AN OPTICAL AND X-RAY TOPOGRAPHIC STUDY OF GIANT SCREW DISLOCATIONS IN SILICON CARBIDE

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A 6H SiC crystal plate exhibiting three large growth spirals on its (0001) face was examined. One spiral maintained a simple step structure over an area 0.8 mm in diameter, enabling its step height to be measured interferometrically as 31.7 nm corresponding to 21 repeats of the 6H structure. X-ray topography confirmed the presence of a giant screw dislocation with Burgers vector parallel to [0001] at the centre of each spiral and showed that these dislocations had hollow cores running their lengths. Diameters of the hollow cores within the crystal were measured non-destructively by X-ray section topographs. The values found ( ~ 10 µm) were an order of magnitude greater than the diameters of the microscopically visible holes at the surface outcrops of the dislocations. A possible reason for this discrepancy is discussed. Attempts were made to measure the magnitude of the giant screw dislocation Burgers vector by X-ray diffraction methods, firstly from the size of the diffraction-contrast image of the dislocation and secondly from the tilt of the lattice planes close to the dislocation. Both methods were subject to much uncertainty, the former underestimating and the latter overestimating the Burgers vector magnitude by factors of more than two relative to the magnitude inferred from step-height measurements.

1. Introduction

Single crystals of many substances are known to exhibit growth spirals of large step height on their growth faces and more than a dozen crystal species behaving thus were listed some time ago [1]. A prominent example is silicon carbide [2,3], for which the step height on (0001) in the case of the 6H polytype can range from half of the structural repeat along the c-axis to many tens of this repeat distance. Now, it was predicted by Frank [4] that dislocations having a Burgers vector exceeding about 1 nm in magnitude should in equilibrium have hollow cores. In the case of a screw dislocation of Burgers vector magnitude b, the equilibrium radius of the hollow core r₀ is given by

\[ r₀ = \frac{\mu b^2}{8\pi^2\gamma} \tag{1} \]

where \( \mu \) is the modulus of rigidity of the material and \( \gamma \) is the specific surface free energy. Surface evidence of such "hollow cores" at the centres of spirals of large step height was soon afterwards reported, good examples being exhibited by SiC [5]. Recent experimental observations of holes at the centres of spirals of large step height in SiC have been reported by Tanaka et al. [6] and by Sunagawa and Bennema [7], with the latter work being followed by further theoretical studies of the conditions leading to the formation of hollow cores [8,9]. However, there is some conflicting evidence on hollow dislocations in SiC. For example, in an optical interferometric study of a hollow at the origin of a spiral of step height 48 nm, Bhide [10] concluded that the hollow (diameter 200 µm at the surface) extended to a depth of only 2.3 µm, whereas on Frank's criterion an extension (but with a smaller diameter, of order 10 µm) throughout the length of the screw dislocation would be expected. On the other hand Knippenberg and Gomes de Mesquita [11], reported that one or more holes parallel to the c-axis were
microscopically visible (diameters 1 to 10 µm) in all their specimens of columnar SiC crystals, but only in one single case was a spiral observed on the (0001) face. There is thus good reason for applying X-ray topography to this problem. Not only will the method easily detect giant screw dislocations (and give an estimate, albeit only roughly, of the magnitude of their Burgers vectors) but it also provides a non-destructive method for detecting hollow cores (if their diameter exceeds 1–2 µm, say) and for tracing their course in the interior of the crystal.

Among published X-ray topographic studies of SiC, some have aimed at mapping polytypes [12,13], but most have been concerned with dislocations and other lattice defects in single-polytype specimens, usually 6H. Among the latter types of investigation, e.g. refs. [14–17], the early experiments of Amelinckx et al. [14] succeeded in correlating etch pits with outcrops of dislocations of normal strength. None of the studies cited [12–17] was concerned with giant screw dislocations in SiC.

2. Optical measurements

The specimen, a commercial product, was a complex intergrowth of two or more crystal orientations. One uniformly oriented component possessed a well-developed (0001) face which displayed three large growth spirals near its centre, as shown in fig. 1. This (0001) face had only one good straight edge, that bounding it on the upper left corner over a length of 11 mm where it met a pyramid facet whose orientation was determined by optical goniometry to be (1013). The re-entrant invading the left side of the (0001) face, below the large spirals, is occupied by growth in another orientation whose facets have determined the directions of some straight segments of the encircling edge of (0001). Thus is explained the apparent non-low-index nature, relative to the crystal growth photographed in fig. 1, of these edge segments. The growth in different orientation protrudes above the level of the (0001) face but it does not show in fig. 1 because it has no facets sufficiently close in orientation to the major (0001) face to reflect the incident illumination. Details of the spirals are seen in the Nomarski differential interference contrast micrograph, fig. 2. The regularity of the single spiral, A, and the uniformity of the step height of its first six turns, contrast with the complicated patterns of fusion and division of steps associated with the centres B and C.

The growth protruding above the level of the face prevented the close approach of a reflecting surface as is needed for multiple-beam interferometry, so only two-beam fringe patterns could be recorded. That reproduced in fig. 3 uses widely-spaced white-light fringes to show the height scale of the steps near A, B and C, whereas in the case of fig. 4 a narrow-band interference filter with peak transmission at 506.6 nm was employed. Photographs of the latter type of fringe pattern were used to measure the profile across spiral A as...
Fig. 2. Nomarski differential interference contrast micrograph of central region of face shown in fig. 1, including the spirals A, B and C. Width of field 0.9 mm.

Fig. 3. Two-beam white-light interference micrograph of field of fig. 2.
accurately as the limitation to two-beam interference allowed. In the measuring technique adopted, identical negatives (on 100 mm by 125 mm sheet film) were superimposed emulsion to emulsion so as to produce an X-shaped pattern from each fringe passing over hillock A. With the help of this superimposition it was concluded that the height increase per turn of spiral A was very close to \( \lambda/16 \), i.e. 31.7 nm. The error in measurement was believed to be not greater than about 1/20 fringe per unit fringe displacement. The measured height increase per turn corresponds to 21 cells of the 6H structure (\( c_0 = 1.511 \) nm), or 126 times the interlayer repeat of 0.2518 nm. With the measurement accuracy claimed, height increases per turn corresponding to 20 or 22 cells of the 6H structure are by no means excluded, but values corresponding to 19 cells or lower, or 23 cells or higher, are considered improbable. In figs. 3 and 4 the straightness of the fringes between steps testifies to the flatness of the areas between steps. Neither Nomarski nor two-beam interference revealed any small steps comparable with the \( c_0 \) repeat in this region. Elsewhere, however, a few very fine steps were detected by the Nomarski technique. These were associated with the two other growth centres recognizable in fig. 1, viz. that \( \sim 1/2 \) mm above and to the left of A, and that about 1/2 mm from the bottom edge of the face. These fine steps had a visibility \( 10^{-2} \) or less of that of the 31.7 nm steps, and they might well have been of 1.5 nm height, but without multibeam interference experiments this height guess could not be tested. In a description of spirals on SiC crystals exhibiting step heights of 1 \( \mu \)m and greater, Buckley [18] referred to them as being “on a scale almost visible to the naked eye”. On the present crystal we found both the single hillock A and the double hillock of B and C to be easily visible to the naked eye using suitable illumination, but a hand lens was needed to resolve the individual steps of A.

Microscopically visible holes were present at the summits of A, B and C. Those at B and C were both round and about 1.2 \( \mu \)m in diameter. The hole at A was elongated, its long axis being inclined about 13° anticlockwise from the [1210] direction and its major and minor diameters were \( \sim 1.4 \) and \( \sim 0.4 \) \( \mu \)m, respectively.
3. X-ray topography

3.1. Lattice perfection

The thickness perpendicular to (0001) of the crystal regions near A, B and C ranged between 1/3 mm and 1 mm, so it was necessary to use Mo Kα radiation in order to obtain transmission X-ray topographs corresponding to conditions of low absorption. (Thicknesses giving the value unity in the product of linear absorption coefficient and crystal thickness are 0.66 mm for Mo Kα and 0.07 mm for Cu Kα radiations.) The strong reflections from the prism (1120) and pyramid (1126) were recorded (the latter also in second order), and settings were adopted with the (0001) face acting...
either as entrance or exit surface for the X-rays. The projection topograph, fig. 5, gives an idea of the overall perfection of the crystal, and it is apparent that defects are abundant. The image registers reflection from the volume below (0001). Hence its outline is considerably wider than that of the surface micrograph, fig. 1. However, the same crystal corner forms the extreme left-hand point in the images of both figs. 1 and 5. Using this landmark it is easily verified that the strong diffraction contrast image of a giant dislocation appears in fig. 5 at the position corresponding to A in fig. 1. Diffraction contrast images of B and C also appear in fig. 5, but partly obscured by other lattice defects nearby: they can be better seen in topographs taken with other crystal settings. The Burgers vector direction [0001] was confirmed for the dislocations A, B and C.

The lattice defects can be categorised as follows: (a) surface strains, (b) internal strain centres, (c) dislocations and (d) long-range elastic deformation. The surface strains due to scratches and percussion damage greatly interfere with the visibility of internal defects, especially in projection topographs. Abrasion damage is strong at crystal edges. The internal strain centres may be mainly small inclusions. In many parts of the crystal they seriously degrade the overall lattice perfection and hence reduce individual dislocation visibility. Dislocations seen in the topographs other than the giant screws are present either as straight segments parallel to (1010) and (2110) directions extending out to the crystal periphery, or as families of concentric loops parallel to the basal plane. The former are interpreted as grown-in dislocations, the latter as arising from plastic deformation. The dislocation density is quite variable and in much of the crystal is too high to permit individuals to be resolved, even in section topographs. The long-range elastic strains manifest themselves either as dynamical diffraction effects, viz. strong variations of diffracted intensity above or below the average value, and reversals of dislocation contrast, or as long-range warping of the crystal. The warping can amount to about 1 mrad/mm. Both the long-range strains and the cumulative effect of the unresolved internal strain centres seriously impeded attempts to estimate the Burgers vector magnitudes of A, B and C from the dimensions of their diffraction contrast images.

3.2. Mapping the hollow dislocations

Projection and section topographs showed that the dislocation A runs from the (0001) face perpendicularly down to an opposed (0001) facet 0.35 mm below (0001). On the other hand, B and C lie in a much thicker region, nearly 1 mm thick, not bounded by an identifiable facet opposed to (0001). Moreover, B and C (which have the same sense) diverge from a common parent dislocation at a depth 0.62 mm below (0001). Above the bifurcation point they separate, at first rapidly, and then more slowly, their separation being 0.06 and 0.08 mm at depths of 0.55 and 0.05 mm below (0001), respectively. By a geometrical coincidence, the diffracted beam of the 22412 reflection of Mo Kα radiation issues from the (0001) face nearly normal to it. Thus projection topographs taken with this reflection give a good view down the hollow dislocations, as seen in fig. 6. The sharpness of definition of the white discs seen in this and all other topographs leaves no doubt that they delineate cylinders of missing diffracting material, rather than of increasingly misoriented material, surrounding the dislocations A, B and C. Examples of the section topographs used for mapping the course of A, B and C within the crystal are shown in figs. 7a and 7b. These also show how variable from point to point is the diffracted intensity from the crystal matrix, and how greatly the interference of other strain fields causes the diffraction-contrast images of A, B and C to depart from being a simple pair of lobes disposed symmetrically above and below the plane of incidence as would be expected from models of dislocation image contrast in X-ray topography [19,20]. In 1126-type reflections, the intense diffraction-contrast generated by the dislocation strain field tends to cause halation of the image which impairs measurement of the diameter of the hollow core. In the higher-order, 22412 reflections, the dynamic range of contrast is smaller, and measurement of hollow core diameters is rather easier. Figs. 8a–8c show 22412 sections which may be compared with the corresponding projection topograph, fig. 6.
Fig. 6. Part of the X-ray projection topograph of the second-order reflection, 22412, from the (1126) planes, taken with Mo Kα₁ radiation for which θ₂ = 32.8° and the diffracted beam leaves the crystals at θ₁ = α = 32.8° - 31.4° = 1.4° to the c-axis. Magnification the same as in figs. 2, 3 and 4. The three screw dislocations A, B and C have hollow cores which project as white discs, using this diffraction geometry.

Fig. 7. Parts of X-ray section topographs, 1126 reflection, Mo Kα₁ radiation. Projection of diffraction vector points horizontally to the left, and the (0001) surface forms the vertical, right-hand boundary of the image. The ribbon incident beam is about 20 μm wide. Arrows point to the white dots which correspond to the hollow cores of dislocations A, B and C where they are intersected by the incident beam. The height of image reproduced is 1 mm. (a) Incident beam intersecting A about midway between its outcrops on (0001) and (0001). (b) Incident beam intersecting B and C about 0.07 mm below (0001).
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Fig. 8. Parts of X-ray section topographs, 22412 reflection, Mo Kα1 radiation. Projection of diffraction vector points horizontally to the left. Height of image reproduced corresponds to 425 μm: the magnification is the same as figs. 2, 3, 4 and 6. (a) Incident beam intersecting hollow dislocation A near the middle of the specimen and the pair B and C near the (0001) surface. (b) Specimen translated 188 μm relative to (a), so shifting to the right the intersection of the incident beam with A, and eliminating the intersections with B and C. (c) Specimen translated 315 μm relative to (a), causing a further rightwards shift of the intersection with A.

These and other sections provide no definite indication that the hollow cores are faceted rather than circular in cross-section. Geometric and other instrumental factors limit the topographic resolution to between 1 and 2 μm, so that all diameter measurements carry an uncertainty of about ±1 μm. Within this uncertainty, hollow A appears to maintain a constant diameter of 10½ μm. From close to the (0001) surface down to within a short distance above the bifurcation point, the diameters of B and C are 12 and 10½ μm, respectively. The parent hollow dislocation, measured a few tens of μm below the bifurcation point, has a diameter of 14 μm. Above the bifurcation, B inclines more steeply away from [0001] than does C, and when so inclined it has a smaller hollow diameter than C: about 75 μm above the bifurcation point the hollow of B has a diameter of only about 7 μm whereas that of C has the same diameter, 10½ μm, that it maintains from there upwards. The discrepancy between hollow diameters inside the crystal and at the surface is discussed below in section 4.

3.3. Burgers vector magnitude

The X-ray methods for estimating the magnitude of the Burgers vectors of the giant screw dislocations depend upon the difference in orientation between lattice planes near and far from the dislocation. Consider first a screw dislocation in an infinite medium. Take the Burgers vector, \( \mathbf{b} \), parallel to the z-axis of cylindrical polar coordinates (\( r, \theta, z \)) with origin on the dislocation line. The displacement \( u_z = (\mathbf{b} / 2\pi)z \) due to the dislocation causes a shear, \( S_\infty \), given by

\[
S_\infty = \frac{1}{r} \frac{\partial u_z}{\partial \theta} = \frac{b}{2\pi r}.
\]

Let the dislocation line lie in the plane of incidence of the X-rays (which is horizontal in the experiments). We are concerned with the (1126) planes in the regions vertically above and below the dislocation line, i.e. where (1126) contains the radius vector \( r \). The construction in fig. 9 shows that the planes above are rotated anticlockwise about an axis perpendicular to the plane of incidence by the small angle

\[
\frac{EA'}{CA} = S_\infty \sin^2 \alpha.
\]

In addition, the Bragg angle is changed by

\[
\Delta \theta_h = - \frac{\Delta d}{d} \tan \theta_h = - \frac{DD'}{OD} \tan \theta_h
\]

\[
= - S_\infty \sin^2 \alpha \cot \alpha \tan \theta_h.
\]
Fig. 9. Construction showing the tilt and change of interplanar spacing of (1126) planes due to a screw dislocation lying parallel to [0001]. Case of infinite medium. The plane of OABC and of the sketches (r) and (r) coincides with the plane of incidence of X-rays. OC is parallel to the z-axis of the right-handed cylindrical polar coordinates used in eq. (2), and hence is parallel to \( u_0 \). The origin of coordinates lies below the plane of the paper and is perpendicularly below O, so that OA is normal to the radius vector \( r \) and is parallel to \( u_0 \). In the crystallographic orientation adopted, [0001] is parallel to CO and [1120] is parallel to AO. The cell OABC of the 6H structure is deformed to OA'B'C' by the shear BB'/CB': 
\[
S_- = \beta(\pm n) S_\infty \sin^2 \alpha.
\]

The sketches (r) and (r) on either side of the construction in fig. 9 illustrate the diffraction geometries under which the positive and negative signs in (4) apply, respectively. In the symbol \( \beta(\pm n) \) denoting the quantity in the brackets in eq. (4), \( n \) is the order of reflection from the (1126) planes. The values of \( \beta \) for Mo K\( \alpha \) radiation are \( \beta(-2) = -0.05 \), \( \beta(+1) = 1.46 \), \( \beta(-1) = 0.54 \) and \( \beta(-2) = -0.05 \). The last value shows that in the case of the \( \overline{2}2412 \) reflection with the geometry depicted in the sketch (r), the \( \Delta d/d \) and rotation contributions to \( T_\infty \) almost cancel out. In the experiments, the geometry (r) was mainly used since it was desired that the diffracted ray should make small angles with [0001] in order to give the least distorted projection of the (0001) surface. The geometry (r) also applied to the \( \overline{1}1\overline{2}6 \) and \( 22412 \) reflections when \( 0001 \) served as X-ray entrance surface: the arrangement is then like (r), but with ray directions reversed, and this again gives the positive sign in eq. (4).

Next consider the strain field in the vicinity of the outcrop on (0001) or (0001) of a screw dislocation parallel to [0001]. Eshelby and Stroh [21] investigated the cases of a screw dislocation normal to the surface of a semi-infinite body, and of a screw dislocation normal to a plate. In the present work we are concerned with the strain field at values of \( r \) lying mainly between, say, 30 and 100 \( \mu \)m in a crystal of minimum thickness 350 \( \mu \)m (for dislocation A). This approximates sufficiently closely to the case of the semi-infinite body. Let the surface of the body be the plane \( z = 0 \), the body occupying the region \( z > 0 \). The elastic image field which annuls the traction on the plane \( z = 0 \) gives the additional displacement
\[
u_0 = \frac{b}{2\pi} \frac{r}{R + z},
\]
where \( R^2 = r^2 + z^2 \), \( u_0 \) being parallel to OA in fig. 9. (Here, in eq. (5), the sign of \( u_0 \) is opposite to that which appears incorrectly in eq. (2) of ref. [21]. Later equations for \( u_0 \) in ref. [21], and a re-statement of eq. (2) in Cartesian coordinates by Yoffe [22], have the correct sign, that in eq. (5) above.) Differentiating eq. (5) gives
\[
\frac{\partial u_0}{\partial z} = -\frac{b}{2\pi} \frac{r}{R(R + z)}.
\]
Thus, instead of the shear \( S_\infty \) which occurs at infinite depth below the surface, we have at depth \( z \) the weaker shear
\[
S_\tau = \frac{1}{R} \frac{\partial u_0}{\partial \theta} + \frac{\partial u_0}{\partial z}.
\]
Fig. 10. Plot of the “effective tilt”, $T_z$ (eq. (8)), in units of $b/2\pi$ for relative depths below the specimen surface, $z/r$, in the case of a screw dislocation normal to the surface. Range of $z/r$ from 0 to 2.

plus the anticlockwise bodily rotation

$$R_z = \frac{-\partial u_\theta}{\partial z},$$  

with $R_z + S_z = S_{z_e}$, and $S_z \to 0$ as $z \to 0$. The “effective tilt” $T_z$ at depth $z$ is thus

$$T_z = R_z + \beta(\pm n) S_z \sin^2 \alpha.$$  

In fig. 10, $T_z$ is plotted (in units of $b/2\pi r$) in the range $0 \leq z/r \leq 2$, for the $(+2)$, $(+1)$, $(-1)$ and $(-2)$ diffraction geometries previously discussed. (Since the magnitude of $\beta(-2)$ is very small, as noted earlier, the curve labelled “$-2$” is a very close representation of the $R_z$ component of $T_z$ alone.) Asymptotic values of $T_z$ for large $z/r$ are 0.557, 0.396, 0.146 and $-0.014$ for the $(+2)$, $(+1)$, $(-1)$ and $(-2)$ cases, respectively.

Models for the widths of the X-ray diffraction contrast images of dislocations in nearly perfect crystals, when X-ray absorption is low, suggest that the diffracted intensity rises significantly above the “perfect crystal” background level at distances from the dislocation line where the “effective tilt” reaches a value about equal to the full angular width at half maximum intensity of the “perfect crystal” reflecting curve [19,20]. A simple expression for the latter width is $2d/\xi_\theta$ where $d$ is the interplanar spacing and $\xi_\theta$ is the X-ray extinction distance [20]. In the case of the 1126 reflection from 6H SiC, $d = 0.131$ nm, and, with Mo Kα radiation, $\xi_\theta = 41$ μm, giving $2d/\xi_\theta = 6.4$ μrad.

From an average of ten measurements of the image width of dislocation A in section topographs cutting A at points remote from the crystal surfaces (so that $T_{z_e}$, eq. (4), applies) a Burgers vector magnitude of about 13 nm was deduced, less than half that deduced from the optically measured step height. Considering the level of background intensity arising from strains other than that due to A, such an underestimate of the Burgers vector, which would result from the apparent width of the dislocation image being smaller by a factor of more than two compared with that which would be seen in a perfect crystal matrix, is not surprising. Estimates of image widths of B and C were even harder to make than in the case of A; all that can be said is that they did not differ greatly from that of A.

The second X-ray method for estimating Burgers vector magnitude applies in regions where the spatial rate of change of lattice orientation is sufficient for the crystal to act as “ideally imperfect” from the diffraction-theoretical point of view. Then, a simple geometrical-optical analysis can be applied to find the Bragg-plane tilts close to the dislocation. The technique adopted in the present work was a modification of that used by Mardix, Lang and Blech [23] on thin, blade-like, platelet crystals of ZnS which contained a giant screw dislocation running down the middle of the crystal, parallel to the long axis of the blade. Section topographs of the ZnS crystals were taken in symmetrical transmission, i.e. using Bragg planes perpendicular to the thin crystal platelet and perpendicular to the screw dislocation. In such circumstances the diffraction geometry was well-defined and it was a straightforward matter to derive lattice tilts from the linear displacements of Bragg diffracted beams because the displacements recorded were large compared with the width of the section topograph image. In the present case
the diffraction geometry is altogether less favorable: the dislocation runs perpendicular to a thick plate rather than parallel to a thin plate. In order to record the displaced image produced by tilted regions in the presence of the wide section topograph image given by the whole crystal it was necessary to take section topographs with narrow incident beams which intersected the dislocation at its outcrop at one of the crystal surfaces. Fig. 11 shows the diffraction geometry and explains the measurement technique. The angles in fig. 11 correspond to the 1126 reflection with Mo Kα radiation. Both the 1126 and 22412 reflections were used in these experiments, examples of 22412 section topographs are shown in fig. 12. Burgers vector measurement could be attempted only on dislocation A. In fig. 12a a pronounced “spike” protruding outside the width A'B' of the section topograph image is produced by rays Bragg reflected from regions just above the dislocation outcrop, i.e. by rays such as B'(a). Quantitative interpretation of this type of image is only possible if the exit surface is reasonably flat and free from
gross damage in the vicinity of S'. These conditions are fulfilled by the (0001) face in the neighbourhood of A. By rotating the crystal 180° about the goniometer axis and translating it so that S' is straddled by the beam entering the crystal at O, images such as those shown in fig. 13 are obtained. In fig. 13 it is now crystal regions just below S' which produce the spike protruding outside A'B', to the left of A', as expected.

Eq. (2) et seq. show that the product of effective tilt, \( T \), and distance from the dislocation, \( r \), is constant. Thus, taking the horizontal displacement of the margin of the section topograph image as proportional to \( T \), it follows that the curves traced by the margin as a function of \( r \) should be rectangular hyperbolae having the horizontal line through S' as one asymptote and the vertical line coinciding with the margin far above and far below S' (i.e. the vertical line through B') as the other asymptote. However, rays which are deviated towards the interior of the section topograph image, i.e. rays such as B' (b) in fig. 11, appear to get largely "lost" in a background of rays from less deviated regions. The images thus lack the symmetry that obtained in the experiments on ZnS [23] when a conjugate pair of hyperbolic "spikes" could be recorded. The present asymmetry makes it very difficult to locate precisely the level of the outcrop S' on the topograph images. In addition to the impossibility of measuring the inward displace-

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**Fig. 13.** Section topographs with the incident beam intersecting dislocation A at its outcrop on the X-ray entrance surface, obtained by rotating the crystal 180° about the goniometer axis so that (0001) becomes the X-ray entrance surface instead of being the X-ray exit surface (as in X-ray topographs previously shown). Radiation Mo K\(_\alpha_1\), reflection 1126, projection of diffraction vector points to the left. Height of image reproduced corresponds to 450 \( \mu \)m. Plate-to-specimen distances: (a) 100 mm, (b) 10 mm.
ment B'B'(b), it was found that the outward displacement B'B'(a) could only be assessed with any confidence over a small range of $r$, from about 20 to 60 $\mu$m (on the crystal). Since the ribbon incident beam does not sample depths greater than 10 to 20 $\mu$m under the surface at S', the values of $T_z$ appropriate are those applying in the range of small $z/r$ (see fig. 10). Indeed, the locus of the apparent margin of the section topograph image is determined in practice by the most strongly deviated rays. Hence the value of $T_z$ at the surface, i.e. $T_0 = b/2\pi r$, was adopted. Values of $T$ were derived as follows. If $x_i$ denotes the linear displacement of B'B'(a) when the specimen-to-plate distance is $L_i$, then

$$T = (x_2 - x_1)/(L_2 - L_1).$$

(10)

The minimum practicable values of $L_1$ were 10 and 5 mm for the 1126 and 22412 reflections, respectively. The maximum practicable value of $L_2$ was 100 mm: larger values of $L$ gave too diffuse an image. The principal measurements were made on photomicrographs of the section topographs (magnification 200). The mean values of $b$ obtained were 63 nm from the 1126 reflections and 80 nm from the 22412 reflections. Greater weight should be attached to the former value because of the better definition of the section topograph image edge in the lower-order reflection, and for other reasons, discussed below.

4. Discussion

Comments will now be made on three topics: (a) the justification for assuming that the optically-measured step height equals the Burgers vector, (b) the poor agreement between the step-height value and the Burgers vector magnitude derived from the “geometrical-optical” X-ray measurement of lattice tilt, and (c) the noteworthy difference between the diameters of hollows at the surface and in the interior of the crystal.

With regard to (a), it should be borne in mind firstly that the steps on a basal-plane surface of a polytypic layer-stacking-structure crystal do not have to be integral multiples of the unit cell height. In SiC the thickness, $h$, of a unit monomolecular layer parallel to the basal plane is 0.2518 nm. Thus there exist very nearly equivalent, low-energy, basal-plane surfaces spaced $h$ apart: any of such surfaces might serve as the external surface of the crystal. However, the structure of the risers of steps, i.e. the structure of non-basal planes, influences which multiples of $h$ are preferred in steps on (0001), at least in the case of the simpler polytype structures: with the 6H structure step heights of 3$h$ and 6$h$ are observed, the latter more commonly [3,5]. Secondly, it should be remembered that in the case of a spiral step surrounding a screw dislocation outcrop (such as seen at A), the observed macrostep may be less than the Burgers vector. The invariant quantity is the number of layers of height $h$ counted when a Burgers circuit is made. Consider a perfect dislocation in the 6H structure. Then $b = 6mh$, $m$ being an integer, but the 6$m$ unit steps could be distributed in any manner between the limits of complete bunching into a single macrostep of height 6$mh$ or complete dispersal as 6$m$ steps of height $h$. In an intermediate state with, say, a fraction $r$ of the 6$m$ units bunched into one big step and the remainder distributed as unit steps of height $h$, the Burgers vector inferred from macrostep height would be only 6$rmh$ unless unit steps distributed between the big steps could be resolved. The growth hillock A of present concern is believed to represent the first extreme possibility: complete bunching into a single macrostep. This belief is founded on the fact that the common minimum step height in 6H SiC is 6$h$, and such steps should have been visible in the careful, high-contrast Nomarski photomicrography (though, as explained in section 2, the sensitivity of stepheight detection could not be quantified in experiments on this crystal surface). Certainly steps of height 12$h$ or 18$h$ would have been detected. Nowhere on growth hillock A are any such small steps visible.

Some of the difficulties attending the X-ray measurement of lattice tilt have already been mentioned. Here the uncertainties in the method will be examined further. Firstly, consider the validity of the model illustrated in fig. 11. It is assumed that the incident radiation is monochromatic but divergent. Note that if the experiment had been done with X-rays from a synchrotron source some
tens of metres from the specimen then the opposite conditions, i.e. polychromatic radiation but strict collimation, would have applied. In the first case the deviation of the diffracted beam equals the effective tilt, $T$, of the lattice planes. In the second case the deviation is $2T$. From the source and slit sizes and distances given in the caption to fig. 11 it is seen that the incident beam divergence is about $4.2 \times 10^{-4}$ rad. This is about the same as the largest values of $T$ measured, in both the 1126 and the 22412 reflections. To test the validity of the model, “spikes” of deviated rays were recorded with the crystal deliberately set off the Bragg angle which gave maximum diffracted intensity from the crystal as a whole. Mis-setting in the appropriate direction brought extra intensity into the spike image at low values of $r$, but did not greatly affect hyperbola-matching for values of $r$ within the 20 to 60 $\mu$m range. Nevertheless, the consequences of natural wavelength spread of the Mo $K\alpha_1$ radiation are not negligible. The value of $\Delta\lambda/\lambda$ corresponding to the full width at half maximum intensity of the $K\alpha_1$ emission line profile is $4.1 \times 10^{-4}$. The corresponding ranges of Bragg angle, $\theta_B$, are $1.1 \times 10^{-4}$ and $2.6 \times 10^{-4}$ rad for the 1126 and 22412 reflections, respectively. In the case of the 22412 reflection it is clearly possible that a significant fraction of the intensity contained in the more strongly deviated rays could come from wavelengths in the flanks of the $K\alpha_1$ emission line profile. For example, deviated rays which on the assumption of a unique $\theta_B$ give an apparent $T$ of $3 \times 10^{-4}$ rad via eq. (10), could also arise from the combination $T = 1.5 \times 10^{-4}$ rad with an equal departure, of $1.5 \times 10^{-4}$ rad, from the $\theta_B$ value corresponding to the peak of the $K\alpha_1$ line. Thus the effects of wavelength dispersion are sufficient to account for the larger value of $b$ estimated from the 22412 reflection, and further justify attaching more weight to the lower value of $b$. While considering wavelength dispersion, it should be pointed out that even the largest values of $T$ concerned in the tilt measurements are much smaller than the difference in Bragg angles for the $\alpha_1$ and $\alpha_2$ components of the $K\alpha$ doublet (these are $1.7 \times 10^{-3}$ and $3.8 \times 10^{-3}$ rad for the 1126 and 22412 reflections, respectively). On the other hand, if the white discs seen in figs. 6 and 8, and interpreted as hollow cores of dislocations, denoted, instead, regions of high tilt, then reflection of the $\alpha_2$ component would have fallen within the white discs either in their upper or lower halves, depending upon the sense of misorientation. The absence of the $\alpha_2$ reflection confirms the “hollow-core” interpretation of the white discs.

Another geometrical-optical complication arises from the helicoidal configuration of the lattice planes surrounding the giant screw dislocations and the consequent rotation of misorientation vector of Bragg planes as a function of the polar angular coordinate about the dislocation line. The incident beam straddling $S'$ (fig. 11) will also cut volumes to right and left of $S'$ where $T$ has a component in the plane of incidence. This component will deviate rays in the vertical plane on the section topographs: the resultant vertical smearing of the image will broaden the base of the “spike” BB' (a) and tend to fill in the “trough” BB'(b). Such smearing is believed to be a principal cause of the lack of inversion symmetry about $S'$ in the pattern of deviated rays, noted above in section 3. The broadening of the spike will perturb hyperbola-fitting so as to give falsely high values of $T$.

A diffraction effect also deserving mention is a diffusion, or “flaring”, of the spike at the smallest $r$ values. This precluded measurement of $T$ with any confidence at $r$ values less than about 20 $\mu$m, as previously mentioned. The flaring suggests high lattice distortion and/or small effective crystal thickness (less than, say, 1/2 $\mu$m) in the immediate vicinity of the outcrop $S'$. Since the hollow core diameter below the surface is about 10 $\mu$m and the hole diameter seen at the surface is about 1 $\mu$m, diffraction broadening arising from a thin crystal layer partially closing the hole is a possibility. The idea of such a layer is taken up below.

Finally, the applicability of Frank’s equation (1) and the problem of hole diameter variation will be considered. Inserting observed values of $b$ and $r_0$ into (1) yields the ratio $\gamma/\mu$. A spread in $\gamma/\mu$ indicates variations in $\gamma$, such as might occur due to presence of liquid in the hollow core during crystallisation, provided that the elastic model underlying (1) applies. The present experiments provide one $b$, $r_0$ pair: the optical interferometric measurement of $b$ and the internal X-ray measure-
ment of 2\( \gamma_0 \) in the case of dislocation A, with uncertainties believed to be about \( \pm 10\% \) in \( 2\gamma_0 \) and \( \pm 5\% \) on \( b \). These measurements yield \( \gamma/\mu = 0.0024 \text{ nm} \). The elastic constants of 6H SiC are accurately known: measurements by a resonance method and by a double-pulse echo method give \( C_{44} = (1.68 \pm 1\%) \times 10^{11} \text{ N m}^{-2} \) and \( (1.70 \pm 1.5\%) \times 10^{11} \text{ N m}^{-2} \), respectively [24]. Thus \( \gamma/\mu = 0.0024 \text{ nm} \) corresponds to a surface free energy of \( 400 \text{ mJ m}^{-2} \). Note that the behaviour of dislocations B and C above and below the bifurcation is not consistent with (1), if a constant \( \gamma \) is assumed. The X-ray topographs suggest Burgers vectors of roughly equal magnitude for B and C, as does the similarity in their hole diameters. Then the combined Burgers vector below the bifurcation point should produce a hole diameter about \( 2^2 \) times those observed above the bifurcation, instead of roughly \( \sqrt{2} \) times, as observed.

The value \( \gamma/\mu \) found for A is not greatly different from the value 0.0033 nm adopted by Sunagawa and Bennema [7] as fitting best measurements by themselves and others on macrosteps and surface holes in 6H SiC. It is much more discordant with the measurements of Tanaka et al. [6]. Values of \( \gamma/\mu \) derived from their plotted \( b \) and \( \gamma_0 \) magnitudes range from 0.01 to 0.17 nm. Included in their measurements were four holes each associated with a spiral step close to 50 nm in height. Diameters of these four holes ranged from 2.6 to 0.8 \( \mu \text{m} \), with corresponding \( \gamma/\mu \) values ranging from 0.024 to 0.076 nm. In earlier experiments, Golightly [25] cut thin sections of 6H crystals containing growth spirals in order to facilitate the measurement of hole diameters microscopically. His log-log plot of \( b \) versus hole diameter (the latter ranging from 0.3 to 100 \( \mu \text{m} \)) was compatible with the \( \gamma_0 \propto b^2 \) relation of (1) but with a wide scatter in individual measurements. Golightly assessed the range of possible \( \gamma/\mu \) values to lie between 0.025 and 0.127 nm. These values are more like those derived from the measurements of Tanaka et al. than the lower values of the present work and of Sunagawa and Bennema. Note that with the present crystal an estimate of \( \gamma/\mu \) obtained for A, B or C using hole diameters seen at the surface rather than those measured in the interior by X-rays would raise the value of \( \gamma/\mu \) by an order of magnitude.

The large difference between hole diameters at the surface and in the interior which the joint use of optical microscopy and X-ray topography has disclosed raises the question, well deserving further investigation, of how common such occurrence is. As far as could be ascertained by optical microscopy, the edges of the holes at A, B and C were sharp: no trumpet-like profile of the openings (such as reported in ref. [7]) was detected. Since (0001) is a low-energy surface, a mode of growth in which the junction of (0001) with the tubular surface of the hole has no more rounding than at any natural crystal edge is possible. Suppose that the radius of curvature of the hole edge in a radial plane containing [0001] is much less than the radius of the hole and is so small as to be comparable with the common minimum growth layer height, \( 6mh \). Then a fluctuation may initiate the inward growth of a thin sheet parallel to (0001), forming a shelf partially closing the hole. The inward growth of a thin shelf is energetically possible because with both upper and lower surfaces free the strain energy density in the shelf at a given radius \( r \) due to the dislocation is less than that in bulk material distant \( r \) from the dislocation. How thin the shelves partially closing the holes of A, B and C are is unknown, and would be difficult to measure non-destructively if their thicknesses were of sub-micrometre magnitude. Only in the case of C is there any X-ray section topographic evidence available, and it shows that the shelf thickness does not exceed a few micrometres.

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