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Dislocation Structures Observed in High-purity Recrystallized Aluminium by X-ray Diffraction

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The x-ray diffraction method of observing dislocations within crystals (Lang 1958) allows examination of sections up to several millimetres in thickness in the case of light elements. Hence the dislocation density found this way should be typical of the material in bulk. We wish to report preliminary results of a survey of crystals of especially pure aluminium grown in this laboratory. X-ray examination indicates a quite low dislocation density, in the range of 10^4 to 10^5 lines per cm^3 within sub-grains. A variety of interesting dislocation configurations has been observed, and from the quality of dislocation contrast some general conclusions can be drawn about the perfection of the crystals.

The starting material, 99·999% pure Al, was zone-refined with twenty passes. Portions taken from the purest region of the ingot were cold-rolled into strips approximately 5 cm by 1 cm by 2 mm, After etching and washing, these were annealed overnight in air at 495°c. During the anneal they rested on a bed of alumina powder. The strips were then given a 2% extension in a Hounsfield tensometer, re-annealed overnight at 495°c and electropolished. The resulting grains averaged about 1 cm^2 in area.

Figure 1 (*a*) and (*b*) (Pl. 92) shows a stereo-pair of x-ray diffraction micrographs taken by the method of 'projection topographs' (Lang 1959) using AgKα radiation and the 111 and $\overline{111}$ reflections. These figures are photomicrographic prints from the original x-ray images, hence dislocations appear light on a dark background. The field covered is about 700 by 950 microns in area. The particular region shown has the shape of a blunted wedge, about 20 μ thick at the sub-grain limit in the upper right and increasing to about 100 μ in the lower left. The near surface of the wedge is the surface of the specimen, the far surface is a sub-grain boundary. Figure 2 provides a key to some of the features which are, of course, more clearly seen on the original x-ray images. The sub-grain boundary outcrops at the specimen surface along the line A: a fair amount of extra x-ray reflecting power is irregularly distributed on this boundary and can be seen as a sheet dipping into the crystal when the micrographs are viewed stereoscopically. Quite frequently observed are irregular helical dislocations B. Their pitch varies widely, 50 μ being about an average value.

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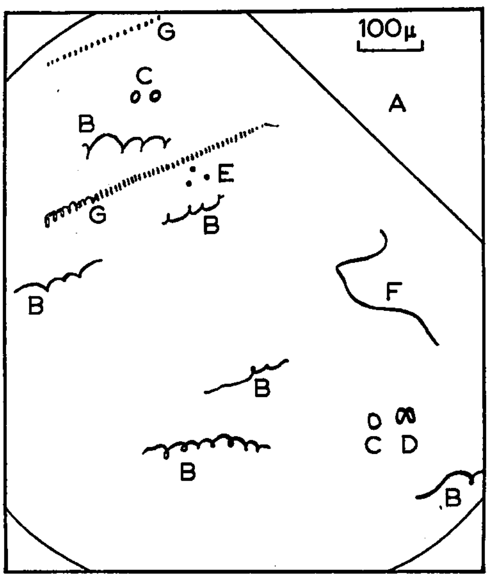
Isolated loops are seen at C, and D appears to be a distorted figure-of-eight loop. The three features E are believed to be smaller loops. (The present resolution of the x-ray method does not resolve loops as such when their diameter is 5 μ or less.) A single dislocation leaving and returning to the low-angle boundary is seen at F. Features of interest which are fairly frequently observed are indicated at G. As far as they can be resolved, they appear to be mainly lines of regularly spaced loops of diameter and spacing in the range 10 to 15 μ, but regular helices, figures-of-eight, and multiple figures-of-eight also can be seen. There is a close resemblance to the features observed in silver chloride crystals by Jones and Mitchell (1958) and Parasnis and Mitchell (1959).

Fig. 2



Key to dislocation features in x-ray topographs.

By comparison with silicon, germanium and good quality halide crystals, the dislocation contrast is very poor. Some type of lattice imperfection is present other than the dislocations seen in the figures. The x-ray evidence does not rule out the presence of large numbers of dislocation loops of 1 μ diameter or less, which could not be seen individually. However, we believe the principal imperfection to be small variations in lattice parameter caused by irregularly distributed impurities. In nearly perfect crystals local variations from the mean lattice parameter of the order of 1 part in 10^6 cause a recognizable increase in x-ray reflecting power. Very noticeable in the x-ray topographs of aluminium are diffuse sheets and lenticles of increased reflectivity. Some of these appear to be associated with concentrations of slip lines prior to recrystallization.

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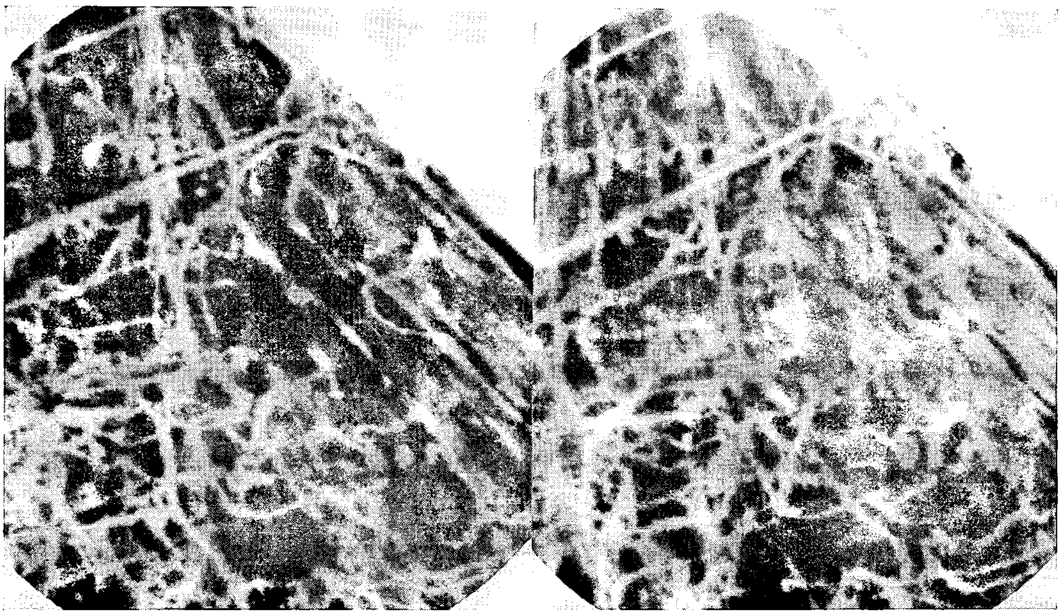
Fig. 1



(a) (b)

Stereo-pair of x-ray projection topographs of aluminium.