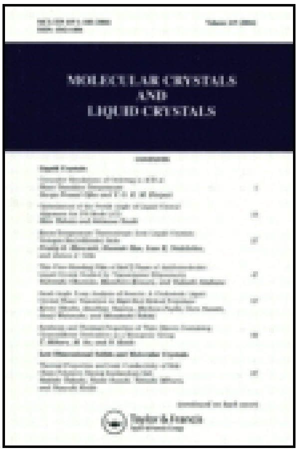

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Dislocation Line Direction Determination in Pyrene Single Crystals

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DISLOCATION LINE DIRECTION DETERMINATION IN PYRENE SINGLE CRYSTALS

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Abstract A new technique for the unambiguous determination of dislocation line directions in macroscopic single crystals is presented. The technique is based on analysis of the projected directions of the images of the dislocations on synchrotron white beam topographs. Calculation is carried out in a pseudo-cubic axis system which is related to the crystal axis system by a transformation matrix. Final line directions are presented referred to the crystal axes. An example of the application of this technique to the analysis of growth dislocations in pyrene single crystals is presented.

Keywords: dislocation line direction, synchrotron topography, pyrene single crystals, synchrotron white radiation, growth dislocations, protective properties

INTRODUCTION

Determination of the line directions of dislocations in crystals is important from diverse points of view. For the case of mechanically induced dislocations, full characterization of the operating slip system requires determination of the line direction as well as the Burgers vector of the dislocations.[1] In the case of growth dislocations, line direction determination is of interest since it has been shown that dislocations adopt low energy directions in the lattice.[2] In cases where dislocations might potentially play some role in either chemical reactions or phase transitions, the detailed molecular configurations surrounding dislocations, which are a function of the dislocation line direction (and Burgers vector), become important.

Dislocation line direction determination can be conveniently carried out using synchrotron white beam x-ray topography (SWBXRT). This and other related x-ray topographic techniques are generally superior to transmission electron microscopy (TEM) due to the fact that macroscopic (cm^3) specimens can be studied. This means that significant lengths of dislocation line can be observed allowing for unambiguous line direction determination, unlike the case of TEM where, generally speaking, only short lengths of dislocation can be observed due to the necessarily small specimen size.

Information on the line directions of dislocations in crystals is generally obtained from x-ray topographs via analysis of the projected directions of the associated dislocation images on at least two topographs recorded with different reciprocal lattice vectors. In the case of SWBXRT, this can be conveniently carried out since several images are recorded in a single exposure. To date, two techniques have been developed to facilitate this. The first, based on analytical geometry and called the projective property method, consists of determining the projected directions of known line directions in the crystal on several independent topographic images, and then comparing with observed line directions in an iterative process.[3] Agreement between calculated and observed projected directions on at least two different topographic images may then lead to unambiguous assignment of dislocation line direction. The problem with this technique is that when dislocations lie along irrational directions, as they frequently do in molecular crystals, predicting possible line directions becomes difficult. This is contrary to the case in many elemental crystals where dislocations often lie along rational directions enabling this technique to be put to good use.[4] The second technique used to date is based on a stereographic projection analysis of dislocation line directions.[5] This involves plotting the poles of at least two sets of diffracting planes (for example, corresponding to two images recorded on a Laue pattern) on a stereographic projection which has the incident beam direction as its center pole. The diffracted beam directions associated with each of the reflections, $\mathbf{S}_{g1}$ and $\mathbf{S}_{g2}$ are also plotted on the same projection, as well as the poles of the line directions of interest $\mathbf{l}_1$ and $\mathbf{l}_2$, respectively (images are recorded with the detector perpendicular to the incident beam, so that this latter step becomes trivial). Great circles running through both $\mathbf{S}_{g1}$ and $\mathbf{l}_1$, and $\mathbf{S}_{g2}$ and $\mathbf{l}_2$ are then constructed with the aid of a Wulff net. The intersection of these two great circles is the pole of the dislocation line direction, which can then be assigned an index. The main problem with this method arises from the low accuracy associated with the graphical nature of the technique.

The purpose of this paper is to describe an analytical method for the determination of unknown dislocation line directions, from analysis of their projected directions on at least two different topographic images. This method is devoid of the inaccuracies inherent in the projection technique. Application of the technique to the determination of dislocation line directions in pyrene single crystals is also described.

EXPERIMENTAL

Materials

A pyrene crystal, grown by slow cooling of a seeded, saturated toluene solution by Prof. J. Sherwood's group at Strathclyde University, was used to demonstrate the utility of the calculation. The specimen was cut from the central region of crystal which contained the seed. The detailed crystal morphology has been described previously.[6] The surface orientation of the specimen was (001), and the growth dislocations of interest emanated from the seed to the $\{110\}$ growth faces. The reflections utilized were $\bar{1}10$ and $\bar{1}\bar{1}0$. Figure 1 shows a schematic representation of the crystal shape showing the twelve different line directions observed. Four regions of crystal, as indicated on the diagram, were studied independently using both $\bar{1}10$ and $\bar{1}\bar{1}0$ reflections.

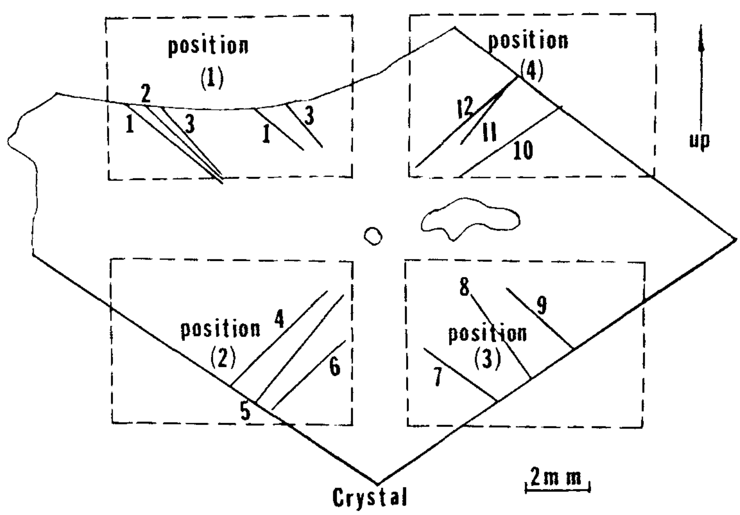

FIGURE 1 Schematic representation of crystal shape, showing the four regions of crystal that were separately imaged, along with the twelve different dislocation line directions observed in this crystal.

Synchrotron White Beam Topographic Imaging

Synchrotron white beam topographic imaging was carried out at the Stony Brook Synchrotron Topography Station (beamline X-19C) at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. The experiment basically consists of placing the crystal of interest at a preset orientation in the path of an area-filling, synchrotron white x-ray beam. Area-filling diffracted beams, forming a diffraction pattern, are then collected on a large area film detector (8.5"×11" Kodak SR5), also placed at a preset orientation. For the experiments described here, the detector was placed normal to the incident x-ray beam. The projected directions of dislocation lines were measured on enlarged reproductions of the original topographs using a protractor and cross-checked with trigonometric analysis. The measurement error was around ±0.2°.

Line Direction Analysis[7]

The relationship between a line direction in the crystal and its projected directions on x-ray topographs is illustrated in figure 2. The idea is to convert measured projected directions on two topographs into real directions inside the crystal.

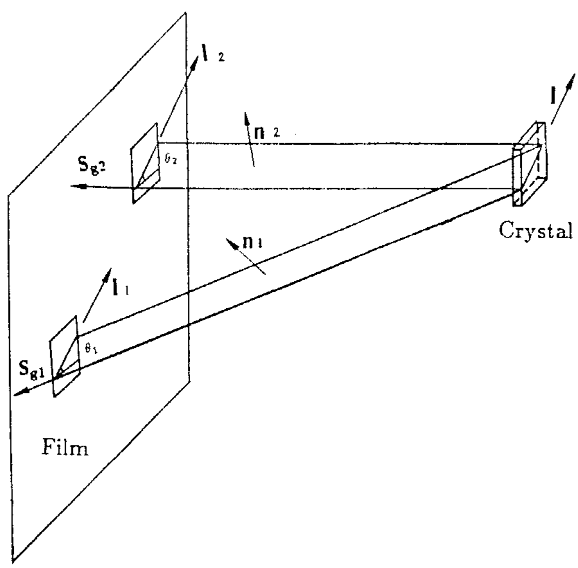

FIGURE 2 Schematic showing relationship between the projected directions of dislocation images and the dislocation line direction itself in the crystal.

All calculations are carried out in a pseudo-cubic reference axis system, and are valid for any crystal system. Real space and reciprocal space vectors in any crystal system can be converted to corresponding vectors referred to a pseudo-cubic axis system, X_0,Y_0,Z_0 using simple transformation matrices.[8,9] A second orthogonal axis system related to the laboratory frame of reference is then defined, with the Z-axis parallel to the incident beam direction (which is horizontal in space), the Y-axis pointing down in the vertical direction, and the X-axis orthogonal to both of these. Note the X-Y plane defines the detector plane. The relationship between the X,Y,Z and X_0,Y_0,Z_0 systems is defined by a rotation matrix.

Suppose we have a dislocation line that lies on both $(h_1k_1l_1)$ and $(h_2k_2l_2)$ images, projecting at angles θ_1 and θ_2, respectively to the horizontal X-axis, which lies in the detector plane. The line directions of the dislocation on $(h_1k_1l_1)$ and $(h_2k_2l_2)$ can be expressed as vectors $\mathbf{l_1}$ and $\mathbf{l_2}$ referred to the X,Y,Z axis system. Similarly the diffracted beam directions associated with $(h_1k_1l_1)$ and $(h_2k_2l_2)$ can be expressed as vectors $\mathbf{S_{g1}}$ and $\mathbf{S_{g2}}$, referred to the X,Y,Z system. Now suppose that $\mathbf{n_1}$ is the normal to the plane defined by $\mathbf{l_1}$ and $\mathbf{S_{g1}}$, and $\mathbf{n_2}$ is the normal to the plane defined by $\mathbf{l_2}$ and $\mathbf{S_{g2}}$. As can be seen from figure 2, the line direction of the dislocation inside the crystal is simply the direction of intersection between these two planes, i.e. $(\mathbf{n_1} \times \mathbf{n_2})$.

All that remains is then to convert this direction, which is expressed as a vector referred to the X_0,Y_0,Z_0 system, to one referred to the crystal axis system, using the transformation matrices referred to earlier. All calculations described above are carried out with the aid of a personal computer.

RESULTS AND DISCUSSION

Figure 3 shows white beam topographs recorded from region 1. Figures 4, 5 and 6 show similar sets of images recorded from regions 2, 3 and 4, respectively. By comparison with figure 1, a total of twelve different dislocation line directions can be identified. The line directions calculated from the measured projected directions of the various images are presented in Table 1, along with their respective angles with the growth direction in the growth sector of interest.

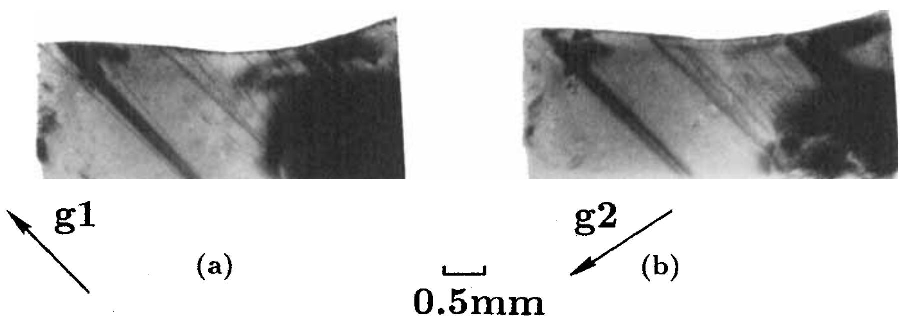

FIGURE 3 White beam topographs recorded from region 1. (a) $\bar{1}10$ reflection (λ=1.52Å); (b) $\bar{1}\bar{1}0$ reflection (λ=1.56Å). Dislocations 1-3 are visible.

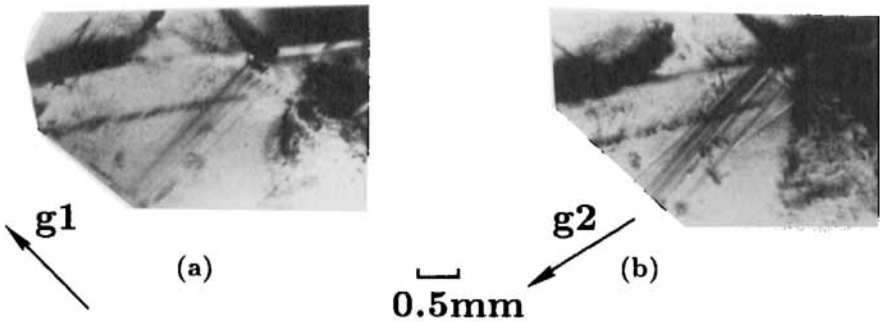

FIGURE 4 White beam topographs recorded from region 2. (a) $\bar{1}10$ reflection (λ=1.52Å); (b) $\bar{1}\bar{1}0$ reflection (λ=1.56Å). Dislocations 4-6 are visible.

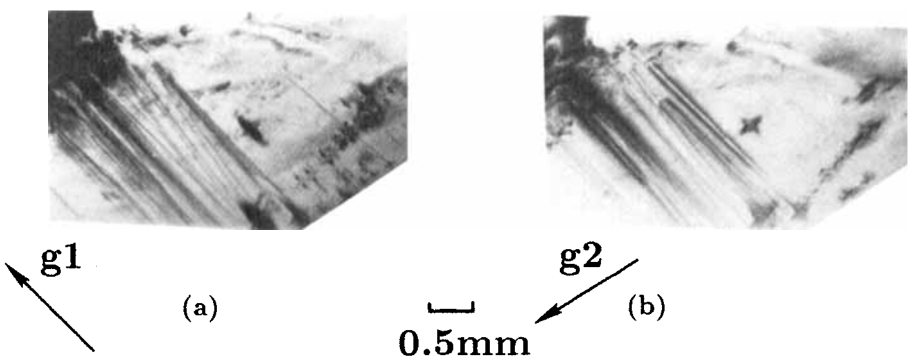

FIGURE 5 White beam topographs recorded from region 3. (a) $\bar{1}10$ reflection (λ=1.52Å); (b) $\bar{1}\bar{1}0$ reflection (λ=1.56Å). Dislocations 7-9 are visible.

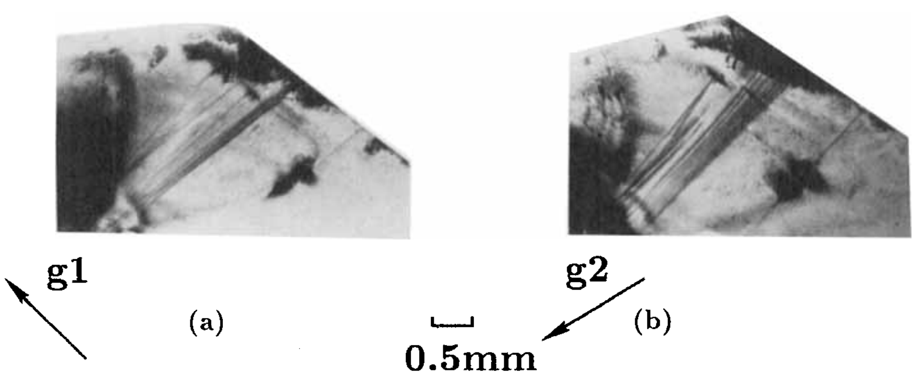

FIGURE 6 White beam topographs recorded from region 4. (a) $\bar{1}10$ reflection (λ=1.52Å); (b) $\bar{1}\bar{1}0$ reflection (λ=1.56Å). Dislocations 10-12 are visible.

TABLE 1 Results of Line Direction Calculation.

Line Number	Angle with X-axis	Calculated Line Direction	Growth Sector	Angle with Growth Direction
1	221.5°	$[\bar{4}.\bar{0}\ \bar{5}.\bar{2}\ \bar{0}.\bar{9}]$	$(\bar{1}\bar{1}0)$	13.66°
2	225.0°	$[\bar{2}.\bar{0}\ \bar{3}.\bar{0}\ \bar{0}.\bar{2}]$	$(\bar{1}\bar{1}0)$	10.45°
3	228.5°	$[\bar{4}.\bar{0}\ \bar{7}.\bar{1}\ 0.9]$	$(\bar{1}\bar{1}0)$	12.25°
4	133.0°	$[\bar{4}.\bar{0}\ 6.2\ \bar{1}.\bar{1}]$	$(\bar{1}10)$	8.43°
5	139.0°	$[\bar{4}.\bar{0}\ 5.0\ \bar{1}.\bar{1}]$	$(\bar{1}10)$	14.43°
6	133.5°	$[\bar{5}.\bar{0}\ 8.0\ \bar{9}.\bar{0}]$	$(\bar{1}10)$	33.55°
7	42.5°	$[4.0\ 5.0\ 2.3]$	(110)	16.35°
8	51.0°	$[4.0\ 6.9\ 1.9]$	(110)	9.38°
9	43.0°	$[3.0\ 4.1\ 0.9]$	(110)	12.43°
10	313.0°	$[3.0\ \bar{4}.\bar{1}\ 2.2]$	$(1\bar{1}0)$	16.64°
11	327.0°	$[4.0\ \bar{8}.\bar{9}\ 1.1]$	$(1\bar{1}0)$	1.58°
12	321.0°	$[4.0\ \bar{7}.\bar{2}\ 1.1]$	$(1\bar{1}0)$	4.43°

Clearly, growth dislocations in pyrene lie along irrational directions, presumably chosen on the basis of energy minimization. Standard $\mathbf{g}\cdot\mathbf{b}$ analysis does not permit Burgers vector assignment in this system since most of the dislocations never exhibit contrast extinction. This, along with the irrational line directions, is indicative of the fact that the dislocations studied are of mixed screw and edge character. It is interesting to note that none of the dislocations observed actually lie in the (001) basal plane, contrary to observations in *p*-terphenyl.[10]

In summary, detailed line direction analysis of dislocations in pyrene single crystals has been successfully carried out using a direct analytical approach. Most dislocations are found to lie below an angle of $\approx 15°$ to the growth direction, which is in agreement with observations in many other systems.[2] Work is currently under way to determine the significance of the determined line directions with respect to the molecular structure surrounding the dislocations, using an approach similar to that adopted by Scheffen-Lauenroth *et al.*[11] It should be noted that the angular errors inherent to the stereographic projection technique (sometimes as large as 4-5°) are greater than some of the angles between closely oriented dislocations in this crystal, thereby precluding this kind of analysis. This new method provides a much more precise determination of dislocation line direction.

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