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Studies of graphite surface after laser irradiation

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Abstract. Graphite was irradiated by CO_2 laser of energy 10 J and pulse width 4 μs. SEM and XRD examinations were carried out before and after laser irradiation. It was observed that XRD pattern changed after irradiation and new entities were seen on surface. Amorphous graphite was also irradiated and it was found that it became partly crystalline. The changes were explained on the basis of phase changes induced by high pressures and temperatures during laser irradiation.

Keywords. Graphite; laser irradiation; SEM; XRD.

1. Introduction

The graphite materials have been regarded as one of the promising candidate materials for fusion reactors. They are being widely used in present fusion devices and are also being considered for plasma facing component. However, several problems are being encountered in their use. The emission of particles or gases from them caused by high heat load can cause plasma contamination. It may also cause surface erosion. Work has been reported in literature regarding desorption studies after ion beam irradiation of graphite (Atsumi *et al* 1985). Davis and Haasz (1991) reported work on irradiation of graphite by helium, hydrogen along with the release of these gases after thermal treatment. The heat load effect can also be simulated by exposing the graphite surface to high heat fluxes, when laser beams of high energy impinge on the surface. Some studies on laser irradiation of graphite to give high thermal fluxes have also been reported (Muroga 1989; van der Laan *et al* 1992). The behaviour of other materials like B_4C (Deschka *et al* 1991) after high heat load or TiC coated graphite after laser irradiation (Benz *et al* 1987) have also been studied.

The laser beam of high energy, apart from building high heat load on the surface, can also cause instantaneous pressure built upon it, because of very short duration of the laser beam. This high heat load and instantaneous pressure build up by laser beam on the graphite surface can cause surface changes. They may also cause crystallographic transformation. Under high temperature and pressure, there is a possibility that diamond like structures are formed on the surface. This paper reports the results on the effect of laser beam on crystalline and amorphous graphite.

2. Experimental

Both graphite and amorphous graphites, were obtained from Graphite India, Bangalore. Samples of sizes 0·02 × 0·02 × 0·005 m were cut from graphite obtained

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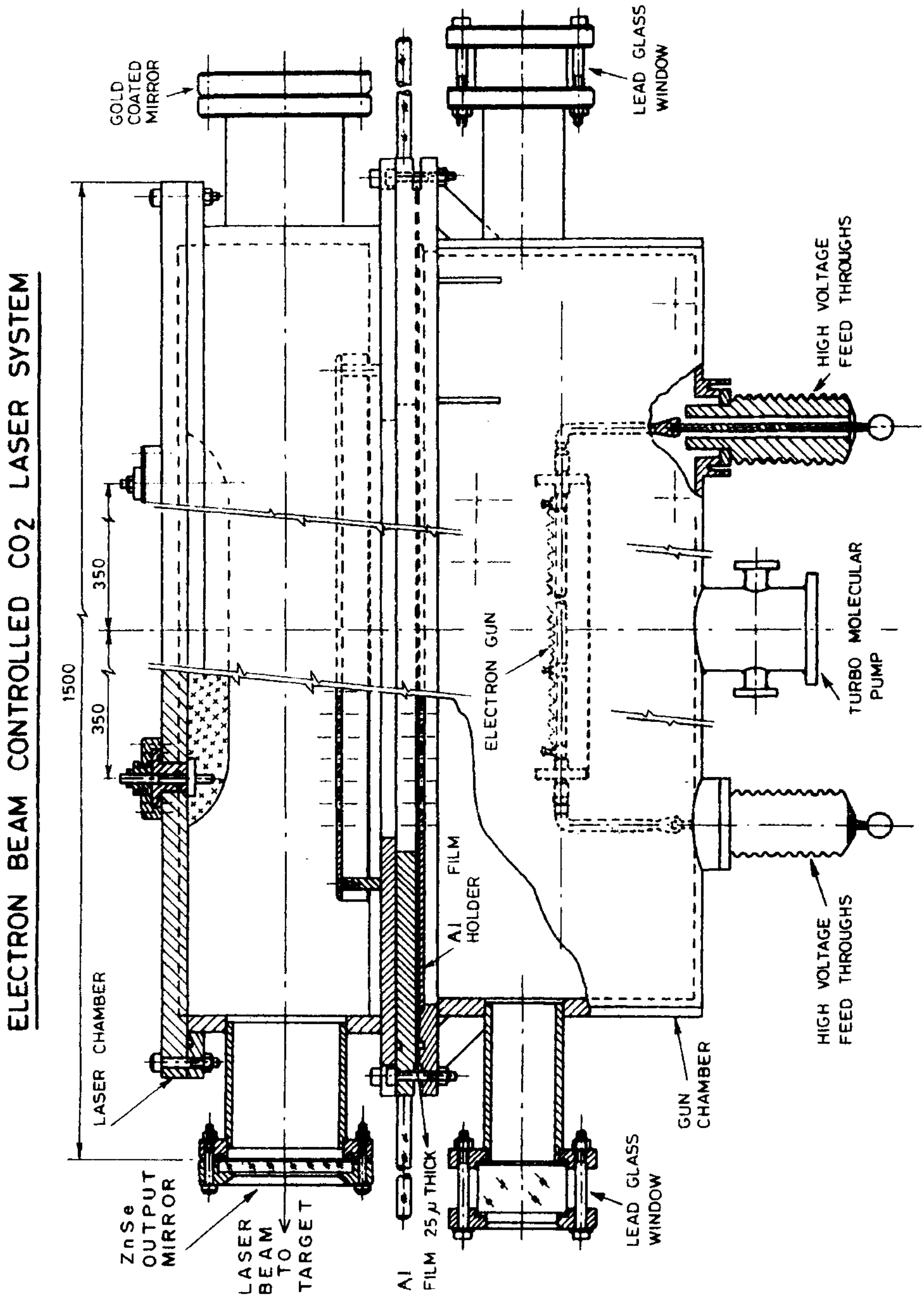

Figure 1. Schematic diagram of the CO_2 laser set up.

from vendors. In case of amorphous graphite the samples were of sizes nearly 0·01 × 0·01 × 0·003 m. The samples were polished on carborundum paper uptill 600 grit. They were washed with distilled water and alcohol and then dried thoroughly. Scanning electron microscopy examination (Jeol-330A) and X-ray diffraction (Phillips PW 1729) patterns of the sample were taken. Samples were then mounted on to the laser system for irradiation. The electron beam controlled CO_2 laser system was developed in BARC. Figure 1 gives a schematic diagram of the laser set up. It has a pulse energy of 10 J and pulse width of 0·4 μs (HMFW). The laser beam was focused on to the surface of graphite target with the help of a concave metal mirror. The mirror was gold plated and has a focal length of 50 cm. By adjusting the distance, the spot size on the sample was made to be 5 to 8 mm in diameter. Different samples were mounted on to the target and different number of laser shots were given to the different samples. It was ensured that after initial shot, subsequent laser shots fell almost exactly on the same spot on the sample. Weight of different samples of crystalline graphite was taken before and after irradiation. SEM examination of samples were carried out after laser irradiation and XRD pattern taken.

3. Results and discussion

Scanning electron micrograph of unirradiated graphite showed a smooth surface except for some polishing lines. XRD pattern of the unirradiated graphite is given in figure 2a. Here intensity vs 2θ is shown. It is seen that maximum in the intensity occurs

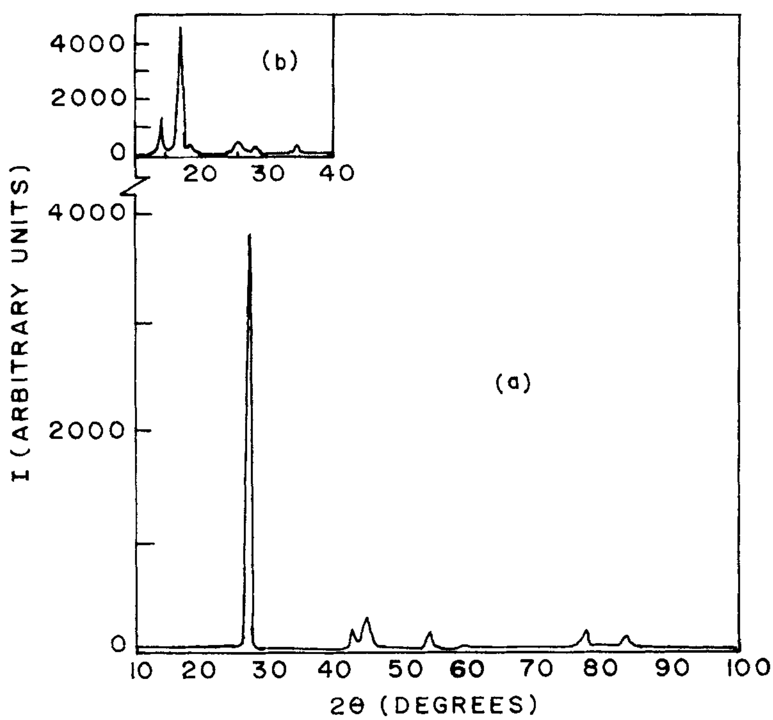

Figure 2. XRD pattern of unirradiated (**a**) and irradiated (**b**) graphite sample.

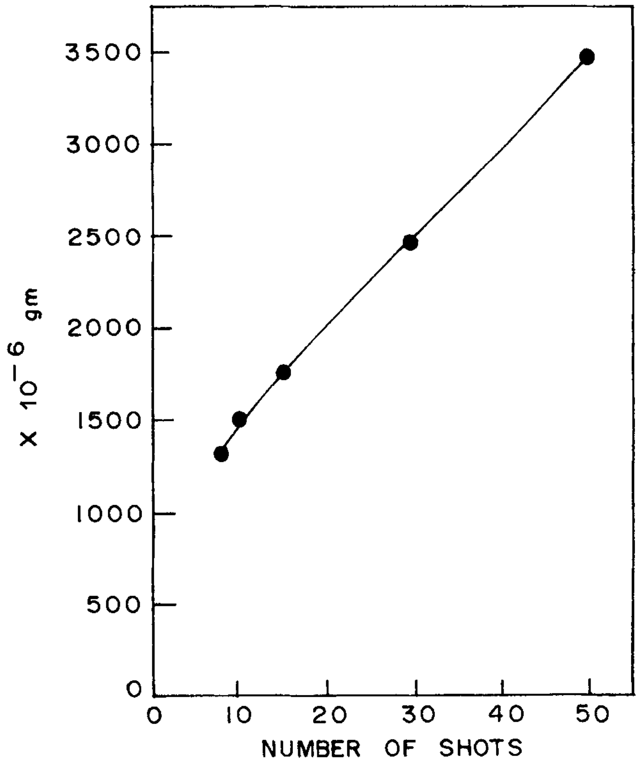

Figure 3. Weight loss of graphite vs number of laser shots.

at 26·4°. This corresponds to a d-spacing of 3·37 Å. Other smaller peaks are also seen. This is a typical graphite pattern.

In the present experiment, graphite samples were irradiated with different numbers of laser shots varying from 10 to 50. The weights of different samples were taken before and after irradiation. It was observed that there was no linear correlation between weight loss and number of shots incident on the sample (figure 3). This means that in the case of laser irradiation, the power deposited in each shot is not exactly the same. This implies that power built up in the system is not the same everytime. The weight loss in the present case occurs predominantly by sublimation because of high energy of laser beam (Franzen *et al* 1995).

After laser irradiation, XRD patterns showed variation from that of the unirradiated sample. After 50 laser shots, XRD pattern looked as in upper part of figure 2b. It was seen that 100% peak was observed at angles of 17·10°. This corresponds to a d value of 5·1 Å. The intensity of the preirradiated peak at 26·4° was reduced and there was a peak before 20°. These results imply that a new phase has been formed because of laser irradiation. Figure 4 shows the SEM micrograph of graphite surface after 50 shots. It is seen in this figure that some new entities have been formed on the surface. Raman spectra taken at this point did not show the typical diamond peak and as such the formation of diamond was ruled out. The X-ray peaks obtained were compared with X-ray data base available in CD–Rom for different types of

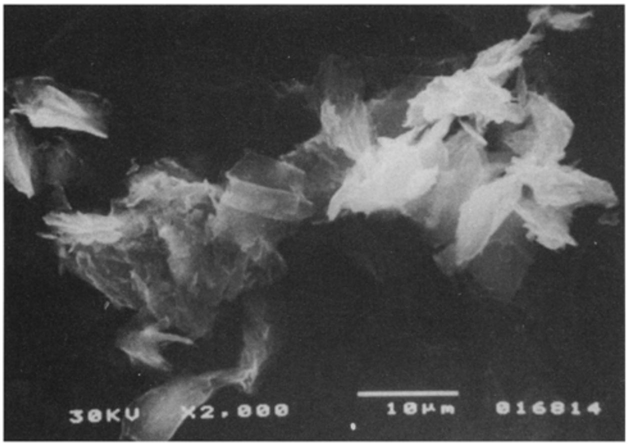

Figure 4. SEM micrograph of irradiated graphite sample with diamond like particles.

carbon. All peaks do not match with one given structure. They have been indexed by fitting peaks of different structures. In case of metals and alloys, it has been observed that laser irradiation produces phase changes on the surface of intermetallics (Goswami *et al* 1991; Weston and Wright 1989). In these cases phase changes occur due to change in temperature. Similarly for carbon, which has many phases, phase changes could occur.

To see whether the changes in XRD pattern after irradiation were intrinsic due to phase changes, few samples of amorphous graphite were irradiated by laser beam in the same system as in figure 1 by giving different number of shots. XRD pattern of amorphous graphite before irradiation is shown in figure 5a. There are no well defined peaks and the sample is amorphous. Upper part of figure 5b shows the XRD pattern of amorphous graphite which was irradiated to 50 laser shots. In this figure some definite peaks can be seen and they have been indexed with help of CD–Rom for carbon and match with choite type carbon. This fact confirms that new crystalline phase has been formed on the surface. SEM micrograph of same laser irradiated amorphous graphite is shown in figure 6. Some crystalline entities are seen here. These changes are arising due to the fact that during laser irradiation, in the present case, there is build up of stress due to sudden heating of surface in few microsecond. These stresses propagate into the surface and pressures are exerted onto different grains. These pressures were estimated to be 50 MPa at laser intensity, (I) of 10^8 watt/cm^2 and $\lambda = 10{\cdot}6\,\mu$, using the Manheimer *et al* (1982) formula

$$P = \frac{2{\cdot}4 \times (I/10^{13})^{2/3}\,\mathrm{Mb}}{\lambda^{(2/3)}}.$$

Furthermore, in addition to these pressures, there is also a sudden increase in temperature of the surface up to a few thousand degrees.

The temperature was estimated at laser intensity of 10^8 watt/cm^2 by comparing with Tarasov's (1983) estimation of temperature using laser's intensity dependence. It was of the order of 5000 K on graphite surface. In fact there is sublimation on surface, as

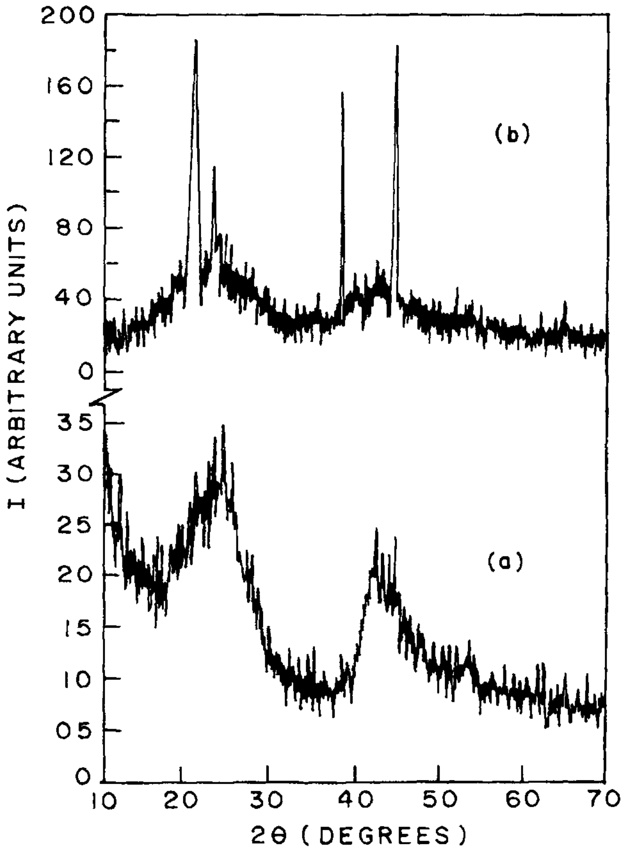

Figure 5. XRD pattern of unirradiated (**a**) and irradiated (**b**) amorphous graphite.

Figure 6. SEM micrograph of irradiated amorphous graphite.

observed by decrease in weight of the sample. At these pressures and temperatures graphites are known to get transformed into different structures and sometimes into diamond like entities. Vaidya (1995) also observed diamond formation in graphite (as confirmed by Raman line of diamond) under a high pressure of 5·5 GPa and 1300°C.

4. Conclusions

In the present study, it has been observed that there is a change in phase of crystalline graphite on laser irradiation to large number of shots. In case of amorphous graphite after laser irradiation, it gets transformed to crystalline structure. These changes arise because of rise in temperature and high pressure build up.

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