Laser Heated Diamond Anvil Cell facility for Synthesis of Novel Materials

**EXECUTIVE SUMMARY**

In the recent decade the Laser Heated Diamond Anvil Cell (LHDAC) technique has become the preferred tool to study materials at extremely high pressures and temperatures on a wide range not possible by conventional methods; (b) ultrapure sample environment; (c) highly localized heating of the sample. Applications of the LHDAC include exploration of P-T phase diagrams of materials, search for novel phases like superhard and harder-than-diamond materials, simulation of planetary interiors etc. Keeping some of these in mind, a state-of-the-art LHDAC facility has been set-up successfully to study materials under very high pressures (~1 Mbar) and temperatures (~5000 K). There are some unique features in this facility, like a fully automated scan routine to achieve uniform areal heating of the sample using nanomotion systems and an indigenous imaging arrangement.

**OUTLINE**

The Diamond Anvil Cell (DAC) is a tool par excellence to compress matter to very high static pressures. High strength of the diamonds coupled with their excellent transmittance to almost the entire electromagnetic spectrum has led to extensive in situ studies on a myriad of pressure induced phenomena in materials using X-ray diffraction and various spectroscopic techniques. This wide-range transmittance of diamonds can be exploited to focus high power IR laser beams onto the sample squeezed in the DAC, thereby subjecting it simultaneously to very high temperatures of over 5000 K and pressures of over many megabars. This technique, called the Laser Heated Diamond Anvil Cell (LHDAC) is emerging as an unique route for investigating materials and synthesizing novel phases in the hitherto unexplored P-T region. The LHDAC technique can be utilized for simulating, in the laboratory, the extreme environments such as those existing in Earth and other planetary interiors. At such extreme conditions, nature of the chemical bonding in elements changes and the chemical reactivities generally increase such that direct elemental reaction between even inert species becomes possible. This can lead to the formation of novel and exotic phases. Synthesis of nitrides of C, B, Si and Ge that are expected to rival diamond in terms of hardness is one of the frontline problems addressed by LHDAC researchers. Other applications of the LHDAC include study of melting phenomena and structural transitions in a variety of materials.

At Materials Science Division, IGCAR, a state-of-the-art LHDAC facility has been set-up recently for investigating materials behaviour up to ~1 mbar and ~5000 K simultaneously (Figs. 1 and 2). This facility essentially consists of an indigenously developed DAC, a 120 W CO2 laser (λ=10.6 μm; continuous wave; single mode), a X-Y Nanomotion system, and IR compatible optical components. In this technique, the challenge lay in heating the ultra tiny sample (~75 μm diameter) that is “squeezed” between the two diamond anvils to very high-temperatures using an invisible high power IR laser without causing damage to the diamonds and other supporting materials. This has been achieved using simple and cost effective ideas like a webcam–microscope combination for real-time imaging (now, upgraded with a CCD imaging system), indigenously designed vibration free tables and optical mounts. The use of the nanomotion system, for which the control software was developed inhouse, played a critical role in achieving the above successfully. A CCD based spectrometer has been included in the set-up to (a) record the grey body spectrum emitted by the sample for temperature estimation using Planck distribution; and (b) to record luminescence from a ruby chip, located inside the DAC, excited by 514.5 nm line from an argon ion laser for pressure determination.

Valuable experience in sample synthesis at high-pressures and high-temperatures have been gained using this IGCAR LHDAC facility. In particular, two experiments that demonstrated the capability of this facility and mastery over the difficult technique are conversion of graphite to a diamond-like translucent phase at ~14 GPa and ~2000 K and successful synthesis of the semiconducting material InSb by direct heating of In and Sb at ~2000K and ~0.5 GPa.
**EFFECTS OF PRESSURE AND TEMPERATURE ON MATERIALS BEHAVIOR**

Apart from expected changes in the unit cell volume, dictated by the Equation of State of the material under study, high-pressure affects the material in principally two ways, namely induction of structural and electronic changes. Structural changes include increased densification and phase transitions from one structure to another, generally involving change in crystallographic symmetry. Electronic changes result from increasing overlap of the electronic orbitals, leading to transitions like metal-insulator, normal-superconducting, magnetic-nonmagnetic, etc. Expectedly, structural and electronic effects are often interrelated and influence each other. Simultaneous application of high temperature enhances the kinetics and brings about interesting interplays with the pressure induced perturbation of the electronic and crystal structures, resulting in novel ground states of the material.

**PHASE TRANSITION OF GRAPHITE TO DIAMOND-LIKE MATERIAL**

It is well known that diamond is a metastable phase of carbon and can be synthesized, even without the aid of any catalyst, in the laboratory by subjecting carbon in one of its myriad forms like graphite or fullerene to very high pressures and temperatures. This experiment was taken up as the first problem in order to test the IGCAR LHDAC facility and gain experience in handling the difficult LHDAC technique.

A high purity graphite chip (linear dimension ~75 μm), sandwiched between two KBr layers was loaded in the DAC (Fig.3). The KBr layers were used to thermally insulate the sample from the diamond anvils as also provide optical windows. At a pressure of ~14 GPa and at a CO\(_2\) laser power of ~15 Watts, exothermic reaction characterised by flashes of very bright light got initiated. The temperature was estimated to be ~2000 K. Using the nanomovers, the sample was rastered by the CO\(_2\) laser beam. After the heating event lasting for nearly 45 minutes, it was seen that the entire graphite chip had become translucent to light, indicating formation of a new diamond-like translucent phase (Fig. 3). Photographs at the bottom depict views of the graphite sample through the table of one of the diamond anvils. The three photos correspond to the state of the sample before heating, during laser heating and after laser heating. It can be seen that after laser heating, the sample has become translucent to light.

In Fig.3, the red beam represents the heating CO\(_2\) laser (10.2 μm) while the green beam represents the argon ion laser (514.5 nm) used to excite ruby luminescence for pressure measurement.

**WHAT IS A DIAMOND ANVIL CELL (DAC)?**

DAC is a device used to generate very high pressures on materials. It essentially consists of two identical gem quality diamonds shaped in the form of anvils and aligned perfectly (Fig.3). One of them is mounted on to a piston and the other on to a cylinder. A pre-indentated 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. IGCAR DACs have been designed to do high-pressure experiments up to a maximum of 1 million atmospheres. To get a feel for the pressure, it may be noted that the pressure at the Earth’s core is about 3.5 million atmospheres.

**ACHIEVEMENT**

Very few laboratories across the world have mastered the LHDAC technique, owing to the intricacies involved and risks of frequent, irreversible damage to the diamond anvil cells (DAC). In IGCAR, several LHDAC heating experiments, each lasting for few hours have now been done. Careful inspection of the DAC has not revealed any damage to the cell or its constituents, demonstrating the high-level of perfection in the optics that has been designed and set-up. With this success, IGCAR is looking forward to establishing a similar facility centered around the XRD beamline at INDUS-II synchrotron (Indore). The published report on IGCAR LHDAC has attracted widespread attention and has received compliments from eminent scientists from within the country and abroad.

**PUBLICATIONS ARISING OUT OF THIS STUDY AND RELATED WORK**


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