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Recent Studies of Lattice Defects by X-Ray Diffraction Topography

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In X-ray topographic methods point-by-point differences in X-ray reflecting power are utilised to detect the local strain fields due to lattice defects. The usefulness of the X-ray methods is based upon the following characteristics. (1) Sufficiently thick specimens may be examined to ensure that their imperfection content is representative of the bulk material. With light elements the specimen thickness may be several millimetres, but it is usually preferable to work with specimens not more than a few hundred microns thick in order to obtain the best contrast and resolution. (2) Experiments are non-destructive: hence repetitive experiments may be made to show changes of imperfection content such as dislocation multiplication and movement. For example, X-ray topographic studies are being made on specimens before and after irradiation and annealing and before and after deformation and annealing. (3) The method is a general one and the same technique can be applied to a wide variety of crystals. (4) Under usual experimental conditions the identification of Burgers vectors of dislocations is straightforward and unambiguous. (5) The spatial relationships of imperfections in the crystal specimen may be revealed by taking stereo-pairs of topographs. The potentialities of the X-ray topograph are best realised in transmission methods, and such methods are almost exclusively used at Bristol. The technique employed in the majority of experiments is that of the "projection topograph"[1]. Occasionally this is supplemented by taking "section topograph"[2]. The resolution is a few microns in most experiments: it depends upon the X-ray wavelength and the X-ray scattering power of the material. In favourable cases (longer wavelength, higher scattering power) the resolution is about one micron for localized defects such as small precipitates, and under similar conditions individual diffraction images of dislocations separated by three microns can be well resolved. The maximum density for resolution of individual dislocations depends upon the specimen thickness in projection topographs, being roughly 10^4 lines cm^{-2} for a 1 mm thick specimen, and 10^5 lines cm^{-2} for one 0.1 mm thick. In section topographs the limiting density is $10^6 \sim 10^7$ for dislocations and somewhat more than 10^7 for precipitates. The limited resolution is in fact the principal shortcoming of the X-ray method. The resolution is comparable with etch-pit methods for the study of dislocations. It does not fall very far short of the resolution of dislocation decoration techniques, but it is very poor compared with the performance of the electron microscope.

Present investigations at Bristol can be divided into the following categories.

1. Dislocations

Grown-in dislocations are being studied in diamond, lithium fluoride, aluminum, silicon, iron+3% silicon alloy, germanium, and indium antimonide. In the case of melt-grown crystals the dislocation configurations observed indicate a complex history of dislocation movement during the cooling of the crystal: silicon[3] and lithium fluoride show much evidence of high-tempreature slip. Many large loops are found, especially in lithium fluoride,

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the plane of the loop often being not of low index. A striking feature of the dislocation pattern in strain-anneal grown crystals of pure aluminum is the frequent occurrence of sets of coaxial loops. The loop axis is always a [110] direction, and the Burgers vectors of the loops lie in the same direction. Loop diameters range upwards from about 1 micron (the threshold of visibility) to about 50 microns. Loop separations are usually a few times the loop diameter. As many as 40 loops will occur on a single axis, and the total length of the axis may approach a millimetre. In melt-grown Fe+3% Si alloy, on the other hand, loops are not seen. Individual dislocations in low-angle boundaries are resolved in lithium fluoride for boundary tilt angles up to 20″ of arc, at which the dislocation separation is just under 3 μ. An example of a lithium fluoride low-angle boundary with tilt angle only about 5″ is shown in Fig. 1. Here the dislocations are not lined up together regularly: their alignment improves when the tilt angle increases and the dislocations lie closer to each other.

2. Precipitates, Inclusions and Impurities

The role of these defects in generating dislocations during or after growth is being studied, as well as the function of dislocations as nucleation centres for precipitates. Cases of decoration of dislocation have been observed in natural crystals of diamond and calcite. In heavily doped germanium which has solidified under conditions giving rise to "cellular growth" the impurity cells can be detected both by diffraction contrast and absorption contrast, and when segregation occurs the strain field and dislocation distribution around segregated material can be studied. Another application of the X-ray topographic method involves taking topograph using the diffuse "spike" reflections from diamonds, and correlating these with normal X-ray topographs and ultraviolet absorption topographs. For a long time an outstanding problem in diamond research has been the origin of the ultraviolet and infrared absorptions that distinguish the common Type I diamonds from the rarer Type II diamonds, and the origin of the diffuse "spike" X-ray reflections exhibited by Type I diamonds. A few years ago Kaiser and Bond[4] showed that nitrogen was a major impurity in diamond and that there was a linear relationship between the nitrogen content and the strength of the UV and IR absorptions peculiar

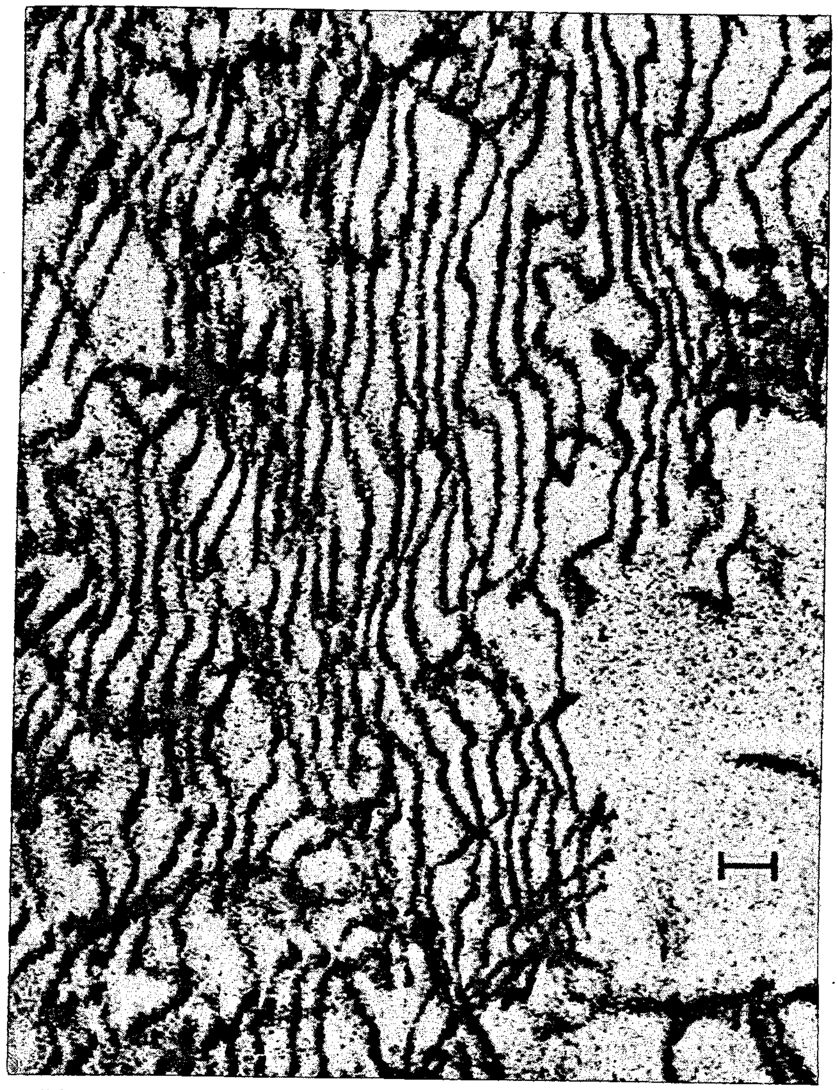

Fig. 1. Dislocations in lithium fluoride. Scale mark 25 μ.

to Type I diamonds. Also, it has been pointed out that the diffuse reflections could be accounted for by double layers of nitrogen segregated on cube planes. It was thus of interest to establish quantitatively the connection between optical absorption properties and the intensity of the diffuse "spikes". This investigation, conducted by Professor Mieko Takagi, has shown that great point-by-point variations in both UV absorption and "spike" intensity are commonly found within a single stone, thereby clearly demonstrating the need for the "topographic approach" to this problem. However, the investigation has shown that, point-by-point within the diamond, there is a linear relationship between UV absorption and "spike" intensity. Thus the X-ray experiments indicate the either all or a constant fraction of the nitrogen has been precipitated in the form of the very thin platelets that cause the diffuse "spikes".

3. Radiation Damage

Work so far has been mainly concerned with X-irradiation of lithium fluoride. Heavy irradiation causes an overall increase in lattice imperfection and loss of dislocation contrast. The most notable effects of lighter doses, when a selected area of a plate of lithium fluoride has been irradiated, is to cause strong intensification of diffracting power at the boundary separating the irradiated and non-irradiated areas. This effect appears to be due mainly to the dilation of the irradiated part. Some experiments have been performed on pile-irradiated diamonds. This material shows a very homogeneous expansion under neutron irradiation so that sufficient long-range order of the lattice is maintained to give good dislocation contrast even when the overall expansion has increased the average lattice parameter by more than 1%.

4. Magnetic Domain

Strains associated with magnetostrictive effects enable the configuration of magnetic domains to be studied in sheets of ferromagnetic material. Domains, dislocations and low-angle boundaries can be seen simultaneously on the same topograph. The X-ray topograph does not depict solely the surface domain configuration, for domain structures in the specimen interior can be seen which do not appear on the colloid pattern. Also, repetitive experiments can be made showing domain move-

Fig. 2. Magnetic domains in Fe+3.5% Si alloy. Scale mark 25 μ.

ments and no physical contact with the specimen is necessary for such observations. A variety of domain patterns has been studied in Fe+3% Si alloy[5], an example is shown in Fig. 2. This is a topograph of part of a thin (112) plate, thickness about 50 μ. The pattern, though complicated, is simpler than the colloid pattern. The X-ray pattern averages the strains of surface closure domains over a range of several microns, producing a stripe pattern with stripe repeat period in the range 10 to 15 microns. The stripes lie either along $[20\bar{1}]$ or along $[0\bar{2}1]$, these directions representing the outcrops of the two cube planes most steeply inclined to the (112) surface. The boundaries separating areas of differently directed stripes are 90° Bloch walls and they lie along $[11\bar{1}]$ or $[1\bar{1}0]$. Extra strong stripes can be seen leading from corners in the 90° walls: they show the location of 180° Bloch walls.

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