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Dislocation Movement and Slip Systems in β-SiC

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Plastic-carbon replicas taken off the fracture surfaces of self-bonded SiC were found to have transparent flakes of β-SiC attached. Certain of the flakes contained dislocations which were shown to have moved during the fracture process. The dislocations were shown to move on a $\{111\}$ slip plane and to have a Burgers vector $a/2$ $\langle 110 \rangle$.

1. Introduction

Silicon carbide is known to exist in two forms, the α (hexagonal) and β (cubic), the hexagonal form exhibiting many polytypes. Both crystallographic forms have been the subject of intensive investigation [1-3], into the methods of growth and into the measurement and characterisation of their resulting properties. Because of the extreme difficulty in obtaining thin sections that can be used for observation of dislocations by electron microscopy, experimentation in this field has been somewhat limited. Thus observations on dislocations have been restricted to examination of surface effects in the form of etch-pit arrays.

Attempts have been made to deform both α and β silicon carbide at elevated temperatures [4, 5], although dislocations were only observed to move at 1900° C under creep condition in the α form and then in limited numbers. Hence electron microscopic observations on deformed silicon carbide would appear to have little chance of success in determining Burgers vectors and possible slip planes for dislocations. During the course of an investigation into the mechanical properties of a self-bonded SiC [6], carbon replicas were taken from the surface of fractured specimens and flakes of material were seen attached to the carbon film. Observations on these flakes formed the basis of the present investigation.

2. Experimental Techniques

Specimens of self-bonded silicon carbide, 5 cm $\times$ 5 mm square were fractured under four-point bending. Two stage plastic replicas were taken from the fractured surface and examined in an Hitachi HU 200 electron microscope equipped with a specimen stage capable of tilting $\pm$ 30° in two directions. With this, fracture chips could be oriented with their thinnest section perpendicular to the electron beam, and together with the deeper penetration available from the 200 kV [7] electron potential a small percentage of the fracture chips were found suitable for examination using transmission techniques.

3. Results and Discussion

A typical area of replica containing a fracture chip is shown in fig. 1. Smaller flakes of material were usually examined for reasons of transparency, and these were found to be mainly β-SiC. Larger chips which were not transparent,

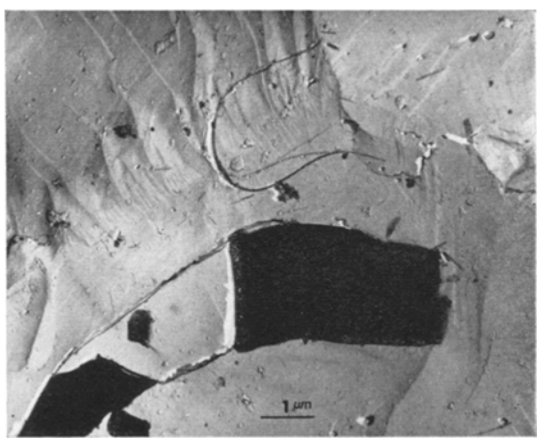

Figure 1 Two-stage plastic carbon replica of the fracture surface. The brittle nature of fracture is evident from the conchoidal appearance of the fracture surfaces. Approximately 1 to 2% of the area consisted of fracture chips.

were found to be α-SiC by obtaining diffraction patterns from their edges. The preponderance of thin chips of β-SiC is probably a consequence of the metallographic structure of the self-bonded SiC (fig. 2) which can be seen to consist of three distinct phases. The primary grains of SiC are surrounded by a thin coating of secondary SiC

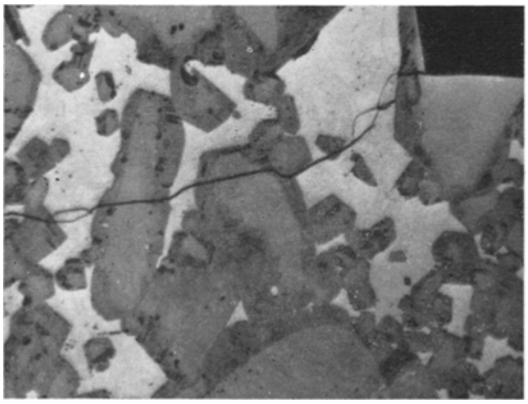

Figure 2 Optical micrograph of self-bonded silicon carbide showing the branching of the primary crack. The material was diamond polished and electrolytically etched in saturated KF + 2% HF before fracturing under four-point loading (×105).

which etches darker, the individual grains being held together by the free silicon phase. Removal of the secondary SiC from the primary grain by the carbon replica would produce a chip of fairly even thickness whereas if the primary SiC were picked up it would more likely be irregularly shaped and excessively thick, hence quite unsuitable for transmission electron microscopy.

Large numbers of flakes of β-SiC were examined individually, and very few were found to contain dislocations, such as those shown in fig. 3. Hence this micrograph, although containing only a fairly low density of line defects ($10^7 \sim 10^8$ cm cm^{-3}) should not be considered as representative of chips from over the whole fracture surface.

Of particular interest in the micrograph are the dislocation configurations at A and B. At A particularly, the configuration strongly suggests that a Frank-Read source has emitted three or four loops. A portion of the outermost loop appears to overlap one of the inner loops and suggests that part of the dislocation loop has moved on a cross-slip plane. The oscillatory effect at the ends of the loops even on the innermost loop suggests the ends of the dislocation loop are inclined at a small angle to the foil

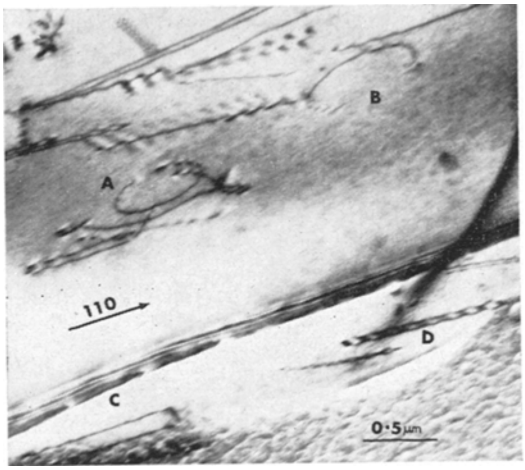

Figure 3 Transmission electron micrograph of dislocation sources and other features in a β-SiC fracture chip.

surface [8], indicating that the dislocation source occurred at or just beyond the surface of the fracture chip. The presence of the loops in the foil is probably due to the very high Peierls force retarding motion, preventing the loops shortening themselves after the generating stress due to the crack-tip has been relaxed by the fracture process.

A small-angle grain-boundary is evident at C, a misorientation of approximately 2° being indicated by the diffraction pattern. Free dislocations were found to lie along the $<110>$ directions as at D where the dislocation appears to have dissociated into partials separated by a stacking-fault. Unfortunately no tilting experiments were done on this particular fracture chip to obtain the Burgers vector of the dislocations. Later work on other fracture chips using the invisibility criterion [8] $\mathbf{g}.\mathbf{b} = 0$ showed the dislocations to have Burgers vectors consistent with $a/2<110>$, which is the shortest available slip vector in the $<110>$ direction.

Observation of the stacking-fault at D is in itself not indicative of a low stacking-fault energy, since the stress system generating the partials is unknown and is probably due to the nature of the foil. What are believed to have been stacking-faults have been observed previously in β-SiC as etched surface effects [9], and although the stacking-fault energy of SiC has not been measured, it is expected to be low because of the large numbers of known polytypes [2] and the nature of its crystal structure [10].

It can be seen in fig. 4 that the arrays of dislocations have produced slip traces, which is

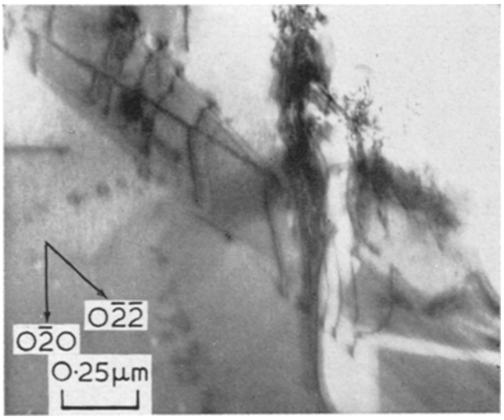

Figure 4 Slip traces in a (100) type fracture chip of β-SiC

direct evidence for dislocation movement in the fracture chip. The slip trace is still visible because of the method of preparation of the specimen. Usually slip traces are visible only for a short period of time after a specimen has been deformed in the microscope [8], depending on the strength of the oxide surface layer. Since the slip trace is a surface effect, the trace must have been formed at a free surface, indicating that the dislocation array moved in the direction of and produced the slip trace during passage of the crack in their vicinity, i.e. during the fracture process.

The slip trace of the dislocation array lies along the [0$\bar{2}\bar{2}$], with the projection of the length of the dislocation lying along [0$\bar{2}$0]. Surface trace analysis of these observations is consistent with the dislocations moving on the (001), ($\bar{1}\bar{1}$1) or ($\bar{1}\bar{1}\bar{1}$) planes. Since the diffraction pattern is a (100) type, the (001) slip plane can be eliminated, as all {100} planes must be parallel or perpendicular to the foil.

β-silicon carbide has the fcc zinc blende (sphalerite) structure (fig. 5), which can be seen to be closely related to the fcc diamond structure, each silicon atom taking alternate positions in the carbon lattice. Dislocations in the diamond lattice have been investigated theoretically by Hornstra [11], who concluded that there were three possible slip planes, the {001}, {110} and {111}, and that the {111} was likely to be most important. The shortest lattice vectors that are allowed as Burgers vectors are $a/2\langle 110\rangle$ or $\langle \frac{1}{2}.\frac{1}{2}.0\rangle$, i.e. half the diagonal of the cube face. These conclusions have been confirmed experimentally for the particular case of silicon and germanium [10].

Previously, dislocations in SiC have not been observed to be mobile below ~1900° C [4] and even then mobility was limited. Plastic deformation of vapour-deposited polycrystalline β-SiC has recently been reported [5] at temperatures in the range 900 to 1400° C. However, Gulden [5] believed a mechanism other than dislocation motion to be operative due to the temperature-insensitivity of the yield stress. Similarly in Al_2O_3, the bonding of which is also mainly covalent, macroscopic deformation by dislocation motion has been shown to occur at temperatures above ~1000° C [12]. However, dislocation motion has been observed [13] in thin flakes taken from the surfaces of specimens fractured well below this temperature, and the observations used to explain details of the mechanical properties.

It can be readily shown that the dislocation movement is produced under the special circumstances of stress concentration and geometric constraint at or near the crack-tip. The shear stress τ necessary to operate a Frank-Read source is given by

$$\tau = \frac{Gb}{l} \tag{1}$$

where G = shear modulus $\sim 1.68 \times 10^5$ kg mm^{-2}; b = dislocation Burgers vector = $a/2$ [110] $\sim$ 3Å; l = distance between pinning points of the Frank-Read source.

Substituting values of these constants into equation 1 and using a value of $l \sim 600$ Å (fig. 3), we have

$$\tau = \frac{1.68 \times 10^5 \times 3 \times 10^{-8}}{600 \times 10^{-8}} \sim 84 \text{ kg mm}^{-2}.$$

The maximum outer fibre stress calculated from measurements made on specimens broken under four-point bending was 13.35 kg mm^{-2}. Since, due to the nature of the chip, some relaxation must have occurred after passage of the crack, a minimum stress concentration of ~6 must have been operative to generate the dislocations at the sources, which is not an unreasonable observation.

4. Conclusions

(i) Dislocation generation and motion can occur at room temperature in β-SiC.

(ii) Generation and motion of dislocations occur only on a very limited scale and are probably due to the special conditions of stress concentra-

tion and geometric constraint occurring at the crack front.
(iii) Dislocations have been identified as having $b = a/2 <110>$, and the operating slip plane to be $\{111\}$.

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