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William Jones [a], John M. Thomas [a], John O. Williams [a], Michael J. Gorringe [b] & Linn Hobbs [b]
[a] Edward Davis Chemical Laboratories, Aberystwyth
[b] Department of Metallurgy, University of Oxford, Oxford, U.K.
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# The Characterization of Dislocations in Organic Molecular Crystals by Transmission Electron Microscopy

WILLIAM JONES, JOHN M. THOMAS, and JOHN O. WILLIAMS
*Edward Davis Chemical Laboratories, Aberystwyth*

and

MICHAEL J. GORRINGE and LINN HOBBS
*Department of Metallurgy, University of Oxford, Oxford, U.K.*

The role of transmission electron microscopy (TEM) for the characterization of dislocations in materials has been extensively described in the literature (see, for example, Hirsch *et al.*[1] and references therein). Previous studies of defects in organic molecular crystals have utilized etch-pit studies, x-ray topography and simple microscopic deformation.[2] It is the purpose of this paper to indicate how TEM serves as a valuable addition to this list of techniques.[3]

The theory governing the imaging of dislocations and other extended defects has been discussed fully elsewhere.[1] To a very good approximation the condition for the disappearance of dislocation contrast in an isotropic material may be given by

$$\mathbf{g} \cdot \mathbf{b} = 0 \quad \text{and} \quad \mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u} = 0$$

where $\mathbf{g}$ is the operating diffraction vector, $\mathbf{b}$ the Burgers vector of the dislocation and $\mathbf{u}$ a unit vector along the dislocation line. In the case of strongly anisotropic materials the establishment of $\mathbf{g} \cdot \mathbf{b} = 0$ only is not absolute, but may produce an image of lower intensity than, or at least close to, the surrounding background. Using this assumption, therefore, all that is required is the tilting of the sample, with respect to the electron beam, into several Bragg conditions until a disappearance condition is met; this then allows a determination of the direction of the Burgers vector of the dislocation.

Specimens for observation by TEM must be thin (usually less than *ca* 500 nm) and suitable samples of *p*-terphenyl are obtained by allowing a

dilute solution in xylene ($5 \times 10^{-3}$ M) to slowly evaporate on a water substrate. The crystals then grow as platelets with (001) as the basal plane. The TEM image then consists of many bend extinction contours.[1] These result from the bending of the thin foil, and they map out areas of the foil at the same Bragg angle.

Figure 1(a) and (b) shows the establishment of a suitable condition for Burgers vector analysis. The contrast from one of the dislocations (which are almost certainly basal, and therefore satisfying $\mathbf{g} \cdot \mathbf{u} \cdot = 0$) disappears on tilting from (a) to (b). The diffraction condition (shown in the insert)

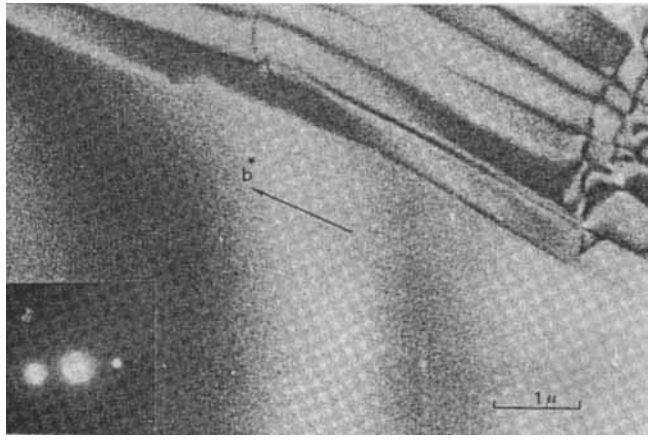


FIGURE 1(a)

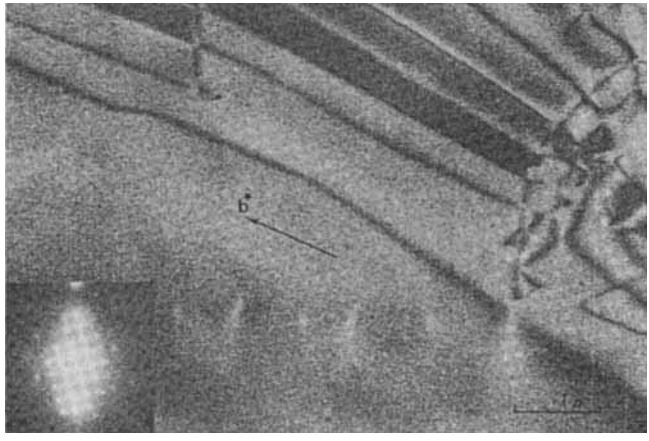


FIGURE 1(b)

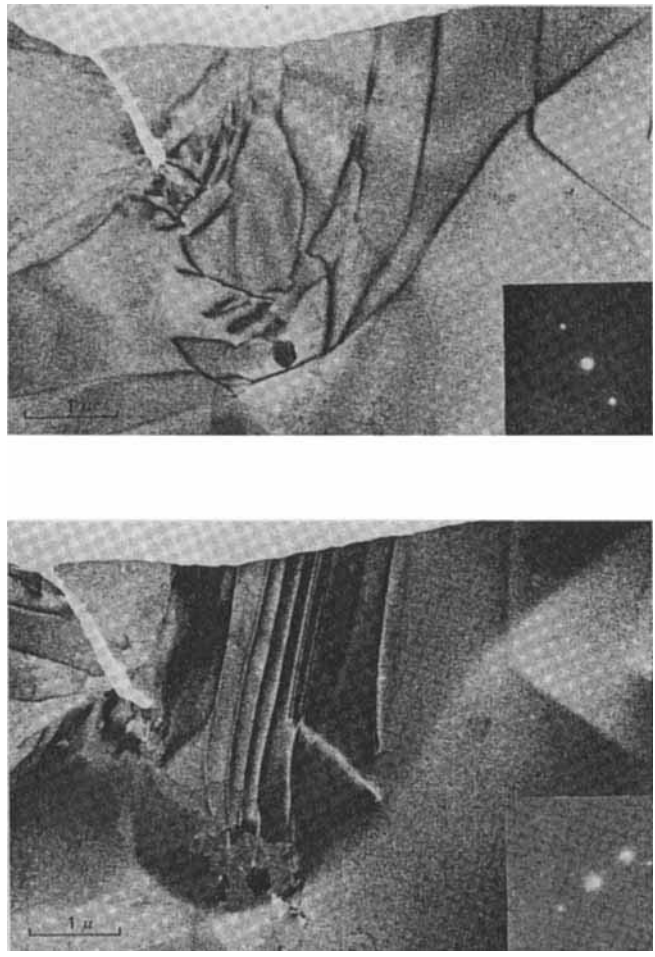

FIGURE 2(b)

for this disappearance is $\mathbf{g} = 110$, meaning that $\mathbf{b}$ must be along $[\bar{1}1w]$†. Further evidence for this Burgers vector is given in Figure 2(a) and (b) where contrast from one particular family is lost, whilst another family is brought into the diffracting condition. Burgers vectors along [010] and [120] have also been identified by this technique. The vectors along [110] and [120] may be of particular relevance to the explanation of the apparent non-topochemical photodimerization of anthracene[4] (which crystallizes

† In the *ab* plane the directions 110 and $\bar{1}10$ are equivalent and the conditions $\mathbf{g} = 110$ and $\mathbf{g} = \bar{1}10$ cannot easily be distinguished. For dislocations gliding in (001) then $w = 0$.

FIGURE 3

in the same space group as *p*-terphenyl). Regions having several non-basal dislocations have also been recorded, and contrast from these dislocations is exemplified by Figure 3.

Any study of organic crystals by TEM will usually be hampered by the instability of the material to the obligatory dose of high energy electrons that they receive.[5] Within a few minutes the area being observed will become irreparably damaged. We have obtained value for the maximum observation time for an area before complete loss of the diffraction pattern—these values are shown in Table I. Information from the image of this area is effectively lost after approximately 50% of this dose; giving a reasonable time of 10 minutes for *p*-terphenyl, but only 2 minutes for $Cl_{18}Me_{10}An$.

TABLE I

Experimentally determined lifetime of various aromatic crystals at room temperature in an electron microscope operated at 100 keV. (Current density at sample $3.8 \times 10^{-4}$ A cm$^{-2}$)

| | Approximate lifetime (minutes) | Critical dose for sample As cm$^{-2}$ |
|---|---|---|
| Anthracene | $5 \pm 1$ | 0.1 |
| *p*-terphenyl | $22 \pm 3$ | 0.5 |
| $Cl_{1,8}Me_{10}An$ | $4 \pm 1$ | 0.1 |
| $Cl_{1,8}Me_{9}An$ | $4 \pm 1$ | 0.1 |
| $Cl_{1,8}An$ | $5 \pm 1$ | 0.1 |

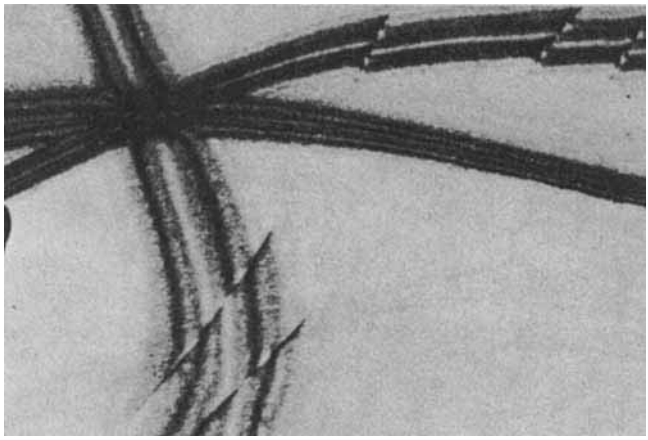

FIGURE 4

With this time limitation in mind we have recently been attempting to devise ways of gaining as much information (if not all) to complete a Burgers vector analysis from one micrograph. Figure 4 shows three intersecting bend extinction contours in *p*-terphenyl—the electron beam is then parallel to the pole established by these intersecting Bragg diffraction planes. Interaction is obvious with two of the contours, but not with the third. Since the extinction contours represent regions of the foil at particular (common) diffracting (**g**) conditions, it follows that no interaction must be a result of the $\mathbf{g} \cdot \mathbf{b} \cdot = 0$ condition. In this case the contour is of the type 111 and the Burgers vector of the dislocation therefore lies in a direction perpendicular to this plane. This image plane technique has subsequently been utilized in several ways, and a more thorough discussion has been given elsewhere.[6]

## References

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