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INSIDE

From the Director's Desk

Spent Nuclear Fuel Reprocessing at Indira Gandhi Centre for Atomic Research

A strategy to sustain nuclear power in India was visualized by Homi Jehangir Bhabha. When he envisioned the three stage program for meeting energy demand for centuries, the charter for the nuclear fuel reprocessing had been embedded, as the recovery of fissile material produced in the reactor is an integral part of this vision. In Dec 1958, the decision for setting up a reprocessing plant at Trombay to process spent fuel discharged from CIRUS reactor was taken, giving expression to the ambition of Bhabha and Pandit Jawaharlal Nehru for achieving self reliance in energy security through the nuclear route.

Genesis of Fast Reactor Fuel Reprocessing Programme

In sixties, the reprocessing group at Bhabha Atomic Research Centre (BARC) led by N.Srinivasan, which had built and operated the Plutonium Plant, was also carrying out R&D work on the chemistry of solvent extraction of plutonium at high concentrations. This experience proved very useful when Dr. Vikram Sarabai concluded an agreement for transfer of know-how from France, for setting up a Fast Breeder Test Reactor at Kalpakkam similar to Rapsodie. He thought it prudent to have a dedicated Centre with R&D programmes focused around fast breeder reactor with closed fuel cycle. He constituted a project implementation committee under the Chairmanship of Dr. Raja Ramanna, which culminated in the formation of Reactor Research Centre (RRC). N. Srinivasan was chosen as the first Project Director. Among the other facilities of the

The beginning of Reprocessing in the country

The Plutonium Plant in Trombay was the first plant to reprocess spent fuel in India. It reprocessed the spent fuel discharged from CIRUS Reactor. The plant was designed and commissioned mainly by engineers from the first three batches of BARC Training school with H.N.Sethna as Project Engineer and N.Srinivasan as design engineer. The plant and equipment designs were based on first principles of chemistry and engineering. All the equipment were indegeounsly fabricated with minimal pilot plant studies. The plant, which was commissioned in 1964 within the scheduled time and cost, established the capability and credibility of Indian engineers internationally. The plant operation offered an opportunity to solve many challenges and provided valuable inputs to design subsequent reprocessing plants.

Center, a Reprocessing Development Laboratory (RDL) for carrying out R&D activities relating to the reprocessing of fast reactor fuel and a plant for reprocessing of spent fuel from Madras Atomic Power Station (MAPS) were planned.

Leader Identified

G.R.Balasubramanian, a member of the team which designed, erected and operated the plutonium plant at Trombay was chosen for leading the activities related to the development of fast reactor fuel reprocessing at Kalpakkam. He was also an important member of the group that produced the first button of plutonium metal in the country as early as 1965. In 1969-70, he was deputed to Fontenay Aux Roses in France as an IAEA fellow for studies related to the novel solvent trilauryl amine for Pu separation. He was also engaged in Trombay in the work relating to the solvent extraction chemistry of plutonium at high concentrations, separation chemistry of thorium and uranium. His work also included R&D studies related to airlifts and other equipment. He led the group which designed and operated a pilot facility in Trombay for recovery of U^{233} from irradiated thorium using equipment such as thermosyphon dissolver and air pulsed mixer settlers using 5% TBP as solvent.

G.R.Balasubramanian moved to Kalpakkam in 1972 with one draughtsman, P. K. Banerjee, from the Reprocessing Group of BARC in response to his new assignment and he drew up the master programme, identifying the short term and long term needs and started implementing his vision. The team got nucleated with the regular induction of engineers and scientists from the 14th batch of BARC training school.

Reprocessing Development Laboratory construction in 1974

Emergence of Fast Reactor Fuel Reprocessing Philosophy

Generally information dissemination on reprocessing is limited. Very few publications, that appeared in the proceedings of the conferences held in Geneva on peaceful uses of Atomic Energy in the sixties, focused on the subject. They only gave insight into the global scenario which prevailed then.

Initially many countries evinced interest in the field. This number drastically dwindled to a very few subsequently. Aqueous processes, based on solvent extraction and non-aqueous processes based on pyrochemical and pyrometallurgical methods have been studied. Even in aqueous processes, a large number of solvents figured. After careful consideration of all processes, it was decided to defer the work on non-aqueous processes to a later date when we would have gained reasonable experience in remote handling technology and material suitability for high temperature and corrosive environment.

Hence our efforts were launched towards the development of aqueous processes, based on solvent extraction using Tributyl phosphate in dodecane type of diluent. This was due to the confidence arising out of satisfactory experience in Trombay on thermal reactor spent fuels. Limited published literature available on this field from other countries also indicated that PUREX process can be adapted for fast reactor fuel reprocessing.

Formulation of the Programme

Considering plutonium rich solutions to be handled with high levels of radioactivity, various areas for development were identified, such as chemistry of solvent extraction of Pu rich nitric acid solution, critically safe batch type pulsed thermosyphon and continuous rotary dissolvers, high speed feed clarification centrifuges, short residence time centrifugal contactors, air pulsed type mixer settlers for extraction cycles with low doses to solvents and diluents, fuel wrapper cutting systems, single and multipin choppers, continuous precipitators, feed metering devices etc. Since high concentrations of Pu were to be handled, remote maintenance constituted the core feature of the design. While evolving a candidate process flow sheet, it was decided to avoid heating of plutonium rich aqueous solutions. These solutions were known to invariably carry entrained and dissolved solvents that could lead to safety related problems. The difficulties that were likely to be encountered when handling plutonium loaded TBP solvent were also identified as an area of concern. For high integrity, containment boxes with concrete or lead shielding were conceived. With stress on remote maintenance, cell design became radically different from the hot cells used for thermal reactor fuel reprocessing. Thus, remote handling equipment such as manipulators, remote viewing cameras, shielded windows etc., were also identified for development, in synergy with efforts of BARC.

It was quite clear, right from the beginning, that approaches to the thermal and fast reactor fuel reprocessing are quite different though the same solvent is being used in both the plants. The difference in the routes for fast and thermal reactor reprocessing widened further, when the choice of fuel for FBTR fuel was made in favour of mixed carbide fuel of high plutonium (70%) concentration. Thus, the need for a hot cell facility was conceived for testing of the equipment and systems, so that the process as well equipment designs could be validated for future plants. Thus, the infrastructure for carrying out engineering scale R&D in non-radioactive as well as radioactive conditions was realised in the conceptualization itself. Accordingly, the reprocessing programmes were formulated

Reprocessing Development Laboratory Goes Hot

With the completion of construction of RDL and with all the infrastructure available, clearance was obtained for commissioning the laboratory with Pu in 1980s. The first lot of plutonium needed for initial phase of R&D work in the Center was transported to Kalpakkam. This operation enabled training in Pu handling by personnel of RDL. Simulation experiments with mixer settler were carried out with the proposed fast reactor process flowsheet conditions with a 16 stage mini mixer settler. The results gave confidence to adopt the flowsheet for the hotcell conditions.

U^{233} Campaigns precede Pu^{239} Campaigns

When C.V Sundaram, the then Director of the Centre asked G.R.Balasubramaniam to undertake the separation of U^{233} from CIRUS J-Rods, for the Kalpakkam MINI reactor (KAMINI), G.R. Balasubramanian accepted the challenging task, as

Engineering Facility Commissioned

The project approval for setting up the Reprocessing Developing Lab (RDL) was obtained in 1972, as a part of the master plan of Reactor Research Centre. The engineering development laboratory for carrying out the non radioactive experiments on the equipment and systems for qualifying deployment in hot cells with the infrastructure such as the radioactive laboratory storage facility for different levels and types of liquid radio active waste and other services facilities like filter room, plant room, workshop etc., was completed progressively from 1976 onwards. These facilities were used for various development activities. Single solvent extraction cycle loop with natural uranium was conceived, designed and installed in concrete cells in 1980s, which provided much needed operational experience to the team, apart from testing of some of the equipment like air pulsed mixer settlers, metering airlifts, dissolver, sintered filter and air lifts.

Dr Anil Kakodkar, Chairman-AEC visiting CORAL during erection

J-Rod campaign

Contribution to the third stage of Indian Nuclear Programme

The concrete cell in RDL housed the facility. Since the operation was planned to be carried out in campaign mode, wherever required PVC hoses were used inside the cells also. The flow sheet already tested in Trombay was used. A vertical thermo-siphon dissolver was designed and fabricated in RDL workshop. A few stainless steel tanks fabricated at Trombay were also brought to RDL. These equipment and tanks were installed in concrete cells. Centrifugal extractor with eight stages and of belt driven bank type was used for the extraction. An air pulse mixer settler was used for stripper. Air driven centrifuge was used for feed clarification. All solvent contactors, centrifuge, diverters, sampling systems and sample conveying systems were located inside the cell. These were designed for remote operation, maintenance and viewing. The facility took up the reprocessing of J-Rods in 1987. The J-Rods were brought from CIRUS. The fuel was transferred from the cask to the cage liners through a pair of remotely operated tongs with the help of cameras. This is the first time this concept was tested under radioactive conditions.

this assignment would provide an opportunity to train the staff in β, γ handling and validate the special equipment developed for Fast Reactor Fuel Reprocessing. V.R. Raman was transferred from PREFRE to RDL in 1987 to guide the young team in the plant operation.

The campaign, apart from providing sufficient amount of U^{233} with the desired purity level for KAMINI reactor, provided operating experience and feed back to the designers. It was a progressive step towards acquiring independent capability in reprocessing of fast reactor fuel as equipment used in this campaign were of similar design to the ones planned for fast reactor fuel reprocessing plants. The individuals who were leading the different shift crews in the first campaign, took up independent responsibilities like plant design, plant operations, R&D and plant construction subsequently and have risen to positions of important responsibilities, in Indian spent fuel reprocessing industry, today. S.R.Paranjpe who steered the Prototype Fast Breeder Reactor (PFBR), including its fuel requirement, initiated a program for testing PFBR MOX fuel composition with U^{233} and Pu^{239} in FBTR. This called for a second campaign of reprocessing thorium rods. Safety clearances were obtained to restart the facility for the second campaign which was carried out with Thorium rods from CIRUS and DHRUVA Reactor. This campaign provided new challenges, like higher dose, difficulty in dissolving denser pellets etc. This campaign had many innovative features, such as the design and operation of a temporary under water fuel handling system to reduce radiation exposures while handling fuel rods.

These campaigns provided opportunity for validating various design philosophies that would be vital for fast reactor fuel reprocessing. They also aided in providing the fissile material required for Fast Reactor Fuel Development. With the material supplied from the first campaign, KAMINI which is the only operating U^{233} fuelled reactor in the world, is working at high levels of performance.

When the reactor designers chose mixed carbide fuel with plutonium of 70% concentration for FBTR, the candidate fuel had to be assessed from reprocessing considerations. This type of fuel has not been reprocessed anywhere in the world. Degree of solubility during dissolution, complication in solvent extraction due to dissolved carbon in form of organic acids and safety perceptions due to hydrocarbons, were to be examined. The group considered a few methods, such as dissolution after pyrohydrolysis or voloxidation followed by dissolution in nitric acid and direct dissolution in strong nitric acid with chemical oxidants for destruction of organic acids. Experiments with unirradiated pellets were carried out and electro-oxidative dissolution methodology was formulated for dissolution of this unique fuel. Based on the results of these experiments, Reprocessing team expressed confidence in reprocessing Pu rich carbide fuels. This enabled finalisation of mixed carbide fuel rich in Pu as the driver fuel for FBTR. Based on these studies as well as studies related to third phase formation, the flow sheet, prepared earlier for the previous oxide fuel was modified. Today it is a matter of immense satisfaction that the assessment has come true. It is to be noted that reprocessing of spent mixed oxide fuel from power producing large Fast reactors, would not pose such a difficulty compared to the Pu rich mixed carbide fuel of FBTR. Thus, it can be said that facing difficult challenging assignments has helped reprocessing group to develop credence and confidence to take up larger challenges and responsibilities in the coming years.

By the time, the thorium campaigns were completed in RDL, FBTR was operating with mixed carbide fuel with an excellent performance in terms of burn-ups etc., It was decided to upgrade the hot cell facility for reprocessing FBTR mixed carbide fuel. Process flowsheet as well as equipment designs had to be validated for Fast Reactor Fuel Reprocessing.

Mixed Carbide Fuel Reprocessing in Hot Cell - CORAL is Born

With the validation of most of the equipment and system designs in J-Rod facility, the hot cell erection could be taken up with confidence. Initially the facility was planned for gram scale operation of irradiated fuel, which was later enhanced to kg capacity, when mixed carbide was chosen as the driver fuel for FBTR. Thus, Lead Mini Cell (LMC) was born which was later rechristened as CORAL (COmpact Reprocessing facility for Advanced Fuels in Lead cells).

It was decided to test all the process equipment developed for fast reactor fuel reprocessing in this facility. Also, the cell design, which was conceptualized for future FBR fuel reprocessing plants, is being validated in CORAL facility. Thus, some of the key equipment such as single pin chopper, dissolver, centrifugal extractors , etc, are being evaluated. A high speed centrifuge similar to the one tested in J-Rod campaign, was installed with amenability for remote maintenance, after extensive testing. The contributions of Ashok Kumar of Fast Reactor Technology Group, in finalizing the rotating bowl design was commendable. M.S. Ilangovan, provided the much needed expertise in instrumentation systems of CORAL. He validated several concepts such as use of Programmable Logic Controllers (PLC) and was instrumental in meeting the control & instrumentation requirements of the plant in a limited space and to the satisfaction of the regulators.

The design and commissioning of the Pu reconversion laboratory was completed with the cooperation from BARC colleagues.

The erection of in-cell crane with α-tightness and the design and operation of horizontal α-tight transfer system were accomplished for the first time in the country. The design features of HEPA filter without breaching α-tightness during filter change is unique. Quality Assurance Division provided the much needed quality control support during fabrication and erection of CORAL facility.

Inactive commissioning trials with acid runs in CORAL were started in 2002. The regulatory clearance for Pu with low burn-up fuel was obtained and the plant was hot commissioned in May 2003. The first batch of irradiated fuel pins with 25 GWd/t burn-up was taken inside CORAL in Dec 2003.

Successful reprocessing of 25 to 50 and 100 GWd/t mixed carbide fuel is a testimony to the dedication of the design, operation and maintenance teams. The designers continue to be involved in ensuring and accumulating safe and successful experiences.

Road Ahead

Successful reprocessing of highly irradiated fuel, and the closure of the fuel cycle is a tribute to the interdisciplinary technological approach of the Centre and the Department. The Plant has registered several benchmarks of excellence. Versatility and flexibility to suit challenges has been amply demonstrated. Further, these campaigns have boosted the confidence level of the entire group, conditioning them to accept tougher challenges in the design, construction, operation & maintenance of the future fast reactor fuel reprocessing plants.

The next challenge for the Reprocessing Group, is the Demonstration Fuel Reprocessing Plant (DFRP). The main objectives of this plant are to reprocess the fuel of FBTR on regular basis and that of PFBR on experimental basis. Because of the logistics of the design, the civil construction of this plant was taken up along with that of KARP and was completed in early 1990s.

DFRP is now in advanced stage of

Codes and Standards

With the assimilation of knowledge, it is time to write the codes and standards. For reactor systems, the fabrication codes, quality control & inspection procedures are well developed and documented. In the absence of codes and guides specific to reprocessing, the regulators are inclined to apply reactor codes, though the requirements are quite different. Application of codes in reactor systems is related to high temperature behavior of the material coupled with neutron fluence, whereas, the reprocessing is mostly related to corrosion and gamma environments. As the requirement of an independent code was realised, a taskforce was formed in the year 1991. This task force prepared the first design and fabrication code as applicable to reprocessing requirements of fast reactor fuel reprocessing plants. This has been revised, updated and now independent documents for several aspects of the design, material selection, fabrication for fuel reprocessing are available.

Compact Reprocessing facility for Advanced Fuels (CORAL)

CORAL facility is a unique hot cell with an α-tight containment box. All the operations involving open solution handling are carried out inside the containment box. All the process tanks are kept below the containment box. α, β shielding is provided by lead bricks. The facility has about 2 kms of piping with more than 3000 bends and 2000 radiography qualified weld joints. With more than 500 penetrations in the containment box, commissioning of CORAL was indeed a challenge, since locating the leaks during vacuum hold tests, was a real challenge, with the lead bricks around. As the box leak tightness was progressively increased, detection of very small leaks proved to be a very difficult task. The interactions between those involved in design and erection were very interesting as very little information on this type of novel plant is available. There used to be many 11 'O' Clock meetings' in which all designers and erection engineers took active part to thrash out all issues to arrive at expeditious decisions. Many innovative ideas crystallized in those meetings.

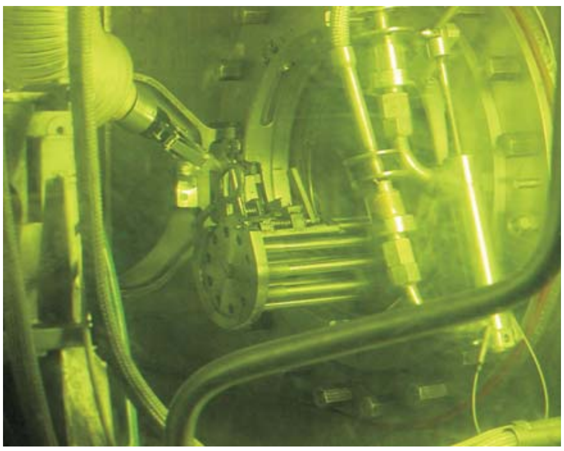
erection. Most of the design options are closed since CORAL experiences on many issues are available. Wherever inevitable retrofitting is being done in the light of operational experience of reprocessing FBTR fuel in CORAL. The plant has flexibility to take up the reprocessing of PFBR fuel subassemblies on demonstration scale, since large scale experience with irradiated mixed oxide fuel is essential for optimizing the design and operational parameters for large scale reprocessing of PFBR fuel.

Human Resources

Robust Technology is based on rigorous science and the synergy of cross-disciplinary engineering efforts in challenging situations. Knowledge acquired by the Reprocessing group in designing, operating and maintaining CORAL, is getting enhanced through large collaboration with groups within the Centre, DAE, Universities and other Research Institutes.

Coordination With Other Groups of IGCAR & Other Units of DAE

Success of reprocessing programme is a closely woven fabric of teamwork within the group at various levels. The limited manpower has provided adequate challenges and motivated our colleagues to play more than one role. All Directors of the Centre, starting from the founder director N. Srinivasan and subsequently C.V. Sundaram, S.R. Paranjape, Placid Rodriguez and S.B. Bhoje have provided crucial support to the activities of the group.



CORAL goes hot – Irradiated fuel pins taken into the cell in 2003

N. Srinivasan initiated the fast reactor reprocessing program in the Centre to realise the vision of Homi Bhabha and Vikram Sarabhai. With his rich experience in the reprocessing technology, he guided the initial phase of the activities so that it could mature to a robust technology in future and provide necessary trained and experienced manpower for this crucial activity.

C.V. Sundaram was instrumental in guiding the Reprocessing Group to take up the assignment of processing Thorium rods from CIRUS and DHRUVA. This approach has provided the much needed confidence to the group to embark on higher challenges of fast reactor fuel reprocessing.

S.R. Paranjpe was responsible for providing direction to ensure that the CORAL facility is truly a pilot plant. This approach has enabled in getting the required plant level experience in complete flow sheet.

Placid Rodriguez has motivated the group through the difficult phase of construction and erection phase of CORAL and DFRP by regularly conducting project review meetings. This approach enabled the progress of the construction of the CORAL facility, and DFRP providing solutions to problems of interdisciplinary nature.

S.B. Bhoje continued the review process and gave full support towards completing the construction activities of CORAL and DFRP.

As the Director of Reprocessing Group from 1999 to 2005, I have provided the motivation to the team for meeting the challenges during the commissioning phase of CORAL. As Director, IGCAR from 2004, several task forces have been constituted with the best experts in the centre to bring synergy among different groups of the Centre with the purpose of making IGCAR, a mature organization in fast reactor fuel reprocessing technology.

Fast Reactor Fuel Reprocessing -Mission of IGCAR

A full fledged R&D team is carrying out process modeling, equipment development for the DFRP and future Fast Reactor Reprocessing Plants.

The PFBR reprocessing plant is a part of integrated fast reactor fuel cycle facility, being set up at Kalpakkam to close the fuel cycle. A mega challenge to the Reprocessing Group of IGCAR is to design and operate the plant for reprocessing spent fuel from the PFBR matching with the discharge rates of PFBR spent fuel. It is a great opportunity to provide the technology for the recovery, meet specifications of the product and in tight time schedule. Many innovative targets such as fissile material monitoring by neutron interrogation, non invasive fissile material analysis with K-edge X-ray densitometry, actinide and group partitioning of high active raffinate streams etc. are being pursued for implementation in Fast Reactor Fuel Reprocessing Plants. The aim is to develop a robust science based technology for the reprocessing of fast reactor oxide fuels.

All the groups of IGCAR, namely, Chemistry Group, Metallurgy and Materials Group, Reactor Engineering Group, Electronics and Instrumentation Group and the Safety Group have given their unstinted support for all the reprocessing group activities and enabled successful completion of various reprocessing campaigns in CORAL, design and construction of DFRP and design of fuel reprocessing plant for PFBR.

Fast Reactor Fuel Reprocessing - Now a national mission

Central Workshop, of BARC gave substantial support for the remote handling equipment development. The development of special motors and laser systems for inspection of dissolver and dismantling of fuel subassembly, development of coated anodes for electro oxidative dissolution are a few notable contributions by the national institutes. A large number of private industries have participated in fabrication of special equipment such as centrifugal extractors, remote handling equipment and other sophisticated systems.

Conclusion

All the challenging research work of the Reprocessing group cannot be published because of the strategic nature of the work. Still there are more than 300 publications in national, international journals, conferences, IGCAR reports, inspection guides and codes, which bears the testimony to the documentation of work and a policy of sharing science and technology, nationally and internationally.

The success of the fast reactor fuel reprocessing is not only due to the efforts of the group but also due to the synergy of many experts and their experiences, provided by diverse groups at IGCAR, other units of DAE as well as national research institutes, academia and industries.

In India, public support for reprocessing technology for fast breeder reactor fuels is one strong reason for steady progress. Breakthroughs based on science and experiences, have been obtained. We have gained experience based on successful reprocessing of the novel indigenous mixed carbide fuel of uranium and plutonium of high burn-up in our plant, for the first time in the world. We have made an international benchmark. The present team in the Reprocessing Group, IGCAR has matured to face the future challenges and are working with commitment and passion.

One is reminded of the quotation of Robert Frost which Pandit Nehru had on his table: "woods are lovely, dark and deep. I have promises to keep and miles to go before I sleep". It is seen when you work with faith, dedication and mission that God honours your smart work, though he normally takes time.

Science, technology, management, inspiration, strategy, mission and vision are the important ingredients of this challenging and at times unchartered path. We are all convinced that our country needs this success and we deem it as the distinct honour to provide this success to India.

The Reprocessing Group of this Centre is confident of meeting the challenges with elegance.

(Baldev Raj)
Director

Reprocessing Changes Gear Technology development concurrently with Regulatory clearances for CORAL

Safety reviews and regulatory clearances of emerging technologies is always a challenging task. Initial safety reviews with A.K. Ganguly, leading the safety review team, had the holistic approach to the regulatory practices, namely, vision of growth of science without losing the respect for the safety. He was a unique leader who motivated and inspired his colleagues for excellence. Subsequently, T.N. Krishnamony L.V. Krishnan, A.R. Sundararajan and V.M. Raghunath pursued his approach.

CORAL being a first of a kind of plant, Atomic Energy Regulatory Board (AERB) accorded stage-wise clearances. The clearance for commissioning was given based on the recommendation by safety reviews conducted by AERB committee with A.R.Sundararajan as the Convener. This team was supported by various working groups, which carried out detailed safety analyses in specific areas such as fuel handling, process, laboratory, radiological safety and ventilation.

Once the plant went operational, Safety Review Committee for Operating Plants along with IGCAR safety committee carried out safety reviews. Clearances were given in steps of increasing levels of radioactivity starting with pins with very low burn-up to those of burn-up upto 100 GWD/t. The campaigns were successfully conducted achieving good product purity and concentration of Pu with quality levels meeting the specifications acceptable for the refabrication.

Simulated Corrosion Testing Facilities for Reprocessing Plant Materials

As a consequence of the multiplicity of corrosion phenomena involving various parameters and mechanisms, the number of potential corrosion testing methods is large and test procedures have to be carefully designed to generate reliable and useful data. Test facilities to simulate the plant operating conditions with respect to the corrosiveness of the process fluids and other process parameters, have been built and corrosion assessment been made for the reprocessing plant materials at IGCAR.

Corrosion resistance properties required for materials to be used in the various developments of the Purex process are crucial in the field of nuclear fuel reprocessing, since equipment inspection, decontamination, repair or premature replacement would result in procedures much more complicated than in conventional chemical industries.

Austenitic stainless steels are widely used in components of fuel reprocessing plants because of their good corrosion resistance to the solutions encountered. However, several incidences of failures of components made of AISI type 304L SS have been reported in spent nuclear fuel reprocessing plants when they were used in HNO_3 medium beyond 8N concentration and temperatures beyond 353K. The failures have been attributed to (i) intergranular corrosion due to sensitization, (ii) intergranular corrosion due to impurities segregation at grain boundaries, (iii) transpassive dissolution of passive films, and (iv) selective corrosion of welds.

Comprehensive corrosion assessment of AISI type 304L by ASTM A 262 practice C test is impossible, taking into consideration the ranges of concentration of nitric acid used in the plant, temperature of operation, impurities and redox chemicals present and the metallurgical state of the materials used in the reprocessing plants. In the plant, acid is transferred from one component to another after every operation. Thus there is a need to evaluate the flow induced corrosion properties of the materials to be used in the plant. Component geometries and high temperature boiling also induces turbulence in

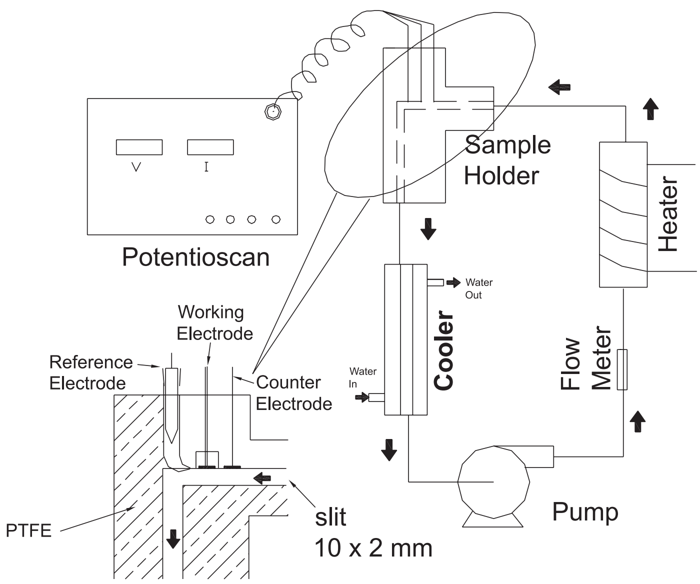


Fig. 1 : Schematic of the MINIL test facility

the system which can influence the corrosion properties of austenitic stainless steels. Also, in the reprocessing plants, mixing operations are extensively used during solvent extraction for mixing of aqueous and organic liquids. In the centrifugal extractors very high rpm's are used for effective separation of the organic and aqueous phases. All these operations introduce high level of turbulence in the liquid phase, which lead to different corrosion mechanisms.

These limitations of the current approach motivated us to design dynamic simulated testing systems namely the Mini Loop (MINIL), Nitric Acid Loop (NAL) and the High Temperature High Concentration test systems (HTHC). The detailed features of these systems and the results obtained so far from these systems are presented in this article.

2.0 MINI LOOP (MINIL)

A special laboratory scale test facility, MINIL (Mini Nitric acid Loop), has been established at CSTD for the testing of the materials under flowing condition (Fig. 1). The special features of this facility are the flexibility of controlling the flow rate of the medium to the required rate and also controlling the temperature of the medium up to a maximum of 348 K. The specimens were fixed in a manner such that the face of the specimen is parallel (flushed) to the flow direction. The flow was in a rectangular slit of size 10 mm x 2 mm through the PTFE holder. Electrochemical polarisation experiments were carried out in this facility using nitric acid of 1M concentration at different flow rates (velocities) namely 0.42 m/s, 0.61 m/s, 0.74 m/s, 0.88 m/s and 1.01 m/s (Reynolds Number of 2824, 4102, 4976, 5917 and 6791 respectively) in order to study the effect of increasing flow rate on the corrosion of type 304L SS.

2.1 Effect of flow

Potentiodynamic polarisation tests were carried out in 1M HNO_3 in the MINIL facility. The velocity of the flow was varied from 0.42 m/s to 1.01 m/s in steps. It was observed that acid flow had a significant effect on the corrosion behaviour of type 304L SS. In the case of the flowing acid at a velocity of 0.42 m/s, the passive current was marginally higher and also the passive to transpassive transition occurred at a lower potential compared to that in the static condition (Fig. 2). Also, in both the cases secondary passivation could be observed at a potential of about 1V, though in the case of flowing medium the secondary passivation current density was one order of magnitude less as compared to the static condition. The motion of the electrolyte thins quiescent passive layers at the metal surface so that corrosive species could easily reach the metal. Corrosion rates were found to increase from 0.004 mpy to 0.373 mpy with increase in velocity from 0.42 m/s to 1.01 m/s (Reynolds Number from 2824 to 6791) as shown in Fig. 2.

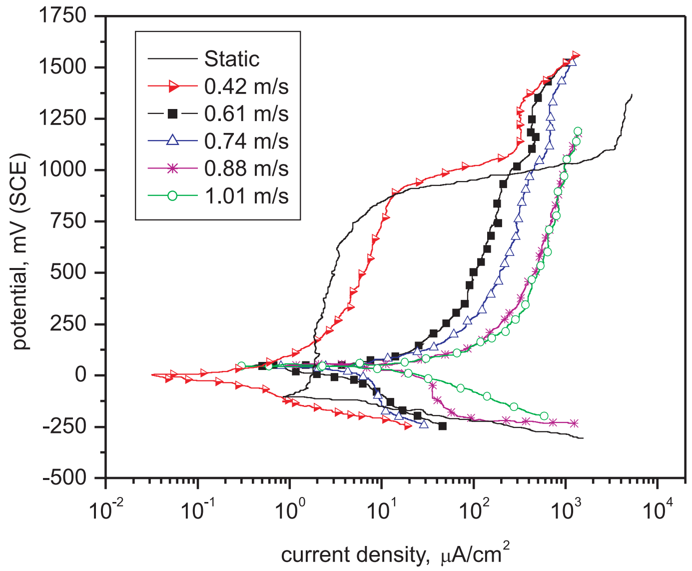


Fig 2: Anodic polarisation curves for 304L SS in 1N HNO_3 at different flow velocities at RT.

It is well known that the oxide layer (Cr_2O_3) formed on the surface of stainless steel protects it from corrosion. In a flowing medium, a hydrodynamic boundary layer is established adjacent to the metal surface. When corrosion takes place, the Cr_2O_3 oxide layer dissolves into the nitric acid and the rate of this oxide layer removal is controlled by the rate of diffusion of this species through the boundary layer of the acid near the surface into the bulk acid. This diffusion (or mass transport) of chromium away from the surface depends directly on the concentration of soluble species at the oxide surface and inversely on the thickness of boundary layer. When the flow changes from laminar to transition and then to turbulent flow, the boundary layer thickness reduces. Thus, a decrease of the boundary layer thickness due to increased acid flow rate or because of local turbulence, causes an increase in corrosion rate.

3.0 NITRIC ACID LOOP

A 400 litre capacity dynamic nitric acid loop has been designed and constructed at IGCAR, Kalpakkam for evaluating the corrosion performance of candidate materials over long operating periods under plant simulated conditions (Fig.3). Corrosion resistance of the materials in different metallurgical conditions, namely, cold worked, solution annealed, sensitized, alloy composition, etc. will be evaluated in this loop in flowing nitric acid at different flow velocities (upto

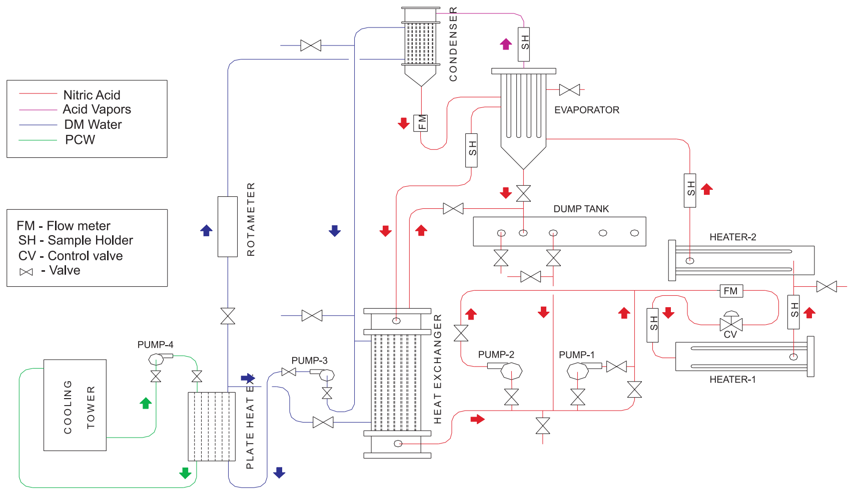


Fig.3: Schematic flow diagram of the nitric acid loop

1.55 m/s) and temperatures (313, 333, 353, 380 K and vapour phase). The data generated over a long period of exposure (up to 10,000 h) would be used to arrive at an acceptable corrosion rate for AISI type 304L SS used for applications in nitric acid service. The availability of such a reliable and useful data will be helpful in predicting the remnant life of the components used in the reprocessing plants by developing suitable analytical and modeling tools of the corrosion processes.

Currently, the loop is operating with AISI type 304L SS samples of solution annealed, sensitized (675°C/ 1h) and welded conditions, used in demonstration fast reactor reprocessing plant (DFRP) along with indigenous NAG 304L SS (from M/s MIDHANI). Results obtained after 100,250,500, 1000, 2x1000, 3x1000 h and 4x1000 h of operation indicated that the samples showed very low corrosion rates at all temperatures of testing. Non-destructive testing of the samples using eddy current and ultrasonic methods did not reveal any significant defects on the surface of the samples investigated. Examination of the tested samples by scanning electron microscopy (SEM) showed the grain boundary attack, and the samples tested in vapour phase condition showed significant deposits on the surfaces.

It is proposed to continue testing of samples up to 10,000 h of exposure. The long term operation of the loop and the resultant corrosion data of the materials, will provide valuable information on the life and performance of the actual components in the operating plants, and in qualifying materials under realistic conditions for reprocessing plant applications.

Results of the AISI 304L SS samples in different metallurgical conditions exposed to 6N nitric acid for 4850 hours under flowing conditions at a velocity of 1.25 m/s indicated an average corrosion rate of about 12 mpy at boiling temperature (Fig.4). The corrosion rates at lower temperatures of 80°C and 60°C were 0.4 mpy and 0.03 mpy respectively.Though flowing acid is seen to accelerate the corrosion rate of AISI 304L stainless steel specimen, the average corrosion rate is still within the acceptable limits. Samples of nitric acid grade (NAG) 304L SS showed much lower corrosion rates under similar operating conditions. The average corrosion rates were found to be in the range of 6.5 mpy under boiling conditions. The SEM images of the exposed DFRP & NAG SS samples upto 4850 h (Fig.5) in boiling and vapour phases showed progressive intergranular mode of corrosion attack indicating that IGC was common mode of attack. The analysis of the loop acid after each campaign indicated an increase in the concentrations of the corrosion products, namely, Fe, Cr and Ni ions. The rate of increase of these corrosion products was in direct correlation to the corrosion rate of the specimen (Fig. 6). As can be seen the corrosion rates directly follow the Fe concentration profile in the loop acid. It is an indication that the loop material is also corroding leading to the increase in the corrosion product concentration. This also simulates the actual plant condition, where in the corrosion products from the SS fuel clad and equipments can have an influence on the corrosion of the subsequent plant components.

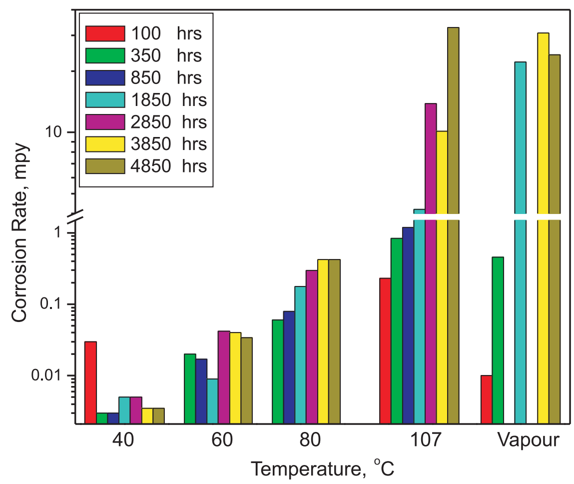


Fig.4 : Corrosion rates of AISI 304L SS exposed to flowing 6N HNO_3 at different temperatures.

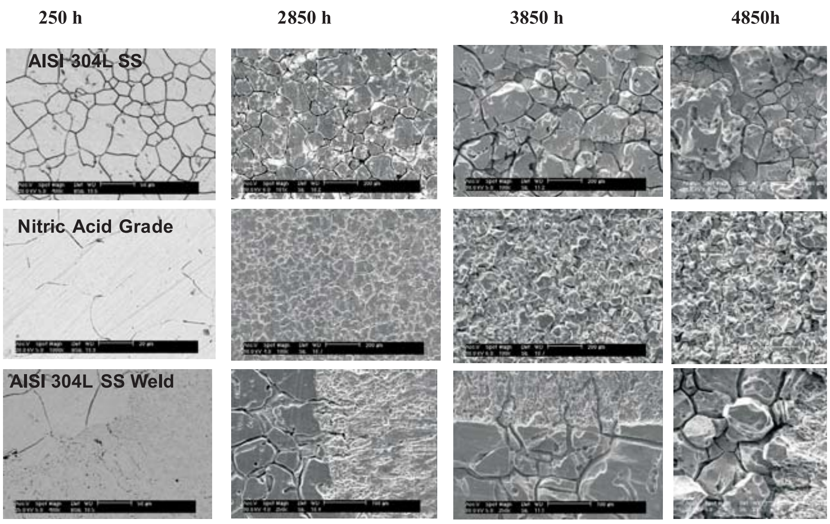


Fig.5 : SEM micrographs of AISI 304L SS specimen exposed to flowing 6N nitric acid at boiling temperature after 250h, 2850h, 3850 h and 4850 h.

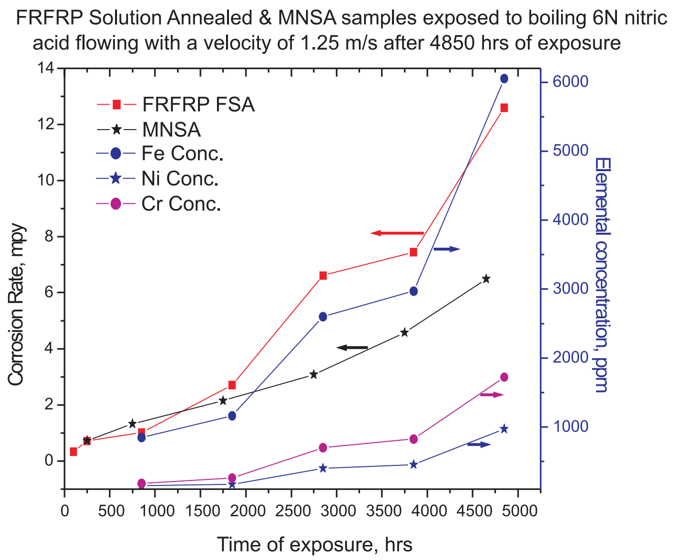


Fig.6 : Change in the corrosion rate and elemental concentration of corrosion products in the acid with time of exposure

Fig. 7: Titanium Gr-2 and Zircaloy–4 Corrosion testing systems

4.0 HIGH TEMPERATURE HIGH CONCENTRATION TEST SYSTEMS

In a fast reactor fuel reprocessing plant, higher concentrations of nitric acid (11.5 M) under boiling conditions are used for fuel dissolution unlike in the thermal reactor fuel reprocessing plants. Since stainless steels are prone to higher corrosion in such conditions, materials like titanium, zirconium and their alloys are considered for such applications. However, these materials undergo specific corrosion problems in the plant, though they exhibit excellent corrosion resistance in nitric acid medium. One such condition is the high corrosion rates observed in Titanium in the concentration range ~ 40-55% and also under condensing acid conditions. Zirconium has been reported to be susceptible to stress corrosion cracking in nitric acid medium under specific operating conditions.

In order have a greater understanding of the corrosion mechanism in these alloys and also to test candidate materials under plant simulated conditions, two test facilities have been set up at IGCAR (Fig. 7). One system is made of CP titanium Grade-2 with provision for exposing test samples to boiling nitric acid in liquid, vapour and condensing conditions. Provision is also made to test corrosion monitoring techniques based on Laser Raman Spectroscopy and electrochemical noise as online corrosion monitoring tools.

The second system made of Zircaloy-4 material has been fabricated using the electron beam welding technique at NFC, Hyderabad. In this vessel provision is made for exposing zirconium based test samples for their corrosion property evaluation. Both the systems have been commissioned at CSTD and testing of the first set of samples is in progress.

(Reported by V.R. Raju, R.K. Sole, U. Kamachi Mudali, R.K. Dayal Corrosion Science & Technology Division, MMG)

Knowledge Management Policy for IGCAR

The 21st century is ushering in a new phase of economic and social development which can be referred to as "Knowledge Economy", in which knowledge is the key factor in determining the organization's economic success or failure.

Knowledge Management is defined as a conscious strategy implemented in an organization to create, gather, store and disseminate the knowledge to those who need it in a timely manner to achieve organization goals. Knowledge Management has created a paradigm shift in the thinking that "Knowledge is Power to Knowledge Sharing is Power". Strength of the organization comes from effectively combining knowledge with the technological advances and the creative capacities of it's employees.

It is needless to emphasize that in R&D Organizations like IGCAR involved in complex and demanding technologies, the knowledge is considered as an asset as much as facilities and human resources. The strategy is to convert this knowledge in to the intellectual capital and leverage it for competitive advantage and achieving our mission.

We, in our Policy incorporate both the forms of Knowledge viz. explicit and tacit knowledge. Explicit knowledge is in the form of design reports, training manuals, log books, journal publications, internal reports etc. Tacit knowledge is in the minds of the people as experiences, observations etc. It is well known that capturing and managing the tacit knowledge is a much bigger challenge.

In IGCAR, we are leveraging the Knowledge Management Policy to achieve the following:

- Raising the quality of work by making available the right knowledge to the right people at the right time by reducing the search for the right information.
- Improving the productivity through higher level of reuse without redoing, relearning etc.
- Meeting growing demands by recruiting people and training them in the shortest time.
- Meeting the targets and achieving the mission, in spite of employee attrition.
- Managing task forces by collating workers from different environments.

Knowledge Management Strategy for IGCAR:

Knowledge Management strategy of IGCAR is structured around the answers for the basic questions. a) Where are we now? b) where do we want to be? and c) How do we get there ?

IGCAR being a more homogenous Centre, the knowledge resources and needs are well understood. The knowledge flow in the organization is known and requires an open approach. Appropriate authentication systems for accessing confidential information are being implemented.

The management's vision is to achieve world class leadership in the fields of Fast Reactor Technology and associated fuel cycles through the synergy of knowledge, resources, facilities and employees and use it for higher productivity and quality.

To achieve this a vibrant Knowledge Management Policy is essential.

Knowledge Management Policy for IGCAR:

IGCAR is a front line R&D Organization working in various advanced areas of Science and Technology with a lot of in house developments and innovations. Also the knowledge / information created has a longer life cycle.

About 1640 employees who had

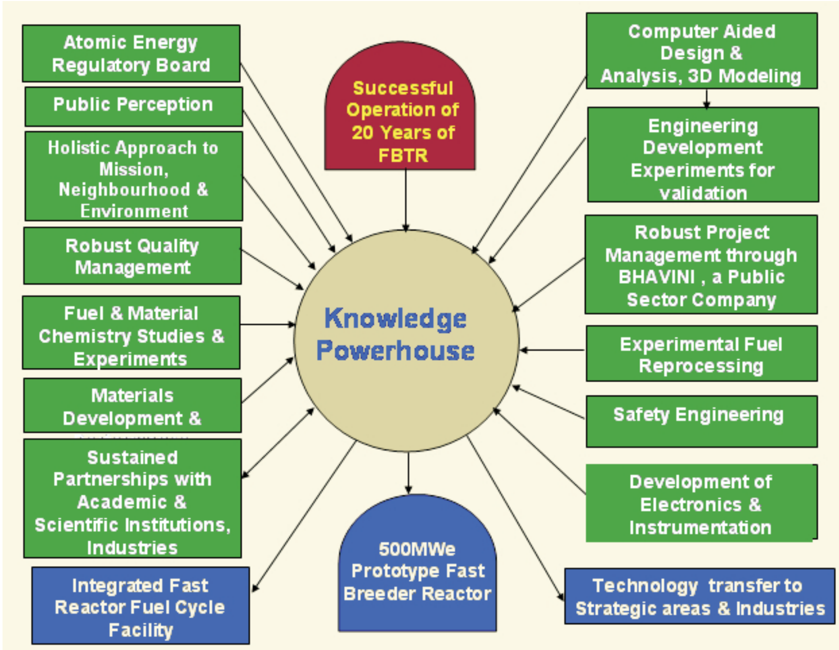


Fig. 1 : Knowledge Melting Pot at IGCAR

grown with the centre and contributed significantly to the development of various programmes had superannuated/voluntarily retired/resigned from RRC/IGCAR from it's inception till date. It is known that about 450 of our employees would superannuate in the coming 5 years and about 880 employees would superannuate in the coming 10 years. With them the invaluable expertise, experience and the knowledge would be lost from the organization. Hence, adopting and implementing a Knowledge Management policy is very important.

Knowledge Management Policy:

The Knowledge Management Policy of IGCAR is formulated as below :

"Indira Gandhi Centre for Atomic Research (IGCAR) will consistently endeavor through concerted efforts of all it's employees to generate, archive, manage and disseminate the valuable Knowledge for improving it's productivity and achieve & sustain world class Leadership in all it's Scientific & Technological Research and Development activities."

The Knowledge Management Policy in IGCAR is implemented with the participation of all the employees, management guidance and support. A mechanism is worked out(a model is given below) and is implemented by all Divisions/Sections for collecting, codifying and disseminating the knowledge which is in the form of both Explicit and Implicit forms. Efforts are made to codify and archive the knowledge generated by various groups from the inception of the Centre. This helps in reusing the valuable knowledge and avoiding redoing & relearning, thus improving productivity. These efforts go in parallel with the management of knowledge being generated now. The knowledge generated in various groups and through collaborations is being effectively utilized to achieve the mission of the centre as depicted in Fig. 1. The Knowledge Management Policy will be reviewed every year to find out any inadequacies and to strengthen the strategies for successful implementation of the Knowledge Management Policy.

At IGCAR we concentrate on both the forms of knowledge viz. a) Explicit Knowledge b) Tacit Knowledge.

Explicit Knowledge:

The explicit knowledge is in the form of design reports, internal reports, training notes, journal publications etc. This knowledge is being collected and stored in the form of IGC Information Management System (**http://igcims**) and the respective Information Management Servers of the groups. The knowledge which is available in the form of hard copy design reports, training manuals, publications etc. are scanned and converted in to electronic form and then stored in the respective servers. Most of the FBTR design reports, training manuals and publications prior to about 1990 are in hard copy form only. Hence all these documents are converted in to electronic form in "Mission mode" and archived in the servers. The present System of Information Management servers for each group is being taken forward and implemented more effectively. Towards this, every Division / Group has a Knowledge Management Officer designated, who will ensure that all the information generated is documented, stored in the servers and disseminated. At present, the Information Management System Committee members are collecting and storing the information in the respective group servers and the system is working fine.

The Heads of the Divisions ensure that all the project proposals, results, reports are documented. This shall include both success stories and otherwise. All the employees are encouraged to document all their efforts in the form of internal reports / publications.

Authentication of Explicit Knowledge:

Since we already have a system of checking and approving the design reports, publications etc. this information does not require special authentication. However, Division Heads will ensure that the correct revision of the report or publication is posted on the information server.

Tacit Knowledge:

Collecting the Tacit Knowledge, codifying it and storing requires lot of efforts. But with concerted efforts of all the employees, it is achievable.

Nonaka and Takeuchi advise that effective organizational knowledge creation best occurs through a spiral process of socialization, externalization, combination and internalization and through this process, the tacit knowledge is converted to explicit knowledge. The following system is planned for IGCAR to bring out the knowledge, expertise which is in the minds of the people and document it.

a) Every Division will conduct a technical lecture program for one hour every week on a specific day, where an employee shall explain the results of the findings, the methodology followed, the problems faced, the way they were overcome, the reasons for the options which he chose to solve the problem. This way the knowledge which is in the minds of the people and which is otherwise not documented will come out. The employee shall be encouraged to give salient points of his talk in the form of a document. This document or the power point presentation will be stored in the information management server.

b) Every discipline / domain shall have a colloquium like Chemistry Colloquium, Metallurgy Colloquium, Computers Colloquium, Electronics Colloquium etc. (some of them already exist and are active). The employees from respective domain will be encouraged to give a technical lecture in the colloquium at least once in a month where knowledge transfer takes place and the presentation which was made will be archived in the information management server.

c) The Section / Division head shall prepare precise and incisive quarterly progress reports of activities, results, achievements and failures etc. and post them in the information management servers.

d) The quality circles are encouraged and the deliberations recorded and stored.

e) The employees are encouraged to publish all their work / findings. If some of these works are not published in the standard journals, they can be in the form of Internal Reports. It is also planned to start at IGCAR an internal e-journal portal in different disciplines where various unpublished works will be posted. Also employees can forward their ideas , suggestions and problems which are relevant to the Centre's mission to the editors of this e-journal for porting.

f) A scheme is planned to collect the expertise and the knowledge from the employees retiring and retired in the form of reports, interviews, guest lectures etc. The Divisions will start the process of collecting this information at least a few months before the employee retires.

Authentication of Tacit Knowledge:

Verification for the correctness of the tacit knowledge being codified is very essential. The presentations made by the employees in the Division meetings / colloquiums can be authenticated after the lecture through discussions and the corrected version will be put on the information management server.

Various Groups of Experts in every domain like Physics, Chemistry, Metallurgy, Electronics, Computers, Reprocessing etc. will be formed to codify the knowledge collected from the employees (retiring / retired) in the form of lectures, reports and publications to e-journals. The editorial board of the e-journal will forward the articles to these expert groups for authentication.

In case, any of the findings / knowledge are not sufficiently backed by analysis/experiments , the information will be ported on to the server depending on its relevance in a folder called "observations".

Dissemination of Knowledge :

A clear policy for the dissemination of the knowledge among the employees is planned. All the Information Management Servers are connected to the Campus Intranet of the Centre and are accessible only to it's users. This information is not accessible to the Internet Users. The publications, general information, news about the achievements of the Centre , other utility information like user manuals, Accounts and Administration forms etc. would be available to all the employees.

The knowledge like Design reports, Training Manuals, Internal reports etc. are made available to those employees on **"Need to Know"** basis. Proper authentication mechanisms through Finger Print Reader ,User Id/Password etc. are planned to ensure that only the authorized users have access and that to for the required information. A special system would be worked out to archive "confidential" information and disseminate it to the selected authorized employees through proper authentication mechanism. These authentication mechanisms would be audited periodically like what is done for network information security.

Motivation for Sharing the Knowledge:

Contribution of every employee towards Knowledge Management initiative in the form of divisional lectures, presentations in the colloquium, contributions to internal e-journals will be permitted to be reported in the confidential reports, in addition to the journal publications, design reports etc. A certificate of appreciation is given by the organization to the Knowledge Workers who have contributed significantly.

Conclusion:

Knowledge Management plays a pivotal role in shaping the destiny of any organization in this challenging world of technological changes, requirement for faster design cycles, employee attrition etc. However, it is a challenging task worth taken ,to change the mindset of the people to share the knowledge instead of hoarding it. It is possible to achieve the objective with the cooperation of all employees and support and guidance from the Management. The employees are encouraged to share the knowledge and those who make significant contributions will be acknowledged and appreciated in a clear manner.

Suitable hardware and software technologies exist for smooth implementation. An empowered taskforce is entrusted with the responsibilities to identify and commission the hardware and software required (in the form of servers, software packages, new tools for storing and retrieving the information).

The Project will certainly succeed with the participatory support of all the stake holders from employees to the management at different hierarchies.

(S.A.V.Satya Murty, P.Swaminathan, Electronics & Instrumentation Group)

The α (*bcc*) to γ (*fcc*) transformation in steels: some less well-known basic aspects of a technologically important solid-state transformation

Steels, as a class of engineering materials are unique in many ways. Not withstanding their extensive industrial appeal, they are also storehouses of many an interesting physical phenomenon spanning diverse length and energy scales. The present report deals with one such issue that is generic to almost all grades of low carbon steels including the Cr-Mo variety that is of interest to reactor technology.

It is well known that steels derive their properties from material or application specific heat treatments; the foremost of all such treatments is the so-called homogenisation or solutionising step, in which the starting ingot or the raw piece from the melting shop characterised by severe compositional and microstructural inhomogeneity, is taken to a suitable high temperature single phase region and kept at this temperature for sometime, before finally cooled to room temperature. Albeit routinely carried out in labs and industries alike with quite reproducible end results, the physical phenomena behind this step are rather complex, and in a sense, not yet fully understood. The reason being, three possibly synergistic sub issues are involved in the realisation of a good homogeneous cast or wrought steel. They are:

(i) the $\alpha \rightarrow \gamma$ transformation that takes place upon crossing the *bcc/fcc* stability domain during heating,

(ii) concurrent dissolution of carbides (and other phases depending on composition) and its indirect effect in subtly modifying the $\alpha \rightarrow \gamma$ transformation kinetics in enabling a complete realisation of a single *fcc*-austenite region, and finally

(iii) the $\gamma \rightarrow \alpha$ reverse transformation on cooling, which in turn is considerably affected by the cooling rate, in addition to the holding time at the homogenisation temperature. It must be remarked that there is no simple way to enumerate a straightforward strategy to do a parametric modelling of the homogenisation treatment taking due account of all the key factors, for each one of these sub issues in themselves deserves a detailed study. The present report intends to highlight some of the less well-known aspects connected with the kinetics of $\alpha \rightarrow \gamma$ solid state transformation in low carbon, moderately alloyed iron alloys. The experimental technique involved in *Differential Scanning Calorimetry* (DSC), which for the present purpose may be defined as a sensitive calorimetric device, intended to provide accurate estimates of transformation temperatures and energies, besides offering useful estimates of the reaction kinetics.

In *Fig. 1*, a typical DSC thermogram of a 9Cr-1Mo-0.1C (all in

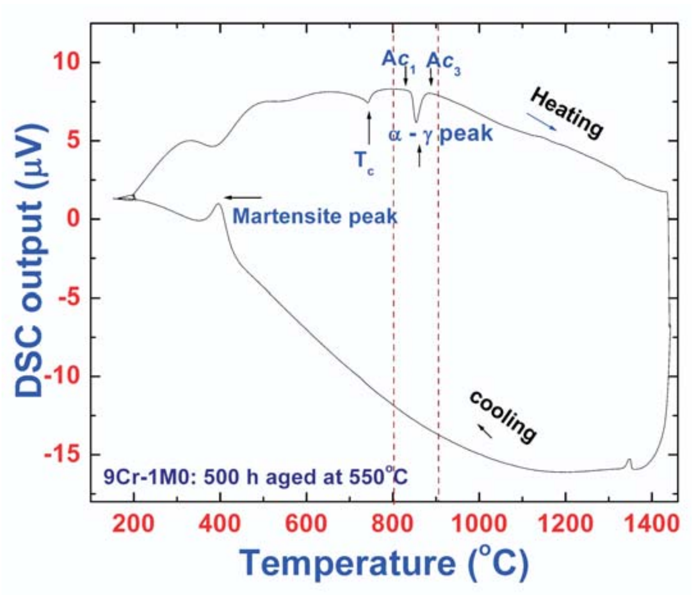


Fig. 1 : DSC thermogram of a 9Cr1Mo steel heated to 1400 °C and cooled to room temperature. The discussion focuses only on the α→γ transformation observed during heating.

wt.%) steel that is taken during a typical heating and cooling schedule is shown. Two transformation peaks corresponding respectively to the magnetic transformation (ferro magnetic to paramagnetic change at the curie point) and the α-*bcc ferrite + carbides* → *γ-fcc* austenite structural change are readily apparent. Focussing on the latter phase transformation, it may be said that in pure iron, this structural change occurs at a single temperature. But in Fe-0.1 %C alloys, the presence of carbon, opens up the $\alpha+\gamma$ two phase field quite a bit and this induces a phase transformation domain rather than a sharp transition point. In steel metallurgy, this transformation start temperature is known as Ac_1, and the possible transformation finish or arrest temperature is known as Ac_3. It is useful to note that both Ac_1 and Ac_3 are really kinetic quantities, in the sense that at very low rates of heating these points approach the equilibrium phase boundaries of a Fe-C phase diagram. But coming to practical experimentation possibilities, the austenite start and finish temperatures are very sensitive to the heating rate, in fact, lower the heating rate, smaller is the value of Ac_1 and Ac_3. But what is important to note is that this heating rate dependence of the transformation temperatures is strongly nonlinear, as exemplified in *figure 2*. The important point that emerges from this graph is that the existence domain of $\alpha+\gamma$ two phase mixture is widened for higher heating rates. The practical implication of this point is that higher homogenisation temperatures are needed, if faster heating rates are employed in the industrial homogenisation process. Processes like laser annealing etc., involves fast heating and cooling as well.

From a materials physics point of view, the possibility of superheating of alpha ferrite by employing faster heating rates suggests that at high temperatures, when the non-equilibrium overly heated ferrite is finally relieved of its instability, it transforms in a catastrophic manner to austenite, the transformation mode in this case being distinctly different from the normal one. This again has a telling industrial implication in that the austenite so formed is highly inhomogeneous (*defeating the very purpose of homogenisation*) and with considerable stress built up due to volume mismatch, the resulting product is prone to cracking upon cooling. One added problem is that the carbide dissolution is seldom realised under very fast heating. This brings us to the second less well-known or appreciated aspect of α→γ transformation and this has to do with heating rate dictated reaction kinetics.

In figure 3, a cryptic portrayal of the transformation kinetics is highlighted. The coordinates of the plot are $In(\beta/T_P^2)$ *and* $1000/RT_P$. β stands for the heating rate and T_P for the corresponding peak transformation temperature. For the purpose at hand, it is not necessary

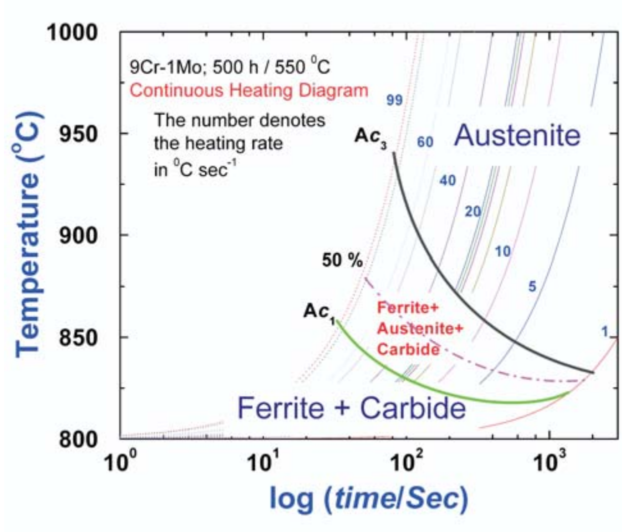


Fig. 2 : The continuous heating diagram for the a®g transformation in 9Cr1Mo steel. Note the pronounced nonlinear variation of austenite start (Ac_1) and finish (Ac_3) temperatures with heating rate, which incidentally is represented by thin dotted lines on a time versus temperature plot.

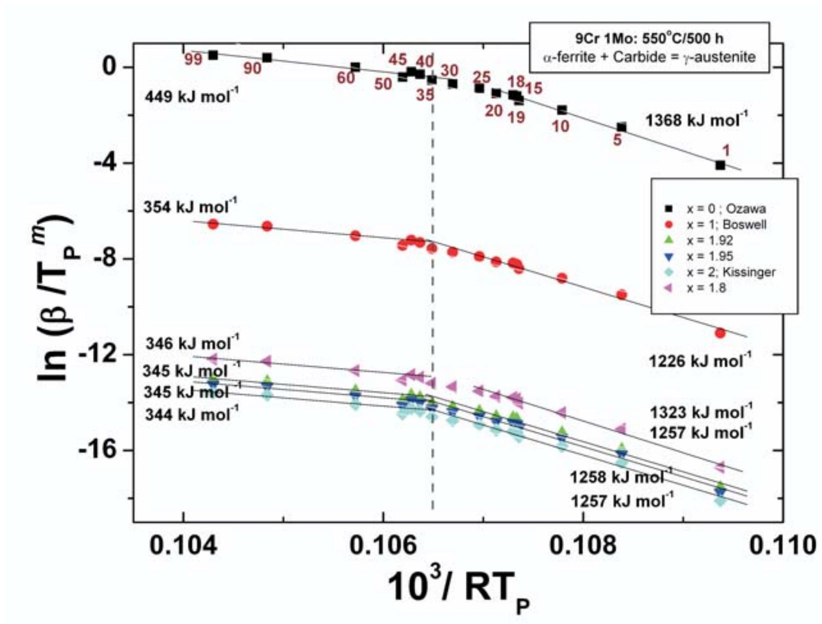


Fig. 3 : The activation energy scenario for the ferrite + carbide to austenite transformation in 9Cr1Mo steel. Note that there is a natural separation in to linear domains pertaining respectively to low and high heating rates, characterised by high and low activation energy values. The slope = $n E_{act}$, with n » 3 represents a measure of overall activation energy for the transformation.

to go into the details of the physics behind this graph. It is sufficient to observe that the slope of various straight lines in *figure* 3, gives a measure of the *overall* activation energy (E_{act}) of the primitive reaction step involved.

As can be seen that there is a huge difference in the activation energies estimated for higher and lower heating rate values. In fact, it is higher for smaller range of heating rates and a ramification of this paradox requires a clear enunciation of the unitary steps involved in the transformation process itself. For a given starting piece, irrespective of the heating rates, industrial homogenisation aims at achieving a fully single-phase austenite, *devoid of any carbide particle* that can be subsequently slowly cooled to get a homogeneous room temperature microstructure. But as the sample is getting heated during the process, two things take place. At about two-thirds of the Ac_3 temperature limit, first the carbide particles begin to dissolve due to the outward movement of carbon from the carbide into the matrix; this carbide dissolution is necessarily a slow carbon diffusion controlled process. Meanwhile, as Ac_1 is approached, the α-ferrite grains slowly begin to transform into γ-austenite, by what is known as small scale interface diffusion controlled process. This latter process, albeit a diffusion mediated one is comparatively faster as it involves only a few coordinated iron atoms movement *across* the transformation front. This kinetics is faster, as it is basically initiated at higher temperatures. Note that these two processes are concurrent and proceed with their respective velocities. But what is actually measured as a net activation energy is a complicated sum of these two mutually influencing processes. The manner in which they influence each other is sensitively dependent on heating rate. For slow heating, considerable carbide dissolution takes place, even before the initiation of austenite formation. But as the heating rates go up, carbides are rather stagnant and stubborn and only they serve as potential sites for austenite nucleation at appropriate temperatures. For intermediate rates of heating, both carbides dissolution and subsequent austenite nucleation are realised to different extents. The activation energy derives in this case from both carbide dissolution and austenite nucleation and growth. Finally, at very high heating rates, virtually no carbide dissolution is noticed at all, leaving only a trace of inhomogeneous austenite that is strewn with metastable carbides. The activation energy in this case corresponds to that of interface diffusion of iron atoms. Putting in a nutshell, the presence of a small fraction of carbide in the starting microstructure has a profound influence on the heating rate sensitivity of the homogenisation step.

Scientifically therefore, lower heating rates are preferred; but the process times and the heating power requirements are enormously large that they eventually eat into the overall process economy. Faster heating rates are commercially attractive and surprisingly easily implemented as well. Laser and pulsed direct energy related rapid heating sources are order of the day. The challenge then is to evolve an innovative hybrid of the two possibilities to yield an economically viable process option. With the advantage of hindsight gained by systematic lab scale experiments, it is possible to evolve a pulsed or cyclic homogenisation process, in which a stepwise rapid heating ramp is followed by a short isothermal anneal. The work piece is then heated to the next temperature increment, followed by even a *shorter* isothermal hold. Such a series of rapid heating + holding steps can lead to a better as well quicker way of realising a good homogeneous ingot. It remains of course, that the design of such processes need be wetted by careful simulation experiments, before the actual process line is initiated.

(S. Raju, B. Jeya Ganesh,
E. Mohandas, M. Vijayalakshmi,
PMD, MMG)

Forum for Young Officers

Determination of the hydrolysis products of KBF_4 in aqueous waste generated in the electro winning process of boron

Shri N. Ramanathan

N. Ramanathan obtained his M.Sc (Chemistry) from St. Joseph's College, Trichirapalli, Tamilnadu in 2001. He is from the 45th batch of BARC Training School and joined IGCAR in September 2002.

The electro winning process is employed for the production of enriched boron, which is converted to boron carbide and used as a control rod in PFBR. In this process, molten mixture of KBF_4, KF and KCl is electrolysed using mild steel cathode and graphite anode. The boron deposit obtained from the cathode is crushed, milled and leached with hot water to remove the residual salts from the deposit. The resultant effluent solution contains BF_4^- and its hydrolysis products. The determination of boron in effluent solutions is necessary, in view of the cost of enriched boron and the environmental impact of boron. While, ion selective electrode is ideal for the determination of BF_4^-, it cannot estimate boron present as the hydrolysis products or as boric acid. Various other methods such as inductively coupled plasma mass spectrometry, inductively coupled plasma atomic emission spectrometry are therefore used for the estimation of total boron.

Spectrophotometric methods using various dyes have been reported for the estimation of the boron species. In these methods, complex or ion-pair formed between boron species and the dye is extracted into the organic phase and boron is determined using calibration. In the determination of boron as tetrafluoroborate, none of these methods considered the hydrolysis of BF_4^-, as the analysis of the BF_4^- solution was carried out immediately after preparation. Hydrolysis of tetrafluoroborate results in fluoroboric acid species, such as BF_3OH^-, $BF_2(OH)_2^-$, $BF(OH)_3^-$ and finally to boric acid. When the analysis of the solution is not carried out immediately after preparation, the concentration of the hydrolysis products can be expected to increase; in such cases, neglecting the hydrolysis products of BF_4^- will result in underestimation of boron. Therefore, the real challenge involves the estimation of boron as hydrolysis products.

In this study, a spectrophotometric method for the determination of BF_4^- and its hydrolysis products is developed using malachite green (MG) and mandelic acid (MA). BF_4^- forms an ion-pair with MG (ion-pair A). In the presence of mandelic acid, MG forms a complex with boric acid (ion-pair B). Both the ion-pairs can be extracted into toluene for the spectrophotometric study.

Both the ion-pairs have the same absorption maxima at 632 nm. The concentration of the boron species in the organic phase is decided by the distribution coefficient of the ion-pairs, between the aqueous and organic phases. In order to calculate

TABLE 1

Characteristics of ion-pairs

Characteristics	Ion-pair A	Ion-pair B
λ_{max} (nm)	632	632
Beer's law range (M)	$6 \times 10^{-5} - 5 \times 10^{-4}$	$2 \times 10^{-5} - 2 \times 10^{-4}$
Distribution coefficient in toluene	0.031	0.099
% Extraction in toluene	3	9
Molar absorptivity ($Lmol^{-1}cm^{-1}$)	$6.67 \pm 0.65 \times 10^4$	$6.67 \pm 0.41 \times 10^4$

the distribution coefficient and molar absorptivity, the boron in the organic phase was back extracted into the aqueous phase and determined using ICP-AES. The distribution coefficient of ion-pair B was found to be three times higher than that of ion-pair A (Table1).

The calculated values of molar absorptivity are same for both the ion-pairs eventhough their distribution coefficients are different. This observation indicates that the absorptivities in both cases are basically due to that of MG. Boron, as boric acid, can be determined in the range of 2×10^{-5} M – 2×10^{-4} M, while boron in the form of tetrafluoroborate has a relatively poorer detection, the range being 6×10^{-5} M – 5×10^{-4} M.

Determination of the hydrolysis products

The strength of this method is in the estimation of hydrolysis products. Freshly prepared tetrafluoroborate solution was found to have the same absorbance when extraction with toluene was carried out using either MG or MA-MG whereas solutions of tetrafluoroborate which were allowed to stand over a period of time showed different extraction behaviour towards these reagents. With increase in storage time of the tetrafluoroborate solution, extraction carried out using MG was found to show a decrease in absorbance whereas extraction with MA-MG showed an increase in absorbance. This behaviour indicates that the hydrolysis products produced on standing form complexes with MA which were extracted with MG. Higher absorbance in MA-MG extraction suggests the distribution coefficients for these mandelate complexes are higher than that of tetrafluoroborate.

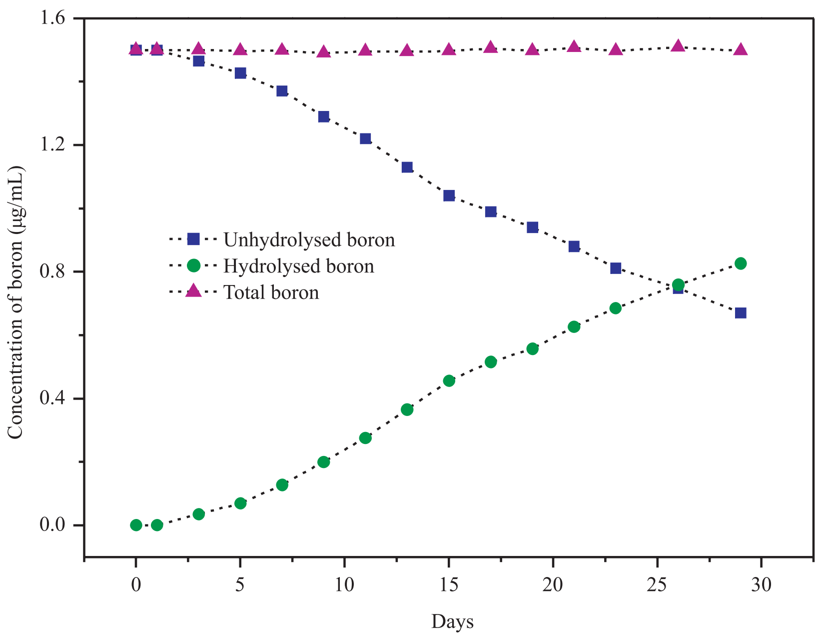
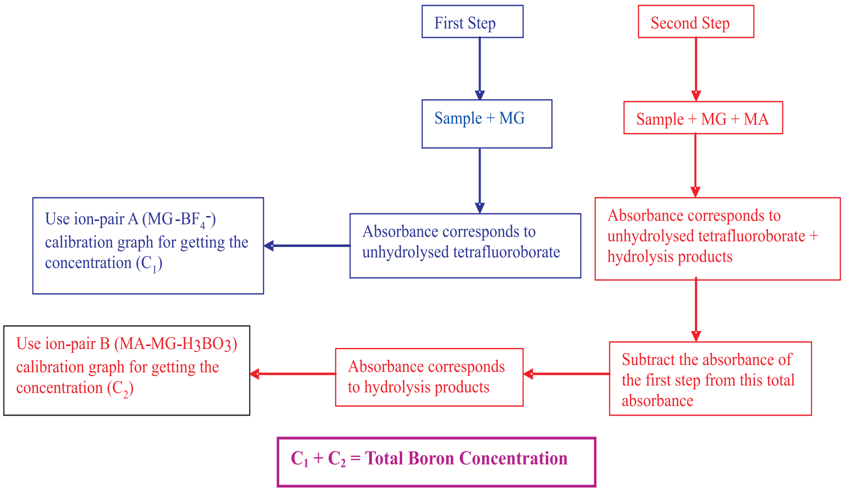


Fig. 1 Variation of concentration of boron, present as BF_4^-, hydrolysis products and total boron with time.

If the distribution coefficient and molar absorption coefficient of these hydrolysis products (BF_3OH^-, $BF_2(OH)_2^-$, $BF(OH)_3^-$) are same as that of boric acid, then the total concentration of hydrolysis products can be conveniently determined by using boric acid for calibration. Considering the difficulty in the isolation of individual hydrolysis products (BF_3OH^-, $BF_2(OH)_2^-$ and $BF(OH)_3^-$), the similarity in their extraction behaviour was further examined by monitoring the concentration of total boron, tetrafluoroborate and hydrolysis products with time. The absorbance of the organic phase measured with MG corresponds only to BF_4^-. Using the KBF_4 calibration plot (ion-pair A), the unhydroylsed tetrafluoroborate concentration was calculated. The difference in the absorbance between the extractions carried out with MA-MG and MG yields the absorbance due to hydrolysis products. The concentration of these hydrolysis products was estimated using calibration graph of boric acid (ion-pair B). The measured concentrations of tetrafluoroborate and the hydrolysis products as a function of time are given in Fig. 1.

As can be seen in Fig. 1, tetrafluoroborate solution stored for a day gives negligible concentration of hydrolysis products. On further storage, the concentration of the hydrolysis products increased and the concentration of BF_4^- decreased; however the total concentration of boron remains constant. This study implies the identical behavior between the hydrolysis products and boric acid in this extractive spectro-photometry using MA-MG. Also, this observation clearly suggests the



Scheme 1. Methodology of sample anaalysis

need to estimate both BF_4^- and the hydrolysis products especially when solutions containing tetra-fluoroborate are allowed to stand over a period of time.

Methodology of sample analysis

The determination of total boron in effluent solutions was carried out in two steps as given in Scheme 1.

Concentration values of boron measured in effluent solution samples were found to show standard deviation of less than 5 %, confirming the good precision of this method. The same solutions were also analyzed for boron using ICP-AES. The matrix effects in these effluent sample solutions were also checked by the method of standard addition and good recoveries (96%) were obtained. For all the samples, spectrophotometric results agree well with ICP-AES confirming the accuracy of this method.

(N. Ramanathan and Colleagues, Materials Chemistry Division, Chemistry Group)

Visit of IBSA delegation on Nanotechnology - April 6, 2007

A twelve member overseas delegation, comprising of six scientists from Brazil and six from South Africa led by Prof. J.A. Brum and Dr. T. Hillie respectively, visited IGCAR on April 6, 2007 under India-Brazil- South Africa (IBSA) initiative on Nanotechnology. Dr. Baldev Raj, Director, IGCAR, is the co-ordinator of IBSA nanotechnology initiative from India. The delegation also visited Indian Institute of Technology (IIT), Delhi and Inter University Accelerator Centre (IUAC), Delhi on April 2nd 2007, Bhabha Atomic Research Centre (BARC) and Tata Institute of fundamental Research (TIFR) at Mumbai on April 3rd and 4th 2007 and Indian Institute of Science (IISc),Bangalore on April 5th 2006. These visits were coordinated by Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam on behalf of DST, India. It was ensured that experts in nanotechnology from all parts of India participated in the discussions in at least one of the above mentioned centres, to present and evolve their ideas for collaborative projects under this initiative.

Prof. J.A. Brum, Brazil, Dr. Baldev Raj and Dr. T. Hillie, South Africa during the meeting at IGCAR on April 6, 2007

The IBSA nanotechnology initiative is a trilateral collaborative program between the Departments of Science and Technology of three participating countries, namely, India, Brazil and South Africa. The objective of this initiative is to formulate tri- and bilateral mega collaborative programs in the area of nanotechnology, of mutual interests to the participating nations. The accepted priority areas of common national interest to all three countries under this initiative include, advanced materials, energy systems, sensors, catalysis, health (TB and Malaria), water treatment, agriculture and environment.

(A. K. Tyagi, Metallurgy and Materials Group)

International Symposium on ADVANCES IN STAINLESS STEELS 2007 (ISAS 2007)

April 9-11, 2007, Convention Centre, Chennai Trade Centre, Chennai, India

Prof. E, Roos, MPA Stuttgart, lighting the lamp for inauguration of ISAS 2007. Dr. Sanak Misra, CEO, Mittal Steel Ltd., Shri S.K. Jain, CMD of NPCIL, Dr. S. Banerjee, Director, BARC, Dr. Baldev Raj, Director, IGCAR, Dr. K.Bhanu Sankara Rao, Convener, and Dr. P.R. Vasudeva Rao, Director, MMG, are also seen.

An International Symposium on **Advances in Stainless Steels 2007 (ISAS 2007)** was organised during April 9 – 11, 2007 by the Indian Institute of Metals (IIM), Kalpakkam Chapter in association with Metal Sciences and Ferrous Divisions of the IIM and Indira Gandhi Centre for Atomic Research, Kalpakkam, at Convention Centre, Chennai Trade Centre Complex, Chennai. The symposium was inaugurated by Prof. E. Roos, Managing Director of MPA Stuttgart, Germany in the presence of Dr. S. Banerjee, Director, BARC, Mumbai, Dr. Baldev Raj, Director, IGCAR, Kalpakkam, Shri S.K. Jain, CMD, NPCIL, Mumbai and Dr. Sanak Misra, CEO, Mittal Steel India Ltd. on 9th April 2007. Dr. P.R. Vasudeva Rao, Co-Chairman, welcomed the invitees and delegates. Dr. K. Bhanu Sankara Rao, Convener, explained the genesis and organization of ISAS 2007. Dr. S. Banerjee, Director, BARC and Vice-President and Chairman, Metal Sciences Division of IIM delivered the presidential address. Dr. E. Roos, Managing Director, MPA, Stuttgart lighted the *Kuthuvilakku*. The soft copy of proceedings and Souvenir was released by Shri S.K. Jain, CMD, NPCIL, Mumbai, and the exhibition was inaugurated by Dr. Sanak Misra, CEO, Mittal Steel India Ltd., Vice President and Chairman of Ferrous Division of IIM. Dr. Baldev Raj, Director, IGCAR Kalpakkam and immediate past president of IIM who is bestowed with Padmashri Award a few days ago by the Government of India, delivered the Plenary Address at the symposium. Dr. U. Kamachi Mudali, Co-Convener, proposed vote of thanks.

In the technical program, about 200 papers including 60 invited papers were delivered on various topics of importance including science and technology of stainless steels, manufacturing, welding, corrosion, mechanical properties, design, codes, modeling and applications of various types of stainless steels. Invited talks were delivered by experts from USA, Japan, Germany, UK, France, Sweden, Australia, Korea, Finland, China, Singapore, Hong Kong and India. An exhibition comprising 35 stalls with display of products, services, instruments, and applications dealing with stainless steels was also organized during the symposium.

About 400 delegates from 50 industries, 35 academic institutes and 40 research institutions participated in the symposium from 12 countries. 35 students working in the area of stainless steels, for their masters and doctoral programmes, were specially invited by providing financial assistance. The symposium covered a broad spectrum of topics spanning the entire life cycle of stainless steel - beginning from alloy design and characterisation; to engineering design, fabrication, quality assurance of components and finally in-service performance assessment, life prediction and failure analysis of materials and components, giving useful feedback for further developments for effective utilization of this class of materials. The technical programme was broadly classified under eight major themes including: Trends in alloy development and applications; Processing technology and characterization; Mechanical properties; Manufacturing technology; NDE methods; Surface modification; Corrosion and Component design, structural integrity and performance.

The eight themes were structured into 3 parallel sessions containing 10 oral sessions each, in addition to a poster session containing 54 papers. The technical presentations provided the opportunity for the delegates to appreciate the state of art R&D work ongoing, facilities available with industries for stainless steel melting, fabrication and manufacturing. The interaction among academia-industry-R&D laboratories also brought out areas where complementary expertise and facilities could be effectively utilized. The symposium also brought out case studies where the technology developed in R&D laboratories was transferred to industry for successful production of components to the stringent quality requirements. There is no doubt that ISAS 2007 would pave the way for many collaborative ventures between R&D and industry to meet future challenges.

A felicitation function was conducted on 9th April evening to honour Dr. Baldev Raj on his 60th birthday. Dr. P.R. Vasudeva Rao made the opening remarks highlighting the significant achievements and contributions of Dr. Baldev Raj. This was followed by felicitation speeches from Dr. P. K. Iyengar, Former Chairman, AEC, Dr. P. Rodriguez, Former Director, IGCAR, Dr. S. P. Mehrotra, Director, NML, Jamshedpur, Prof. B.S. Murthy, IIT-Madras, Dr. P. Barat, VECC, Kolkata, Prof. Frank Garner, USA, Prof. M. Kroening, Germany and Dr. S. Banerjee, Director, BARC. Dr. Baldev Raj and his family members participated in the cake cutting ceremony. Dr. Baldev Raj responded to the felicitation speeches at the end of the function.

The valedictory session of ISAS 2007 was conducted on 11th April evening. Dr. Farhad Tavassoli, CEA, France delivered an excellent technical talk on "Materials Data to Design". Dr. T. Jayakumar, Convenor, Technical Committee gave a brief summary of the papers presented at the symposium. Dr. L.K. Singhal, Director, Jindal Stainless presented awards to the winners of best technical papers. Dr. K. Bhanu Sankara Rao, Convenor of ISAS 2007 proposed vote of thanks.

(Reported by K. Bhanu Sankara Rao, Metallurgy & Materials Group)

Training Program on "Disaster Management" held at Ramanna Auditorium at IGCAR, Kalpakkam during June 22-23, 2007

For carrying out well coordinated rehabilitation works in the event of any natural calamity, a two day Training Programme on disaster management was jointly organized by IGCAR and the Center for Disaster Mitigation and Management, Anna University, Chennai. The members of all the rehabilitation committees attended the training program. The members of the local working committee to handle radiation accidents also attended the training program. The training program covered topics on Emergency health management, Psycho-Care of disaster affected communities, Management of man-made disasters, Coastal disaster management, etc.

The training program was inaugurated by Shri B.Bhattachargee, Member, National Disaster Management Authority, Delhi. Shri Bhattacharjee explained the structure of National Disaster Management Authority and the importance of the training program to be organized for members of various rehabilitation committees.

Shri K.Muralidhar, Member-Secretary, Crisis Management Group detailed the procedure to be followed to mitigate the crisis in the event of any calamity. Shri Muralidhar further explained the role of Emergency Response Centres, which are established at different parts of the country. He further emphasized the importance of public awareness, which play a key role in the emergency preparedness, and response plans for any type of emergency/disaster where participation/role of public is envisaged.

Shri P.R.Baidya from India Meteorological Department explained the causes for Tsunami and different types of Tsunami warning systems which are being commissioned for India.

Prof.A.R.Santhakumar, Anna University, detailed the simple procedures to be followed to strengthen dwelling houses to withstand earthquake or Tsunami.

Prof S.Rajarathinam, Director, CDMM, Anna University gave an overview on coastal disaster mitigation and management measures.

Prof J.S.Mani from IIT(M) explained about the various turbulences in the sea and protection features to be followed in shore.

Shri S.Vijayasekar, Dy. Director, Fire & Rescue Services, Tamilnadu explained about the procedures to be followed in the event of major fire.

Dr.Rajendiran from Govt. Hospital, Chennai spoke at length about the emergency health management during disasters. He detailed about the Triage sorting of the victims, viz. Red-Critical; Yellow-Urgent; Green-Less serious & Black-Moribund . Dr.Rajendiran further explained about the measures to be taken to revive the victims with vivid examples.

Shri B.Asir Vijayakumar, DSP illustrated different types of man-made disasters and the method of management of the same.

Shri N.Sathiyamurthy, Retired Home Guard spoke at length regarding role of civil defense and home guards in disaster management.

All the lectures were well received by the participants. The audio video modules are also prepared for all the lectures.The same will be played through cable TV to the residents of Kalpakkam and Anupuram townships.

(Reported by P.Swaminathan
Electronics & Instrumentation Group)

Fire Service Week Celebration - 2007

Shri P. Swaminathan, Director, EIG handing over prize to one of the winners

On 14th April, 1944, a major explosion took place on board ship S.S. Fort Stikline carrying explosives and other combustibles and berthed at Bombay Docks. The explosion and the fire that followed had resulted in massive destruction of ships, port facilities and residential area in the neighbourhood besides claiming lives of over 68 fire service personnel, about 150 port personnel and unaccounted number of civilians. In commemoration of the precious lives lost in the event, April 14 is being observed as the Fire Service day and the week following the day as the Fire Service Week (FSW).

The FSW is a grim reminder of the destructive potential of fire and need for fire prevention efforts. Every year fires take a heavy toll in terms of human lives and property loss. The estimates are 25,000 deaths and Rs.2, 700 crore worth property lost annually. It is expected that all sections of society, business and industry would review their fire safety needs, develop suitable strategies for fire prevention, fire control, rescue and rehabilitation and undertake a campaign for fire safety during the FSW.

In IGCAR a Fire Service Week was observed from 14th to 20th April 2007. The objective of celebration was to 1) create an awareness of the fire safety in all spheres of human activity, 2) Focus attention on fire prevention in work place, 3) Check operability of fire protection equipment and 4) Enable employee to control fire effectively. The program was attended by a large number of IGCAR employees including trainees from IGCAR Training School & BHAVINI.

Dr. K.K. Satpathy Head, E&ISS welcomed the gathering and emphasized the need for promotion of Fire Safety culture in IGCAR. Shri. S.K. Saxena, Deputy Chief Fire Officer, Kalpakkam gave a brief introduction about the Fire Safety Week. An inspiring technical talk on Fire Safety was delivered by Shri. V. Krishnakmurthy, Gen. Mgr. (Retd.), Safety, Environmental & Regulatory Affairs, International Flavour & Fragrance of India Ltd., USA, CA&ME.

As part of this celebration various competitions like Slogan, Essay, Poster, Quiz & Self Content Breathing Apparatus (SCBA) demonstration were conducted among employees of IGCAR. Twenty seven prizes were awarded to various winners.

Shri. P. Swaminathan, Director, EIG distributed prizes and in his brief remark emphasized the need for fire safety at work place.

The following won the First Prizes in various categories.1. Slogan Competition – Mr. B. Velayudham, ITG (Tamil), Mrs. Santhi Parathasarathy, RSD (English). 2. Essay Competition – Mr. K. Kalyanakumar, ROMG (Tamil), Mr. D. Ravindran, QAD (English), Mr. Umashankar, QAD (Hindi). 3. Poster Competition – Mr. D. Ganesan, ITG. 4. Quiz Competition – Mr. P.K. Chourasia & Mr. Rajesh Saxena, IDEAS. 5. SCBA Competition for DAE Fire Service Personnel – Mr. E. Sankar & Mr. A.G. Abdul Rasheed.

(Reported by K.K Satpathy, E&ISS, Sadety Group)

Forthcoming/Symposium Conferences

Recent Advances in Information Science & Technology (READIT-2007)

July 12-13, 2007

MAdras **L**ibrary **A**ssociation – **K**alpakkam **C**hapter is planning to conduct a two day National Conference on REcent Advances in Information Science & Technology" (READIT-2007) during July 12-13 2007. MALA-KC and Scientific Information Resource Division (SIRD), IGCAR have been conducting a series of conferences on "Recent Advances in Information Science & Technology" from 1995. The proposed conference will have invited talks by experts in the field and contributed paper presentations from Researchers. A pre-conference tutorial on *"Web Tools and IT Enabled Services"* is also being arranged for the benefit of Information Professionals on **July 11 2007.**

Call for Papers

The conference will consist of invited talks and contributed papers. Contributed papers, on any of the following theme sub-topics are welcome.

Theme:

Information to Knowledge: Technology and Professionals

Sub-topics

- Library as Knowledge Centers
- Digital Infrastructure and Technology Integration
- Document Management and Resource Generation
- Digital Preservation Issues
- Information Security
- Knowledge Environment and Professionals
- Knowledge – A Commodity

Important Dates

Pre-registration	: May 23, 2007(with abstract)
Intimation of acceptance	: May 30, 2007
Receipt of completed papers	: June 22, 2007
Last date for registration	: June 29, 2007

Address for Correspondence:

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AWARDS AND HONOURS

Dr. Anish Kumar, Metallurgy & Materials Group has won the INSA Young Scientist Award for the year 2007

Mr. Chittaranjan Das, Metallurgy & Materials Group has been selected for the ASM young metallurgist award, Chennai Chapter

Dr. Vidhya Sundararajan, Strategic & Human Resources Planning Section has received the Tamil Nadu Young Women Scientist Award for the year 2006