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Direct Observation of Dislocations in Organic Molecular Crystals by Transmission Electron Microscopy

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Abstract—Dislocations in anthracene have been identified for the first time by diffraction contrast, transmission electron microscopy. Burgers vectors in the [010] direction are prominent.

Two main approaches are available for the observation of dislocations in organic solids by electron microscopy. The first is the so-called lattice-imaging procedure, which may be used either indirectly (utilizing pseudo-replica techniques) as demonstrated by Dawson⁽¹⁾ for materials such as β -lactoglobulin which have large lattice dimensions, or directly using two-beam tilted imaging techniques first demonstrated by Menter⁽²⁾ for the phthalocyanines. The second involves diffraction contrast and dark-field microscopy (3) and is inherently more widely applicable provided the sample can withstand electron irradiation during examination. The results that have been so far obtained on the relatively stable polymers (4) indicate that the frequently exploited diffraction-contrast approach, which has proved so successful in the study of line defects in metals, anisotropic chalcogenides and minerals holds much promise for the study of rather unstable organic molecular crystals. In this communication we illustrate how dislocations in anthracene, the paradigm, of a wide range of aromatic solids, may be identified by utilizing dark field electron microscopy at low temperatures.

Anthracene, like many other organic materials, belongs to the low-symmetry, anisotropic, monoclinic crystal system and the characterization of dislocations in crystals of these materials is important because of the influence of such defects in governing the chemical reactivity⁽⁵⁾ and mechanical properties⁽⁶⁾ of aromatic solids. Previous studies of defects in anthracene by optical microscopic⁽⁷⁾ and X-ray topographical⁽⁸⁾ methods have indicated that dislocations may glide both in basal (001) and in non-basal planes, and that a predominant Burgers vector is [010].

Anthracene crystals, which were grown from solution in subdued light, varied in thickness between 25 nm and 100 nm. Examination of the specimens, sometimes supported on perforated carbon films, was made on the goniometer stage at low temperatures (ca 90K) in a high resolution electron microscope (Phillips EM 300). The operating conditions were such that the emission current was extremely low at 100 kV, and by recording the images on high speed film it was not necessary to incorporate image-intensifying devices.

Figures 1(a), 1(b) show an anthracene crystal taken in bright-field and dark-field respectively. The reflection employed to produce the dark field image is encircled in the diffraction pattern, and the corresponding reciprocal lattice vector g indicated. Immediately obvious are the extinction (thickness) contours, e.g., AB, CD in the brightfield photograph and the absence of several of these in dark-field. The dislocations at E and F give interesting contrast effects in brightfield when traversed by these extinction contours but disappear in dark-field. These dislocations terminate at the edges of the crystal, and from the invisibility criterion g, b = 0, the Burgers vector is deduced to lie along [010]. It is significant that extensive areas of these crystals are dislocation free—a conclusion substantiated by further tilting and dark-field experiments. Figure 2 shows another bright-field photograph of anthracene with the diffraction effects characteristic of dislocations again apparent. The precise identity of the dense regions, typified by those demarcated G, H, is at present not known. These regions could arise from either slight contamination of the specimens, or correspond to localized thick patches within the crystals. The selected area diffraction apertures are not sufficiently small to provide useful information on such features. The dislocations may be subdivided into two groups: (a) those radiating from or terminating at point 0; and, (b) those constituting linear arrays and terminating within the crystal. The non-uniform contrast along some of the dislocations in group (b) suggests that they lie on planes other than the basal (001), whereas dislocations visible in

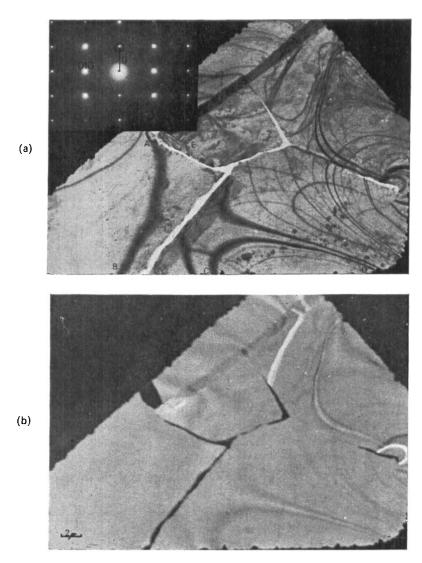


Figure 1. Electron micrographs of an anthracene crystal taken in (a) bright-field, (b) dark-field. Note that the dislocations in (a) are out of contrast in (b). The anthracene electron diffraction pattern is shown (inset) and the reflection used in producing the dark-field image and the corresponding reciprocal lattice vector g indicated.

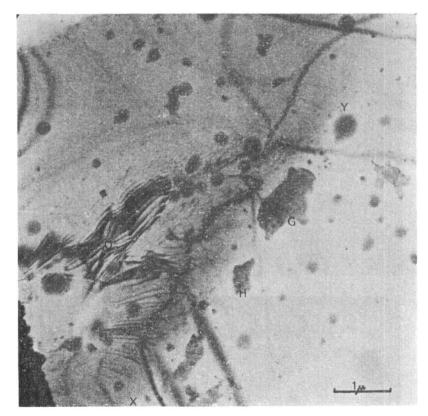


Figure 2. Electron micrograph (bright-field) of an anthracene crystal showing an area containing dislocations.

Fig. 1a, terminating at the crystal edges, are probably basal in origin. The step XY in Fig. 2, where a few extinction contours end, delineates the termination of molecular planes within the crystal. It was not possible to determine the Burgers vector of the dislocation shown in Fig. 2. Further work to characterize dislocations in aromatic hydrocarbons and other organic solids is in progress.

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