrate over thorium nitrate. Experimental: Sc-CO₂: 2.5 mL/min; P: 300 bar, T: 40°C; Ligand: D2EHIBA.

MODIFIER FREE DELIVERY PROFILES OF VARIOUS LIGANDS

Investigation was carried out to modify the exponential delivery profile of ligands in to a linear delivery in order to achieve a uniform and controlled delivery during SFE. Among the various parameters studied for this purpose, the ligand delivery vessel geometry was investigated (Figure 3). The modification of the ligand delivery vessel geometry involves changing from normal geometry to the restricted geometry. In the normal geometry, the inlet and outlet arrangement are in the opposite faces of a cylindrical ligand delivery vessel and Sc-CO_2 is passed once through. However, in the restricted geometry vessel, the inlet and outlet are arranged in the same side and hence the outgoing Sc-CO_2 carrying the dissolved extractant always meets and is in equilibrium with the incoming fresh Sc-CO_2 stream before its release into either extraction vessel or back pressure regulator. Hence the controlled and sustained delivery of the extractant is more probable with the restricted geometry in comparison with the normal geometry vessels. Hence studies were carried out using vessel with restricted geometry design.

The extraction of ligands was studied using a 10 mL vessel (LDV-RG). The SFE delivery profiles were obtained for ligands at different initial amounts, pressure as well as flow rates of carbon dioxide. The delivery profiles were elucidated for an extraction period of 6-8 hrs. The delivery rate of ligand was found to be almost uniform and independent of the initial amount taken. The uniform delivery rate observed in this case is strikingly different from the generally observed exponential extraction profile of various compounds by SFE technique. The rate of extraction at different intervals was found to be almost identical and this meets the delivery criteria of supplying the required amount of ligands for metal extraction. However the amount of ligand delivered remains constant and not altered by the initial amount variation. But the Sc-CO_2 flow rate as well as pressure was observed to vary the ligand delivery rate. The results from the extraction profiles of CMPO reveals that the increased pressure of CO_2 increases the amount of CMPO delivered.

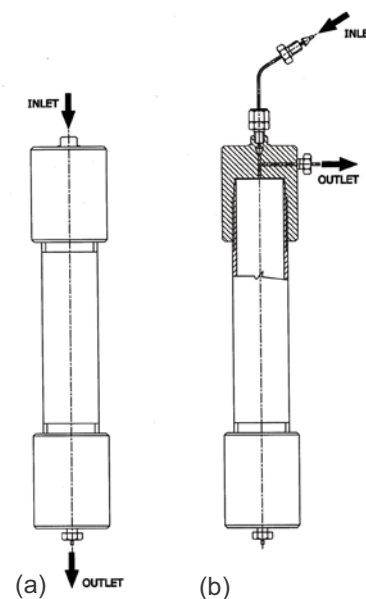


Fig. 3: Schematic of ligand delivery vessels geometry with 10mL capacity. Fig.3a: Normal geometry vessel (LDV-NG) and Fig.3b: Restricted geometry vessel (LDV-RG).

ADDITIONAL INFORMATION

The modifier free mode of delivering the various ligands was applied to extract the uranium and was compared with the extraction of uranium using the same ligand in a typical modifier mode i.e. ligand dissolved in methanol. In both these studies, the quantity of CMPO delivered was maintained constant. The extraction profile of uranium using CMPO delivered by modifier free mode established its extraction pattern, similar to the trend observed with the CMPO in methanol, in which a complete extraction of uranium was established.

GENERAL INFORMATION

Application of modifier free delivery mode of ligands e.g. CMPO for the supercritical fluid extraction of uranium from tissue paper matrix was demonstrated. Uranyl nitrate was preferentially extracted from thorium nitrate using modifier free delivery mode with D2EHIBA and the results indicated that the extraction is found to be as efficient as the one obtained using modifier method. Extraction of neodymium nitrate was also demonstrated using modifier free delivery method using CMPO.

ACHIEVEMENT

A novel technique has been developed for modifier free delivery of ligands for supercritical fluid extraction of actinides. The technique eliminates use of co-solvent leading to minimization of secondary waste during SFE of actinides from waste matrices.

PUBLICATIONS

"A Novel technique for Modifier Free Delivery of Ligands for Supercritical Fluid Extraction", R.Kumar, N.Sivaraman, K.Sujatha, T.G.Srinivasan and P.R.Vasudeva Rao, *Radiochimica Acta* 97 (2009) 441.

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Bubble Detector Based Criticality Monitor

EXECUTIVE SUMMARY

Criticality Monitors are important for rapid evaluation and reduction of operators' exposure. A bubble detector based neutron dosimeter, which offers instant alarm has been developed for nuclear criticality accidents. The system consists of an elastic polymer throughout which millions of tiny droplets of a superheated liquid (Freon-12) have been dispersed. When neutrons strike these droplets, they form small gas bubbles, which remain fixed in the polymer. Instant alarm is achieved using an array of optical sensors, which consist of coupled IR diode transmitter and photodiode receivers. When bubble nucleation occurs due to neutron irradiation, there is increased scattering of IR light and this leads to reduction in light intensity received by photodiodes that in turn activates the buzzer. The performance of the device was tested in KAMINI reactor upto the neutron flux of 10^8 neutrons/cm²/sec. This bubble detector based system holds promise as a practical approach to criticality monitoring. Multiple numbers of such systems can be installed and suitably networked for monitoring a particular area. This system will be very much useful in confirming the occurrence of a criticality event and also ensuring the absence of criticality during re-entry of personnel to the locations where criticality event had occurred earlier.

OUTLINE

Criticality Monitors are important for rapid evaluation and reduction of exposure to operators. Many neutron area monitors are currently being used as criticality monitors in nuclear facilities. A bubble detector based criticality monitor (Figure 1) that offers instant alarm has been developed (first of its kind) for detection of a criticality event. The device is meant to be installed in reprocessing plants in the plutonium separation stage or in fuel fabrication facilities, where plutonium and enriched uranium are handled. They will be affixed to the walls or the ceiling of fuel processing plants in order to detect the criticality events.

The system consists of a neutron bubble detector prepared in specially designed polycarbonate vial, stepper motor with programmable controller unit and incident alarm system.

Bubble Detector consists of an elastic polymer in which droplets of superheated Freon-12 liquid has been uniformly dispersed. These droplets are photon insensitive, whereas neutron triggers the boiling of the drops, nucleating visible bubbles that remain fixed in the polymer.

A reversible synchronous motor with torque of 10 kg-cm along with its controller unit was selected to deliver desired pressure to the gel. The bubble detector is pressurized during forward rotation of the stepper motor and depressurized on reverse rotation.

The alarm system consists of a set of infrared diode and photodiode as transmitter and receiver arranged in transmission-reception geometry and placed in a cylindrical Perspex container along with associated electronics. IR light from diode passes through the bubble detector (placed at the centre of the container) and gets collected at the photodiode. The system is provided with a blinking green LED indicating the normal function and continuous glowing of a red LED along with buzzer activation indicating the event of criticality. Interference from ambient light is avoided by operating IR diode in pulsed mode.

During the criticality event, 10^{16} to 10^{17} fissions in 1 milli second (as initial burst) occurs and the fission neutrons interact with the superheated droplets resulting in clusters of bubble nucleation in the bubble detector. This process decreases the intensity of light reaching the photodiodes and activates the buzzer.



Fig. 1: Criticality Monitor

NEED FOR CRITICALITY MONITOR

A supercritical system releases energy rapidly, of the order 10^{16} to 10^{17} fissions in 1 millisecond for the initial burst, until thermal expansion, boiling or other effects reduce the reactivity. The total dose from several past criticality accidents at a distance of 2 meter from the reacting materials was of the order of 10 Gy in few seconds. The installation of criticality accident alarm system is recommended by ANSI for all activities in which the inventory of fissionable materials represents a nontrivial risk of a criticality accident. Such an alarm system can provide a significant reduction in radiation exposure for personnel in the vicinity of an accident by signaling a person for evacuation. Anyone very close to the accident may still receive a lethal dose before having time to respond to the alarm. Thus the alarm system is not expected to protect every individual, but rather to minimize collective dose.

PERFORMANCE OF BUBBLE DETECTOR BASED CRITICALITY MONITOR

Performance of the prototype criticality monitor system was tested in KAMINI reactor. The response time was measured by placing in the irradiation port of south beam tube of KAMINI reactor. After stabilizing the reactor power, the bubble detector was sensitized using the stepper motor device and the time required for activation of the buzzer was measured. Experiments were repeated at various neutron fluxes (10^3 to 10^8) by operating the reactor at given power levels (0.5, 1, 2, 5 & 30 kW) and the corresponding response times were measured (Table 1). Activation of alarm occurs within nine seconds for the neutron flux of 10^8 and it is expected to get reduced further at higher neutron flux.

Table 1: Response time

Neutron flux (ϕ_n /cm ² /sec)	Response time (sec)
10^3	180
10^4	95
10^5	53
10^6	37
10^7	20
10^8	9

BRIEF DESCRIPTION OF STEPPER MOTOR WITH CONTROLLER UNIT

A reversible synchronous motor with torque of 10 kg-cm along with its controller unit was selected to deliver desired pressure to the gel. The bubble detector is pressurized during forward rotation of the stepper motor and depressurized on reverse rotation. Forward and reverse rotation of the motor is achieved by activating the appropriate coil in the motor. An embedded system has been designed using micro controller PIC16f877a to carry out the task. The entire rotation is divided into small steps to have fine control. The control action involves forwarding and reversing the steps, number of rotations, coarse or fine control of steps. Control action is enabled by pulsed operation from the micro controller. Optically isolated 240 V AC, 10 A rated solid state relays are used to switch the motor. The firmware for the above task is done in cross compiler C language.

ACHIEVEMENT

This system promises as a practical approach to criticality monitoring. Multiple numbers of such systems can be installed and suitably networked at fuel cycle facilities. This system will be very much useful in confirming the occurrence of a criticality event and also in ensuring the absence of criticality during re-entry of personnel to the locations where criticality event had occurred earlier.

PUBLICATION

D. Ponraju, H. Krishnan, V. Gopalakrishnan, N. Ramesh and K.M. Somayaji, "Bubble detector based criticality incident detection and alarm system", Radiation Protection and Environment, 31 (2008) 418.

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Measurement of Biomagnetic Fields using Super Conducting Quantum Interference Device Sensors

EXECUTIVE SUMMARY

Facilities have been set up at IGCAR for the Super Conducting Quantum Interference Device (SQUID) based measurement of biomagnetic fields such as those associated with the physiological activities of human heart (Magnetocardiography or MCG) and human brain (Magnetoencephalography or MEG). These magnetic fields (~ 100 fT to 100 pT) are extremely weak and their measurement falls exclusively in the domain of SQUID technology. Unlike the measurement of electric potential (ECG / EEG), measurement of magnetic fields is non-contact and is much less affected by the conductivity of the intervening tissues. Using a four channel system currently operational at IGCAR, measurements of MCG and the α rhythm of the brain have been carried out with high signal-to-noise ratio. Preliminary results show that certain cardiac dysfunctions manifest more clearly in the MCG compared to the ECG.

OUTLINE

Magnetoencephalography (MEG) and Magnetocardiography (MCG) are non-invasive and non-contact measurements of the magnetic fields associated with the physiological activities of human brain and human heart respectively and provide data which is complementary to techniques like Electroencephalography (EEG) and Electrocardiography (ECG). The measurement of such extremely weak magnetic fields (typically 100 fT to 100 pT) is one of the most challenging applications of SQUID sensors. Since the ambient magnetic noise in an unshielded laboratory is typically 1 to 100 nT, a Magnetically Shielded Room (MSR) is essential for the measurement of MCG / MEG signals. A special building using 316 stainless steel rods as reinforcement in concrete (SS-RCC) has been built at IGCAR to minimize the influence of transient magnetic noise, which is believed to be the first load bearing construction using SS-RCC in India. The MSR is erected in this building on a 50 ton SS-RCC foundation block, which reduces the vibration levels to less than 10^{-4} m/sec². The MSR has two layers of μ -metal (2 and 3mm thick) and two layers of Al (8 and 4mm thick) and provides a shielding factor of 70 dB at 1 Hz, which improves to 100 dB at 10 Hz. A Fibre Reinforced Plastic (FRP) liquid helium cryostat housing the SQUID sensors is located inside the MSR (Figure 1); the cable carrying the SQUID output signals is passed through a shielded waveguide to a separate RF Shielded Room (RFSR) housing the necessary instrumentation. The output of each SQUID is digitized with a resolution of 24 bits using an individual ADC in each channel and recorded at any desired sampling rate upto 20 kHz per channel. To provide noise immunity, the digitized data is transmitted over an optical fibre link to a server class PC located in the unshielded area. The overall system noise-floor is measured to be ~ 12 fT/ $\sqrt{\text{Hz}}$, which is adequate for the measurement of the biomagnetic fields. This is the first MEG system in India and its sensitivity is comparable to that of typical systems operating elsewhere.

Measurements of the MCG and α -rhythm of the brain in human subjects has been carried out with a high signal-to-noise ratio. Figure 2 shows typical MCG patterns simultaneously recorded in this facility at four different locations on the chest surface. The signal clearly shows the P-wave related to the depolarization of the atria, QRS peak related to the depolarization of the ventricles, and the T-wave related to the repolarization of the ventricles. The MCG patterns are similar in appearance to the standard ECG since they are the manifestations of the same underlying biological activity; however, they may reveal new information not contained in the ECG since the MCG signals are not much affected by the conductivity of the intervening tissues. Figure 3 shows the α -rhythm recorded in the occipital lobe of the human brain. This is ~ 50 times weaker in intensity compared to the MCG signal and manifests in the Fourier transform as a peak at 8 - 13 Hz when the eyes are closed; the peak gets suppressed when the eyes are open. The system has also been used to investigate the μ -rhythm of the motor cortex and for study of the response evoked on the presentation of an external stimulus.



Fig.1: Liquid helium cryostat with four SQUID channels

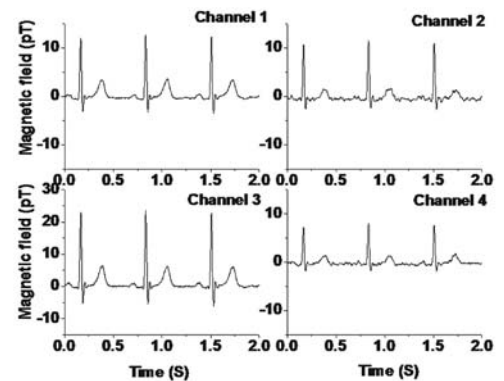


Fig. 2: MCG recorded simultaneously in four channels

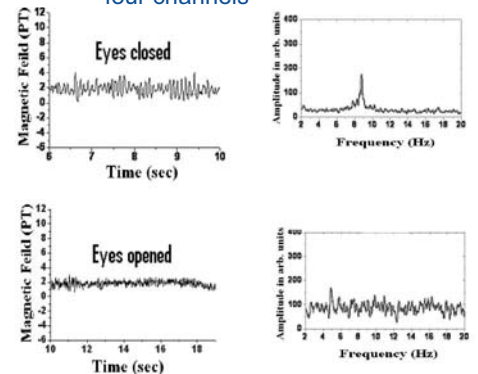


Fig. 3: α -rhythm of the brain recorded using the SQUID system. The left panels show the time series data as recorded, the top for the eyes closed condition and the bottom for the eyes open condition. The right panels show the corresponding Fourier transforms

ADDITIONAL INFORMATION

The system has been used to map the spatial distribution of cardiac magnetic field on a rectangular grid of thirty six points equispaced on the chest surface. A program to assess the potential of MCG in revealing cardiac dysfunctions is presently underway in collaboration with cardiologists; the study will involve recording MCG and ECG patterns of about fifty subjects including healthy volunteers and those with known cardiac dysfunctions. The system will be shortly augmented to nineteen SQUID channels. Using this system, there is a possibility to record the interictal spikes in MEG with a view to localize epileptogenic foci in subjects suffering from drug resistant epilepsy.

COMPARISON OF ECG AND MCG

As an illustration, ECG of a sixty eight years old male subject with a left bundle branch block is shown in Figure 4. The left bundle branch block leads to a delay in the depolarization of the left ventricle compared to the right ventricle, which manifests as a notched QRS complex in ECG with a duration exceeding 120 milliseconds. MCG measurements have been carried out at IGCAR on the same subject at thirty six positions on the chest. MCG at one of the measurement positions is shown in Figure 5. It is evident that the delay in the depolarization of the left and right ventricles manifests very clearly in MCG as a notched and wide QRS. This notched QRS has been seen in MCG at several positions on the chest of this subject. Evidently, MCG has the potential to emerge as a powerful diagnostic tool complementing the standard ECG.

From the measured magnetic field distribution, it is necessary to reconstruct the source current distribution. In general, this inverse problem does not admit of a unique solution. Nevertheless, computational techniques have been developed to reconstruct the sources consistent with the known anatomical and physiological information. It is often possible to model the source as an equivalent current dipole and infer its location, orientation and strength. Techniques such as Singular Value Decomposition and Multiple Signal Classification enable the location of the equivalent current dipole to be determined with an error not exceeding 2 millimeter even in presence of reasonable levels of noise inevitably present in any measurement. The inverse problem for ECG / EEG is more complex than the one for MCG / MEG since the measurements of electric potential are affected by the conductivity of the intervening tissues.



Fig. 4. ECG of a subject with a left bundle branch block. The notched QRS Pattern in V5 and V6 (highlighted in the red oval), the inverted "T" wave in V6, and wide QRS (>120ms) is indicative of a left bundle branch block in ECG

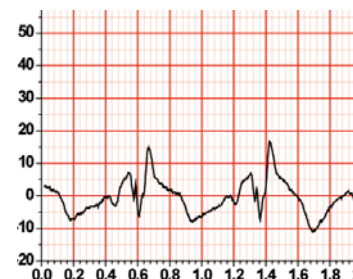


Fig. 5. MCG of the same subject showing the notched and wide QRS complex far more clearly than the ECG

BRIEF DESCRIPTION

Measurement of magnetic fields associated with the physiological activities of human brain and human heart is likely to provide a clearer glimpse of the underlying electrophysiology. IGCAR now houses the first facilities in India, which have a capability to measure the biomagnetic fields using multichannel SQUID system. Work is underway in collaboration with eminent cardiologists and neurologists in the country to harness the potential of this relatively new technique. Development of computational algorithms for reconstruction of sources based on the measured magnetic field distribution is an integral part of this effort. With these developments, SQUID based systems will be increasingly used by Indian researchers for the measurement and characterization of biomagnetic fields.

ACHIEVEMENT

SQUID sensors have been used to measure and characterize the extremely weak magnetic fields associated with the physiological activities of human heart and human brain. These magnetic fields cannot be measured by any other sensor technology

PUBLICATIONS

M.P.Janawadkar, T.S.Radhakrishnan, K.Gireesan, C.Parasakthi, S.Sengottuvel, Rajesh Patel, C.S. Sundar and Baldev Raj, "SQUID based measurement of biomagnetic fields", Current Science ,99 (2010) 36.

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A Novel Variable Temperature Regulator for Measurements at Low Temperatures

EXECUTIVE SUMMARY

Variable Temperature Regulator (VTR) is an enclosure or environment in which the specimen can be mounted to study the temperature dependent physical properties. The variable temperature regulator is a central part of any low temperature measurement system in which the temperature can be varied from 4.2 to 300 K and vice versa with uniform rate of heating and cooling. In some experiments (like magnetization measurements), the temperature of the specimen has to be maintained constant for a user specified duration of time. Achieving a high temperature stability for a long period of time is a difficult task. The variable temperature regulator has been designed, fabricated, characterized and successfully utilized in a high field SQUID magnetometer set-up developed in-house.

OUTLINE

Temperature dependent physical properties of materials such as resistivity, AC susceptibility and magnetization have been measured by varying the temperature of the specimen in a controlled manner and maintaining it constant with a high accuracy. To attain temperatures down to 4.2K, liquid helium is used as a cryogen for cooling the sample mounted in a thermally isolated sample chamber. Usually, a small amount of cryogen is allowed through a narrow capillary tube to cool the sample using a mechanically operated needle valve with a long stem. The reliability of such needle valves is relatively poor and it is difficult to vary the flow rate precisely using automated control circuits. A Variable Temperature Regulator (VTR) has been developed in-house to circumvent the above problems. In this setup, an impedance chamber is used which acts as an electronically controlled valve to precisely control the flow of cold helium gas into the VTR chamber. The desired flow rate can be easily achieved simply by controlling both the heater current in the impedance chamber and the pressure in the VTR chamber.

A schematic cross sectional view of the VTR is shown in Figure 1. The impedance chamber shown on the left acts as a helium valve to control the incoming flow of cold helium gas into the VTR chamber. The liquid helium enters through a fine CuNi capillary tube with an inner diameter of 200 μm and a length of 80 mm. An electric heater is wound over the fine capillary and a thermometer is mounted over the capillary to monitor the temperature of the capillary. On both sides of the fine capillary, two connecting tubes (ID 1.5 mm) in the form of coils (coil diameter 12 mm and length 40 mm) have been connected to reduce the thermal leak between the capillary tube and liquid helium reservoir. The whole arrangement is located inside a vacuum chamber with vacuum level better than 0.001 mbar. An OFHC copper block with a thermometer and a heater is mounted in the VTR chamber to monitor and control the set temperature. Since the viscosity of helium increases with increase in temperature, the incoming helium flow into the VTR chamber can be varied just by heating the capillary. The gaseous helium coming out of the capillary is cooled again to 4.2K by the connecting coil which is in thermal contact with liquid helium, which in turn cools the copper block. The cooling rate at the copper block (sample region) is controlled by controlling the temperature at the capillary and the pressure in the VTR chamber. A capacitance manometer and a stepper motor driven throttle valve is connected in the pumping line with a pressure controller with accuracy better than 0.01 Torr in the VTR chamber.

Temperatures in the VTR have been recorded during the cool down for different capillary temperatures and pressures in VTR. Figure 2 shows that the VTR can be cooled in three modes such as slow, moderate and fast. In the slow mode ($\leq 1\text{K/min}$) the temperature of the capillary is kept between 15 and 20K and the pressure in the VTR chamber is maintained at 5 Torr or less. In the moderate mode (between 2K/min and 4K/min) the temperature of the capillary is kept between 7.5K and 12.5K and the VTR pressure is maintained at 10 Torr. In the high rate of cooling 10K/min, setting the capillary temperature is set as 6K and the VTR pressure is 20 Torr.

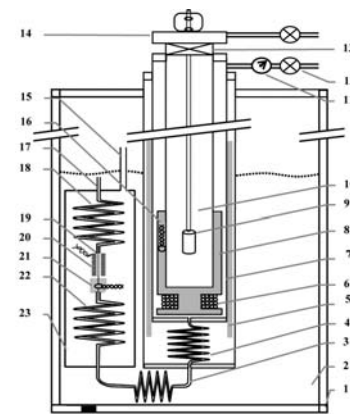


Fig.1: Cross-sectional view of the VTR.

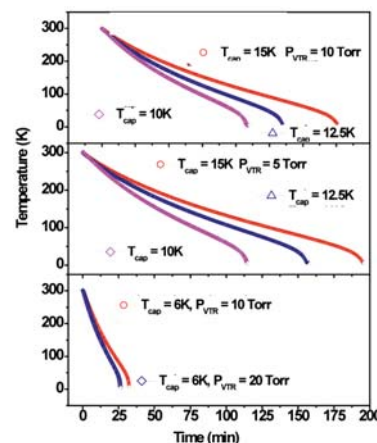


Fig.2: Cooling down characteristic of the VTR chamber as a function of time for different capillary temperatures and VTR pressures

HIGH FIELD SQUID MAGNETOMETER

The variable temperature regulator is a tiny but important part of any physical property measurement system; it enables achievement of different cooling and heating rates and helps in maintaining the temperature constant for any duration of time as desired by the user. The VTR has been incorporated as part of the high field SQUID magnetometer developed in the laboratory. This set-up is being used for measuring the magnetization of the sample at different temperatures and magnetic fields. The high field SQUID magneto-meter comprises of the VTR, a high homogeneity superconducting magnet (0 to 7 Tesla), a NbTi shield to provide magnetically quiet environment, stepper motor driven sample movement across the superconducting gradiometer and the SQUID sensor. This is a complex SQUID based measurement system and substantial effort has been invested towards the realization of the SQUID magnetometer. To calibrate the set-up, the VTR temperature is maintained constant and a superconducting lead sphere is moved across the gradiometer. Figure 3 shows the photograph of the actual SQUID magnetometer set-up and the inset shows the flux profile of the lead sphere recorded by moving it across the second order gradiometer pickup loop while maintaining a constant temperature in the VTR (sample chamber). A sensitivity of 1.2×10^{-7} emu, could be inferred, which is comparable to that achieved elsewhere.



Fig. 3: Photograph of the high field SQUID magnetometer set-up which incorporates the Variable Temperature Regulator (VTR)

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The variable temperature regulator has been designed based on the low value of viscosity of gaseous helium. The flow rate of the helium gas is inversely proportional to its viscosity. The viscosity of the helium gas increases with increase in temperature. The capillary size (inner diameter and length), capillary temperature and the differential pressure maintained between the variable temperature regulator and liquid helium reservoir decides the volume flow rate of helium gas. The flow rate of helium gas through the capillary has been estimated for a wide range of capillary sizes, capillary temperatures as well as the pressures in the variable temperature regulator chamber. The experimentally measured volume flow rate through the capillary was in reasonable agreement with the values estimated by these calculations.

ACHIEVEMENT

Fabrication of variable temperature regulator is a challenging task as it involves the use of a fine capillary of just 200 micrometer diameter to control the flow of helium gas. It also involves fine welding works in joining very thin walled tubes of 1 to 2 millimeter diameter to relatively thick walled tubes. Excellent temperature stability better than ± 10 mK at low temperatures (4.2-100 K) and ± 50 mK at higher temperatures (100 - 300 K) has been successfully demonstrated. The amount of liquid helium required to cool the sample from 300 to 4.2 K is very low and is independent of the rate of cooling down, clearly indicating that the variable temperature regulator is thermally well isolated from the liquid helium reservoir. The variable temperature regulator has been incorporated in the high field SQUID magnetometer developed in-house. In view of the excellent performance of the variable temperature regulator, it can be incorporated in any low temperature measurement apparatus requiring fast and economic cooldown and excellent temperature control.

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Sub-Surface Magnetic Flux Leakage Non Destructive Evaluation using Giant Magneto-Resistive Sensors

EXECUTIVE SUMMARY

Giant magneto-resistive (GMR) sensor based magnetic flux leakage measurement system has been developed to detect feeble leakage fields from sub-surface defects and deeply located defects in carbon steel plates. The tangential (B_x) as well as normal (B_z) components of leakage fields from notches of various depths in 12 millimeter thick carbon steel plates have been measured using two separate giant magneto-resistive sensors. It is found that giant magneto-resistive sensors could reliably detect sub-surface notches located at 11 millimeter below the plate surface.

OUTLINE

Magnetic flux leakage (MFL) technique is widely used for non-destructive evaluation (NDE) of ferromagnetic steels. In this article the study using MFL measurement to detect deep defects in five carbon steel plates (450x150x12 mm³), each having Electro Discharge Machine notches (15 mm length, 1 mm width) of 3.1 mm, 6.2 mm, 8.7 mm, 10.3 mm and 11.1 mm depths is presented. Finite element modeling has been carried out to optimize the spacing between the arms of yoke (70 mm) and the GMR sensor position (middle of yoke arms). The B_x and B_z components of the leakage magnetic flux are measured by scanning the GMR sensor across the notches. The sensor output is amplified, digitized and analyzed. The B_x and B_z components of leakage field of sub-surface notches located at different depths are shown in Figure 1. As can be seen, unique Gaussian type signals and dipolar type signals have been observed for B_x and B_z components of leakage magnetic fields, respectively. The GMR sensor could reliably detect the sub-surface notch located at 11.1 mm (depth 0.9 mm) below the surface. Both the B_x and B_z components of the GMR sensor output are found to decrease with increase in notch location below the surface due to the divergence effect.

MFL images of notches are obtained by scanning the GMR sensor in a raster manner. Figure 2 shows the MFL image of a sub-surface notch located at 6.2 mm below surface in plate. The MFL image (B_x) readily provides the spatial extent of the notch. For enhancing the images, Eigen value based approach has been developed and a significant increase in signal to noise ratio has been achieved. Higher sensitivity of GMR sensors allows them to be used at higher lift-off (spacing between GMR and plate surface) as depicted in Figure 3. As can be observed, the GMR sensor has detected all the notches upto a lift-off of 3 mm.

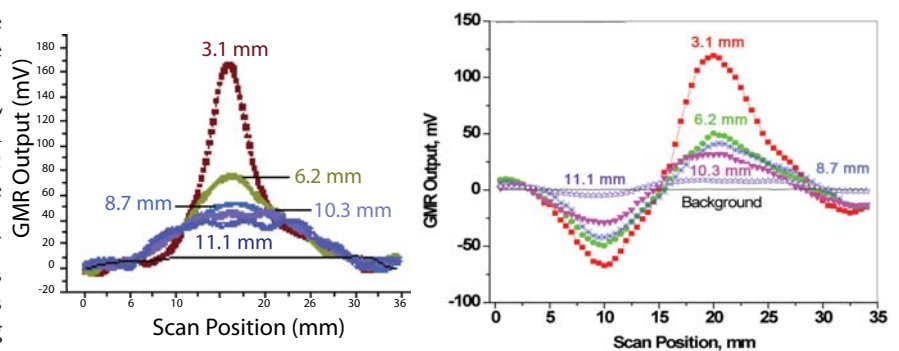


Fig. 1: GMR sensor response for sub-surface notches in 12 millimeter thick carbon steel plates

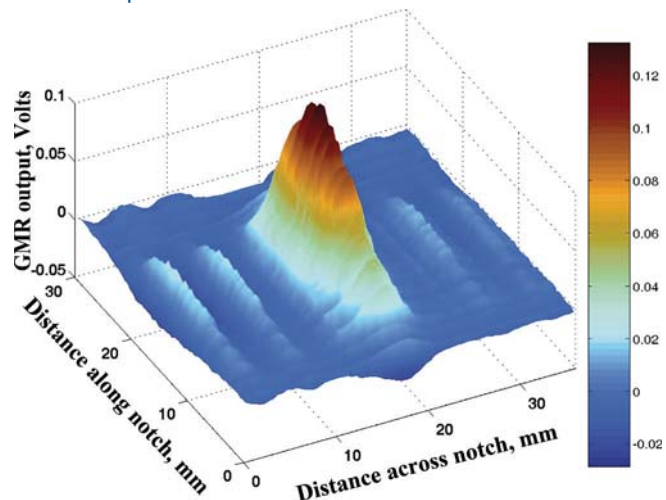


Fig. 2: MFL image (B_x) of a subsurface notch located at 6.2 mm below surface

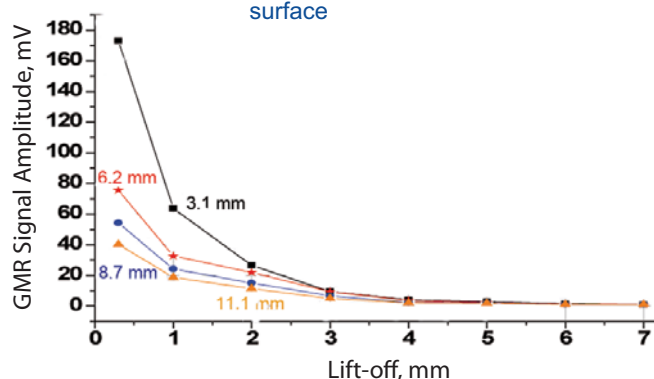


Fig. 3: GMR signal amplitude with lift-off for different sub-surface notches

ADDITIONAL INFORMATION

The tangential and axial components of leakage fields can be computed using the analytical equations derived based on dipole model or image theory and also using finite element (FE) models. In order to understand the interaction of magnetic fields with defects, FE modeling has been carried out using COMSOL 3.4 Multi-physics software package. Typical magnetic field profiles (plots) of sub-surface notches located at 3.10 mm and 6.24 mm (deep sub-surface) in a 12 mm thick carbon steel plate are shown in Figure 4. The intensity of leakage fields for deep sub-surface notch is much weaker than for the sub-surface notch. GMR sensors seem attractive for detection of leakage fields from deep-sub-surface defects. GMR sensor consists of a few nm thick multilayer structures such as Fe/Cr/Fe, Co/Cu/Co, etc. in which ferromagnetic layers are separated by non-magnetic layer (Cr or Cu). It works based on the GMR effect in which there is a large change in electrical resistance of the multilayer, exposed to an incident magnetic field, due to the spin dependent scattering of electrons at interfaces between ferromagnetic films and nonmagnetic interlayer.

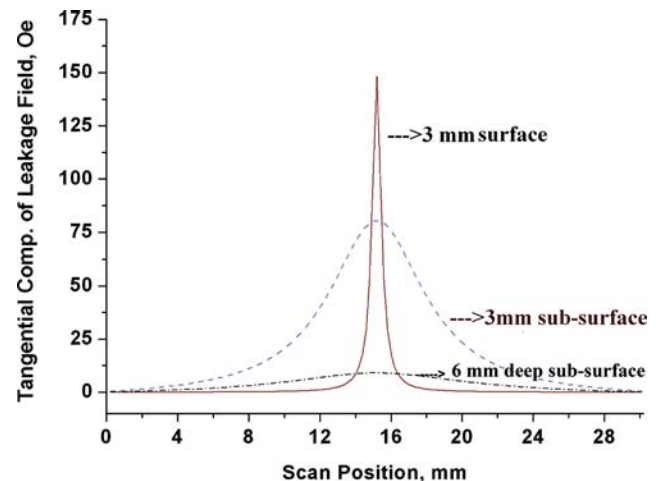


Fig. 4: Model predicted contours for sub-surface notch and deep sub-surface notch

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

MFL technique is widely used for non-destructive evaluation (NDE) of ferromagnetic steels. In this technique, the object is magnetized and the magnetic flux that leaks out of defects is detected using coils, Hall sensor or GMR sensors. As compared to Hall sensors, GMR sensors are attractive as they offer high sensitivity for low-magnetic fields, good signal-to-noise ratio (SNR) and high spatial resolution. They are used in this study to measure the leakage fields from sub-surface defects in carbon steel plates. The MFL test set-up consisting of a C-core yoke, GMR sensor, X-Y scanner, amplifier and personal computer as shown in Figure 5. The sensitivity of the GMR sensor used in this testing is found to be 260 VT^{-1} at 5V biasing voltage and can withstand 120°C . It exhibits a linear response within the range of 0.2 mT to 1.3 mT.

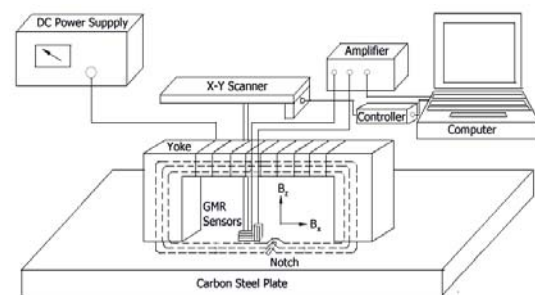


Fig. 5: Set-up for MFL testing using GMR sensors

ACHIEVEMENT

Using the giant magneto-resistive sensor based magnetic flux leakage system we have achieved the detection of defects located even at 11 millimeter below the surface of carbon steel plate. The giant magneto-resistive response is found to decrease with notch location below the measurement surface. Giant magneto-resistive sensor has shown good detection performance used as high as a lift-off of 3 millimeter and giant magneto-resistive sensors have potential for use in magnetic flux leakage testing of components at high temperature.

PUBLICATIONS

1. W. Sharatchandra Singh, B.P.C. Rao, S. Vaidyanathan, T. Jayakumar and Baldev Raj, "Detection of Leakage Magnetic Flux from near-side and far-side Defects in Carbon Steel Plates using Giant Magneto-Resistive Sensor", *Measurement Science and Technology*, 19 (2008) 015702.
2. B.P.C. Rao, S. Thirunavukkarasu, K. Krishna Nand, T. Jayakumar, P. Kalyanasundaram and Baldev Raj, "Enhancement of Magnetic Flux Leakage Images of Sub-surface Defects in Carbon Steel Using Eigen Value based Approach", *Non-Destructive Testing and Evaluation*, 23 (2008) 35.

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Destruction of Organics by Mediated Electrochemical Oxidation

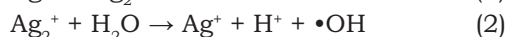
EXECUTIVE SUMMARY

Mediated Electrochemical Oxidation (MEO) is a promising technique for the destruction of organic wastes. Tributyl Phosphate (TBP) in Normal Paraffin Hydrocarbon (NPH) in nitric acid medium containing the electro-generated Ag(II) catalyst was destroyed successfully in constant current mode using this technique. The electrolyte used in the system was 8 M HNO₃ with various concentrations of Ag[II] ranging from 0.05 to 0.5 M at ambient temperature and at 333 K. The results revealed that increase in temperature and current favour the rate of destruction of organics. About 60% of Pu could be recovered from the organic liquid waste, after destroying the solvent and its degraded products by mediated electrochemical oxidation.

OUTLINE

Much attention has been paid recently to the electrochemical processes designed to treat organic wastes containing 30% TBP in NPH, used as extractant in nuclear fuel reprocessing industry. Incineration and caustic hydrolysis are a few methods adopted in the treatment of these organic wastes. A room temperature process which will generate lower volumes of secondary wastes, was developed at the Reprocessing Development Laboratory of IGCAR. This process is called the Mediated Electrochemical Oxidation process where, an electron transfer mediator, typically a transition metal in its highest valence state, is generated at the anode of an electrochemical cell and is dispersed throughout the anolyte solution. Upon oxidation of the organics by the mediator, the reduced mediator species is re-oxidised at the anode and the cycle repeats until all oxidisable organic material in the cell is depleted. In the destruction of degraded organics, Ag(II), a powerful oxidant having E° value of 1.98 V is utilised as the electro chemical catalyst. Ag(II) may react directly with the organic material to be destroyed or it may first react with water to form hydroxyl or oxygen radicals which in turn oxidise the organics to carbon dioxide and water. The otherwise unstable Ag(II) is generated at anode in the electrochemical cell in aqueous solution. The virtual cell reactions are given in equation 1 to 4

At Anode



At Cathode:



Experiments were carried out in constant current mode with 0.05, 0.1 and 0.5 M AgNO₃ in 8 M HNO₃ medium. For comparing the destruction efficiency of the mediator ions, one experiment was carried out with 0.05 M Ce as redox intermediate. Pt mesh anode and Pt plated Titanium cathode were separated by one end or both ends opened diaphragm tubes. Organic solvent containing uranium in ppm level (272, 424 or 680 ppm), pure NPH, 30% TBP (all in 8 M HNO₃ medium) and pure 8 M HNO₃ with out redox intermediate formed the anolyte solutions. Effect of temperature on the destruction of organics was investigated by carrying out experiments at 303 and 333 K in batch and continuous modes. Rate of destruction was determined by analysing the acid strength in the aqueous phase. Figure 1 depicts the rate of destruction of the solvent at 333 K with and without uranium. Anomalous behaviour was observed in the destruction of pure TBP and NPH with the appearance of a greasy layer which got dissolved subsequently. Higher temperatures and other metallic ions viz. uranium favoured the destruction. The reliability of mediated electrochemical oxidation process in destroying the degraded solvent was verified by oxidising the organic material present in the actual radioactive waste solution and the results are shown in Figure 2.

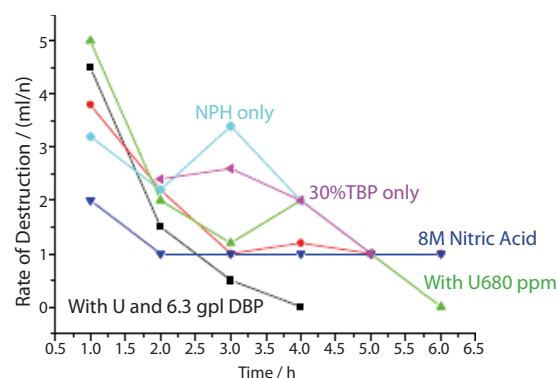


Fig. 1: Comparison of the Rate of Destruction of Organics

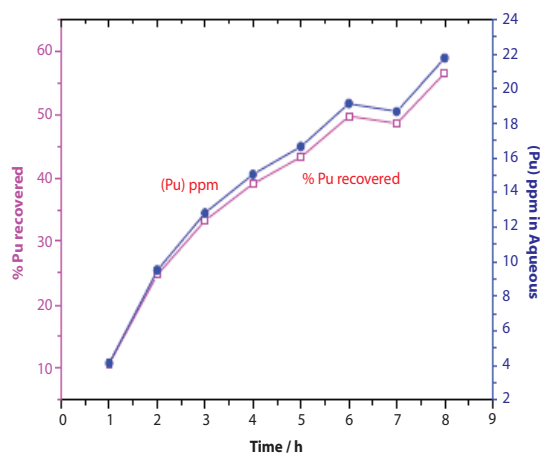


Fig. 2: Recovery of Plutonium from Organic Phase

ADDITIONAL INFORMATION ABOUT MEDIATED ELECTROCHEMICAL OXIDATION - WHAT IS MEDIATED ELECTROCHEMICAL OXIDATION?

Destruction of organics by oxidation can be accomplished with Ozone, Direct Chemical Oxidation method, Fenton reaction, photochemical oxidation and electrochemical methods. The energy efficiency in Direct Electrochemical Oxidation of organic species is very less due to mass transfer and kinetic restraints at the electrode surface. Many of the restrictions can be overcome by the adoption of Mediated Electrochemical Oxidation, in which metal ions such as Ag, Ce and Co having high redox potential, acts as an electrochemical catalyst to increase the efficiency of the oxidation process.

PLUTONIUM RECOVERY FROM RADIOACTIVE SPENT SOLVENT

Five milliliter of active organic sample was added to 100 milliliter 0.5M AgNO_3 in 8 M nitric acid and electrolysis was conducted with a double end open porous alumina diaphragm tube. The specifications of the active organic sample are given in Table 1.

The efficiency of Ce(IV) redox catalyst in destroying the organics was found to be less than 50% of what could be achieved with Ag(II) catalyst. The concentration of Ag(II) started to decrease with time owing to (a) oxidation of water and (b) its migration towards cathode under the influence of an electrical potential. The reduction of nitric acid at the cathode led to a decrease in the volume of catholyte, thereby increasing the cell voltage. To alleviate these problems and to avoid clogging of plutonium in the pores of alumina diaphragm tube (only 60% of plutonium could be recovered from the aqueous solution after destroying the organic phase completely), diaphragmless electrolysis is preferred. However, the destruction efficiency decreases substantially in a diaphragmless cell.

Table:1 Specifications of active organic sample

[Pu]	: 769 ppm
Gross α	: 65.47 mCi/L
Gross β	: BDL
Gross γ	: 0.27 mCi/L
DBP	: 1.8 gpl
[H ⁺]	: 0.022 M
TBP in NPH	: 9.9%

MEO PROCESS - PRINCIPLE

Powerful oxidants like Ag(II) and Ce(IV) mediator ions can be generated at the anode in an electrochemical cell in aqueous medium. Ag(II) itself and the radical species produced by water reaction with Ag(II) attack the organic substrate, progressively oxidise the organics in a series of steps irreversibly to carbon dioxide and water. During the oxidation step, Ag(II) is reduced back to Ag(I) which in turn is regenerated to Ag(II) at the anode. This "Catalytic" use of Ag(II) is utilized in the Mediated Electrochemical Oxidation process for the total destruction of insoluble organics like Tri Butyl Phosphate (TBP) and Normal Paraffin Hydrocarbon (NPH). The application of mediated electrochemical oxidation process in destroying the solvents can be extended to recovering Pu metal from the degraded lean organic solvents.

ACHIEVEMENT

Conventional method of disposal of organic wastes by incineration always leads to hazardous gaseous products. Mediated Electrochemical Oxidation process can be conveniently employed to destroy the radioactive organic wastes safely without polluting the atmosphere. Valuable fissile material also can be recovered from the organic wastes using mediated electrochemical oxidation process.

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Open Source based Graphical User Interface Software for PFBR Operator Display Stations

EXECUTIVE SUMMARY

Distributed Digital Control System (DDCS) is the appropriate design architecture for automating a geographically distributed plant with large number of input / output signals and control loops. DDCS gets plant data from different subsystems located at different buildings and provides all the information to the plant operator in the Control Room (CR). Operator can see any data or status of any of the process signals from the control room. Operator is alerted as and when any alarm condition occurs in the plant. Graphical User Interface (GUI) software to run on PFBR control room operator display station enables the plant operator to monitor and control the plant. GUI software has been developed in C++ to run on Linux OS, using platform independent open source graphics class library - Qt. Qt supports OpenGL extensions using which the plant information is visualized in 3D. Qt has also support for MySQL database in which the plant parameters are stored.

OUTLINE

Figure 1 gives the architecture of DDCS. It is a three tiered architecture with Real Time Computer (RTC) based embedded system at the lowest level connected to field level sensors & final Control elements, Process Computer (PC) at the middle level and Display Station (DS) at the top level, all interconnected by dedicated fiber optic local area network i.e. plant backbone.

Plant information is received from the plant backbone by Process Computers, which do all the post processing, update the centralised plant parameter database and also archive the data in real time. The graphic display stations on the operator console and panels receive the data from these process computers and display the plant information in different display formats. Additionally, dedicated display stations are provided on operator console to receive data directly from SC 1 and SC 2 data highways and display the safety and safety related parameters on-line. Similarly display stations are provided in Handling Component Room for fuel handling operations and Backup Control Room respectively. Graphical User Interface software runs on these display stations, receives real time signal values from the process computer through TCP/IP over Ethernet LAN. It provides menu interface to navigate through the features provided in the GUI. All the sub systems of the plant are listed like primary sodium system, secondary sodium system etc. The operator can select for any of the sub systems information, compare the values of the selected system parameters against their alarm thresholds and display the status.

The GUI software consists of Communication module, Database module and Display module. Display module provides various display formats like Bar graph, Trend graph, Alarm panel display and Tabular format as shown in Figure 2. It also includes critical parameter and color code displays. The critical parameter display is always in the view of the operator, so that at any point of time the status of the signals can be known from the encoded color of the signal. It displays some critical parameters with their current status. Bar Graphs are provided to compare signals against each other. Trend Graphs show the signal values over a period of one hour. Status display is provided to know the status of 128 signals at a time. The operator can move the mouse over any of the signal to know further details about it. In each of the display format ten groups are provided. These signals are grouped as per process logic. Regrouping of the signals also can be done by the operator. Privilege levels and password protection is provided to authenticate for group changing. The changes are recorded by storing the newly grouped signals in the database against respective group names.

3D display of temperature profile of PFBR core is shown in Figure 3. PFBR core map is modeled using 3D libraries. 3D objects can be rotated, viewed in various angles and projected to the screen using perspective projection. The transformation into 2D screen coordinates is done using rasterization techniques.

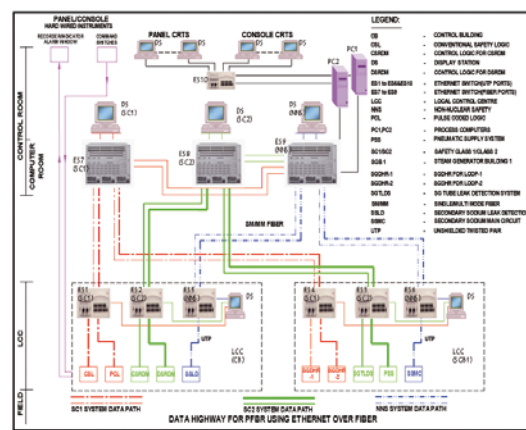


Fig. 1: DDCS Architecture

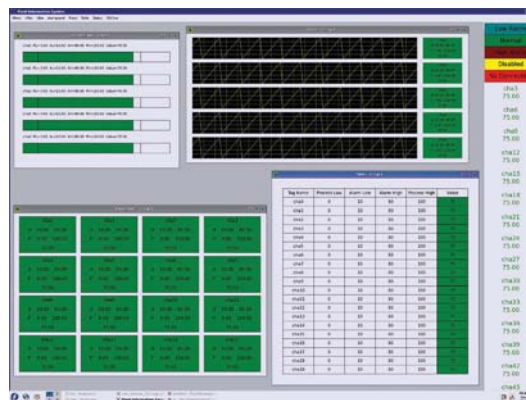


Fig. 2: Displays

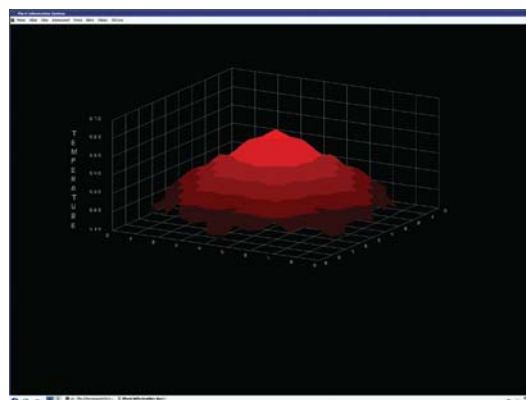


Fig. 3: 3D view of core temperature profile

ADDITIONAL INFORMATION ABOUT QT TOOLKIT AND OPENGL

Qt is a multiplatform C++ GUI toolkit. It provides application developers with all the functionality needed to build applications with state-of-the-art graphical user interfaces. Qt is fully object-oriented, easily extensible and allows true component programming.

Qt is supported on the following platforms: MS/Windows, Unix/X11, Macintosh

OpenGL (Open Graphics Library) is a standard specification defining a cross-language, cross-platform API for writing applications that produces 2D and 3D computer graphics. The interface consists of over two hundred and fifty different function calls which can be used to draw complex three-dimensional scenes from simple primitives.

IMPLEMENTATION OF GUI USING QT

Qt GUI toolkit has been chosen to develop the software for Display station since it is platform independent and the source code is available openly, so that internal verification and validation can be done.

For each of the display format separate classes were used. The displays were drawn using Qt GUI class libraries like QPushButton, QLayout, QWidget, QMainWindow, QMenu, etc.

The communication between the DS and the Process computer was made using QTCPsocket and QServerSocket class libraries. Database connection to the Process computer to get the signal parameter values were written using QSql libraries. Process computer functions as server and display station as client.

The client requests for connection to the Process computer, whenever the user selects any of the system. The server accepts the connection and the connection is established and signal values of the selected system is sent to the display station. These values can be viewed in any of the display format. If there is a communication failure the sockets will be closed. The user is able to see the failure by the displayed color code of the signal values.

The software also provides user interface to carry out any control operations like modifying the controller set points and giving commands to remote equipments like valves, motors etc. History trend logging was also provided with provisions for storing plant data for one shift, one month and one year. GUI also has core temperature profile display in 3D.

The 3D module was developed using OpenGL libraries like GLpolygon, GLcolor, GLrender, GLortho etc. Each subassembly is drawn by giving the subassembly position as x, y co-ordinates and temperature of the subassembly as z coordinate.

ACHIEVEMENTS

Open source based GUI software for PFBR control room display stations using Qt and OpenGL are first of its kind implementation for Nuclear Power Plants in DAE / Nuclear power industry in general. GUI features for 2D and 3D displays were successfully developed and demonstrated on LINUX platform.

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Performance Evaluation of Prototype Fast Breeder Reactor Model Steam Generator in Steam Generator Test Facility

EXECUTIVE SUMMARY

A 5.5 MW capacity model sodium heated Steam Generator (SG) was tested in the Steam Generator Test Facility (SGTF). The heat transfer performance testing of steam generator was carried out at rated conditions. The performance of the equipment is in line with the design expectations. At rated temperature, pressure and flow conditions, steam generator transferred 5.5 MW thermal power with a sodium inlet temperature of 516.5°C. At rated sodium inlet temperature of 525°C, steam generator transferred 6.0 MW thermal power from sodium to water/steam.

OUTLINE

Construction of the 500 MWe Prototype Fast Breeder Reactor (PFBR) is in progress at Kalpakkam. Steam Generator Test facility (SGTF) is set up at our Centre to test sodium heated Once Through Steam Generator (OTSG) of nineteen tubes (Figure 1) similar to the PFBR steam generator such as operating conditions, material of construction, structural layout etc. Rated power of the facility is 5.7 MWt. The material of construction of shell, tubes and flanges is modified 9Cr-1Mo steel. All the tubes are identical and contain expansion bend which is provided to accommodate differential thermal expansion between the shell and tubes. This facility consists of an oil fired heater system as heat source, sodium system as medium of heat transport and a conventional steam water system without turbine.

The steam generator is a 25 meter high middle supported unit with expansion bend and 23 meter heat transfer length. This vertically simulated test article is similar in all respects to the proposed 157 MWt steam generator module for the Prototype Fast Breeder Reactor, with reduced number of tubes. A sodium pump circulates sodium at 105 m³/hr flow rate through steam generator shell side. A multistage centrifugal boiler feed pump which has the capability to develop 220 bar water pressure supplies the feed water to the steam generator at around 180 bar pressure. Steam at 172 bar, produced in the steam generator is dumped to a condenser through a series of depressurising and desuperheating stations and then recycled to achieve a closed water cycle. The design details of Steam Generator are given in Table.

Sodium flow through Steam Generator is measured by permanent magnet flow meter at the steam generator outlet. This flow meter is calibrated in sodium and maximum possible error in the flow meter reading is $\pm 1.5\%$ of the flow. Bulk sodium temperature at steam generator inlet and outlet were measured by 3 millimeter diameter K-type thermocouples inserted in thermo wells. Feed water flow to steam generator is measured by a flow nozzle, calibrated at Fluid Control Research Institute, Palakkad. Feed water inlet temperature is measured by RTD type temperature sensor. Nine numbers of 1 millimeter diameter K type thermocouples measure the steam outlet temperature from the steam generator tubes directly. These thermocouples are inserted 10 millimeter inside the tubes from the top of the top tube sheet. Feed water inlet pressure and steam pressure are measured by pressure transmitters.

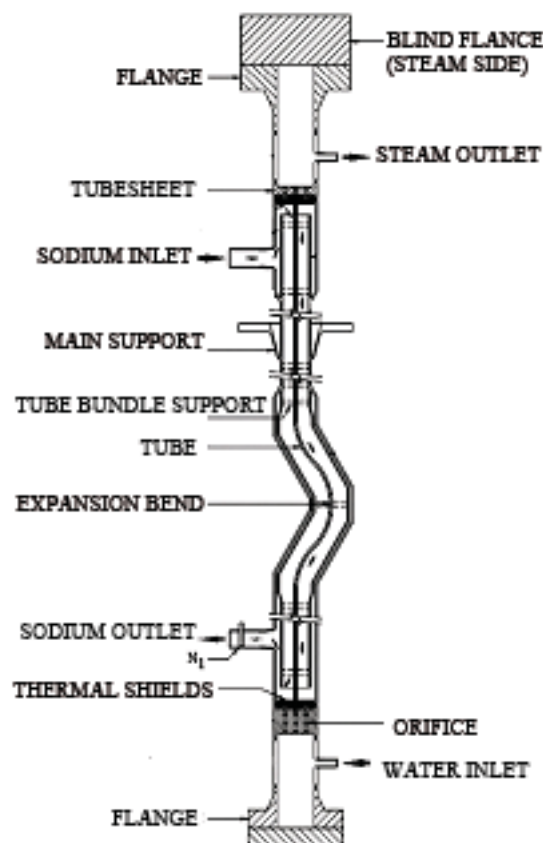


Fig. 1: SGTF Steam Generator

Table.1: The design details of Steam Generator

Thermal Power	5.5 MW
Sodium Flow	104.8 m ³ /h
Water Flow	8820 kg/h
Sodium Temperature	525/355°C
Steam/Water temperature	493/234°C
Steam Pressure	172 bar
Total Length	23 m
Heat transfer area	23.5 m ²
Tube Size	17.2 mm OD ,2.3 mm Thick
Tube Pitch	32 mm,triangular
No. of Tubes	19
Shell ID	164 mm, 10 mm thick
Water side DP	12.46 bar

GENERAL EXPLANATION RELATED TO DESCRIPTION

Steam Generator was operated with the rated sodium flow of 104.5 m³/h and with the rated feed water flow of 8820 kg/h. Feed water inlet temperature to steam generator was maintained as 234°C. Sodium inlet temperature to steam generator was adjusted in order to get the rated steam outlet temperature around 493°C. Rated steam temperature was obtained at a sodium inlet temperature of 516.5°C. In other words, sodium inlet temperature of 516.5°C is sufficient to transfer 5.5 MW of power at rated sodium flow, rated feed water flow and rated water/steam side temperatures. This sodium inlet temperature is less than 8.5°C than the design sodium inlet temperature of 525°C.

Steam Generator was also operated with the rated sodium flow of 104.5 m³/h and sodium inlet temperature to steam generator as 525°C. During the experiment feed water temperature was maintained at 232°C. Feed water flow rate was adjusted to get the steam at temperature around 493°C. It is found that with a sodium inlet temperature of 525°C at 105 m³/h sodium flow rate Steam Generator could produce around 9500 kg/h of steam at 172 bar and 493°C. Average power transferred during this experiment is 6.0 MW. This is 0.5 MW more than the rated power of steam generator.

ACHIEVEMENTS

A model steam generator with nineteen tubes and of 5.5 MW thermal capacity which is simulating the 157MW thermal capacity and five hundred and forty seven tube steam generator for PFBR was designed, fabricated and tested successfully in Steam Generator Test Facility of IGCAR. The steam generator operated at rated temperature and pressure conditions for more than three thousand hours and the performance of the equipment was as expected. At rated temperature, pressure and flow conditions, steam generator transferred 5.5 MW thermal power with a sodium inlet temperature of 516.5°C and with rated sodium inlet temperature of 525°C steam generator transferred 6.0 MW thermal power from sodium to water/steam. With the experimental data it has been estimated that the clean steam generator has 12% extra heat transfer area than required at rated conditions. This ensures the adequate heat transfer capacity for steam generator for PFBR.

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Innovative Differential Pressure Sensor for Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

High precision pulsating type differential pressure sensors have been developed for measurement of differential pressure (DP) in two different ranges (i) 0 – 25 millibar and (ii) 0 – 60 millibar with a precision of less than 0.5 millibar. These pressure monitoring systems of unique design are of industrial grade and they are specially designed to be used in Prototype Fast Breeder Reactor (PFBR). The development includes design and fabrication of rugged differential pressure sensors with associated electronics for pulse counting, data processing to convert frequency to differential pressure in mbar and LED display of pressure in the embedded unit. Highly satisfactory long term performance of this innovative pressure sensor led us to construct a large number of differential pressure sensors which will be used in PFBR.

OUTLINE

High precision DP sensors with reliable performance at very low pressure differentials are required for real time monitoring and control of nitrogen gas space between (i) Main vessel & Safety vessel and (ii) between Safety vessel & Reactor vault during reactor operation or fuel handling. These sensors should be designed in a manner capable of working continuously in industrial environment providing output with excellent precision even at a few mbar of differential pressure. Such sensors are not available commercially. Hence, a new class of pulsating type differential pressure sensors has been developed for use in reactor environment.

Based on the satisfactory performance of many laboratory and prototype units two DP sensors have been designed for two different ranges: 0 – 25 mbar and 0 – 60 mbar, considering the following points (i) to achieve high sensitivity (ii) to choose a material for fabrication, which would impart sufficient robustness and (iii) to evolve a simple method for calibration. These DP sensing devices have been constructed in house mostly out of fabricated stainless steel components. The design is such that, on application of a pressure differential, the level of oil of low vapour pressure in the vertical narrow tube as 'sensing arm' changes considerably in comparison with the corresponding change of oil level in the surrounding cylindrical section of much larger diameter at the bottom as the 'other arm'. A set of equally spaced and vertically mounted rectangular electrodes made up of mirror finished stainless steel plates are placed in the sensing arm which acts like a continuous type level sensor. The level sensors constitute a part of a logic gate oscillator (LGO) circuit. The digital pulse frequency at the output of such pulsating oil level sensor within the narrow tube is governed by the differential pressure. The entire system acts as an oil manometer. Figure 1 shows the schematic diagram of the pressure monitoring system. Suitable calibration devices have been designed and made, which permitted easy and quick multipoint calibration with high reliability.

A laboratory made multi-channel embedded processor with LED display has been configured and connected to the system for on-line monitoring. Performances are excellent with highly reproducible and accurate results. Further, for containment of the oil within the devices in the event of accidental excessive over pressurization or evacuation, an appropriate design with passive feature has also been tested. The work on construction of a large number of DP sensors has already been undertaken for use in PFBR.

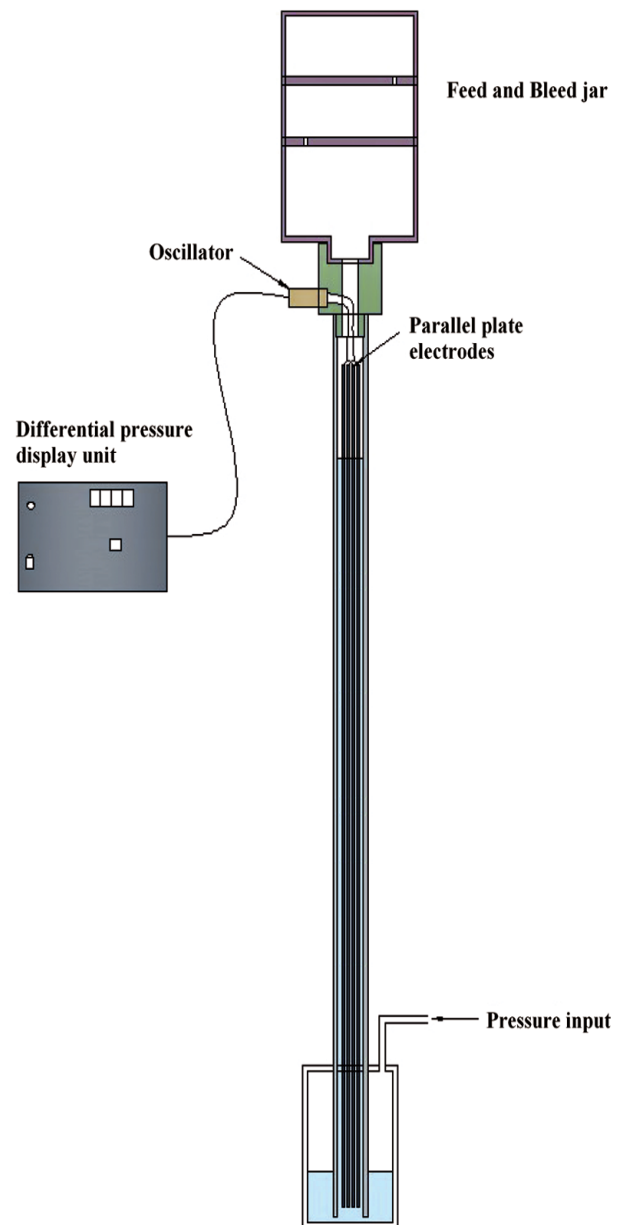


Fig. 1: Schematic diagram of differential pressure monitoring device

ADDITIONAL INFORMATION

Calibration and the Performance of differential pressure Sensor

During calibration the unit has to be connected to the calibration device. By manually pressurizing and evacuating the unit in a controlled manner the change in oil height at several differential pressure levels between 0 and 60 millibar (or between 0 and 25 millibar as the case may be) is determined. The corresponding pulse frequencies are also measured. The difference in oil height in millimeter vs. frequency (f) in kHz are numerically fitted to get a polynomial relation ($\Delta h = af^2 + bf + c$) which is used in the system software to convert online, the output frequency to differential pressure in mm oil height. Further an appropriate conversion factor is also used in the program for direct online display of differential pressure in mbar unit. Calibration plot for a typical differential pressure sensor is shown in Figure 2. Continuous measurement of differential pressure in one of the sensors over a period of one year shows excellent agreement in measurement with less than 0.5 % RSD.

Temperature Effect

Sensors have been tested for a long period to see the influence of temperature on differential pressure. It is observed that up to 60°C the differential pressure values are not affected.

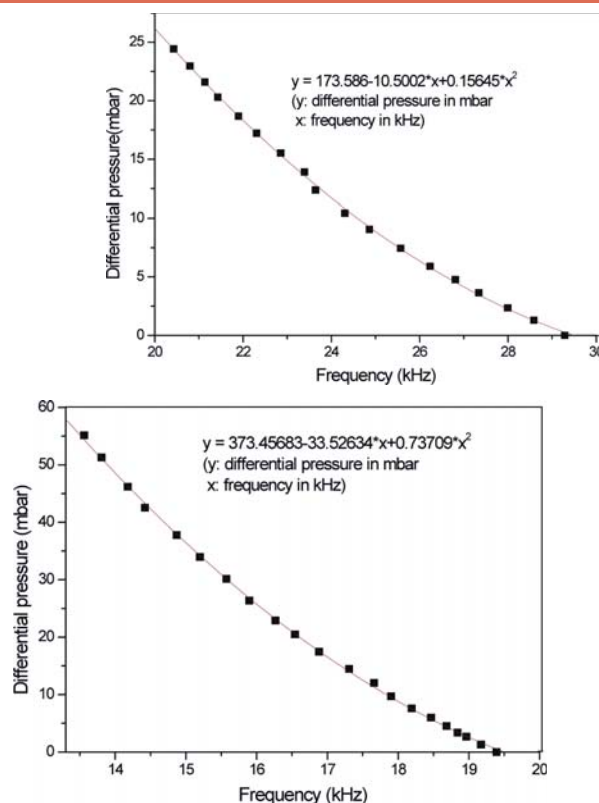


Fig.2: Calibration plots of the two Differential Pressure sensors (A) and (B)

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The differential pressure sensor as described above works as an oil manometer in which an oil of low vapour pressure is used as the sensing fluid. Both arms of the manometer are connected to two different confined zones whose differential pressure has to be measured. A set of equally spaced rectangular stainless steel electrodes constituting a parallel plate capacitor is vertically mounted in the 'sensing arm'. This capacitor assembly forms a part of a logic gate oscillator circuit. The overall capacitance of the capacitor assembly is governed by two dielectrics (i) oil and (ii) air. By pressurizing the unit through one arm the level of oil height increases which in turn shifts the frequency of the logic gate oscillator. Hence the frequency is directly related to the differential pressure.

ACHIEVEMENTS

This innovative approach of sensing differential pressure has been deployed for quantitative assay of hydrogen peroxide in gas evolving reaction (Reaction of hydrogen peroxide and acidified potassium permanganate).

Dash pot oil level sensors for Control and Safety Rod Drive Mechanism of PFBR and continuous type level sensors for Sol-gel plant in Chemistry Group, IGCAR are based on the principle of oil level monitoring as used in this work.

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Establishment of Technology for Sodium Removal from Components

EXECUTIVE SUMMARY EXECUTIVE SUMMARY

Liquid sodium is being used in the experimental facilities at Fast Reactor Technology Group (FRTG). Sodium being highly reactive with atmospheric air and moisture, it needs to be removed before any modification/repair work is initiated. Water vapour-CO₂ (WV-CO₂) process is used for large reusable components. This process deals with the reaction of sodium with moist carbon dioxide in recirculation mode, producing hydrogen and heat. The process eliminates any possibility of stress corrosion cracking (SCC) in austenitic stainless steels (ASS). The process has been used for removal of sodium from PFBR components like Control and Safety Rod Drive Mechanism, Diverse Safety Rod Drive Mechanism, Failed Fuel Location Module, Under Sodium UltraSonic Scanner (USUSS), etc.

OUTLINE

Sodium testing of reactor components is done in FRTG. Sodium adheres to the surface of the components during testing in FRTG test vessels. Safe handling of the component necessitates removal of adhering sodium. The cleaning process involves the four steps, namely sodium draining from the components, vapour phase reaction both with water vapour and CO₂, Rinsing the component with water and drying with hot nitrogen. The draining of sodium from the components is normally carried out in the test vessel itself. The vessel is kept hot at ~200°C for more than 8 hours to enable draining of sodium. Then the component is lifted from the vessel at room temperature and kept in the cleaning facility.

In this method, CO₂ is bubbled through water, maintained at temperature of 323 – 353 K, and injected into a recirculating stream of hot argon / nitrogen (343 - 353 K). Sodium reacts with the water vapour in the CO₂ and forms NaOH with the release of hydrogen (limited to < 2%). Further, this NaOH reacts with CO₂ to form Sodium bicarbonate (NaHCO₃) / Sodium Carbonate (Na₂CO₃) which does not cause SCC on Stainless steel. After complete reaction, which will be indicated by the absence of increase in concentration of hydrogen, the re-circulation is stopped. Finally the carbonates are washed off by water rinsing and dried.

The Sodium Removal Facility (SRF), located in high bay area of Engineering Hall – III, IGCAR, is an independent cleaning facility of 1.6 m x 1.6 m x 13.7 m height with three floors, each of 3.9 m height. Figure 1 shows the flow sheet of the facility. There are two loops namely gas circulation loop and water wash loop. The gas circulation loop consists of a blower (1000 m³/hr) for circulating the water vapour-CO₂-Ar mixture and a finned heater (6 kW) for maintaining the temperature of the gas mixture. A CO₂ bubbler is provided in this loop which injects moist CO₂ into the circulating gas stream. Water temperature in the bubbler is maintained at 323-353 K with immersion heaters (3 kW). Moist CO₂ is admitted into the system by manually opening the solenoid valve. The necessary online monitors for monitoring temperature, pressure, relative humidity (RH) etc. are provided in the loop. The pressure in the system is maintained by Feed-bleed system to regulate the flow of argon / nitrogen using pressure switch (system pressure between 0.2 - 0.3 kg/cm²). is used for flow regulation. Safety relief valve is set to open at 0.5 kg/cm² in case of system is over pressurized. A Hydrogen Sensor is used for online monitoring of hydrogen released during the cleaning process. The sensor output is acquired using a data acquisition system. This same loop is used for drying the component with hot nitrogen after water wash. The second loop is used for water wash of the component after vapour phase reaction. It consists of a centrifugal pump and a Dimineralised (DM) water Tank (1000 liters). DM water from the tank is pumped into the vessel from the bottom until the water level in the vessel is sufficient to immerse the component completely. Hydrogen released during the water wash is also monitored. During the cleaning process, argon was filled and circulated using gas circulation loop for maintaining the inertness and also for heating the component. Figure 2 gives the details of recorded data during sodium removal of USUSS.

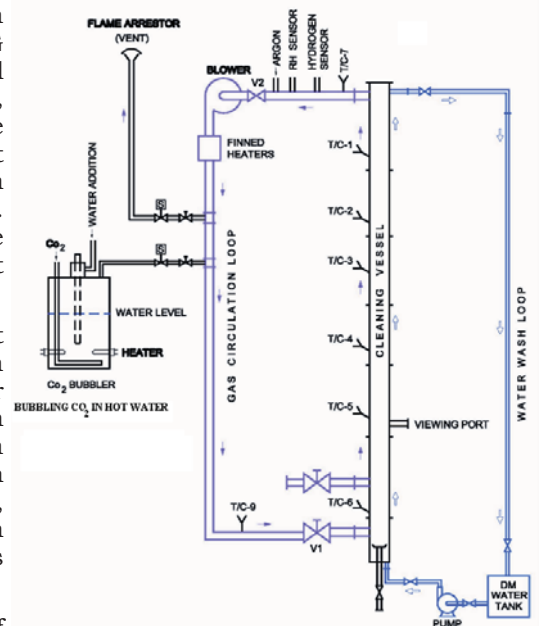


Fig. 1: The Sodium Removal Facility (SRF), located in high bay area of Engineering Hall – III, IGCAR, is an independent cleaning facility of 1.6 x 1.6 x 13.7 meter height with three floors, each of 3.9 meter height

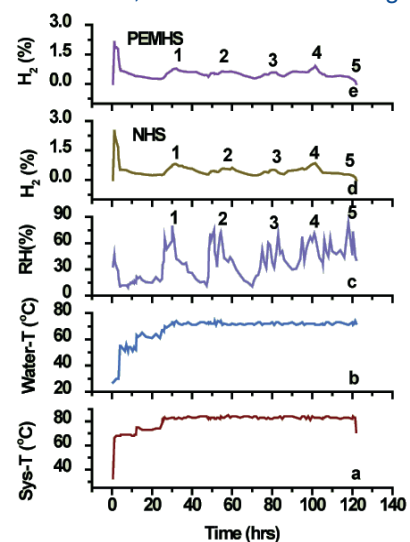


Fig.2: Parameters during sodium removal of USUSS (a) System temperature (b) CO₂ bubbler water temperature (c) System RH (d) H₂ conc. by Nucon sensor (e) H₂ conc. by PEMHS

ADDITIONAL INFORMATION ABOUT VARIOUS SODIUM REMOVAL PROCESSES

Since sodium is a reactive metal various safe removal methods are employed. Methods like alcohol cleaning, steam nitrogen process and water vapour-CO₂ process are preferred depending on the nature of the components used in the reactor. Generally alcohol cleaning method is used for small components like fasteners, bellows sealed valves, etc. while steam nitrogen process, which is a rapid process, is used for non-reusable components like fuel sub-assemblies.

GENERAL EXPLANATION RELATED TO DESCRIPTION

All components, equipments, and pipelines are subjected to sodium removal before any inspection or repair during the service life. Water vapour-CO₂ process is used for large reusable components. This process deals with the reaction of sodium with moist carbon dioxide in recirculation mode, producing hydrogen and heat. The process eliminates any possibility of stress corrosion cracking in austenitic stainless steels, which is the material of construction for most components, and is therefore preferred for reusable components. For reusable components, the steam nitrogen process cannot be employed because the sodium hydroxide (NaOH) produced during the cleaning process can lead to caustic stress corrosion cracking. In the water vapour-CO₂ process, however, the NaOH is converted to carbonates and bicarbonates of sodium which do not cause stress corrosion cracking.

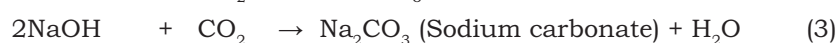
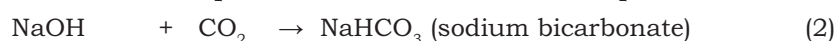
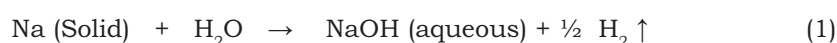
In all the above removal processes, hydrogen is released as a by-product, which forms explosive mixture with air when its concentration is between 4-76%. Hence, during the removal process hydrogen concentration is monitored and maintained below the safe level of 2%. Also monitoring hydrogen concentration reveals the actual progress and completion of the sodium removal process. Table 1 indicates the important parameters recorded during sodium removal works carried out at FRTG.

Table.1: Parameters recorded during sodium removal works carried out at FRTG

	CSRDM	DSRDM	FFLM	USUSS
Reaction Time (h)	~90	~60	~45	122
Sodium Qty (g)	~100	~150	--	643
CO ₂ Qty (kg)	--	< 3	< 2	< 9
Water Qty (l)	92 x 5	540 x 3	--	1600 x 3

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

On injecting moist CO₂ to gas circulation stream, sodium hydroxide is formed predominantly by reaction of sodium with moisture. The formation of sodium hydroxide as per equation 1 is kinetically fast and it subsequently reacts with CO₂ to form sodium carbonate or bicarbonate as per the equations 2 and 3. The possible reactions are given below:



ACHIEVEMENTS

Sodium removal from the prototype components of PFBR was successfully completed and the same component after repair / modification has been tested in sodium. The sodium removal from these components has validated the recirculation WV-CO₂ process design of SRF and the operational experience has revealed the intricacies involved and the safety precautions to be adopted, the time frame involved and the operational parameters that require optimization.

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Weldability Studies of Austenitic Stainless Steels using Gleeble Tests

EXECUTIVE SUMMARY

In Fast Breeder Reactors, fully austenitic stainless steels and its modified version of alloy D9 are chosen as the major structural and fuel clad materials respectively. Hot cracking is a major problem during welding of these alloys and it affects the weldability which amounts to a loss of productivity. Proper evaluation of weldability of these alloys will ensure crack free welds during fabrication. Among the various weldability tests, Gleeble Tests using weld simulation of heat affected zone on the specimens exploits the liquation cracking of the alloys. Vareststraint tests predict the hot cracking susceptibility of the fusion zone. Both these tests are complimentary to each other for evaluation of the hot cracking susceptibility of these alloys and the weldability of these alloys has been established with optimum procedures for future Fast Breeder Recators.

OUTLINE

AISI type 316 and its variants are used extensively as structural material for components of fast reactors operating at temperatures upto 550°C and service life of about 40 years. A new alloy with enhanced nitrogen levels (0.07–0.22(wt-%)) in the austenite matrix is under development, to enhance the service life of the structural components upto 60 years. Similarly, alloy D9 a fully austenitic stainless steel with the composition of 14Cr-15Ni-2.5Mo-0.3Ti is one of the preferred materials for use in core components of Fast Breeder Reactors, in view of its resistance to radiation damage. Improved versions of this alloy are being investigated towards enhancing the void swelling resistance and irradiation creep behaviour, to achieve higher fuel burn-up. The improved versions contain alloying additions such as P, Si, and Ti, as these minor additions have strong neutron absorption cross-section. In newer versions of this alloy, the compositional limits for P and Si are higher than those in the existing versions. Though the composition of the alloys are beneficial for life enhancement and void swelling resistance, the presence of higher amounts of low melting elements are deleterious to its weldability as they contribute very significantly to increasing the hot cracking susceptibility.

In fully austenitic stainless steels, hot cracking (Figure 1) is a severe concern during welding in the absence of a small amount of residual delta ferrite, especially in compositions that contain elements such as S, P, Si, N, Ti and Nb. Its occurrence is attributed to the formation of low melting phases in the solidifying weld metal and in the heat affected zone (HAZ), which causes cracking under the action of shrinkage stresses and restraint imposed on the joint. In the HAZ, cracking can occur by liquation of grain boundaries in the partially melted zone adjacent to the fusion line and in previously deposited weld metal in a multipass weld. Both Hot ductility and Vareststraint tests were used to evaluate the hot cracking susceptibility. Hot ductility tests were conducted using thermo-mechanical simulator and the Nil Strength (NST), Nil Ductility (NDT), and Ductility recovery (DRT) and nil ductility range (NDR) temperatures were determined using specimens of 3mm thickness at various strain (0.5-4.0 %) levels. Brittleness temperature range was also evaluated from these tests. The hot ductility test results showed (Figure 2) that the NDR of the nitrogen enhanced alloy 0.22% is higher (50°C) than the alloy with 0.07-% nitrogen (40°C) and 0.14% (30°C) indicating high susceptibility of the former to hot cracking. Vareststraint test results also showed a favourable condition for 0.22% nitrogen steel. In the case of alloy D9, the NDR values are very high (253-295°C) with increasing Si content (Figure. 3) indicating that these alloys cannot be welded using normal welding procedures. In Vareststraint test also, alloy D9 has very high hot cracking susceptibility during autogenous welding, as the total crack length in both the weld and HAZ and Brittleness Temperature (BTR) values estimated from the tests are very high compared to that of commercial SS 316L. Hence, an alternate welding method is proposed by imposing dilution effects during welding.

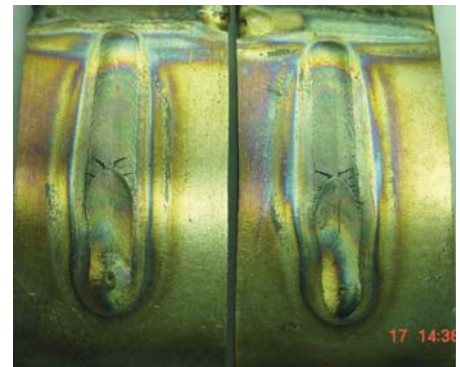


Fig. 1: Typical Hot Cracks of Vareststraint Test in Alloy D9

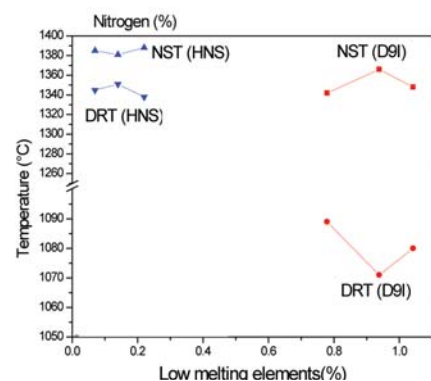


Fig. 2: Effect of Nitrogen/LME with increase in Temperature

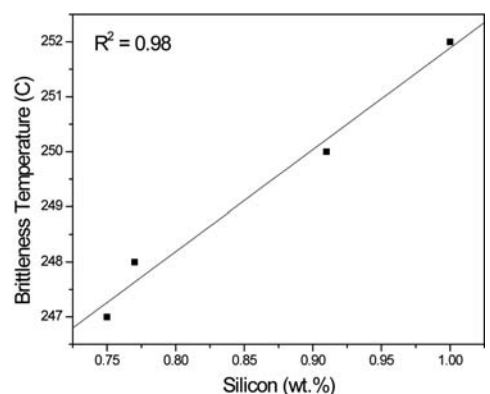


Fig. 3 : Effect of Silicon on Brittleness Temperature

ADDITIONAL INFORMATION

Evaluation of weldability of austenitic stainless steels involves application of strain or some form of restraint during welding, and assessing the deposited weld for cracking. Gleeble test is used to determine the range of temperature at which the material is prone to hot cracking when subjected to a thermal cycle that simulate the actual multi-pass welding. Weld simulation using Gleeble enable to simulate the actual weld thermal cycle on specimen and impose strain in a controlled manner to estimate hot ductility as a reduction in area at the specimen's neck portion after the test.

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The ductility of weldable alloys increases with increase in temperature upto a certain peak temperature; but drops abruptly to very low values at a temperature just below the start of melting and this temperature is referred as nil ductility temperature. The nil strength temperature at which the alloy loses its strength during heating, due to formation of low melting eutectics along grain boundaries, is just above this temperature. However, the hot ductility recovers below the nil ductility temperature during cooling part of the weld thermal cycle. The temperature span from nil strength to ductility recovery is considered to be the brittleness temperature range of the alloy. The Gleeble hot ductility tests are conducted to determine the nil strength, nil ductility temperatures and to compare the susceptibility of hot cracks of different heats of alloys by heating the specimens below to their melting point, to temperatures above 1300°C, and applying tensile load to fracture the specimens.

To compare the hot ductility behaviour of various alloys, the percentage reduction in area of the hot ductility test specimens tested to fracture shall be measured for different alloys and compared. The microstructural features near the fracture surface using optical and scanning electron microscopy respectively. EDX spectrometer analysis carried out to identify the liquated films along the fracture surfaces. Figures 4 and 5 shows the evidence of low melting segregates along the grain boundaries and wetting the fracture surface by incipient melted liquids of low melting eutectics.

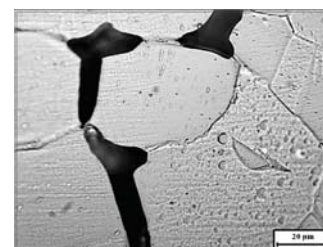


Fig. 4: Presence of low melting segregates along grain boundaries

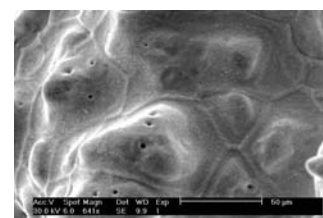


Fig. 5: Fracture surface wetted by incipient melted liquid

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Knowledge on hot cracking behaviour of austenitic stainless steels, such as the 14Cr-15Ni-2.5Mo Ti-modified steels (Alloy D9) and high nitrogen stainless steels is an important consideration for welding as these steels solidifies in primary austenitic mode without any delta ferrite. Hot cracking in these alloys is attributed to the formation of low melting phases in the solidifying weld metal and heat affected zone which in turns leads to cracking due to shrinkage stresses and restraint imposed on the weld joint. Both Gleeble (Hot ductility) and Varestraint tests are among the most versatile methods used to evaluate the hot cracking susceptibility.

ACHIEVEMENTS

A major achievement of this work is in optimizing the composition of new austenitic stainless steel structural material being developed for FBR construction (316LN and D9 alloys) with respect to their resistance to hot cracking during welding. This will in turn ensure crack free welding and reduction in the rejection and repair of the welds. This also improves the understanding of the mechanism of hot cracking in the new generation alloys like IND9 and high nitrogen containing stainless steels. Hence this study can be considered as a technological milestone of high relevance to the Centre.

PUBLICATIONS

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3. G. Srinivasan, M. Divya, S.K. Albert, A.K. Bhaduri, A. Klenk and D.R.G. Achar, "Study of Hot Cracking Behaviour of Nitrogen enhanced Austenitic Stainless Steels using Varestraint and Hot Ductility Tests", 62nd Annual Assembly of International Institute of Welding, 53 (2009).

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Innovative Flow Injection Analyser for Quick Assay of Chemicals

EXECUTIVE SUMMARY

A unique low cost but high performing PC based Flow Injection Analyser (FIA) based on an entirely different approach has been designed and fabricated using in-house built pulsating sensors. The special feature in designing this unit is that it does not have any pump for circulation of liquid which is an essential component in flow injection analyser. The flow of liquid in the flow injection analyser is through gravity and constant flow rate, which is an essential requirement of flow injection analysis, is maintained by using a laboratory made pulsating level sensor. The level sensor senses a preset level of liquid including Demineralised (DM) water and controls the level of the liquid automatically through operation of solenoid valves. Hence circulation of liquid at a constant flow rate using gravity flow is possible. The unit consists of a small injector and a tiny conductance based flow type detector. The unit is mainly designed for rapid off-line analysis of HNO_3 in samples of different PUREX stream. About 5 micro litre of sample is required for each analysis and the analysis is completed within a minute once the unit is calibrated for that particular analysis.

OUTLINE

The Flow Injection Analyser consists of the following components – (i) carrier liquid reservoir (ii) intermediate level controlled reservoir housed with a pair of level sensing electrodes (iii) injection port and (iv) detector. All these components are connected with narrow uniform polyethylene tubes. For control and automated operation of the unit solenoid valves are provided. Figure 1 shows the schematic diagram of flow injection analyser. The flow injection analyser involves injection of a small volume of sample solution to a flowing liquid called carrier liquid within a narrow tube. The carrier liquid either reacts chemically with the desired species in the sample leading to advancement of the reacted solution front along the tube or it is non-reactive one, leading to forward movement of the mixed front (mixed diluted zone) along the tube in the direction of the flow of the carrier liquid. If an appropriate fast responding detector is placed in the downstream, its response can be followed in real time as the reacted or the mixed front passes through the detector. The desired analytical information can be derived from the detector response.

In flow injection analysis two important aspects must be considered in designing the unit in order to get reproducible quantitative information. Flow rate of carrier liquid should be constant and it is made by the use of a special type of pump in all commercially available units. But in our pump-less flow injection analyser the constant flow rate is maintained by gravity flow with controlled level indicator. The level indicator is set to a fixed threshold value depending upon the carrier fluid. Hence the level of carrier fluid is maintained at a constant level with the controlled action of solenoid valves. The 2nd requirement in designing the unit is that the injector and the detector should be properly positioned to generate narrow flow injection peak. If they are placed at a greater distance the response of the detector is flatter which will produce a broad peak. The entire operation starting from making the system ready for analysis, getting the flow injection analyser peak, instruction for next analysis and controlled action of solenoid valves is done by a PC using our own software in C language. Presently the unit is designed for quick assay of nitric acid in Reprocessing plant. With minor modification it can be used for some other application such as rapid analysis of boric acid in heavy water for PHWR.

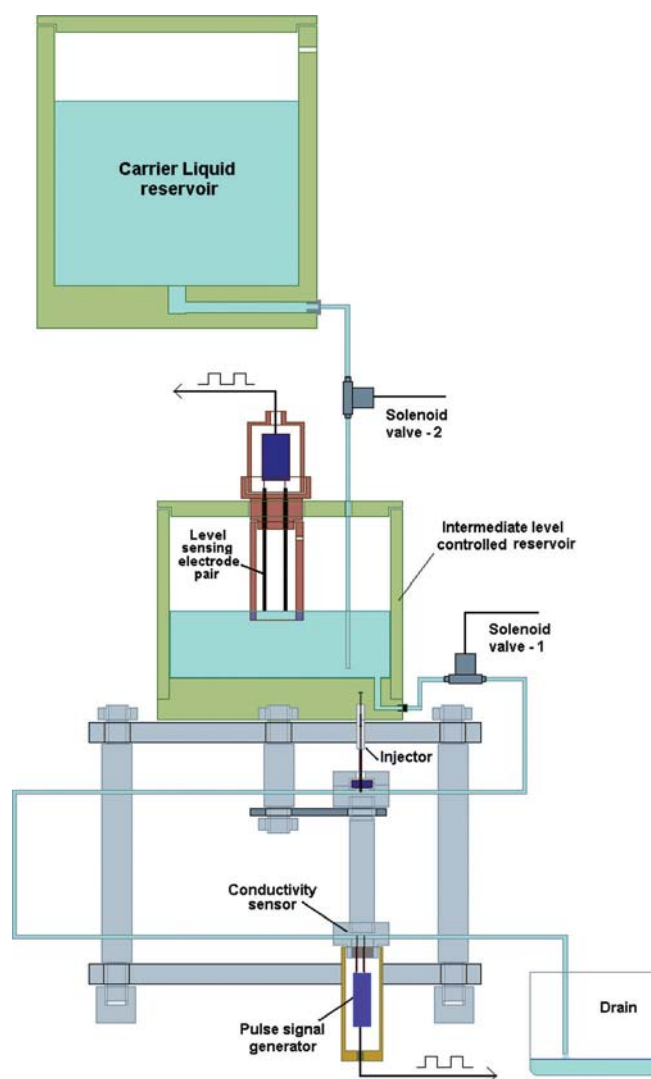


Fig. 1: Schematic diagram of Flow Injection Analyser:pulsating sensors based flow injection analysis

ADDITIONAL INFORMATION

The unit is designed in such a way that the small injector and the tiny flow type detector can be detached from the rest of the unit for housing in a fume board or in a glove box for analysis of radioactive samples

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

Flow Injection Analyser for Quick Assay of Nitric Acid

The performance of the unit was well studied for rapid analysis of HNO_3 with a view to applying this technique for routine monitoring of HNO_3 in PUREX stream of reprocessing plant. About 5 micro litre of sample is injected to distilled water stream which is flowing as the carrier fluid. The flow type conductivity sensor placed downstream picks up the conductivity profile of the moving diluted front. The response manifests the conductivity distribution across the moving front. Before injecting the sample a calibration plot is prepared by injecting HNO_3 of different concentrations. The relation between concentration and area of the peak is fed to the system software to get direct display of concentration of the unknown sample. The flow injection peaks recorded during three consecutive injections of 5 μL of 0.5M HNO_3 solution in distilled water as carrier fluid is shown in Figure 2. HNO_3 in PUREX stream generally contains fission products, corrosion products and other miscellaneous ionic components as impurities. Hence based on a typical data on composition for simulation of such components in HLW, obtained from the Reprocessing Group, the individual contributions to the total conductivity were estimated. The total conductivity is about $6000 \mu\text{S}.\text{cm}^{-1}$. A KCl standard corresponding to that conductivity was prepared. Five microlitres of pure KCl solution as well as same volume of HNO_3 and mixed ($\text{KCl} + \text{HNO}_3$) solutions were separately injected to assess the extent of interference. Figure 3 shows flow injection peaks of KCl as stimulant for total impurities associated with HNO_3 . From the computed peak areas in above measurements it is estimated that the contributions due to impurities would be about 3.5% when HNO_3 concentration is 0.5M and it decreases when HNO_3 concentration goes up (1% at 2M HNO_3). Hence the technique can be easily adopted for reprocessing application. Reproducibility of HNO_3 assay by flow injection analyser using our utility was found to be excellent.

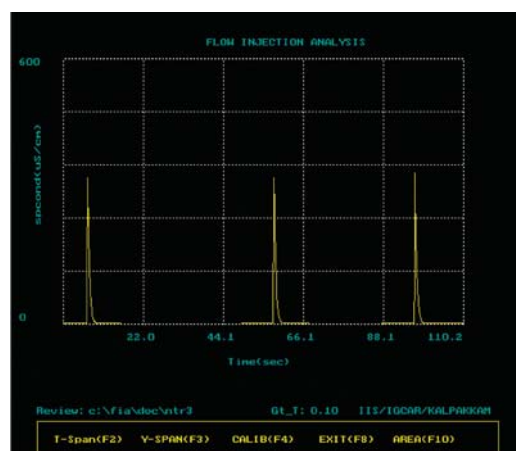


Fig. 2 : Flow injection peaks recorded during three consecutive injections of 5 μL of 0.5M HNO_3 solution using DW as carrier fluid

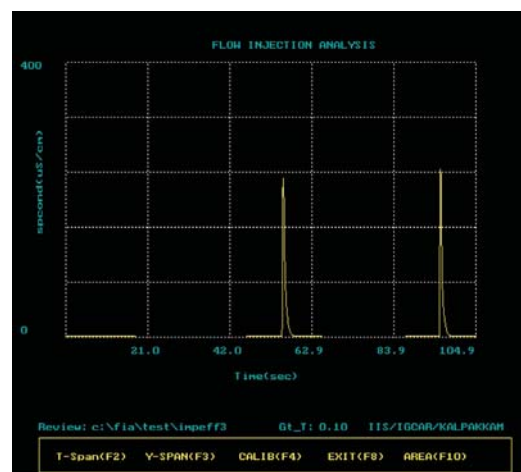


Fig. 3 : Flow injection peaks of KCl as simulated for total impurities associated with HNO_3

ACHIEVEMENT

Realisation of a new kind of inexpensive device for rapid offline chemical analysis could be possible by our team work in Innovative Instrumentation Section.

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A Novel Technique for Determination of Hydrazine in Spent Nuclear Fuel Dissolver Solution

EXECUTIVE SUMMARY

A unique potentiometric titration technique has been developed for rapid assay of hydrazine at ppm (mg/litre) level from a small volume (~2 ml) of aqueous solution in moderately concentrated HNO_3 medium. The methodology involved accurate determination of the end point in redox titration during electrochemical oxidation of hydrazine by potassium iodate solution in HCl medium. The importance of this work is that hydrazine which is difficult to be determined in the presence of nitric acid can be easily analysed by this novel REDOX titration technique. A few microlitre of sample solution is needed for determination of hydrazine and the titration is carried out in a total volume of 2 ml solution after chemical treatment within a couple of minutes. Hence this simple pulsating sensor based REDOX titration technique can be routinely adopted for rapid assay of hydrazine in fuel dissolver samples received from PUREX plants. For the first time a potentiometric titration in such a small volume of solution has been deployed for rapid chemical assay in radioactive solution. This technique has been successfully used to monitor hydrazine loss during electrolysis of fuel dissolver solution.

OUTLINE

We have developed a simple potentiometric titration technique using our laboratory made potentiometric titration facility with pulsating sensor for quick determination of hydrazine in spent nuclear fuel solution handling a small aliquot of sample.

The high performing potentiometric device in Innovative Instrumentation Section is based on the conversion of emf generated in an electrochemical cell into digital pulse frequency using a high sensitive in-house made v to f converter and subsequent capture of digital pulse frequency by a measuring device such as a PC. The digital pulse frequency which is directly proportional to the emf is fed to the Line Printer Terminal Port of a PC without using any interface card. The rapid potentiometric titration facility for chemical assay developed totally in-house, enables continuous online monitoring of emf in a redox system when a reactive chemical species (A) during reaction with another reactant (B) of known concentration, added in small fixed volume steps in quick succession using a Hamilton syringe or a laboratory made reagent dispenser. Figure 1 shows the schematic diagram of the titration facility. Principle of measurement technique with both mini cell and cell with Pt/SCE are presented in the Figure for comparison. A laboratory made typical miniaturized potentiometric titration cell fixed with a pair of bimetallic electrodes is used. Reactant B is added in small volume steps in quick succession into the titration cell. Every addition is followed by momentary generation and communication of a pulsed signal of a fixed frequency to the PC in second channel as the event marker. The redox electrode couple placed within the solution monitors potential once in 0.3 second (in case of automated addition) or in one second (in case of manual addition) as the reaction progresses. The potential change as a function of time is displayed graphically in 3rd channel and the event marker signals with reference to aliquot additions of solution B are recorded on 2nd channel. The titration is completed in less than two minutes and immediately after titration one can get the off line information of titration plot in volume domain i.e. volume of reactant versus emf and the first derivative plot (V Vs. dE/dV) from which the end point can be easily detected (Figure 2). This entire operation is done by using a user friendly software in MS-DOS mode indigenously developed in house.

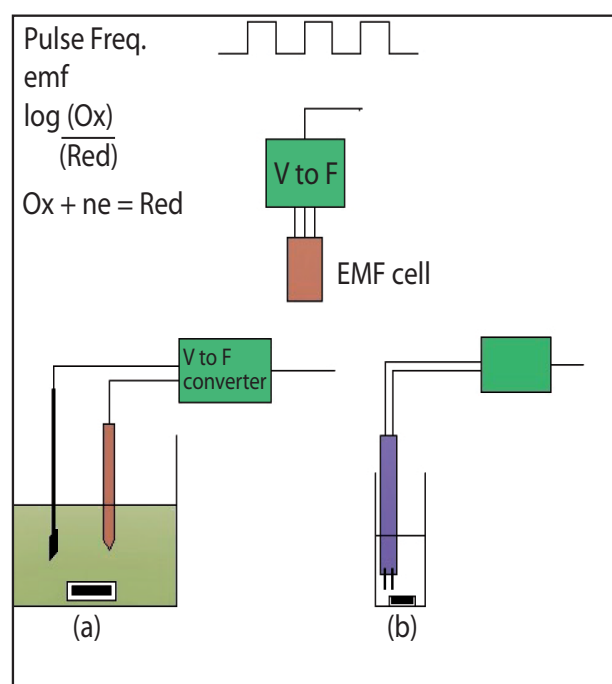


Fig.1: Schematic diagram of Potentiometric titration setup with principle of measurement

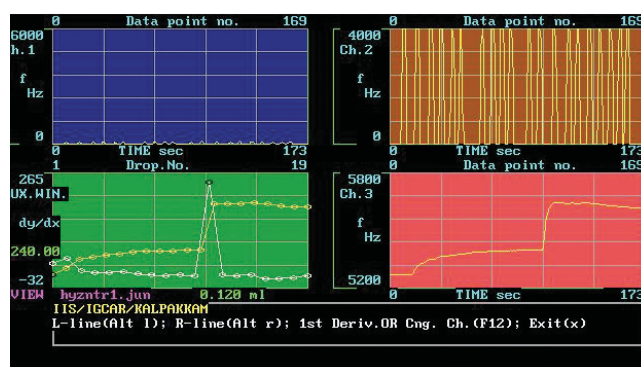


Fig. 2: Screen capture of a typical online titration plot

ADDITIONAL INFORMATION

To a few microlitre of standard hydrazine sample (30 -150 $\mu\text{g N}_2\text{H}_4$) in 1 ml 3N HNO_3 solution, 1 ml conc. HCl was added and allowed to mix well. After five minutes the mixture was quantitatively transferred to the laboratory made potentiometric mini cell. The beaker was rinsed with 0.5 ml distilled water and this was also transferred to the miniaturized potentiometric cell. The solution was allowed to stir well and titrated against 0.005M KIO_3 using potentiometric titration facility. The entire titration is completed within two minutes. From the end point as shown in the first derivative plot of Figure 2. One can easily calculate the concentration of hydrazine present in the sample. A typical online titration plot is also given in Figure 2. Precision of measurement using this technique is given.

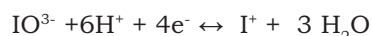
BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Volumetric redox titration technique using potassium iodate solution in 4-6N HCl medium as the titrant is commonly used for assay of hydrazine in hydrazine salts. For the first time we realized the power of this technique for assay of hydrazine in spent nuclear fuel solution using our unique in-house built titration facility. The electrochemical reactions of hydrazine with KIO_3 in HCl medium are given as follows:

Half reaction controlling the emf before end point:



Half reaction controlling the emf after end point:



ACHIEVEMENTS

This unique potentiometric titration technique using a minicell has been exploited for:

- (i) assay of uranium in fuel dissolver solution
- (ii) standardization of suitable conditions for electrolytic partitioning

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Development and Testing of High Performance Secondary Cold Trap

EXECUTIVE SUMMARY

Cold trap is an online purification unit which is used in sodium system of FBRs for maintaining the oxygen/hydrogen level in sodium within acceptable limits. A new design of secondary cold trap has been conceived to improve the trapping capacity and efficiency. The mathematical modeling of new design was carried out and results show that the trapping capacity is 54%. The experimental study on model cold trap was conducted and the total trapping capacity of model cold trap was found to be 60% and it is about three times more than the capacity of reference design (20%). The analysis of impurity deposition pattern from Neutron and X-Radiography indicates that model cold trap was not fully loaded/saturated with sodium hydride impurities and capacity of the new design cold trap could be higher by another 10-15%. This new design will minimize the frequent regeneration or replacement of cold trap and to improve the plant availability.

OUTLINE

In secondary sodium circuit cold trap, hydrogen impurities are more compared to oxygen, due to diffusion of hydrogen through tube wall into sodium in the steam generator. The cold trap load capacity of PFBR is designed for five years. The cold trap is regenerated and reused after every five years. In the reference design of secondary cold trap (Figure 1), the shell of the cold trap is cooled throughout its height and therefore the end temperature is expected to be at the bottom. Hence there is a tendency for the impurities to precipitate close to the bottom outlet pipe resulting in plugging of cold trap. The present design has a capacity of 20% of total wire mesh volume. A new design of secondary cold trap has been conceived (Figure 2) to improve the trapping capacity in order to minimize the frequent regeneration or replacement of cold trap for improving the plant availability.

The new design consists of a cooling zone at the top (for allowing deposition of sodium hydride) and an isothermal zone at the bottom (for deposition of sodium oxide) which gives an advantage of proper utilization of wire mesh and also avoids unwanted plugging at the outlet pipe. The cooling zone is restricted to the top half of the cold trap, thus allowing more residence time for the impurities to crystallize in the isothermal zone in the bottom half. In this zone, if a single concentric wire mesh is provided, there is a possibility of choking due to deposition in the outer region. With this in view, four concentric wire meshes with gap in between to allow free sodium flow has been considered in the new design. This increases the sodium entrance area to the wire mesh by nearly three times of the present design for crystallization of impurities. Also the wire meshes are graded, outer being coarse and inner being fine. In the cooling zone (upper half), trays with wire mesh having sufficient gaps for sodium flow have been provided. In order to evaluate the new design, model cold trap (Figure 2) has been fabricated and experimentally tested.

Description of work carried out

The experiments on model cold trap were conducted in the SOWART test facility. The flow sheet of the sodium loop with model cold trap in position is shown in Figure 3. In secondary sodium, major impurity load is due to diffusion of hydrogen through steam generator wall. An accelerated test of the model cold trap was conducted by injection of measured quantity of pure hydrogen gas into the sodium. The sodium impurity concentration during experiment was measured by using Hydrogen in Sodium Detection (HSD) and Electro Chemical Hydrogen Meter (ECHM). The hydrogen concentration in cover gas during experiment was monitored by using Thermal Conductivity Detector (TCD). The experiment was continued till the amount of hydrogen injection reaches the cold trap capacity of 60%, maintaining the same sodium flow rate through the cold trap. This shows that the capacity of model cold trap was nearly three times higher than present design (present capacity is 20%). Thus a new design of cold trap has been evolved and found to be performing extremely well which can be used to replace the cold trap for PFBR and also for future FBRs. The impurity deposition pattern in the model cold trap was studied by Neutron and X-radiography. The hydride deposition could be seen in the cooling zone (top trays) and also in isothermal zone. The observation from Neutron and X-Radiography indicates that model cold trap was not fully loaded/saturated with sodium hydride impurities. Based on these radiography images, capacity of the new design cold trap could be higher by 10-15%.

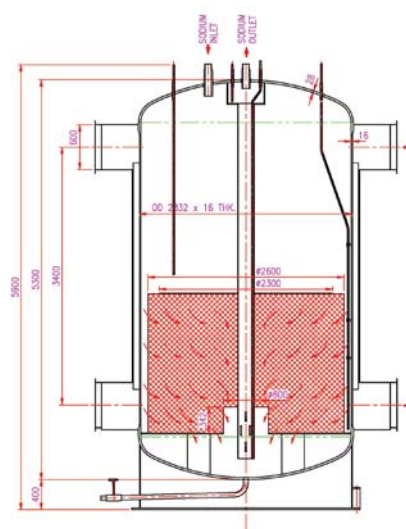


Fig.1: Reference design of cold trap

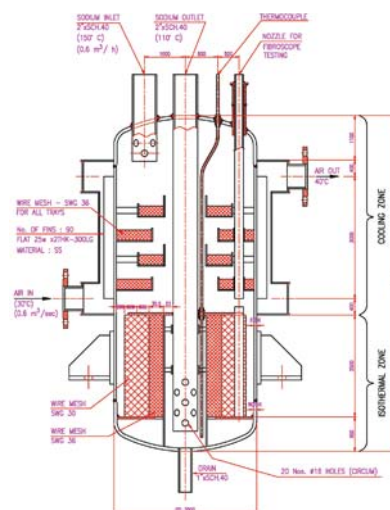


Fig. 2: Model cold trap for testing

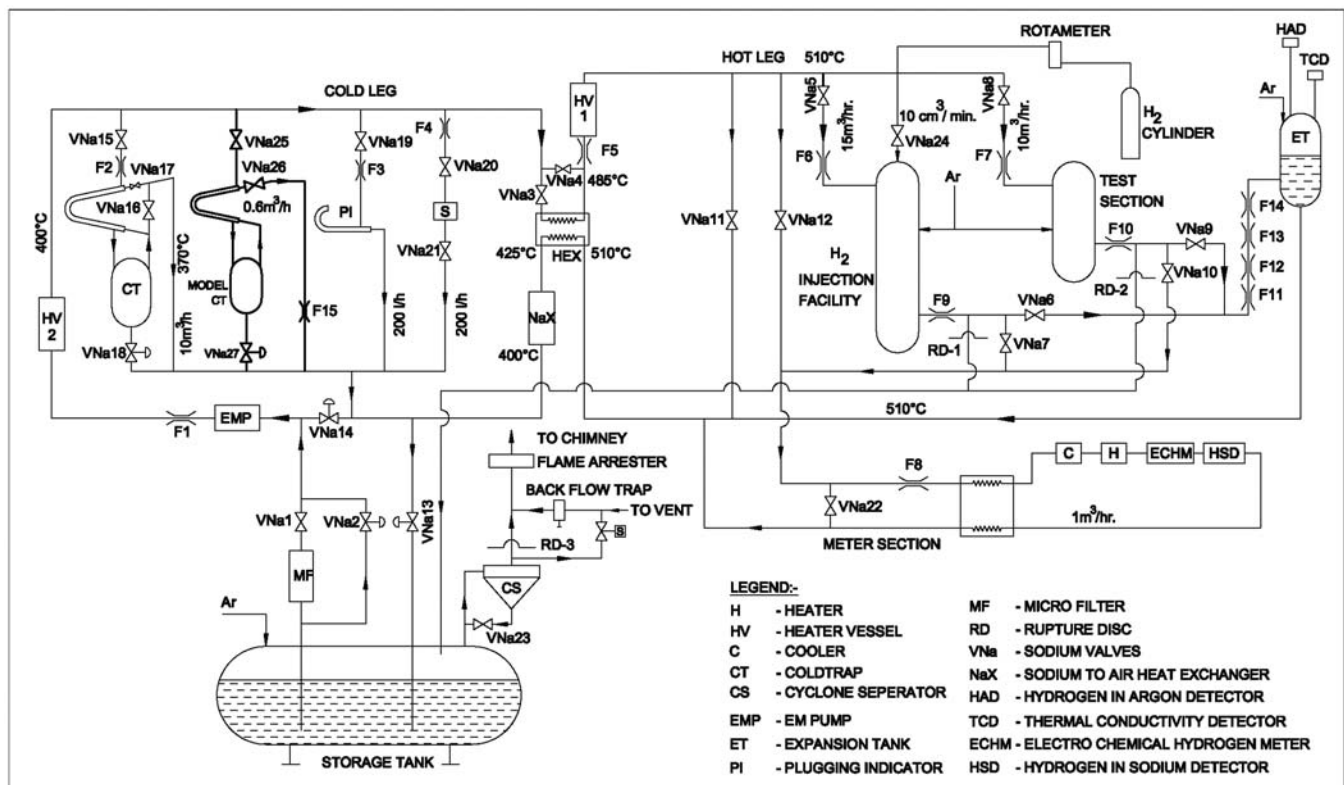


Fig. 3: Flow sheet of SOWART with model cold trap

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The mathematical modeling of scaled down model for the new design was carried out. The mathematical modeling result showed that the trapping capacity of the scale model is 54%. The theoretical results are in good agreement with experimental results.

ACHIEVEMENTS

The experiment on model cold trap was carried out and its trapping capacity was 60%, which is about three times more than the capacity of reference design of cold trap (20%). The observations from Neutron and X-Radiography indicate that model cold trap was not fully loaded / saturated with sodium hydride impurities. Based on these radiography images, capacity of the new design cold trap could be higher by 10-15% of experimentally reached capacity (60%). These new design will minimize the frequent regeneration or replacement of cold trap and improve the plant availability.

PUBLICATIONS

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2. M.G.Hemanath, T.V.Maran, Vishal Paunikar, S.Chandramouli, B.Muralidharan, K.K.Rajan, P.Kalyanasundaram, G.Vaidyanathan, "Manufacturing of cold traps for sodium systems", International Conference on Advances in Manufacturing Technology, ICAMT 2008, Chennai, India

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Qualification of Diverse Safety Rod Drive Mechanism and Diverse Safety Rod for Erection on Pile

EXECUTIVE SUMMARY

Diverse Safety Rod Drive mechanism (DSRDM) and Diverse Safety Rod (DSR) form a part of secondary shutdown system in Prototype Fast Breeder Reactor (PFBR). The safety of the reactor as a whole depends upon the reliable performance of diverse safety rod and diverse safety rod drive mechanism; they are qualified by comprehensive testing carried out in simulated reactor operating conditions. The root cause analysis was carried out for some of the problems encountered during testing and necessary modifications were incorporated. Testing carried out so far has qualified diverse safety rod drive mechanism for ten years of reactor operation and diverse safety rod for its full life in reactor.

OUTLINE

DSRDM was tested in Engineering Hall III (43 m tall building). The mechanism was initially assembled in a vessel, called inner vessel and the prototype DSR was mounted at the lower end of the same vessel. Air testing on DSRDM was carried out in the inner vessel. For carrying out testing in argon and in sodium at various temperatures from 473 K to 773 K, the inner vessel with the mechanism was lowered in a 1.0 m dia x 12.6 m long sodium vessel, called Test Vessel-1 (TV-1). Sodium was filled in TV-1 to a specified height and maintained at different temperatures so as to simulate reactor operating conditions.

Initially extensive testing was carried out on prototype DSRDM and DSR to check and ensure all the intended functions in air and then in hot argon at 473 K. then the whole system was tested in sodium at temperatures starting from 473 K to 773 K. Subsequent to functional tests, endurance testing was carried out. The mechanism was subjected to endurance testing with respect to scram operation and translation operations as the damage occurring in DSRDM & DSR is mainly due to fatigue cycles during these operations.

Qualification testing of DSRDM was completed in three campaigns of sodium testing. Design of DSR was improved based on the experience gained during the first two campaigns.

The required number of cycles for qualification during endurance testing for forty years operations was arrived at based on the guidelines of ASME. During endurance testing, proper functioning of DSRDM & DSR was monitored by measuring the free fall & braking times (during scram tests) and also frictional force. Thus, the endurance testing carried out so far has qualified the DSRDM for 10 years of reactor operation, while DSR is qualified for its full life of 2 yrs.

DSRDM and DSR were taken out of the sodium vessel and cleaned thoroughly using water vapour-CO₂ process, DM rinsing and drying at sodium removal facility. Later, the upper part of the mechanism was dismantled fully for inspection. Inspection reveals that no part in DSRDM upper part related to translation has undergone any deterioration that may affect the satisfactory operation of the mechanism. Figure 1 shows the displacement characteristics of DSR during scram action and Figure 2 shows the variation of free fall time and braking time with respect to drop number during endurance testing.

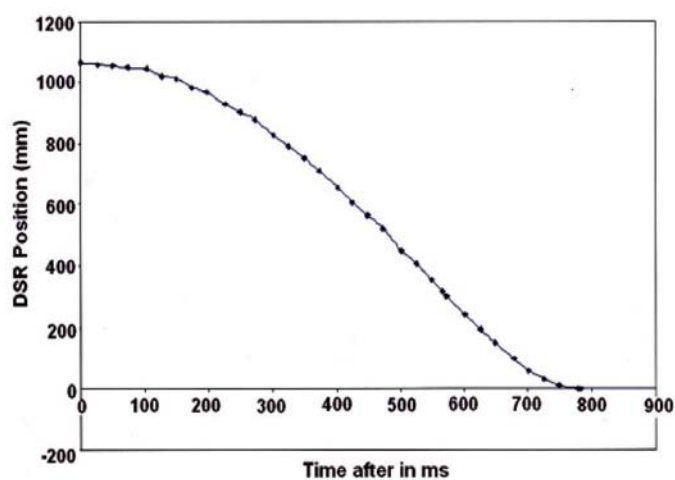


Fig .1: Displacement characteristics of DSR

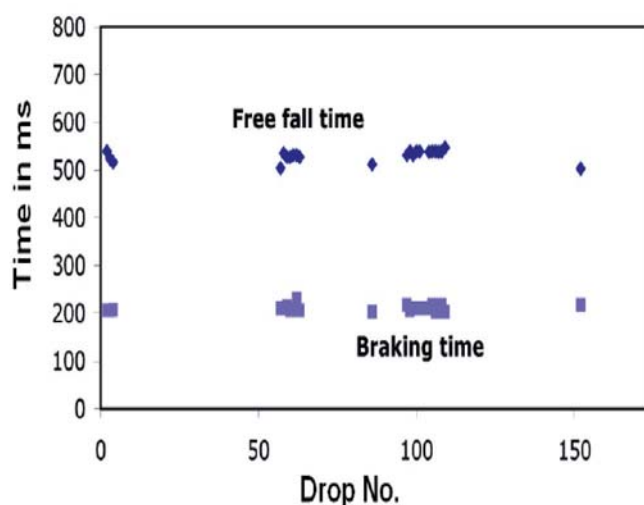


Fig. 2: Variation of free fall time & braking time of DSR

GENERAL DESCRIPTION OF DSRDM

The photograph of DSRDM is provided in Figure 3. Overall length of DSR & DSRDM is about 16.5 m, and the maximum diameter is 250 mm. The length of DSR Subassembly is 4.46 m. Mobile DSR (DSR) moves inside the stationary sheath and there is a nominal gap of 5.45 mm between DSR and stationary sheath. A sodium dashpot is provided at the bottom of the sheath to decelerate the m- DSR during the scram operation. DSR travels upto a maximum of 1075 mm from its bottom most position. The universal joint at the top of DSR allows DSR to hang freely from the Electromagnet (EM) of DSRDM.

Mobile Assembly of DSRDM consists of translation tube & EM. EM is attached to the bottom end of the translation tube. Roller screw-nut mechanism is used for raising & lowering of the mobile assembly of DSRDM. Mobile assembly is connected to the nut of the roller screw. Motor Drive subassembly rotates the Translation Screw to raise & lower the mobile assembly. When DSR is held by EM, it also translates up & down based on the direction of rotation of motor. Mobile assembly slides over two guide columns fixed to support tube. Teflon buses are provided at the contact surfaces. The mobile assembly is also guided by three bushes, two of nitrided SS at the level of V- ring seal block (Top guides), and the other of Colmonoy in sodium at the lower end of Tube Sheath, (sodium guide).

On receiving the scram signal, the EM is de-energised and the mobile DSR is released to fall under gravity. It falls down freely from its operating position. At the end of free fall travel, DSR is decelerated by sodium dashpot for the remaining 250 mm travel.



Fig. 3: Diverse safety rod drive mechanism

ADDITIONAL INFORMATION ABOUT PFBR SHUT DOWN SYSTEMS

In Prototype Fast Breeder Reactor, there are two independent, fast acting, diverse shutdown systems, each comprising of sensors, logic circuits, drive mechanisms and neutron absorber rods having boron carbide pellets. The absorber rod of the first system is called Control and Safety Rod (CSR) and that of the second system is called Diverse Safety Rod. The respective drive mechanisms are Control and Safety Rod Drive mechanism (CSRDM) and Diverse Safety Rod Drive mechanism. There are nine control and safety rods and three diverse safety rods in the core. Each system is capable of bringing down the reactor to cold shutdown irrespective of the other.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Large displacement structural analysis was carried out to estimate the buckling susceptibility of mobile assembly. Bending analysis was carried out to assess the lateral stiffness of DSRDM, reactive forces at various guides and consequently frictional forces on mobile assembly. Thermal hydraulic analysis was done to assess the transient temperature evolution in the electromagnet assembly after scram operation. This analysis forms the basis for calculating the fatigue loads on the dissimilar welds in electromagnet. Analysis was carried out to estimate the margin available against the flow induced vibrations of DSRDM. An in-house program was developed to estimate the pressure drop in diverse safety rod subassembly and consequently diverse safety rod free fall & braking times.

ACHIEVEMENTS

The design of DSRDM is first of its kind in the Indian context. The testing carried out has qualified the system for use in reactor and provided basis for licensing the erection of DSRDM and diverse safety rod on reactor pile. The best performance of the system has been demonstrated by the synergetic efforts of multidisciplinary specialists.

PUBLICATIONS

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2. R.Vijayashree, R.Veerassamy, Sudheer Patri, S.Suresh Kumar, SCSP Kumar Krovvidi, S.K.Dash, T.Logaiyan, N.Ravichandran, S.Chandramouli, K.K.Rajan, Indranil Banerjee, R. Dhanasekaran 'Testing and qualification of Diverse Safety Rod and its Drive Mechanism for PFBR', 17th International Conference on Nuclear Engineering (ICONE-17), Brussels (Belgium) 3 (2009) 979.
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Performance Assessment of Prototype Eddy Current Position Sensor in Sodium for Diverse Safety Rod of Prototype Fast Breeder Reactor

EXECUTIVE SUMMARY

Sodium testing of eddy current position sensor model for diverse safety rod of Prototype Fast Breeder Reactor was carried out successfully. The Diverse Safety Rod Drive Mechanism (DSRDM) is an important safety system of PFBR. During reactor operation, the Diverse Safety Rod (DSR) is held above the reactor core by an electromagnet provided in DSRDM. On receiving the SCRAM signal, the electromagnet de-energizes, thus releasing the diverse safety rod, which falls under gravity in reactor core. It is a safety requirement to detect whether the DSR has reached the bottom-most position or not. Eddy Current Position Sensor (ECPS) which works on principle of variation in inductance is developed to detect diverse safety rod position and its travel time.

OUTLINE

Eddy current position system was conceptualised to detect DSR position and to determine drop time. This report deals with the performance testing in sodium of 1:1 model of eddy current position sensor, that has been developed (Figure 1) to overcome the following challenges: (i) No direct electrical or mechanical contact is allowed as there is no provision of taking out the signal cables from the dash-pot region; (ii) The system is required to provide information for all the three DSRs independently; (iii) The system is required to work in liquid sodium at temperatures up to 600°C.

Eddy current based system called Eddy Current Position Sensor (ECPS) was conceptualised and model was developed to detect DSR position and its drop time. The schematic of the latest version of the system is shown in Figure 2. The system consists of one primary winding excited by an alternating constant current source and two pick-ups which are placed above the electromagnet in the DSRDM. The secondary winding and the sensor winding are placed inside the hexagonal sheath which houses the DSR in the reactor core. There is no direct connection between the windings of the primary side which is on the moving mechanism and those on the stationary secondary side and working of the device is based solely on the electromagnetic coupling between the windings. The bottom of the DSR consists of a ferromagnetic piston and during a SCRAM when the rod falls, this piston gets deposited in the sodium dashpot. The impedance of the sensor coil which is located in the dashpot region changes when the DSR piston having magnetic material gets deposited in the dashpot. This change of impedance in sensor coil induces a change in voltage in the two pick-ups on the primary side and serves as indication of the presence of the DSR in the dashpot region. Two pick-ups are provided to take care of the axial misalignment (up to 40 mm) that takes place between the primary and the secondary side windings. Voltage signal from pick up coils are recorded and change in voltage signal facilitates detection of the position of DSR. The same voltage signal is used for drop time measurement also. The above design was first tested in air in laboratory and then a 1:1 scale engineering model was fabricated and tested in sodium at various temperatures. The sensitivity of the designed system which is a measure of detectability of the system was obtained through the experiments in sodium and is shown in Figure 3. The sensitivity of the system is defined as

$$\text{Sensitivity} = \frac{100 * (\text{Pick up Voltage with DSR Piston} - \text{Pick up Voltage without DSR Piston})}{\text{Pick up Voltage without DSR}}$$



Fig. 1: ECPS set up

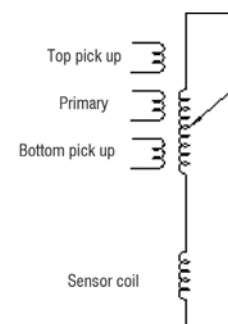


Fig. 2: Schematic of Eddy Current Position Sensor

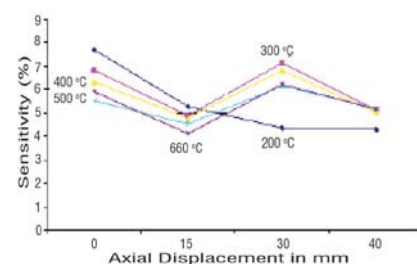


Fig. 3: Sensitivity plot of ECPS

GENERAL EXPLANATION RELATED TO DESCRIPTION

The shutdown system of PFBR consists of two different types of shutdown mechanisms, one the control and safety rod drive mechanism (CSRDM) which is used for controlling the reactor power during normal operation as well as for shutting down the reactor in case of a SCRAM. The other shutdown system is the Diverse Safety Rod Drive Mechanism (DSRDM) which is meant solely for shutting down the reactor in case of a SCRAM. The DSRDM consists of an electromagnet which holds the Diverse Safety Rod (DSR) and a drive mechanism for positioning the DSR. When a SCRAM takes place the electromagnet gets de-energised resulting in falling of DSR under gravity and getting deposited in the dash-pot. The drop time of the control rods can be determined by the limit switches provided in the top of the CSRDM whereas the DSRDM does not have this feature. Ascertaining that the DSR has reached to the bottom most position and the drop time is within limits ensures the healthiness of the system.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The eddy current position sensor works on the principle of change of inductance. The bottom of the diverse safety rod consists of a ferromagnetic piston and during a SCRAM when the rod falls, this piston gets deposited in the sodium dashpot. The impedance of the sensor coil which is located in the dashpot region changes when the diverse safety rod piston having magnetic material gets deposited in the dashpot. This change of impedance in secondary side induces a change in voltage in the two pick-ups on the primary side and serves as indication of the presence of the DSR in the dashpot region.

ACHIEVEMENTS

An indirect method to detect position of diverse safety rod based on eddy current and variation in inductance is developed which overcomes constraints of no direct electrical connection between moving and stationary parts, high temperature sodium atmosphere and space constraints in the reactor. Successful engineering model testing in sodium of eddy current position sensor to detect DSR position is a harbinger towards implementation of this system in future FBRs.

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Compton Profile Spectrometer Facility for Electronic Structure Studies

EXECUTIVE SUMMARY

A ^{137}Cs high-energy γ -ray Compton spectrometer first of its kind in India has been built. A program for computing the Compton profile from the raw data and for transforming the profile yielding the autocorrelation function has been commissioned. The facility has been successfully employed for measurement of isotropic Compton profiles of NaCl, NaF and LiF and for measurement of isotropic and directional Compton profiles of few 3d and 4d metals, GaAs semiconductor, few ionic solids and some shielding materials. To compare the experimental data, we have computed the spherical average Compton profiles using self consistent Hartree- Fock wave functions employed on linear combination of atomic orbital (HF-LCAO) approximation. The directional Compton profiles and their anisotropic effects are also calculated using the same HF-LCAO approximation.

OUTLINE

An indigenous high-energy γ -ray Compton spectrometer consisting of ^{137}Cs radiation source camera, target mount, target scanning facility, shielding system, beam guides, detector platform, vacuum chamber, a planar HPGe gamma spectrometer has been designed, built and in-house deployment has been done and is shown in Figure 1. The achievable momentum resolution of the spectrometer is 0.39 a.u. A Fortran program (PC based) for computing the Compton profile from the raw data generated by a inelastic gamma ray scattering experiment using a solid state detector and a multichannel analyzer has been commissioned. It also Fourier transforms the profile yielding the autocorrelation function. The performance of the spectrometer has been studied and the equipment has been tested for its potential use in Compton profile studies by making a measurement on polycrystalline aluminum. The results for the Fermi momentum is 0.93 a.u. determined from the experimental autocorrelation function, $B(r)$ agrees fairly well with theories.

The spherical average momentum distributions of polycrystalline Al, Ni, Cu, Cd, Pb, CaF_2 , BaF_2 , NaCl, NaF, LiF, Al_2O_3 and the directional profiles of GaAs along the principal direction [100] have been measured at 662 keV gamma energy and compared with the theoretical values. The measured spherical average momentum distributions are in good comparison with theoretical values for LiF. The theoretical spherical average Compton profiles and the directional profiles of NaCl, NaF and LiF along the three principal directions namely [100], [110] and [111] are calculated using the CRYSTAL98 suite program. The anisotropy curves of the NaCl and LiF are shown in Figures 2 and 3 respectively. An analysis of data reveals that the high momentum anisotropy for NaCl, NaF and LiF is mainly due to [100]. The most pronounced effects can be found in the low momenta region. The fact that the [110]-[111] anisotropy in the case of LiF is very small in the high momentum region is due to the fact that both the directions cut the [100] bond at roughly the same angle, and thus to the first-order the bond look same. Further for the difference Compton profile [100]-[111], a significant effect is seen for momentum values $q \leq 1.8$ a.u., where a pronounced minimum is observed around $q = 1.4$ a.u. However, the $J[110]-J[111]$ curve has an amplitude roughly one half of the amplitude of the other two anisotropy curves. Thus, one can conclude that the crystal direction [100] is the nearest neighbor (Li-F), which has been found to be special one. A satisfactory overall agreement is seen between calculated and measured anisotropies. The experimental spherical average profiles are found to be in good agreement with the corresponding HF-LCAO calculations.



Fig. 1: Compton profile spectrometer

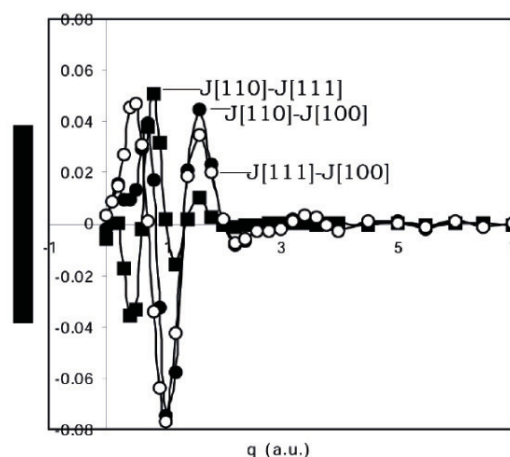


Fig. 2 : Anisotropy curves of the Compton profile of NaCl. circle represents the $J[110]-J[100]$ anisotropy, the line with solid circle represents the $J[111]-J[100]$ anisotropy and the line with square represents the $J[110]-J[111]$ anisotropy respectively

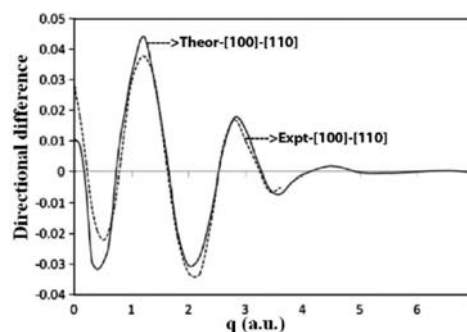


Fig. 3: Anisotropy curves, $J[100]-J[110]$ of the Compton profile of lithium fluoride

ADDITIONAL INFORMATION

The Compton scattered radiation is Doppler broadened because of the motion of target electrons. An analysis of this broadened line shape gives valuable information about the momentum distribution, $n(p)$, of electrons through the Compton profile, $J(p_z)$. Thus the Compton profile, $J(p_z)$, is the projection of the electron momentum density $n(p)$ along the scattering vector (usually z -axis of a Cartesian coordinate system). The Compton profile is directly obtained from the spectral distribution of photons emitted by a monochromatic radiation source and scattered at a fixed angle.

GENERAL DESCRIPTION

Importance of Compton profile measurements

Measurements of the Compton profile provide valuable information about the momentum distribution of the scattering electrons and the ground state electronic wave function in the scatterer. These measurements are particularly sensitive to the behaviour of outer, loosely bound conduction or valence electrons. Since many of the macroscopic properties are determined by the distribution of valence-band electrons, a detailed experimental investigation of the electronic structure is of fundamental interest. The analytical capability of the spectrometer to distinguish and differentiate between different materials particularly low- Z elements is an additional benefit and the measured Compton profiles of each element are found to be distinctly different and are shown in Figure 4.

Present interest and scenario

Increasing interest has been directed towards the experimental and theoretical determinations of isotropic Compton profiles and anisotropies in the directional Compton profiles. The Compton profile studies on ionic solids, particularly alkali halides with NaCl structure, have been the subject of great interest both experiment and theoretically. Amongst all the alkali halides the fluorides have been theoretically predicted to give larger anisotropy and it has been shown that the relative anisotropy of fluorides decreases as one goes from lithium to higher alkali atoms.

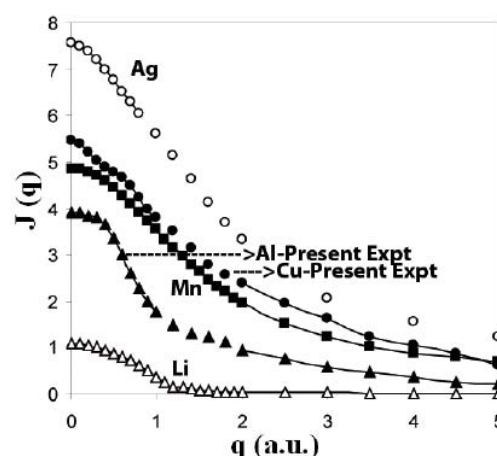


Fig. 4 : Compton profile of Li, Al, Cu, Mn and Ag

BRIEF DESCRIPTION ON IMPACT TO FBR PROGRAMME

The spherical average and directional Compton profiles of LiF are calculated using the CRYSTAL98 suite of program. Crystalline orbitals were generated from self-consistent Hartree-Fock method applied to linear combination of atomic orbitals (LCAO).

The calculation was performed at the characteristic crystal structure (rock salt) using the experimental lattice parameters ($a = 4.0173\text{\AA}$) according to Wyckoff. Default tolerances for the calculations were used in the calculation, with reciprocal space integration being performed at twenty nine points in the irreducible wedge of the first brillion zone. Calculation was performed with high quality all-electron basis sets, namely 6-1G for Li^+ , and 7-311G for F^- . All these basis sets have previously been optimized by the authors of CRYSTAL98 for the systems studied in this work.

ACHIEVEMENT

The first ever ^{137}Cs Compton spectroscopy study of LiF accomplished with improved momentum resolution of 0.39 a.u. and the HF-LCAO theoretical results are tested for the first time at this energy. The analytical capability of the spectrometer to distinguish and differentiate between different materials particularly low- Z elements has been demonstrated.

PUBLICATION

1. R. Vijayakumar, Shivaramu, N. Ramamurthy, M.J. Ford Compton scattering study of electron momentum distribution in lithium fluoride using 662 keV gamma radiations". Physica B 403 (2008) 4309-4313
2. R. Vijayakumar, Shivaramu, L.Rajasekaran, N. Ramamurthy and M.J. Ford "Compton profile of polycrystalline sodium chloride and sodium fluoride". Nuclear Instruments and Methods in Physics Research - B234 (2005) 185 -193

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Single Fiber Grid with Improved Spatial Resolution in Distributed Fiber Sensor

EXECUTIVE SUMMARY

Raman distributed temperature sensor (RDTS) is widely used for temperature monitoring of oil pipelines, power cables, coolant loops of nuclear reactors and in other industrial applications. However, the spatial resolution of raman distributed temperature sensor is limited by the pulse width of the laser used. For laser with pulsewidth of 5 nanosecond, raman distributed temperature sensor has a spatial resolution of one meter. In this paper we demonstrate improvement of spatial resolution using single fiber grid. To improve the spatial resolution of the raman distributed temperature sensor laying fiber as a “single fiber grid” is proposed, and a methodology to reconstruct temperature with high spatial resolution is demonstrated.

OUTLINE

The spatial resolution of Raman distributed temperature sensor is limited by the pulse width of the laser used. For a 5ns pulse width laser, the spatial resolution is 1m. To improve the spatial resolution, a single fiber grid (Figure 1) is envisaged. The grid is of length 2m x 2m, with size of the mesh is 10cm. It is assumed that the size of the grid is always greater than the spatial resolution of the RDTS. The principle is analogous to tomography, where image is reconstructed from many projections. Unlike tomography, here there are only two projections. A single heat spot appears as two measurements along the length of the fiber.

Consider three local regions heated on the grid at (12,20), to 60°C, 90°C and 120°C. Since, the temperature measured in the optical fiber is an integral average over a length equal to the spatial resolution of the Raman distributed temperature sensor, the temperature profile is spread over the grid, as seen in Figure 2a. It can be seen that there are more than three measurement points. Thus, this Raman distributed temperature sensor measurement is said to have true and spurious temperature points. The temperature measurement is underestimated by Raman distributed temperature sensor due to the averaging effect.

The number of true and measurement points is reduced to few points through a minimization procedure, and the true points are identified from the set of true and spurious temperature points using a search algorithm. Temperature is reconstructed using the true measurement points thus found and is shown in (Figure 2b). It can be seen clearly, the three measurement points at (12,20), is reconstructed, and the temperature is close at 60°C, 90°C and 120°C, as expected.

The ratio of the intensities of the Anti-Stokes to the Stokes line shows temperature dependence, and is given by:

$$\frac{I_a}{I_s} = \frac{(n_o + n_k)^4}{(n_o - n_k)^4} \frac{(-hcn_k)}{kT}$$

I_a - intensity of Anti-Stokes-component, I_s - intensity of Stokes-component, n_o - light wave number, n_k - shift of light wave number

h - Planck action quantum, c - velocity of the light within the optical fiber, k - Boltzmann - constant, T - temperature

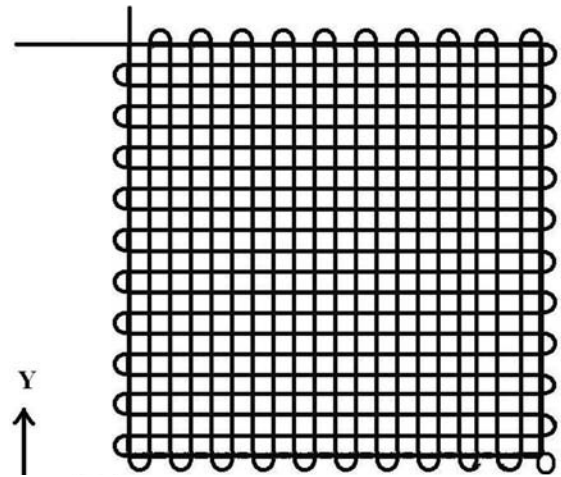


Fig. 1: Fiber laid as Single fiber grid

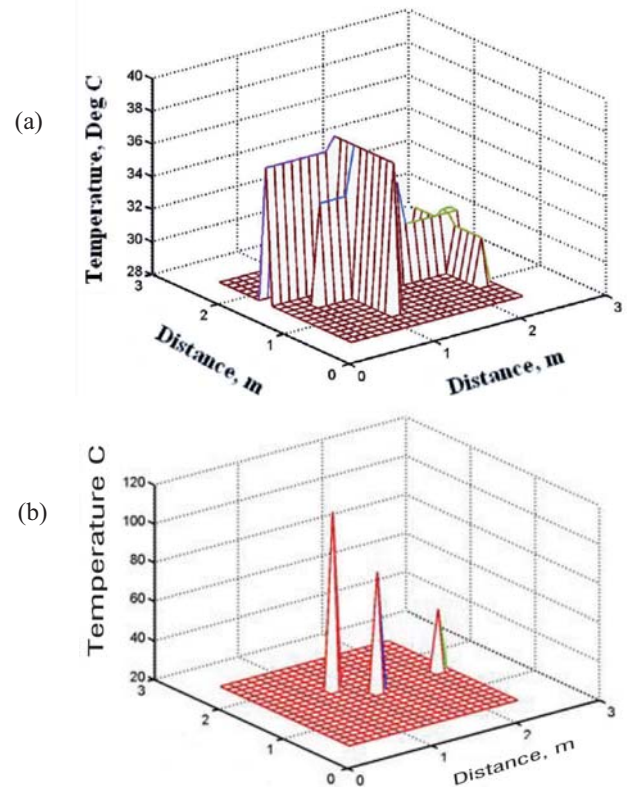


Fig.2: a) Temperature profile on fiber grid as seen by Raman Distributed Temperature Sensor
b) Temperature reconstructed

ADDITIONAL INFORMATION ABOUT FIBER OPTIC SENSORS

Optical fiber based distributed sensor have been widely used to monitor temperature. The main advantage of the system is that the fiber itself is the sensing element. Distributed temperature sensing technology shows real advantages over conventional temperature sensing technology when a temperature profile of the installation is required or when a large number of sensing points is crucial. Therefore this technology lends itself to long length applications (pipelines, tunnels, power cables, conveyor belts), applications where only small sensors can access (oil wells) and safety critical applications where it is important to have all points monitored (refineries, LNG plants, electrochemical processes).

GENERAL INFORMATION

Raman scattering based distributed temperature sensor comprises the following. A pulsed laser is injected into the optical fibre which is the sensing element. In the fibre the photons interact with the molecules of the fibre material. The spectrum of the backscattered light includes the Rayleigh, the Brillouin and the Raman backscattered light. The Raman backscattered light is caused by thermally influenced molecular vibrations. Consequently, the Raman backscattered light carries the information on the temperature of the fibre and can be used to obtain information about the temperature distribution along the fibre. The Raman backscattering light has two components: the Stokes (Is) and the Anti-Stokes (Ia) component (Figure 3). They can be separated from the primary and the Rayleigh backscattered light due to their differences in wavelength. The Stokes component is only weakly dependent on temperature, while the Anti-Stokes component shows a strong relation to temperature. The ratio of the intensities of Stokes and Anti-Stokes components is a measure of temperature. Since the injected light is a pulse of a few nano seconds, the time of arrival of instantaneous back scattered intensity can be correlated with the distance along the fiber length from where it is scattered.

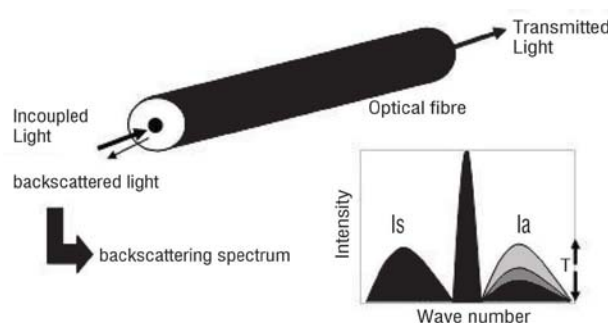


Fig. 3: Measuring Principle

ACHIEVEMENT

Improvement of spatial resolution of Raman distributed temperature sensor upto 10 cm has been demonstrated using this methodology.

PUBLICATIONS

Single fiber grid with improved spatial resolution in distributed optical fiber sensor system. Patent applied

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Design of Innovative Welded Grid Plate for Future Fast Breeder Reactor

EXECUTIVE SUMMARY

An innovative welded grid plate concept has been conceived for future FBRs, as one of the measures to improve economy. The grid plate has sleeves only for supporting and distributing sodium flow for core subassemblies that need cooling and hence the shell and bottom plate are of smaller diameter. For other subassemblies, suitable holders (spikes) are provided that are welded to the top plate. Eight primary pipes are used with smaller diameter. These features have resulted in reduced overall height and diameter of grid plate. The reduction in weight due to the innovative features is about 55%, with reduction in overall diameter by ~700 millimeter, height by 315 millimeter, thus giving significant economic gains.

OUTLINE

Grid Plate of PFBR of 6.8 m diameter, 1 m height & 76 tonn weight has adopted bolted concept, which is selected to indigenously manufacture the same with proven technology present at the time of its conceptual design. For future FBR, many innovative features are incorporated in the conceptual design that has resulted in its overall reduction in diameter (by 700 mm) & height (by 315 mm) and significant reduction in its weight (by more than 50%) that contributes to the economy of the future FBRs. Also, welded concept is selected to exploit the developments that have taken place in Indian industry in the field of non-conventional welding technology.

Core subassemblies such as fuel, blanket, absorber rods, reflector, inner B_4C , etc., and those in storage location require forced cooling, unlike the shielding subassemblies. In PFBR Grid Plate all subassemblies are supported on sleeves. For innovative welded grid plate, sleeves are provided only for support and distributing coolant flow for subassemblies that require forced cooling and for two inner rows of shielding subassemblies. The sleeves for two inner rows of shielding subassemblies act as coolant distribution arrangement to ensure uniform flow distribution in grid plate. Other shielding subassemblies are supported on holders welded to top plate. Also, one row of shielding subassemblies is removed as compared to PFBR grid plate due to the use of ferro boron in shielding subassemblies. The welded concept eliminated the need for flanges and fasteners.

These features resulted in the reduction in overall diameter of the shell assembly and bottom plate from 6.8 to 4.6 m, while the top plate diameter is reduced from 6.8 to 6.1 m (Figures 1 & 2). The top plate has larger diameter than bottom plate to accommodate the spikes for shielding subassemblies. The shell diameter of 4.6 m is selected such that the moment on the top plate due to subassemblies supported on sleeves and that supported on spikes balance each other, which is arrived at based on a preliminary stress analysis. The reduction in diameter, height and replacement of most of the shielding subassembly sleeves by spikes has significantly reduced the weight of grid plate for future reactors by 55% (Spikes weigh about one third of the corresponding sleeves in PFBR Grid Plate).

In PFBR, two primary pipes are provided from each primary sodium pump for entry of coolant sodium to grid plate. For future reactors, four pipes are provided from each pump, resulting in reduction of grid plate height by about 315 mm. This feature has an added advantage that in case of rupture of one pipe the core flow bypassing is reduced, which is also a safety improvement. The in-vessel transfer port (IVTP) is present in the PFBR grid plate for aiding the fuel handling operation, which makes the top plan of sleeve arrangement in grid plate unsymmetrical. For future reactors, the IVTP is shifted to inner vessel, making the sleeves arrangement symmetrical.

A detailed technology development work has been under taken (with all parts in full size, but number of sleeves & spikes reduced (Figure 3)) to validate and confirm the design and manufacture, which involves non-conventional welding processes such as laser beam / electron beam welding to ensure the tolerances on the assembly of grid plate.

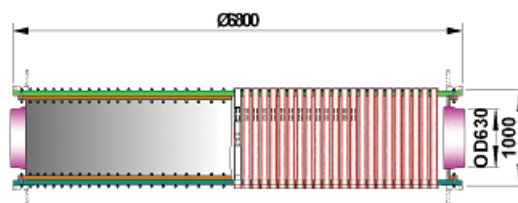


Fig. 1 : PFBR Grid Plate (Wt. 76 t, 1758 Sleeves)

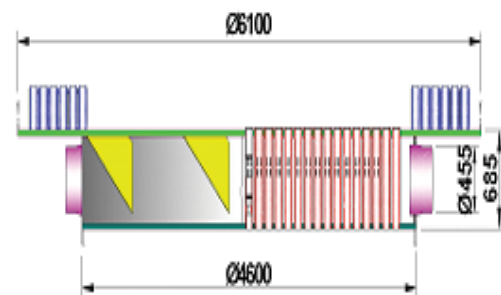


Fig. 2 : Innovative Welded Grid Plate (Wt. 34 t, 929 Sleeves, 746 Spikes)

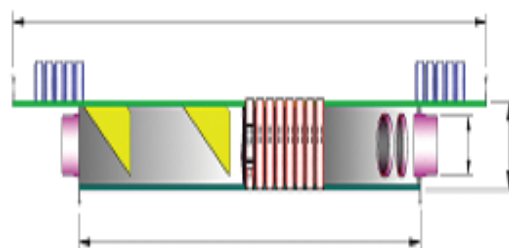


Fig. 3 : Technology Development Component (Wt. 22 t, 238 Sleeves, 57 Spikes)

ADDITIONAL INFORMATION ABOUT GRID PLATE

Grid Plate of a sodium cooled pool type FBR supports the core subassemblies (CSA), maintains their verticality (for proper functioning of absorber rod drive mechanisms and fuel handling machine) and acts as a plenum for coolant distribution to core and for cooling main vessel and supports inner vessel. Grid Plate is supported on core support structure, through which the entire load of core and inner vessel is transferred to main vessel. It consists of perforated top and bottom plates interconnected by shell at periphery and sleeves at perforations. Core subassemblies are supported on sleeves. Sodium from primary sodium pumps enter the grid plate through nozzles provided in the shell.

GENERAL DESCRIPTION OF PFBR GRID PLATE AND INNOVATIVE WELDED GRID PLATE

The PFBR Grid plate consists of a top plate and a bottom plate both perforated and having 6.8 meter diameter (Figures 1, 4 and 5). They are interconnected at periphery by shell assembly using bolted joint and at perforations by 1758 sleeves using threaded joints with plates. There are four nozzles welded to shell assembly. Grid Plate is supported on core support structure by bolted joint at periphery and at eighty two numbers of intermediate supports over core support structure.

The innovative welded grid plate has a top plate of 6.1 meter diameter, bottom plate and shell of 4.6 meter diameter (Figure 2). Shell is concentric and welded to top and bottom plates. Inside the shell, the top and bottom plates are perforated in triangular pattern and are welded to 929 sleeves. 746 spikes are welded to the top side of top plate in the region outside the shell. The overhanging portion of top plate is supported by gusset plates welded between shell and bottom surface of top plate. Eight nozzles are welded to the shell. The periphery of bottom plate of grid plate is welded to the top shell of core support structure.



Fig. 4 : PFBR Grid Plate under Assembly in Nuclear Clean Hall



Fig. 5 : PFBR Grid Plate being unloaded at Site

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

Stress analysis of the innovative welded grid plate has been carried out using finite element method. The component was modeled using quadrilateral element with the perforated top and bottom plates modeled as equivalent solid plates with modified Young's modulus and Poisson's ratio. For top and bottom plates and shell, 40 millimeter thickness was found to be adequate.

ACHIEVEMENTS

Grid Plate for the future FBR has been innovatively designed with large techno-economic benefits and safety advantages. It exemplifies the success of the efforts in minimizing the cost of future FBR. The change in concept of sleeve support to spigot support for the shielding subassemblies has reduced the diameter of shell and bottom plate of the grid plate significantly. The increase in number of pipes from four to eight, which enhances safety, facilitates decrease in overall height of the grid plate. These innovative features resulted in weight reduction by 55%.

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Production of Enriched Elemental Boron for PFBR Control Rod Applications

EXECUTIVE SUMMARY

A pilot plant was created at the Chemistry Group, IGCAR and the viability of a pyro-electrometallurgical process for the large scale production of elemental boron from boric acid was demonstrated. With the help of this plant the optimal flow sheet was arrived at and 15 kg of enriched elemental boron was produced. Part of this enriched boron was converted into high density boron carbide pellets at BARC. These pellets were found suitable for use in PFBR control rods. With the help of the technical know how transferred and the training imparted by IGCAR, the Heavy Water Board could successfully commission and operate an elemental boron production plant at Manuguru.

OUTLINE

The Prototype Fast Breeder Reactor (PFBR) being built at Kalpakkam would use boron carbide (B_4C) in its control rods. The commercial method of producing boron carbide involving the direct carbothermic reduction of boric acid is not suitable for making nuclear grade boron carbide because of its rather abysmally low yields. Thus there was a necessity to develop an indigenous technology for producing boron carbide containing enriched boron. In view of the above a three stage process was devised by DAE for the indigenous manufacture of B_4C . This scheme involves the enrichment of boron, production of elemental boron and its conversion into high density boron carbide pellets. Developmental work on the second step was carried out at Chemistry Group, IGCAR.

The enriched boron was received in two different chemical forms viz., H_3BO_3 and $BF_3(OC_2H_5)_2$. Procedures suitable for the conversion of these compounds into KBF_4 as well as the conversion of the latter into elemental boron were developed (Figure 1).

This was accomplished in two stages. The first stage involved demonstration of the technological viability of this process by designing, constructing and operating a production facility for a couple of years. About 5 kg of natural boron that met the prerequisites for its conversion into B_4C was produced in this plant. In the second stage a comprehensive facility was developed for producing enriched elemental boron. This pyroelectrometallurgical plant was the first boron production facility to be established in the country. The second stage involved development of innovative equipments that were necessary for carrying out the production. Extensive research and development was carried out in order to optimize the process parameters for the production. Our sustained efforts for over four years culminated not only in the production of 15 kg of enriched boron but also in arriving at a viable technology and a comprehensive documentation that made the transfer of technology to the Heavy Water Board (HWB) possible. With the continued support of IGCAR and effective collaboration and guidance, HWB established a plant for the production of enriched elemental boron at Manuguru. Training to a composite team of HWB personnel as well as technical consultancy during the various stages of the construction and commissioning of this plant were also provided by IGCAR.

The chemical conversion procedure for making KBF_4 from $BF_3(OC_2H_5)_2$ was devised. This is an important step that helped interface the enrichment plant with the electrowinning plant. A significant quantity of the elemental boron to be used in fabricating B_4C for the first core of PFBR has been produced with the help of this technology. Insights into the chemistry of production of KBF_4 , electrowinning and post-processing were gained by way of specific and focused R&D carried out at IGCAR (Figure 2).

About 3 kg of high density enriched B_4C pellets were produced at BARC by using the enriched boron produced in our plant.

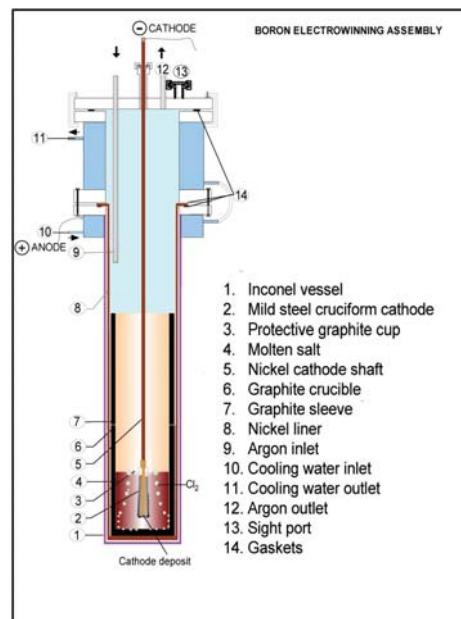


Fig. 1: Boron Electrowinning Assembly



Fig. 2(a): Electrodeposited elemental boron

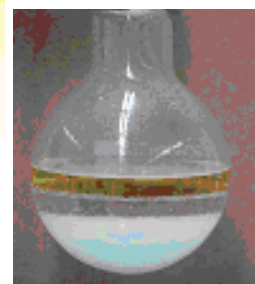


Fig. 2(b): KBF_4 obtained from $BF_3O(C_2H_5)_2$

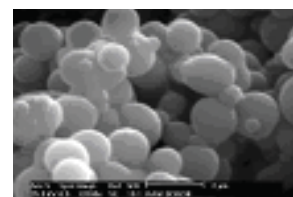


Fig. 2(c): SEM micrograph of the boron powder

ADDITIONAL INFORMATION

One of the major difficulties encountered in the electrowinning operation is the corrosion of the cruciform mild steel cathode. This leads to the contamination of the final product with iron. We minimized this by analyzing the mechanism of corrosion. This contamination was brought to a minimum by optimizing the design of the cathode and by protecting it with a graphite cap. The iron impurity in the final product was brought down to less than 1% through such an optimization. The process parameters in the production of KBF_4 from boric acid were optimized through systematic investigations. About 225 kg of KBF_4 was produced from boric acid through an indigenous polypropylene reactor developed in-house. The process flow sheet for the purification of elemental boron was also arrived at based on investigative trials.

GENERAL EXPLANATION RELATED TO DESCRIPTION

One of the most significant aspects of this developmental work was the post-processing of the deposit. The electrodeposited boron (Figure 3) had to be purified by a rigorous aqueous processing. The soluble anionic impurities were removed by using hot water washing while the cationic impurities such as iron and nickel were removed by leaching with hydrochloric acid. The chemical state of these impurities in the as-deposited boron and the processed boron were understood with the help of systematic investigations of these products. X-ray photoelectron spectroscopy, microstructural characterization and chemical assay were carried out on these products. These results were corroborated with the help of a simple thermodynamic modeling.

Yet another aspect that was investigated in great detail was the vaporisation behaviour of the alkali metal fluoroborates. The systematic dependence of the vapour pressure of these compounds down the group was investigated. These investigations indicated that KBF_4 is the most suitable alkali metal fluoroborate for use in electrowinning.

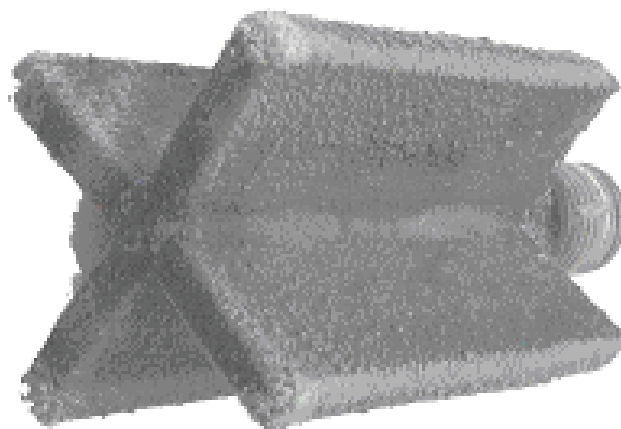


Fig. 3: Mild steel cruciform cathode with the elemental boron deposit

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The electrowinning process involves producing boron by electrolyzing potassium fluoroborate dissolved in a mixture of molten potassium fluoride and potassium chloride. During the electrolysis chlorine evolves at the anode while boron deposits on the cathode. The composition of the molten mixture is very critical for it influences the thermal conductivity, viscosity, density as well as mobility of the ions. Even though the electrowinning of boron is known for a long time, specific information on the ternary melt system used in this developmental work KF-KCl-KBF_4 is rather limited. Further critical parameters like the cathode current density and the residual moisture in the starting material have a strong influence on the electrowinning process.

ACHIEVEMENTS

The technology for the production of enriched elemental boron has been established at IGCAR. The first elemental boron production plant in the country has been built. The technical know-how for the production of enriched elemental boron was transferred to HWB leading to the construction of a similar plant by the latter. 15 kg of enriched elemental boron has been produced in this facility. 3 kg of this was successfully converted into high density B_4C pellets at BARC. Thus this research endeavour played a key role in developing the strategic technology necessary for the indigenous manufacture of enriched boron carbide for PFBR.

PUBLICATIONS

Ashish Jain, S. Anthonysamy, K. Ananthasivan, R. Ranganathan, Vinit Mittal, S.V. Narasimhan and P.R. Vasudeva Rao, "Characterization of electrodeposited elemental boron", *Materials Characterization* 59 (2008) 890.

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Location Specific Ocean Modeling for Extreme Waves, Cyclonic Surges and Effluent Dispersion for Kalpakkam Coast

EXECUTIVE SUMMARY

A comprehensive numerical ocean modeling system to be implemented in operational mode is being developed under a multi-institutional collaborative programme. Local bathymetry data in high resolution, meteorology from site measurements and geography information system data of the topography would be used to estimate parameters of sea water diffusivity, extreme surge height and inundation level. Daily site specific and customised weather model output from radiological safety division cluster is linked as input to ocean state / storm prediction and aquatic dispersion models. The ocean model output would be made available as input to the existing radiological emergency response programme.

OUTLINE

The Radiological Safety Division, IGCAR has initiated R&D activity towards realtime prediction of extreme waves, storm surge and liquid effluent dispersion specific to Kalpakkam. The programme is divided into three modules namely a shallow water wave model (SWAN) implemented for extreme wave prediction, an Advanced Circulation model (ADCIRC) for storm surge and inundation model and a ocean current model (POM) for transport and dispersion of effluents.

The wave model SWAN for simulating waves near shore (Figure 1) has been implemented in collaboration with IIT Kharagpur in a parallel computer system. A typical wind speed of 10 m/s coming from surge effluent was considered for the study and significant waves were simulated as a typical test case. Physical parameterization connecting wave age with drag coefficient has been improved and found with comparable agreement with Janssen parameterisation both for young and mature waves.

A transport-dispersion code was developed and coupled with POM and a case study of thermal pollutant dispersion from Madras Atomic Power Station Jetty was carried out for four seasons of a year using local meteorological data. Currently, a higher version of the research model called ROMS for regional scale current modeling is taken up for implementation. An experimental programme for measurement of currents is initiated in collaboration with NIOT, Chennai.

An Advanced Circulation model for inundation was also adopted for Kalpakkam coast. A case study of the cyclone that crossed near Cuddalore in the year 2000 was carried out in collaboration with IIT, Delhi to test the performance of the model in surge prediction. Simulated surge height (1.4 meter) agrees well with actual observation (Figure 2). The model can be coupled with geography information system to show the inundation hazard on the site map.

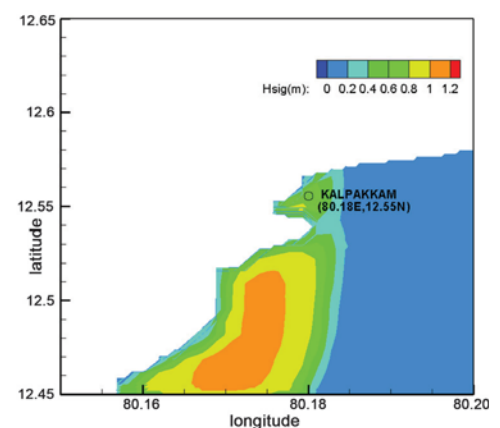


Fig. 1: Significant wave height simulated for Kalpakkam using the wave model

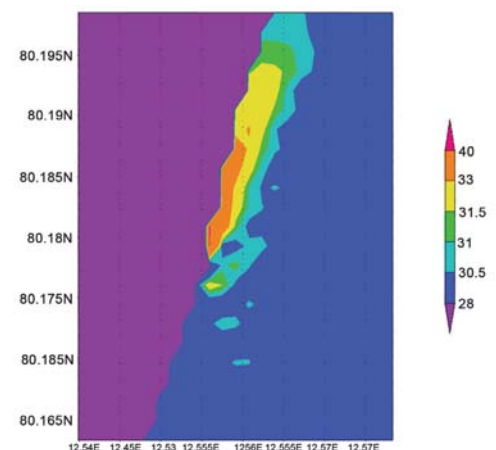


Fig. 2: Simulated Thermal plume pattern in April 2008

GENERAL EXPLANATION RELATED TO THE DESCRIPTION

The finite difference and finite element models for physical oceanography basically deals with solving the system of equations for conservation of momentum and energy for a highly inertial fluid whose state of motion is sensitive to salinity and temperature. Similarly, the air-sea interaction parameterization involved in the wave and atmosphere-ocean current model is still not properly understood and mostly based on analysis of site specific observations. A coupling scheme for atmospheric model MM5, WRF with POM is required to contain the site specific air-sea parameterization. A combined atmospheric-oceanographic model is expected to simulate the storm surge, current and extreme wind and waves in a realistic manner (Figure 3).

In addition, the coupled model performance depends upon the site specific data from an intensive observational network that should be available on-line. The meteorological measurement facility at Kalpakkam provides high quality input on-line to these models.

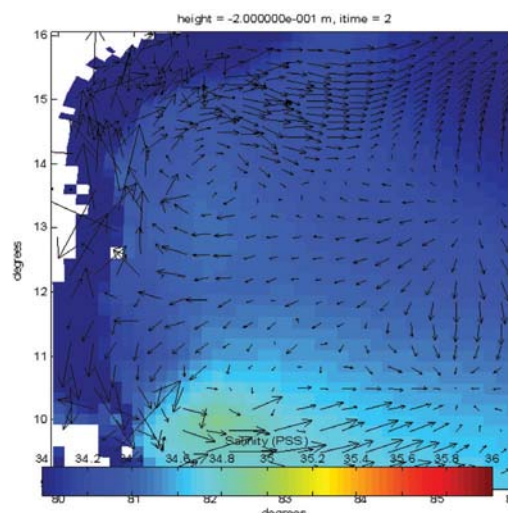


Fig. 3: Forecasted currents and salinity after five days

ADDITIONAL INFORMATION

The ocean models are independently used by various researchers particularly in large scale ocean circulation models. This is the first time an attempt is made in our country to simultaneously run the codes with meteorological input from mesoscale models in an operational mode. The results from the daily run of the models would predict the possible re-circulation of thermal plume from reactor coolant water fallout, the radioactive dispersion and the extreme wave likely to hit the coast. The surge prediction model also gives the forecasted inundation map in GIS software in a ready to use format.

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

The capability to assess and predict the environmental impact of radiation fallout in a postulated emergency event is achieved as an unique outcome of the R&D. Development of a real time coupled atmosphere-ocean model for prediction of extreme waves, surge and dispersion with local site specific data assimilation are the frontier research areas in this field.

ACHIEVEMENT

All the individual modules of the coupled atmospheric-oceanographic modelling has been tested and case studies for Kalpakkam for thermal discharge simulation, wave height and period simulation, cyclonic surge simulation were carried out.

PUBLICATIONS

A. D. Rao, Indu Jain and R. Venkatesan, Estimation of extreme water levels due to cyclonic storms: a case study for Kalpakkam coast, The International Journal of Ocean and Climate Systems, 1 (2010) 1.

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Performance Evaluation of Electro Chemical Hydrogen Meter in Fast Reactor Technology Group

EXECUTIVE SUMMARY

Steam Generator (SG) of a Fast Reactor houses both water/steam at high pressure and sodium at low pressure separated by a tube wall. Any damage to the tube wall initiates sodium water reaction that could badly affect the plant availability. As this reaction generates hydrogen, SG leak monitoring is made possible by monitoring the Hydrogen concentration in sodium. Chemistry Group has designed and developed an Electro Chemical Hydrogen Meter (ECHM) that can detect hydrogen concentrations in Sodium selectively and quantitatively in ppb level. Three such sensors are tested in Sodium Water Reactor Test Facility (SOWART) and Steam Generator Test Facility (SGTF) of FRTG

OUTLINE

Electro Chemical Hydrogen Meter (ECHM), shown in Figure 1, is a concentration cell that measures the potential difference between sample sodium electrode and a fixed reference electrode. This potential difference is a direct measure of the hydrogen concentration in sodium. ECHM measures the absolute concentration of hydrogen in sodium. In the construction of ECHM, an iron thimble houses the electrolyte i.e. a mixture of 60% CaBr_2 and 40% CaHBr . It is a solid electrolyte that conducts H^- ions and produces an EMF in accordance with Nernst equation. $\text{CaO-MgO-CaH}_2\text{-Mg}$ mixture is used as the reference electrode, which fixes the reference hydrogen pressure at the meter operating temperature of 450°C . The EMF generated between the reference and sample electrodes is amplified by a preamplifier unit and is calibrated in terms of hydrogen ion concentration. The output is displayed in volts (0 to 10 V) or ppb (0 to 500 ppb). It is free of spurious signals and steady compared to ion pump based meter.

One of the ECHM sensors was placed in the meter section adjacent to a sputter ion pump based hydrogen meter. In SOWART, steam leak (micro leak and small leak) is simulated and the sodium water reaction experiments on material wastage are carried out. During steam injection, acoustic detectors give an instantaneous response. In 100 seconds time both HSD and ECHM started giving signals. Meters showed similar steep rise in output (Figures 2 & 3). Excluding the transportation lag, the response time of meter is found about 40 seconds which is acceptable as per PFBR specification. In later experiments ECHM preamplifier is set to measure ppm level of hydrogen so that it could be used for measuring leak rate in situ.

Steam Generator Test Facility (SGTF) is equipped with one ECHM, which is wired to safety interlock system (subsequently one more ECHM is also added in SGTF). During SG operation, ECHM showed increase in hydrogen level, due to ingress of hydrogen produced by magnetite formation in the waterside. As soon as SG emptied out of water, it started declining (Figure 4).

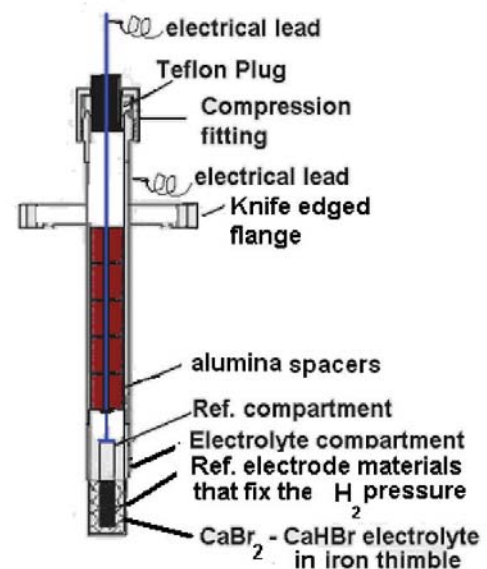


Fig.1 : Evaluation of Electro Chemical Hydrogen Meter details

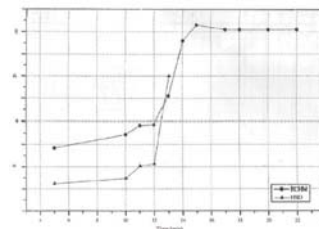


Fig. 2 : SIP & ECHM

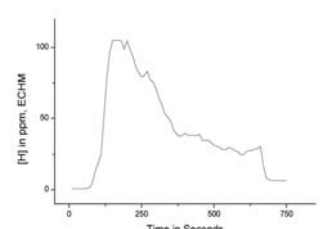


Fig.3: Evaluation of Electro Chemical Hydrogen Meter response during small leak

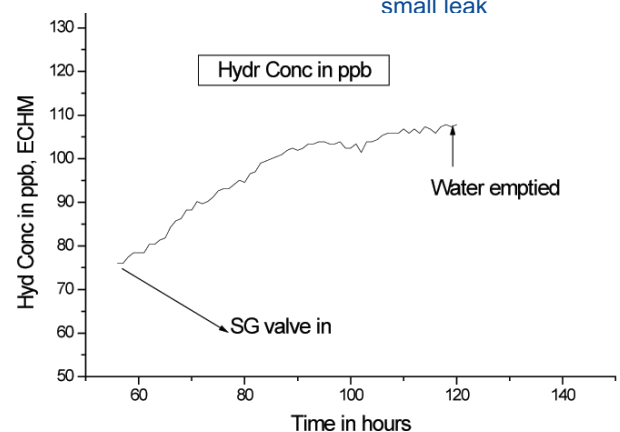


Fig.3: Evaluation of Electro Chemical Hydroge Meter during SG valve in

ADDITIONAL INFORMATION

ECHM output is highly temperature sensitive. The meter is designed for functioning at 450°C. In case of a heater trip, meter should behave in a fail-safe mode; i.e. the output should increase. This was tested by tripping the heater deliberately. It is found the signal was rising as expected. To understand the offset in background reading caused during sodium dumping and cooling, tests were performed by dumping sodium for short duration and long duration. It is understood, that higher the duration of shutdown the more time it takes to come back to original background reading. It is recommended that maintaining sodium at 450°C in the meter section even if other parts of the loop are dumped to avoid this time lag, helps in restoring the background value.

To simulate mechanical failure of iron thimble, the electrodes were short-circuited and found reading is rising to the peak, which is fail-safe. The ECHM was calibrated with known amounts of hydrogen injections and also by varying cold point as shown in Figure 5 & 6. A linear trend in the relationship was obtained. The meter showed a resolution of 5ppb.

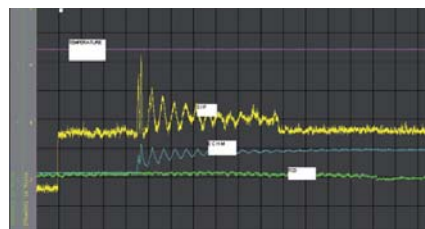


Fig.5: Evaluation of Electro Chemical Hydroge Meter during Hydrogen injection

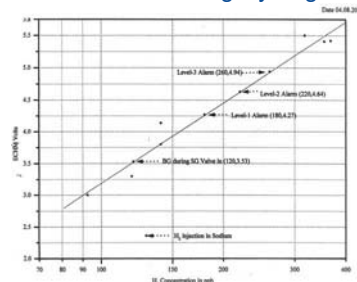


Fig. 6 : Evaluation of Electro Chemical Hydroge Meter calibration

BRIEF DESCRIPTION OF THEORETICAL BACKGROUND

ECHM output is highly temperature sensitive. The meter is designed for functioning at 450°C. In case of a heater trip, meter should behave in a fail-safe mode; i.e. the output should increase. This was tested by tripping the heater deliberately. It is found the signal was rising as expected.

To understand the offset in background reading caused during sodium dumping and cooling, tests performed with dumping sodium for short and long durations.

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To simulate mechanical failure of iron thimble, the electrodes were short-circuited and found reading to rising to the peak, which is fail-safe.

ACHIEVEMENTS

ECHM was installed in FBTR & Phenix reactor after it was tested in FRTG.

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