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The Observation of Dislocations in Yttrium Gallium Garnet by a Photoelastic Method

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A brief outline of the technique for observing dislocations by their photoelastic patterns is given. The method is successfully applied to the determination of Burgers vectors of dislocations in flux-grown single crystals of yttrium gallium garnet, and the origin and elimination of dislocations in this material is discussed.

1. Introduction

The use of the photoelastic method to examine the stress fields associated with individual dislocations was shown to be possible by Bond and Andrus [1]. Further work was done by a number of Russian workers [2-6], and by Bullough [7] who provided a theoretical basis for the method by calculating the intensity distribution of transmitted light around a single end-on edge dislocation situated in an isotropic material. When a slice of such a material is examined between crossed polarisers, an edge dislocation lying parallel to the axis of the microscope gives rise to a characteristic stress pattern in the form of a rosette. The shape of the rosette is determined by the angle between the Burgers vector of the dislocation and the incident plane-polarised light vector in such a way that the Burgers vector may be readily determined. In principle the axis of the dislocation line and the direction, magnitude, and sign of the Burgers vector may be determined from the stress pattern [3].

It was shown by Bullough [7] that contours of equal transmitted intensity are given by the polar equation

$$r = K \cos(2\psi)\cos(\psi - \beta)$$

where K is a constant proportional to the magnitude of the Burgers vector, the photoelastic constant, and the rigidity modulus; (r, ψ) are polar coordinates with $\psi = 0$ lying along the polariser direction, and β is the angle between the slip direction and the polariser direction.

Fig. 1 shows the two special cases which in the determination of the Burgers vector of a dislocation prove to be the most useful. Fig. 1a shows the stress pattern when the polariser and slip directions are coincident, i.e. when $\beta = 0$, while fig. 1b shows the stress pattern when the polariser and slip directions are at 45° to each other. Assuming that the crystallographic orientation of the specimen is known, the slip direction may be determined by rotating the crystal slice with respect to the crossed polarisers until a

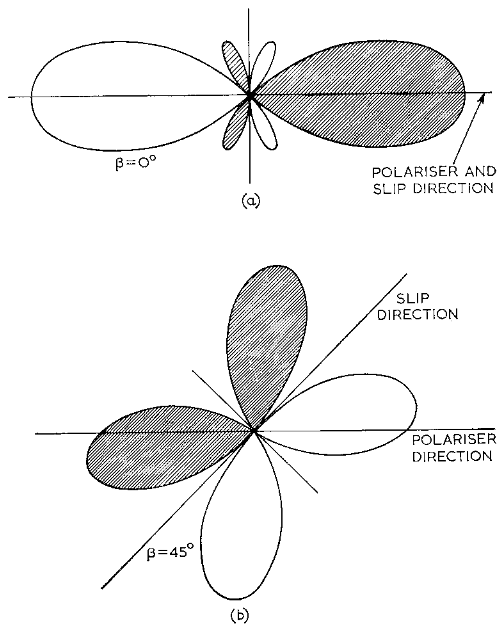


Figure 1 Equal intensity rosettes for an edge dislocation: (a) angle between slip and polariser directions is zero; (b) angle between slip and polariser directions is 45°.

stress pattern corresponding to fig. 1a or fig. 1b is obtained. The black or white appearance of the individual petals of the rosette (represented by cross-hatching in the diagram) is due to the superimposition of the microstresses surrounding the dislocation and the background stresses present in the crystal. It must be noted that, in an isotropic approximation, a screw dislocation does not give rise to an observable stress pattern when viewed along its axis. In the case of a mixed dislocation the Burgers vector may be inferred from the stress pattern associated with the edge component.

All previous investigations in which photoelastic methods have been successfully used to resolve individual dislocations have been on silicon using infrared light with an image converter coupled to the polarising microscope. Other work [2, 9], on corundum and Rochelle salt respectively, does not appear to have resolved the fine stress patterns associated with individual dislocations. The sensitivity of the optical method for the detection of dislocations increases with the hardness, the photoelastic constant of the material, and the magnitude of the Burgers vector of the dislocation. In this paper we describe results obtained from work on yttrium gallium garnet, $Y_3Ga_5O_{12}$, a material which satisfies these criteria to a marked degree. It has a high hardness value, a large lattice parameter and, from the quality of the photographs obtained, appears to have a large photoelastic constant. Furthermore the material is extremely transparent in the visible, so that the full resolution of a polarising microscope may be used.

2. Experimental Procedure

Single crystals of $Y_3Ga_5O_{12}$ having growth faces parallel to crystallographic $\{110\}$ and $\{211\}$ planes were grown from a lead oxide/lead fluoride flux [8]. Slices approximately 1 mm thick were cut parallel to growth faces and polished with 1 μm diamond paste. Their orientations were checked by the Laue back-reflection X-ray technique. Stress patterns produced in the slices by dislocations were examined in a Zeiss polarising microscope using tungsten lamp illumination and the characteristics of the dislocations were determined.

3. Results and Discussion

The axes of the dislocations were found by focusing through the crystal slice. Although none of the dislocations examined was normal to the plane of the slice, most being within 5° of the normal, the stress patterns were remarkably clear. Figs. 2a and 2b illustrate the comparison of dislocation etch pits and photoelastic patterns in a $(1\,1\,0)$ slice. Fig. 2a shows a $(1\,1\,0)$ growth face which was left unpolished in order to preserve the etch pits formed when the crystal was leached out of the solidified flux by a nitric-acetic acid mixture. Fig. 2b shows the stress patterns around the same group of etch pits (the "halos" are the out-of-focus images of the surface etch pits). The one-to-one correspondence of etch pits and stress patterns indicates that all the dislocations present have strong edge components.

3.1. Determination of Burgers Vectors

In order to determine the likeliest Burgers vectors of dislocations in the garnet structure (space group *Ia3d*), it is assumed that only certain simple lattice vectors are possible. The energy of a dislocation is determined mainly by electrostatic and elastic interactions. Because of the complexity of the structure it is difficult to calculate the electrostatic interactions involved in the movement of dislocations with unit Burgers vectors given by simple lattice vectors, or to determine the energies involved in the dissociation of unit dislocations into partials. Since the elastic energy of a dislocation is proportional to the square of its Burgers vector, the likeliest Burgers vectors will be the shortest lattice vectors, in this case $(a/2)\langle 111\rangle$, $a\langle 100\rangle$, and $a\langle 110\rangle$ in that order. As mentioned earlier it is the edge component of a mixed dislocation which produces the birefringence rosette. Thus, the slip direction inferred from such a stress pattern is a projection in the plane of the slice of the slip direction of the mixed dislocation.

The experimental procedure adopted was to rotate the sample until the pattern corresponding to $\beta = 0$ was obtained. The range over which this pattern could be set by eye was usually 10 to 15°. On comparing the angular setting of the specimen stage with the appropriate stereographic projection for the crystal slice, the 10 to 15° range for the slip direction was found to lie close to one, or possibly two, of the projections in the plane of the slice of the simple lattice vectors considered (i.e. $\langle 111\rangle$, $\langle 100\rangle$, or $\langle 110\rangle$). Photographs were taken of the patterns at 5° intervals about these projections, and the accurate position corresponding to the pattern $\beta = 0$ was chosen. Apart from a few special cases discussed later, the method gave consistent

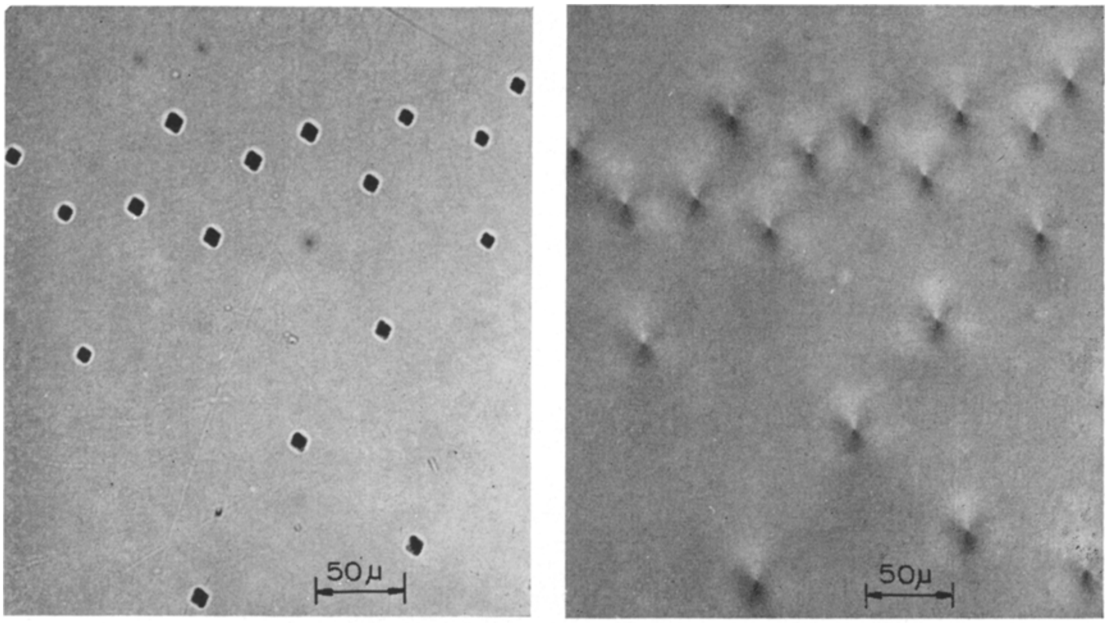


Figure 2 (a) Dislocation etch pits on a (1 1 0) growth face; (b) photoelastic patterns associated with the same group of dislocations.

results for all dislocations examined, and justified the original assumption of simple unit lattice vectors.

The most common Burgers vector was found to be $a\langle 100\rangle$, but some examples of both $a\langle 110\rangle$ and $(a/2)\ \langle 111\rangle$ were observed. Fig. 3 shows stress patterns associated with dislocations emergent on the face of a sample cut parallel to a (112) growth face. The polariser direction is marked as an arrow, and lies along the projection of the [100] direction in the plane of the slice. Several groups of dislocations give patterns corresponding to $\beta = 0$, with their slip directions along the [100] direction (see, for example, the two large groups near the top centre of the field of view, and also the two groups of three at the top right of the picture). The other stress patterns observed in fig. 3 correspond to a number of different values of β, and hence several different Burgers vectors, all of which were determined. The dark areas to the right and upper left of the figure are due to the grown-in stresses which occur in the outer regions of the crystal [10].

Fig. 4a shows a photograph of another group of dislocations in the same slice. The polariser direction (arrowed) is aligned along the projection of the $[\bar{1}01]$ direction in the plane of the slice, and two dislocations exhibiting stress rosettes corresponding to $\beta = 0$ have $a[\bar{1}01]$ Burgers vectors. Fig. 4b is a sketch of the area shown in fig. 4a, and gives the Burgers vectors determined for individual dislocations. The position of each rosette shown in fig. 4a is marked with a symbol, all dislocations with the same Burgers vector having the same symbol. Other members of the group have Burgers vectors of $a[011]$, $a[010]$, $a[1\bar{1}0]$, and $(a/2)\ [1\bar{1}1]$. The large stress pattern at the bottom of the group in fig. 4a is that of an inclusion. The pattern is similar to that of a dislocation and differs only in magnitude [7]. Care must be taken to differentiate between dislocations and inclusions.

Fig. 5 shows a photograph of stress birefringence rosettes associated with a group of six dislocations. The projection of the $[1\bar{1}1]$ direction in the (112) plane of the slice (arrowed) is coincident with the polariser direction. The patterns correspond to $\beta = 0$, the six dislocations having Burgers vectors of $(a/2)\ [1\bar{1}1]$. The dislocation at the top right of the group has been lightly decorated during growth, which results in a slight modification of the pattern.

3.2. Factors Influencing Stress Patterns

Several factors were found to influence the stress

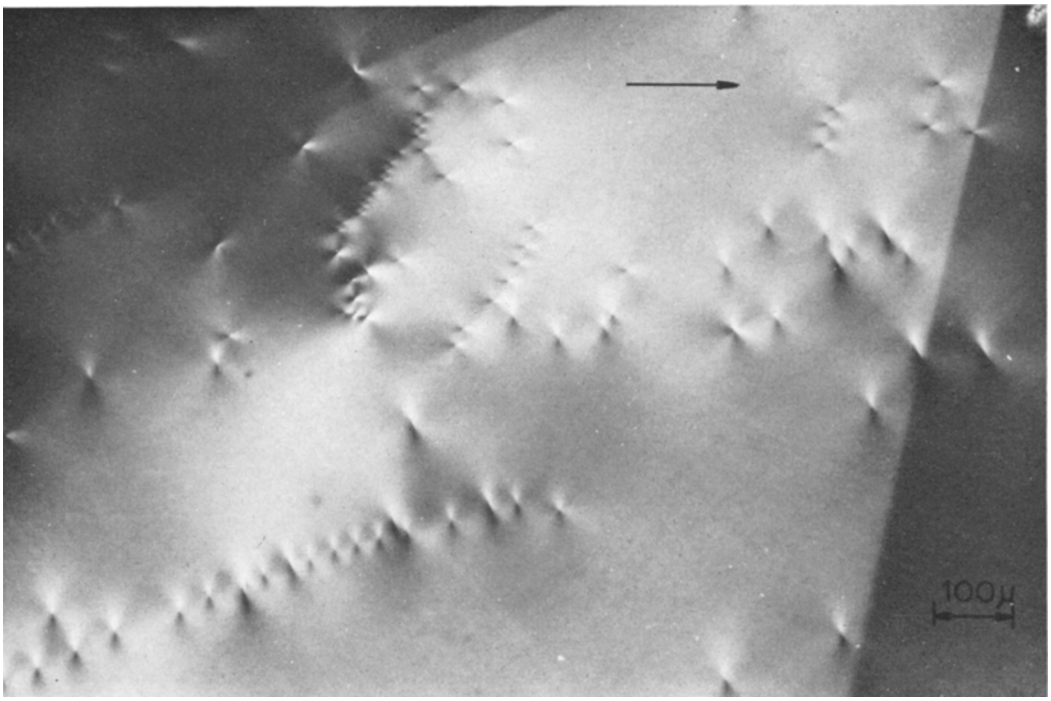
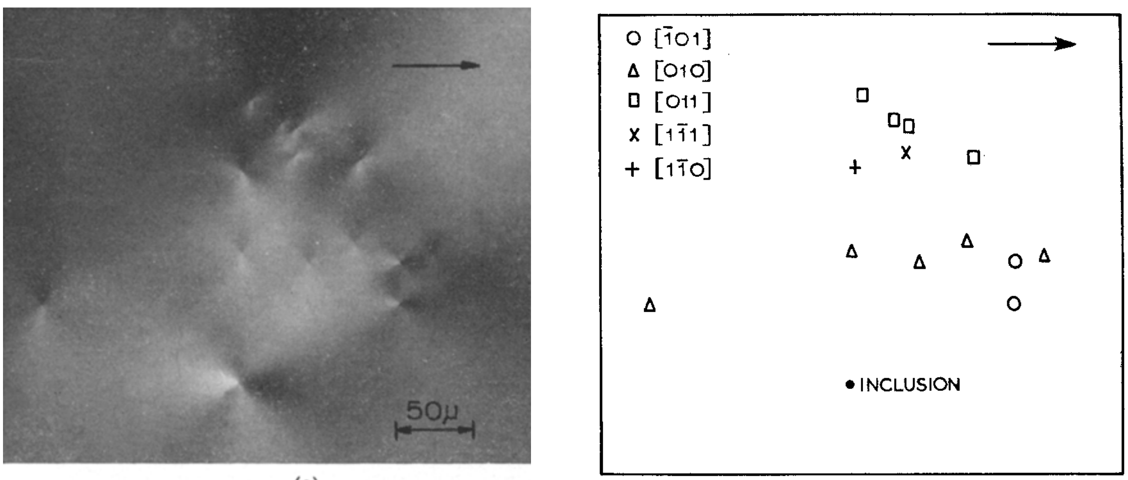


Figure 3 Dislocations seen by stress birefringence in a (1 1 2) slice. The polariser direction (arrowed) lies along the projection in the plane of the slice of the [1 0 0] direction.



(a) (b)

Figure 4 (a) Dislocations seen by stress birefringence in a (1 1 2) slice. The polariser direction (arrowed) lies along the projection in the plane of the slice of the [1̄ 0 1] direction. (b) Diagrammatic sketch showing Burgers vectors of the dislocations in fig. 4a.

patterns. Of these, the most important was the deviation of the dislocation line from the normal to the slice. When this was greater than about 5°, it became difficult to determine accurately the point at which the shape of the rosette corresponded to $\beta = 0$, owing to the lack of definition of the rosette shape. It should be noted that dislocations at greater angles to the normal are

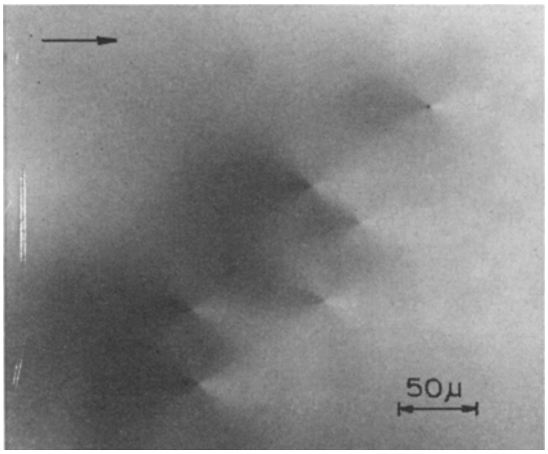


Figure 5 Dislocations seen by stress birefringence in a (1 1 2) slice. The polariser direction (arrowed) lies along the projection in the plane of the slice of the [1 $\bar{1}$ 1] direction.

still readily observable by this method, although it may not be possible to determine accurately their Burgers vectors. For example, in the top left corner of fig. 3 are shown three elongated stress patterns which are associated with dislocations lying at about 10° to the normal to the slice. Difficulty was also experienced when the dislocation line curved in the plane of the slice. Background stresses due to growth strains, large inclusions, other dislocations, etc. tended in some cases to swamp the microstresses around dislocations making it difficult to observe clearly the stress pattern corresponding to $\beta = 0$. Sometimes, in these cases, it was possible to work with the stress pattern associated with $\beta = 45°$ (see fig. 1b). A few dislocations were observed to be decorated with impurities, the decoration taking place during the growth process [10]. As expected in these cases, the rosettes were less distinct (see fig. 5), as the effect of the decoration is to reduce the strain field of the dislocation line itself. Indenbom *et al* [4] have shown that, with sufficient decoration, the microstresses due to precipitation increase, while at the same time the original microstresses due to the dislocation line itself are lost completely.

In general the Burgers vectors and the axes of the dislocations were not perpendicular, and therefore the dislocations were mixed in character. A few dislocations in a (112) slice were found to have $a[1\bar{1}0]$ Burgers vectors, and were probably pure edge. These exhibited larger and more intense stress patterns compared with dislocations having $a\langle 110\rangle$ Burgers vectors lying out of the plane of the slice. In general, dislocations having Burgers vectors of the $a\langle 110\rangle$ type gave more intense patterns than the $a\langle 100\rangle$ or $(a/2)\,\langle 111\rangle$ types. This is probably due to the fact that the intensity of transmitted light is proportional to the square of the Burgers vector, and hence is greater for the $a\langle 110\rangle$ type dislocations than for the other types.

3.3. Improvement in Crystal Quality

The combination of dislocation axes and Burgers vectors did not suggest a consistent set of slip planes, and it is likely that the dislocations in the flux-grown yttrium gallium garnet crystals examined are introduced and propagated during the growth process itself, rather than being produced by slip after growth. It should be possible to produce more perfect yttrium gallium garnet crystals with a fluxed-melt technique by growing onto a seed crystal, rather than relying on spontaneous nucleation to produce the seed. By crystal selection, the seed crystal could become progressively free of dislocations until dislocation-free material is produced. There are difficulties in producing crystals in this way, but in principle the crystal quality could be significantly improved if these were overcome.

4. Conclusion

Photoelastic stress patterns associated with individual dislocations have been observed with great clarity in yttrium gallium garnet using a simple polarising microscope. The Burgers vectors of dislocations normal to the crystal slice were of three types, $a\langle 100\rangle$, $a\langle 110\rangle$, $(a/2)\,\langle 111\rangle$. Since the origin of the dislocations lies in the growth process itself, rather than in dislocation multiplication during the post-crystallisation stage, the possibility of producing dislocation-free material by seeded growth from a fluxed melt is suggested. The photoelastic technique has been found to be equally applicable to the observation of dislocations in yttrium aluminium garnet.

Acknowledgement

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