10. S. RABINOWITZ, A. R. KRAUSE, and P. BEARDMORE, *J. Mater. Sci.* to be published.

Received and accepted
25 February 1972

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Twin morphology in silicon carbide whiskers

Silicon carbide is of interest in many fields of modern technology. In the form of whiskers or filaments it is of possible use as the reinforcing component in fibre-reinforced composites. The high fracture strength and elastic modulus together with the chemical inertness conferred by the surface film of silica, lends the material to this application. The influence of microstructure and crystalline defects on these properties is self evident, and several investigations on this theme have been carried out previously [1-3]. Supplementary results concerning twinning behaviour in silicon carbide whiskers are reported in this publication.

Silicon carbide can exhibit complex crystal structures but is known to exist in its simplest, in the β-cubic (sphalerite) polytype and the hexagonal α-(wurtzite) form. Many more complex hexagonal and rhombohedral structures have been reported, Shaffer [4] having recently reviewed the subject. Optical and electron microscopy [1, 3] has shown silicon carbide whiskers to have a complex morphology although the growth habit appears to be mainly with the axis of whiskers along (1 1 1). Defects in the form of microtwins [1], and contrast bands perpendicular to the whisker axis [2] have been reported previously and explained on the basis of stacking faults or microtwinning on {1 1 1} planes. Further investigation of defects in silicon carbide whiskers has shown larger twins in both cubic and hexagonal crystallographic forms of the whiskers.

Whiskers of silicon carbide in the form of a close-knit matte were obtained from a commercial source. The matte was cleaned by soaking in HF for 30 min and washed in methanol prior to spreading between the faces of a 100 mesh electron microscope specimen double grid. Treatment with HF removed most of the protective SiO_2 surface film and facilitated penetration of the whiskers by the electron beam. The whiskers were then examined in a Phillips EM 300 electron microscope at 100 kV using a specimen stage having $\pm$ 6° tilt about one axis. Because of the large number of whiskers in any given field of view and the fact that the whiskers grow along primary crystallographic directions, whiskers in a suitable orientation for electron diffraction could be readily found.

X-ray powder diffraction analysis of the whiskers indicated approximately 95% α-phase (6H) to be present in the matte. In view of recent observations made on X-ray intensity of lines from both α and β-phases [5] and conflicting optical results, doubt must be cast on this technique when used with crystallites having a large aspect ratio. Nevertheless, nearly all whiskers which were transparent to the 100 kV electron beam yielded diffraction patterns which could be analysed in terms of the β structure. It is worth noting that the percentage of suitably transparent whiskers was small, $< 2\%$.

Of the SiC whiskers examined very nearly all exhibited defects in the form of stacking faults on {1 1 1} planes. Van Torne [1] has shown this type of defect to probably be a microtwin of minimum thickness. Also present, but to a lesser extent, were planar defects perpendicular to the whisker axis. These have been shown by Comer [2] to be multiple twins along the [1 1 1] axis of the whisker, the twin-matrix relationship being a 180° rotation. Such observations were reported on different material and since the type of defect might be expected to be dependent on growth conditions the observation of both types of defect in one whisker would be somewhat unexpected.

During the present investigation both types of defects were observed in the same whisker, Fig. 4. It can be seen that the central portion of the whisker contains numerous defects on {1 1 1} similar to those reported by Van Torne [1], whilst the ends of the whisker contains defects perpendicular to the [1 1 1] axis of the whisker. This observation would suggest that growth conditions changed slightly during this period of the whisker growth and that nucleation of growth

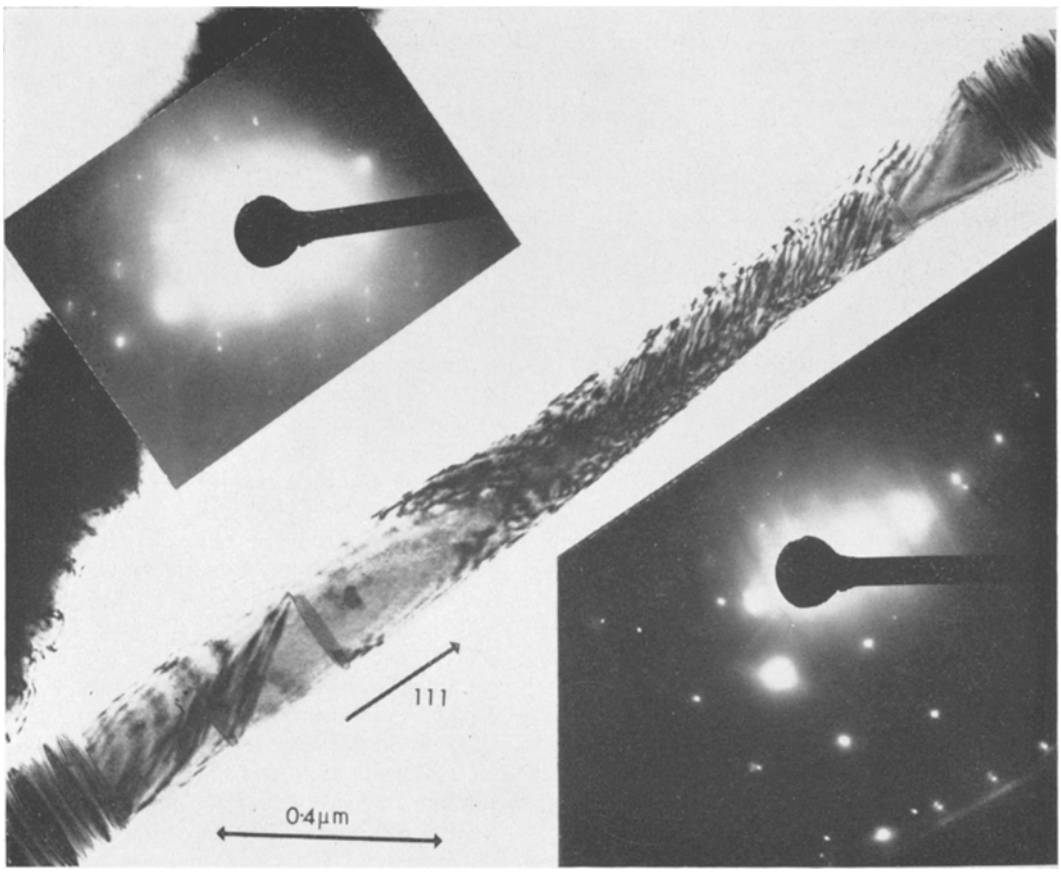

Figure 1 A whisker of SiC showing two forms of (1 1 1) microtwins. The diffraction pattern in the bottom right hand corner is from the end of the whisker, the other was taken from the centre.

twins readily occurs. Further evidence favouring this hypothesis is the low value of stacking fault energy for SiC reported by several authors [6, 7].

In addition to the microtwins which have a thickness $\simeq$ 12.5Å [1], defects having a much greater thickness have been observed, Fig. 2a. The selected-area diffraction pattern shows many extra spots and some streaking which are typical of twinning on {1 1 1} planes, Fig. 2b. Such patterns have been analysed and discussed previously [1]. The curving of the row of spots along the main axis of (1 1 1) can be explained by elastic strain in the whisker. Due to the large aspect ratio of the crystal, distortion is readily introduced during specimen preparation.

Dark field electron microscopy using a (1 1 1) diffracted beam to form the image confirms the nature of the defects as {1 1 1} twins, Fig. 2c. The light and dark bands at the perpendicular to the axis of the whisker at the edge of the twinned regions are evenly spaced thickness contours, the twin-matrix interface forming the wedge section necessary to obtain these contours. Thickness fringes in the twinned regions parallel to the axis of the whisker are also visible. These are not evenly spaced since they are caused by the change in thickness of the whiskers circular cross section. Both effects are more clearly visible under dark field conditions which also allow for more accurate measurement of twin thickness. Such thickness measurements indicate the twins to be one to two orders of magnitude larger than previously reported [1, 2].

A further type of twinning previously unreported has been observed. The twin plane occurs parallel to the axis of the whisker, effectively producing a bicrystalline whisker. The bright-field electron micrograph is shown in Fig. 3a, and at first sight the defect along the

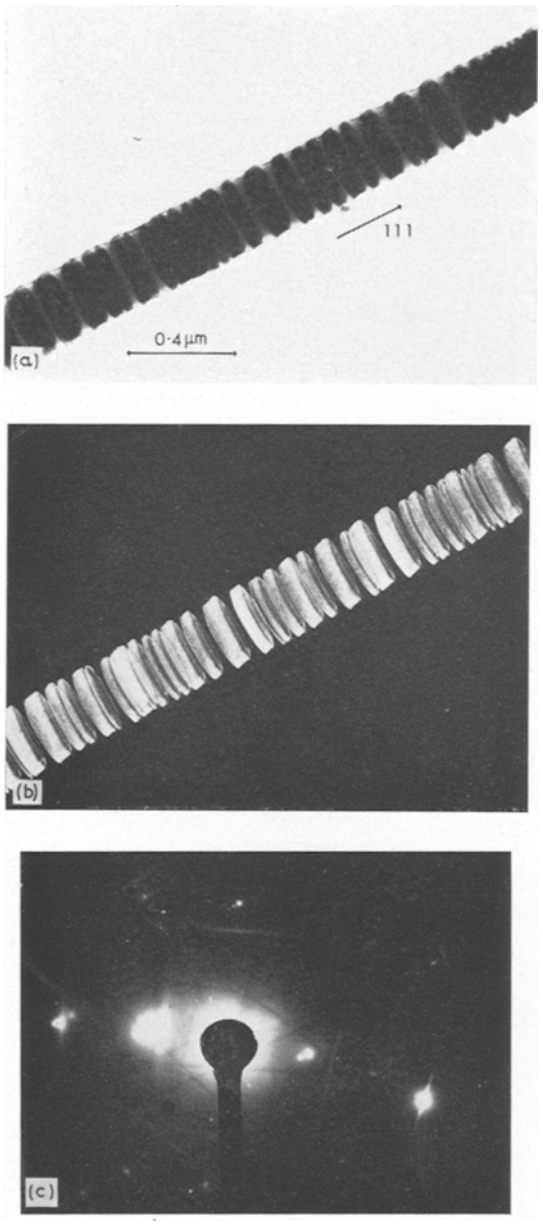

Figure 2 A whisker of SiC showing (1 1 1) twins. (a) Bright-field. (b) (1 1 1) selected-area diffraction pattern from the whisker. (c) Dark-field g = (1 1 1).

whisker axis would appear to be a dislocation. The diffraction pattern is typical of a hexagonal polytype and the micrographs have been indexed as such using three index notation. Using dark field illumination and **g** = (1 1 0) the whole whisker is in contrast, Fig. 3b. If a (1 1 0) diffracted beam is used to produce an image,

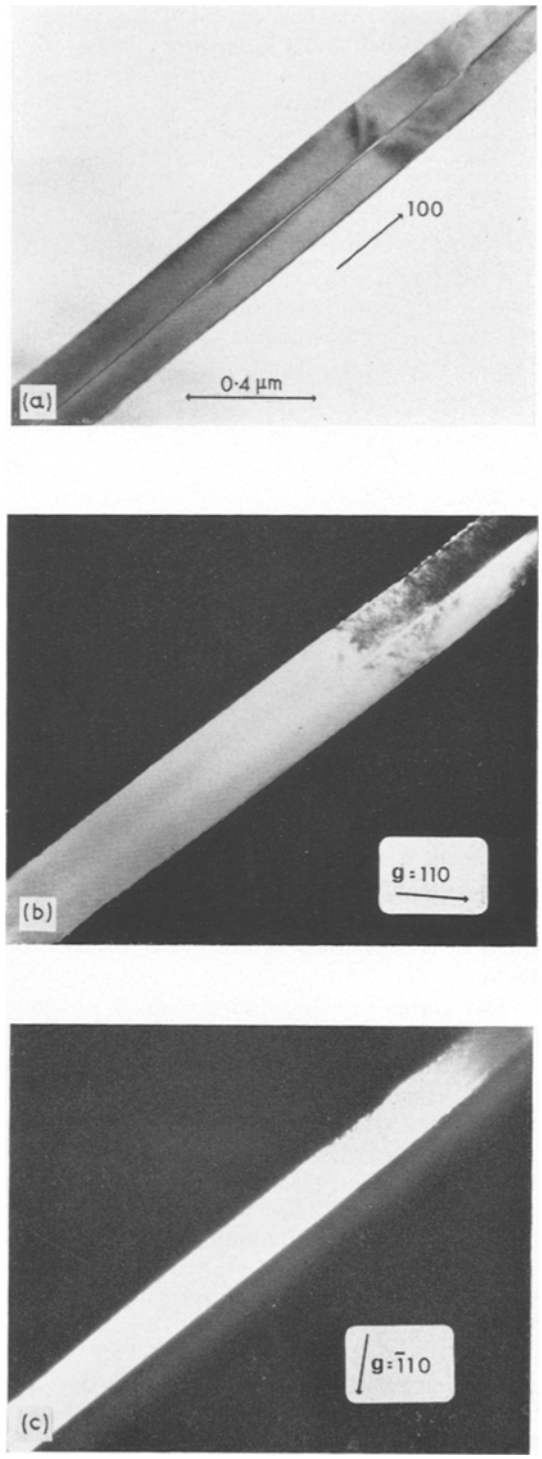

Figure 3 Ribbon-like α-SiC whisker twinned along its growth axis. (a) Bright-field. (b) Dark-field **g** = (1 1 0), (1 1 $\bar{2}$ 0), showing both twins. (c) Dark-field **g** = ($\bar{1}$ 1 0), ($\bar{1}$ 1 1 0), showing a single twin diffracting the beam.

only half the whisker is in contrast, Fig. 3c, indicating conclusively the twinned nature of the whisker. The twin plane is clearly parallel to the whisker axis. Unfortunately only limited tilting facilities were available and with the restricted information it is not possible to determine the twinning plane.

Acknowledgements

The persistent encouragement of Dr P. J. Dyne is gratefully acknowledged as is the support of Atomic Energy of Canada Ltd, Pinawa, where the work was carried out.

References

1. L. I. VAN TORNE, *J. Appl. Phys.* **37** (1966) 1849.
2. J. J. COMER, *Mat. Res. Bull.* **4** (1969) 279.
3. N. SETAKA and Z. INOUE, *J. Amer. Ceram. Soc.* **52** (1969) 624.
4. P. T. B. SHAFFER, *Acta. Cryst.* **B25** (1969) 477.
5. *Idem, J. Amer. Ceram. Soc.* **52** (1969) 51.
6. L. B. GRIFFITHS, *J. Phys. Chem. Solids* **27** (1966) 257.
7. R. STEVENS, *J. Mater. Sci.* **7** (1972) 517.

Received 9 February and accepted 1 March 1972

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Comments on "Order structures and dislocations in bubble-raft grain boundary" by Y. Ishida

The observations made with the bubble model [1] are in basic agreement with our concept of grain-boundary dislocations [2]. There are, however, some misunderstandings in terminology as well as in the implications drawn in our earlier paper.

Two types of grain-boundary dislocations have to be distinguished. First, there are those required to make up the mismatch between the actual orientation relationship and the exact coincidence angle; these boundary dislocations are usually not able to move conservatively. The second type of grain-boundary dislocation proposed by us [2] are mobile grain-boundary dislocations which originate from sources (e.g. grain-boundary junctions) and move in the originally "perfect" periodic structure of the boundary.

The experimental finding by Y. Ishida suggests that grain-boundary dislocations may have Burgers vectors that are smaller than the lattice vector. This is not in contradiction to our observations and the model proposed of grain-boundary sliding. On page 1056 of Ref. [2] it was explicitly stated that dissociation of boundary dislocations may occur, and may result in a Burgers vector smaller than the lattice vector, even if the vector of the coincidence lattice is large. Hence the results of the bubble raft experiments are in basic agreement with our original model as well as with other results obtained by the same method [3]. It may also be emphasized that we agree with Y. Ishida's ideas on the atomic structure of high-angle boundaries, since a similar model followed from recent computer calculations [4-7].

References

1. Y. ISHIDA, *J. Mater. Sci.* **7** (1972) 75.
2. H. GLEITER, E. HORNBOGEN, and G. BÄRO, *Acta Metallurgica* **16** (1968) 1053.
3. R. RAJ and M. F. ASHBY, private communication.
4. B. CHALMERS and H. GLEITER, *Phil. Mag.* **23** (1971) 1541.
5. H. GLEITER, *Phys. Stat. Sol.* (b) **45** (1971) 9.
6. M. WEINS, H. GLEITER, and B. CHALMERS, *Scripta Met.* **4** (1970) 235.
7. *Idem, J. Appl. Phys.* **42** (1971) 2639.

Received 14 and accepted 22 February 1972

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