THE STRUCTURE OF GRAPHITE RESULTING FROM He-ION INJECTION

A.S. Rao and D.J. Bacon
Department of Metallurgy and Materials Science, The University,
P.O. Box 147, Liverpool L69 3BX, England.

1. Introduction

The use of graphite for components in nuclear-fission reactors has resulted in a large number of studies of the effects of neutron irradiation in this material. (For review see [1].) More recent suggestions [2] that graphitic materials could be used in a controlled thermonuclear reactor now require that additional studies of particle injection be made. In particular, the effects of helium atoms within graphite, which could result from either ion injection or the transmutation of fast neutrons and carbon atoms, should be investigated. In contrast to the extensive studies of metals in this area (see [3] for example), very little work has been reported on graphite. Mazey and Barnes [4] bombarded single-crystal flakes with various ions, and showed that twinning occurs. Ekern et al. [5] irradiated graphite-fibre cloths with 100 keV He^+ ions to a dose of 3×10^{18} ions/cm^2 at various temperatures. Scanning-electron microscope observations revealed flaking on room-temperature irradiated samples, but no damage was detected for irradiations at 400 and 800°C.

Transmission electron microscopy is a more sensitive technique for revealing damage, and the aim of the present work is to study by means of optical and T.E. microscopy the effects in graphite of He-ion bombardment for a range of bombardment temperatures, ion energies and ion doses. Damage effects have been observed, and this note presents a preliminary report of the observations made to date; it will be expanded upon at the conference.

2. Experimental Procedure

Both natural, single-crystal and pyrolytic-graphite (supplied by Union Carbide) specimens were used. The former were bombarded in an unthinned state for observation of surface features using optical microscopy. Transmission electron microscopy (at 100 kV) was undertaken on single crystal specimens repeatedly cleaved after injection and on pyrolytic samples which were cleaved, mounted on supporting grids and studied in the TEM prior to injection. The latter treatment ensured that any structural effects observed were due to the ion bombardment.

The specimens were injected with $^4He^+$ ions in the Heavy Ion Accelerator at AERE, Harwell, for ion energies in the range 70 to 100 keV and injection temperatures between 20 and 700°C. No noticeable ion-induced features were observed for ion doses $\approx 10^{16}$ ions/cm^2, and heavy erosion occurred for doses $\geq 5 \times 10^{18}$ ions/cm^2. Accordingly, only doses in the range 5×10^{17} to 10^{18} ions/cm^2 have been used for this study.

3. Results

An optical micrograph of a single crystal injected at 20°C with 100 keV ions to a dose of 10^{18} ions/cm^2 is shown in Fig.1. This unpolished surface was free of imperfections before irradiation, and it can be seen that the ions have resulted in twinning on a fine scale, as reported earlier in [4]. Twinning of this form was observed for all injection temperatures, but contrast associated with bulging of the surface between the twins was apparent for temperatures $\geq$ 300°C. The effect is more marked for higher doses and energies, and an example for 100 keV ions at 500°C and a dose of 10^{18} ions/cm^2 is shown in Fig.2. Numerous observations suggest that increasing the energy and dose widens the twins, and that an increase in the injection temperature increases the twin density, up to 300°C but marginally decreases it above 300°C. These trends are very weak, however, and are not yet supported by quantitative measurements. TEM analysis of the twins shows that most twins intersect the basal plane along either the $\langle 10\bar{1}0 \rangle$ or $\langle 11\bar{2}0 \rangle$ directions, as expected [6].

In the pyrolytic samples cleaved before ion injection, the density of twins produced by ion bombardment is much less than in the unthinned, single crystals, and twins are almost absent from very thin areas. At high irradiation temperatures, large blisters are formed. An example of a pyrolytic specimen injected at 700°C with 70 keV ions to a dose of 10^{18} ions/cm^2 is shown in Fig.3. The blisters take the form of well-defined domes which produce characteristic bend-contour patterns. In addition to the large blisters, ion-induced damage on a much finer scale can be seen in the matrix and blister dome, and this is currently being analysed [7]. Blistering effects are observed at lower bombardment temperatures, but as the temperature is reduced, the blisters become smaller and are less well defined. There is a good correlation between the surface bulging seen in the optical microscope (Fig.2) and the contrast observed in the TEM. An example for a natural-crystal sample bombarded at 500°C with 100 keV ions to a dose of 10^{18} ions/cm^2 is shown in Fig.4; the bend contours between the twins are clearly seen and they correspond to irregularly-shaped domes. It is not yet known if these are actually blisters, nor is it known to what extent twins and blisters are related or how they affect each other. In some areas at 700°C, blisters are observed at twin intersections and twins are seen around the perimeter of individual blisters.

4. Conclusion

The important new result reported here is that blisters are produced in graphite by the injection of the ions; a result now well-established for metals. The blisters are particularly well defined at high injection temperatures and are accompanied by defects distributed on a fine scale. Further analysis of these, the damage produced at low temperatures and the possible inter-relation between gas content, twinning and blister formation is in progress and will be reported at a later date.

Acknowledgements

The authors acknowledge valuable discussions with Drs. D.G. Martin, D.J. Mazey and D. Vesely, and the assistance of Mr. R. Copp with the ion injection. The work is supported by the UKAEA, Harwell.

References

1. P.A. Thrower, 'Chemistry and Physics of Carbon', vol. 5, p.217, Marcel R. Decker, N.Y., (1969).
2. G. Kulcinski and R. Conn, Nucl. Fusion, 15, 327 (1975).
3. Proc. Conf. on Surface Effects in Controlled Fusion Devices, J. Nucl. Mat., 53 (1974).
4. D.J. Mazey and R.S. Barnes, 6th Int. Congress for Electron Microscopy, Kyoto, vol.1, p.365, Maruzen, Tokyo (1966).
5. R.Ekern, S.K. Das and M. Kaminsky, Proc. 6th Sympos. on Engineering Problems of Fusion Research, San Diego, IEEE (1975).
6. E.J. Friese and A. Kelly, Proc. Roy. Soc. (Lond.), A264, 269 (1961).
7. D.J. Bacon, A.S. Rao and D. Vesely, unpublished work.

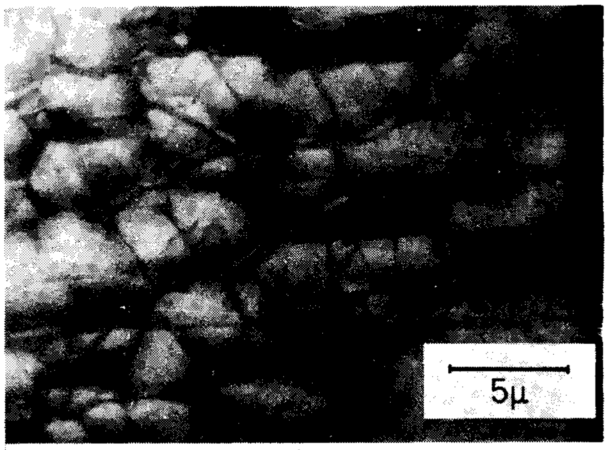

Fig.2

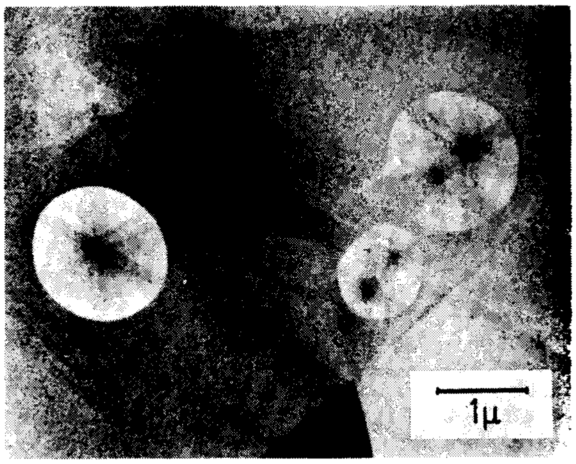

Fig.3

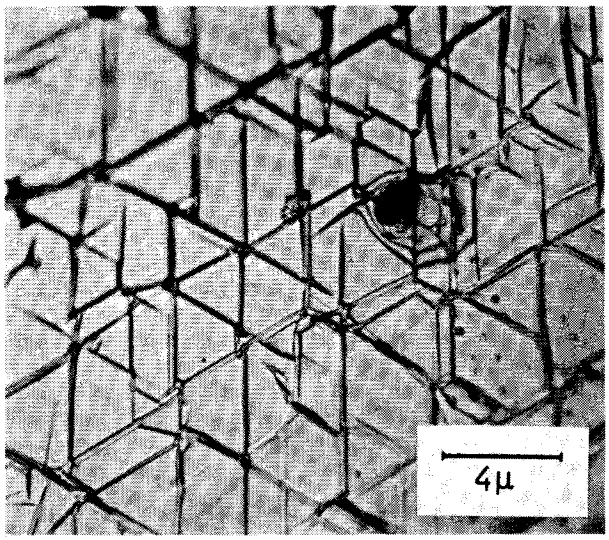

Fig.1

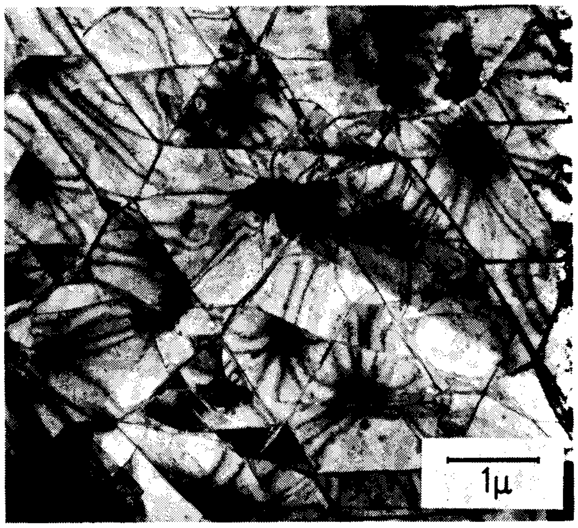

Fig.4