

Birefringence, X-ray Topography and Electron Microscope Examination of the Plastic Deformation of Diamond

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ABSTRACT

The defect structures of types I, II A and II B diamonds were examined by the techniques of birefringence, x-ray projection and section topography and electron microscopy. It is suggested that the higher critical resolved shear stresses required to plastically deform type I diamonds compared with type II diamonds are due to the differences in the initial defect distribution in the two types of diamond. Type I diamonds have an extremely low dislocation density and a high density of coherent nitrogen precipitates in the cube planes, whereas type II diamonds have a high dislocation density (in excess of 10^7 lines per cm^2) with no nitrogen platelets present. There was no evidence for the relief of internal strains by heating the diamonds to 1800°C , although strains at surface scratches appeared to have been decreased by the heat treatment.

§ 1. INTRODUCTION

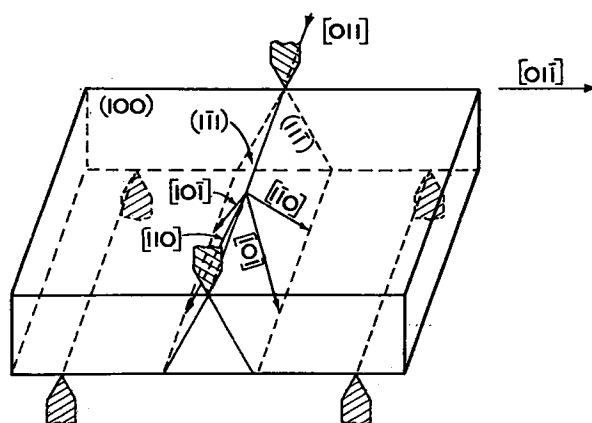
WORK on the plastic properties of single crystals with the diamond-type structure at elevated temperatures has shown that the initial defect structure of the specimens can be important in determining yield phenomena and subsequent deformation characteristics (see, for instance, Bell and Bonfield 1964, for tensile properties of germanium). Recently Evans and Wild (1965) reported experiments in which diamond plates were plastically bent by three-point loading at 1800°C . Type I diamonds (which contain precipitated nitrogen platelets in the cube planes) required a higher resolved shear stress to initiate plastic deformation than did type II specimens (which do not contain nitrogen precipitates). Examination by electron microscopy showed that the nitrogen platelets in the type I specimens hindered the movement of dislocations and thus contributed to the higher critical resolved shear stresses required for the deformation of the type I specimens. Because this type of examination involves the destruction of the specimen it was not possible to obtain information about the initial dislocation distribution in the specimens before they were plastically deformed. This is rather serious because the initial density of

dislocations has been shown to have a strong effect on the yield stress for another material of the diamond-type structure, namely germanium. For this reason diamond plates were examined by x-ray topography and by recording the birefringence patterns before deformation.

§ 2. EXPERIMENTAL METHOD

Eight diamond plates with dimensions of about $5 \times 3 \times 0.5$ mm were used in the experiments. The large faces were approximately $\{100\}$. The plates were classified by measuring the ultra-violet absorption between 2000 and 3500 Å and also by measuring their electrical conductivity. Four of the plates were type II B, two were type II A and two were type I diamonds. The type I specimens were obtained by cutting a larger plate along a $\langle 110 \rangle$ direction into two equal parts. Figure 1 shows a plate and its orientation relative to three tungsten wedges with which plastic bending

Fig. 1



Orientation of diamond plate relative to three tungsten wedges showing the four possible slip systems.

could be produced at 1800°C. The orientation convention shown in the figure will be used throughout the paper to index the various observations. Birefringence patterns were photographed for all the specimens so as to record the distribution of initial strains in the plates. X-ray projection topographs (Lang 1959) of the specimens were then recorded. Two topographs of each specimen were taken, one with the (022) reflection operating and the other with the $(02\bar{2})$ reflection. Dislocations in the diamond structure have a Burgers vector of the type $\frac{1}{2}\langle 110 \rangle$. Thus the use of these two reflections ensured that all dislocations were detected since no dislocation could satisfy the condition $\mathbf{g} \cdot \mathbf{b} = 0$ and be invisible, or nearly so, in both reflections. The four type II B and the two type II A diamonds contained a high density of lattice imperfections and in the case of the type II B specimens there were strong indications of a mosaic-block

type of structure. The dislocation density was too high for clear resolution of individual dislocations in the type II specimens; the density was probably everywhere in excess of 10^7 lines per cm^2 . In such crystals the addition of a large number of dislocations as a result of plastic deformation would be readily measurable by x-ray topography. However, the initial stages of plastic deformation would have been difficult to follow in the presence of the high density of dislocations already present. For this reason no further experiments were done with these specimens. In both of the type I specimens, on the other hand, not more than three or four dislocations were present initially. Previous examinations by electron microscopy (Evans and Phaal 1962, James and Evans 1965) also indicated an extremely low dislocation density in this type of diamond. Attempts were made to deform plastically the two type I diamonds at 1800°C by three-point loading. Previous experiments (Evans and Wild 1965) had shown that very high resolved shear stresses (above about 10^{10} dynes cm^{-2}) were required to produce detectable plastic deformation with this type of diamond. Stresses of this magnitude are close to those necessary to produce brittle fracture in diamond (Bowden and Tabor 1964). Three-point loading experiments were done on the two plates at appropriately high stresses. After the load had been maintained for several minutes, the plates cleaved parallel to and near the centre wedge. The pieces were examined with an optical microscope and the birefringence patterns photographed. X-ray projection and section topographs (Lang 1957) were taken with the $(02\bar{2})$ and (022) reflections being used. Finally the pieces were thinned sufficiently by oxidation for examination by transmission electron microscopy. The thinning procedure has already been described (Evans and Phaal 1962).

§ 3. RESULTS

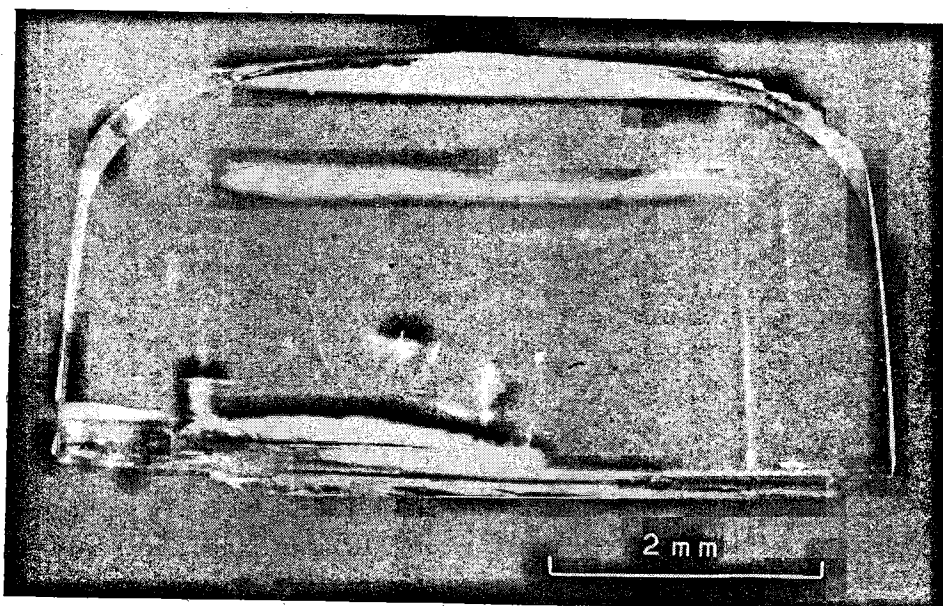
3.1. Examination before Plastic Deformation

3.1.1. First type I plate

The birefringence pattern of the first type I plate is shown in fig. 2. The main feature is a light band representing a strained region in the form of a long side and two short sides of a rectangle. The directions of these sides are $[01\bar{1}]$ and $[011]$, respectively. The x-ray topographs are shown in figs. 3 (a) and 3 (b). Figure 3 (a) was produced with the $(02\bar{2})$ reflection operating and fig. 3 (b) with the (022) reflection. The band of strain recorded in the birefringence pattern is also detected by x-ray topography. When the $(02\bar{2})$ was used the short sides of the band of strain were visible and the long side invisible. When the (022) reflection was used the reverse situation occurred. It is considered that most of the band of strain arises at the intersection of growth layers parallel to the $\{111\}$ planes with the surfaces of the specimen. Lattice displacements due to variation in unit cell dimensions in these layers are constrained to lie in the plane normal to the line of intersection of the layer with the specimen surface.

From this restriction arises their observed visibility behaviour. In both figs. 3(a) and 3(b) the images of surface scratches appear. These are the long curved lines without a definite crystallographic orientation. It is clear that the plate contained only three or four dislocations and so dislocations introduced by plastic deformation would be readily detectable. A section topograph was also taken with the $(02\bar{2})$ reflection operating and showed a high crystallographic perfection of the plate, since clearly defined and undistorted Pendellösung fringes were present. The fringes exhibited a more perfect pattern than has previously been observed in diamond and compares well with that given by essentially perfect silicon (Lang 1964).

Fig. 2



Birefringence pattern of the first type I diamond plate before plastic bending.

3.1.2. *Second type I plate*

The birefringence pattern for this plate shows that a prominent feature was again a band of strain in the shape of two short sides and one long side of a rectangle and is similar to fig. 1. When the birefringence patterns for the two type I plates are compared it is clear that in the original plate from which the two plates were cut, the band of strain was in the shape of the sides of a square. A similar distribution of strain was reported by Phaal (1965) and attributed to the effect of plastic deformation by indentation at the centre of the square. It appears that such a distribution of strain can be incorporated into a diamond during growth and subsequent history without the necessity for postulating that it is produced by plastic deformation in the laboratory. Examination of birefringence patterns,