

## On the Macroscopic Distribution of Dislocations in Single Crystals of High-purity Recrystallized Aluminium

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### ABSTRACT

Single crystals about 1 cm<sup>2</sup> in area and 1 mm thick prepared by annealing both lightly and heavily deformed zone-refined aluminium have been examined x-ray topographically. From stereopairs of various Bragg reflections the orientation and Burgers vectors of dislocations could be determined. Repetitive examination showed dislocation movements after slight deformation and also after long storage. Helices and sets of coaxial prismatic loops are seen with diameters ranging from a minimum observable value of about 1 μm to over 60 μm, and they may extend more than 1 mm along <110> directions. These features result from absorption of vacancies by long screw dislocations which, it is concluded, constituted a major part of the dislocation population grown-in during recrystallization. Dislocation densities of only a few tens per cm<sup>2</sup> are found within 200 to 300 μm of the specimen surfaces and evidence is presented that such low densities arise from loss of dislocations at the surfaces.

### § 1. INTRODUCTION

SEVERAL years ago Lang and Meyrick (1959) showed that the dislocation density in single crystals of zone-refined aluminium grown by the strain-anneal method could be sufficiently low for the dislocation configurations within millimetre thick specimens to be satisfactorily recorded by x-ray topography. Dislocation counts on section topographs (Lang 1957) of single crystals 1 to 2 mm thick indicated dislocation densities of 10<sup>4</sup> to 10<sup>5</sup> lines per cm<sup>2</sup> within sub-grains, and projection topographs (Lang 1959) showed that in thin wedge-shaped volumes contained between the specimen surface and a grain boundary making a small angle with the surface the dislocation density could be as low as some tens of lines per cm<sup>2</sup>. On the other hand, single crystals of similar material grown by solidification in a graphite-coated quartz boat held in a travelling furnace were found by x-ray topography to have dislocation densities of the order of 10<sup>5</sup> to 10<sup>6</sup> lines per cm<sup>2</sup> on the average, and at best only locally as low as 10<sup>4</sup> lines per cm<sup>2</sup> (Meyrick 1959). Since this early work Howe and Elbaum (1961) have

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shown that slender crystals of zone-refined aluminium pulled from the melt can be substantially dislocation-free, and quite recently Nøst (1965) has demonstrated that large strain-anneal grown crystals of zone-refined aluminium can be produced with average dislocation densities of only some hundreds of lines per  $\text{cm}^2$ . Thus the question whether single crystals of pure aluminium can be produced with very low dislocation densities is no longer an unsolved problem, but the dislocation configurations that are observed among the sparse dislocation population in strain-anneal grown crystals are very curious and worthy of study. An account is here presented of the configurations and movements of dislocations in such crystals.

### § 2. SPECIMEN PREPARATION

Most of the material used was zone-refined in the H. H. Wills Physics Laboratory (Meyrick 1959) and was similar to that used in the first x-ray topographic studies (Lang and Meyrick 1959). In addition some zone-refined aluminium was kindly supplied to us by Dr. D. J. Barber, Aluminium Research Laboratories Ltd., Banbury. Cleaned pieces of the zone-refined ingots were cold-rolled into sheets 1 to 2 mm thick. Strips 5 cm by 1 cm in area were cut from the sheets, cleaned in aqua-regia, polished mechanically and electrolytically, and then annealed, usually for 15 hours at  $500^\circ\text{C}$ . The majority of the annealed specimens were then strained by tension in a Hounsfield tensometer. Strains varying from 1 to 8% were introduced: the best values to use appeared to be between 3 and 4%. These lightly strained specimens were then given a second anneal, usually under the same conditions as the first anneal. A final electropolish was given to all specimens before x-ray examination.

Great care was necessary to avoid accidental deformation of the specimen. Two methods were used to reduce the incidence of deformation. In one method the specimen was laid on a strip of aluminium during the anneal so that its bed had the same thermal expansion as itself. Alternatively, the specimen was clamped in an aluminium frame in which it could remain during annealing, final electropolishing and the x-ray examination.

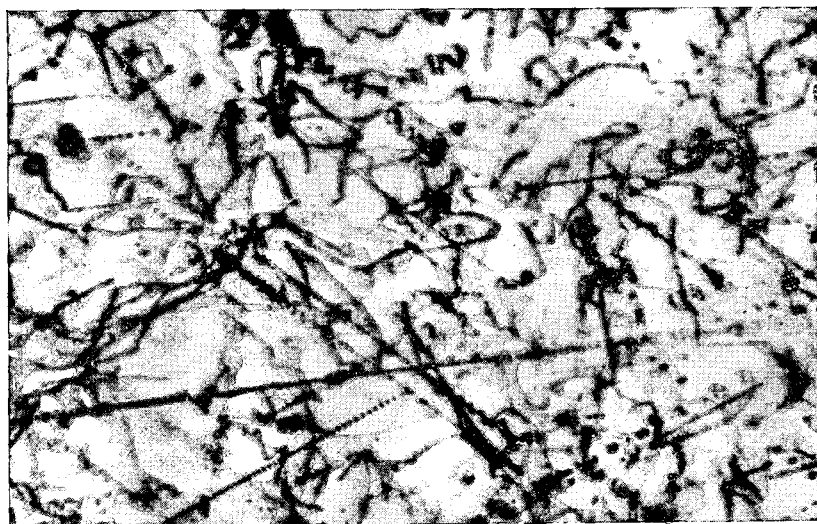
The crystals of highest quality were found in specimens which had been strained in the tensometer and given a second anneal. Topographs both of these crystals and of those formed in a single anneal after cold-rolling are reproduced here. All topographs were taken with  $\text{AgK}\alpha$  radiation.

### § 3. DISLOCATION CONFIGURATIONS

Figure 1 shows part of a projection topograph of a grain about  $1\text{ cm}^2$  in area. The dislocation pattern is typical of the better crystals we have examined. This specimen had been annealed for 16 hours at  $550^\circ\text{C}$ , strained about 2% and then annealed again for 16 hours at  $550^\circ\text{C}$ ; the aluminium used was supplied by Dr. Barber. The crystal thickness in

the field of fig. 1 is  $1\frac{1}{4}$  mm. On projection topographs good contrast is produced by dislocations near the x-ray exit surface of the crystal but the images of dislocations more than about  $\frac{2}{3}$  mm from the exit surface are weak and diffuse. A spreading out of the latter images is an unavoidable consequence of the relatively large angle between the directions of incident and diffracted rays (compared with the electron diffraction case) and similar diffuseness is evident in dislocation images from very pure but thick crystals of silicon (see, for example, fig. 1 of Authier and Lang (1964)).

Fig. 1

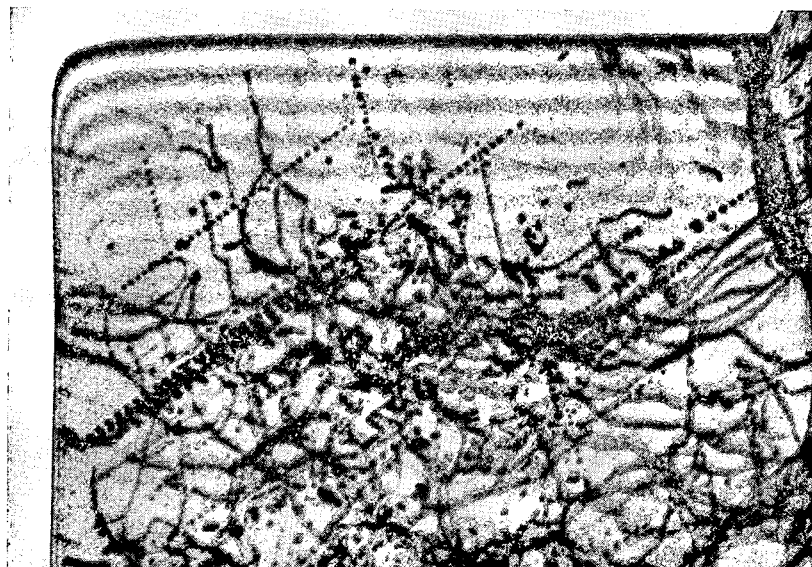


Projection topograph of crystal  $1\frac{1}{4}$  mm thick, 111 reflection,  $\text{AgK}\alpha$  radiation; width of field 1.5 mm. Axes of helices and sets of coaxial prismatic loops are [110], [101] and [011].

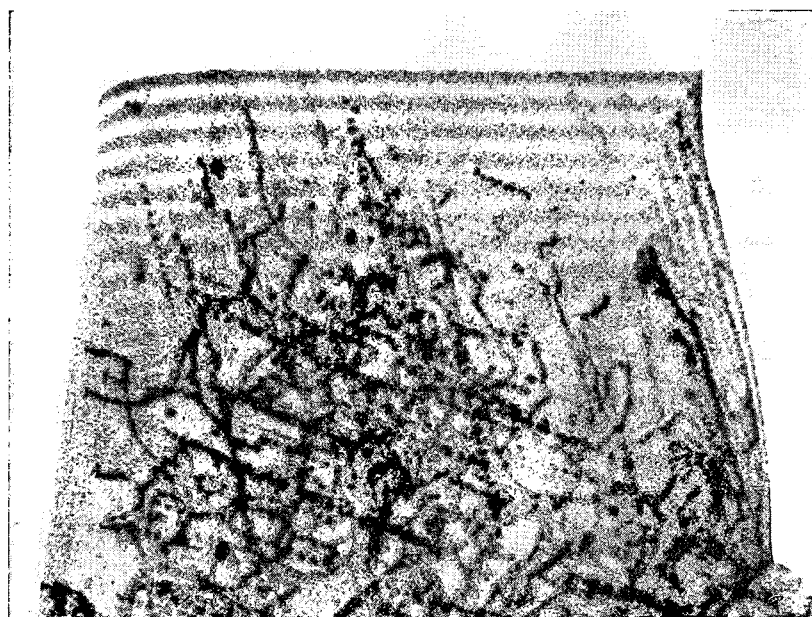
In the present crystal, however, there does appear to be some additional scattering in regions close to the mid-plane of the specimen which reduces the visibility of dislocations lying near the x-ray entrance surface. In section topographs, on the other hand, this crystal shows high diffraction contrast from all dislocations intersected by the direct x-ray beam, and on such sections reliable dislocation counts can be made. Excluding a few local concentrations presumably centred on inclusions,  $10^4$  lines per  $\text{cm}^2$  can be taken as the average density in this specimen as a whole, bearing in mind that the density varies strongly with distance from the crystal surfaces, as discussed below. Image widths of dislocations near the x-ray exit surface and for which  $\mathbf{g} \cdot \mathbf{b} = 1$  are somewhat less than  $5 \mu\text{m}$  in the case of those of mainly screw character, and somewhat more than  $5 \mu\text{m}$  for those that are mainly edge.

The striking features observed in this and other crystals are the long helices and lines of coaxial loops, all lying in  $\langle 110 \rangle$  directions. In each

Fig. 2

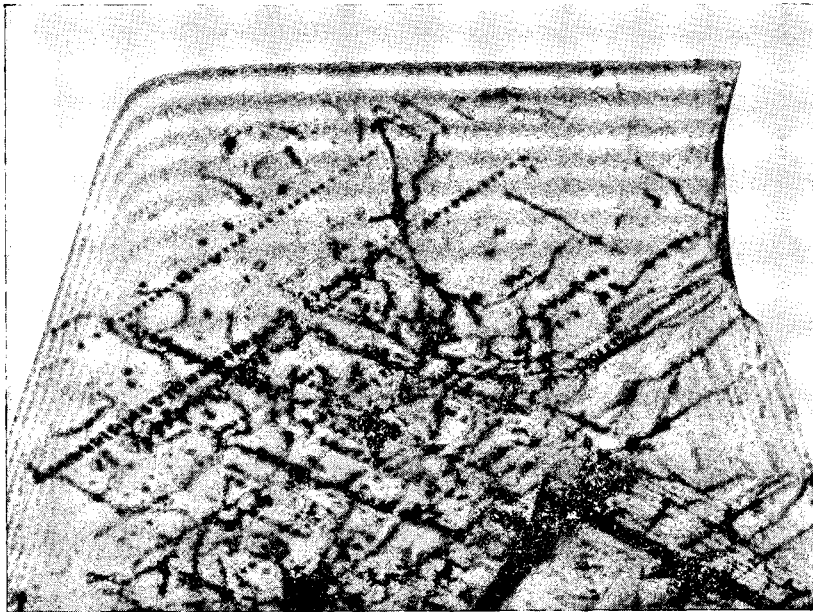


(a)

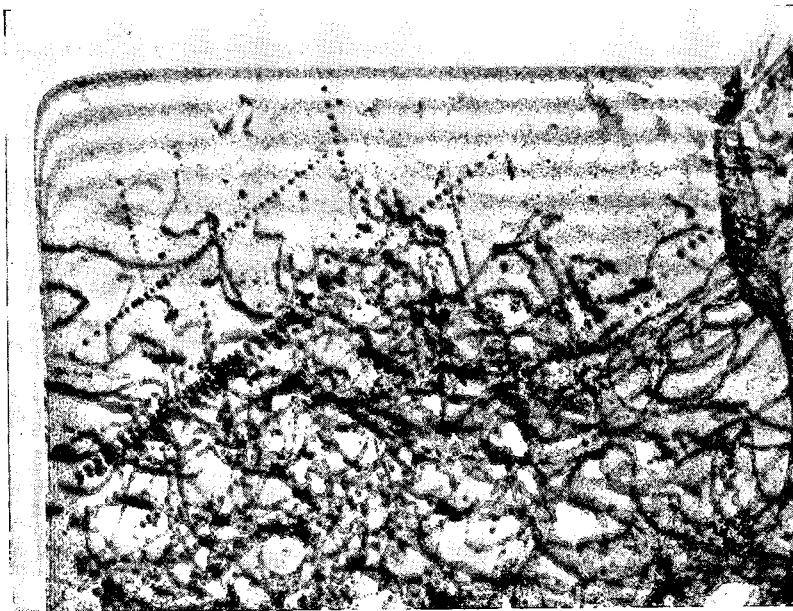


(b)

Fig. 2 (continued)



(c)



(d)

Projection topographs of wedge-shaped region, maximum thickness  $650 \mu\text{m}$ . (See fig. 3 for numbering of features.) (a) 111 reflection, loops (1) and (4) visible; (b) 111 reflection, loops (3) and (4) visible; (c)  $\bar{1}\bar{1}\bar{1}$  reflection, loops (1) and (2) visible; (d) repeat of (a) after  $1\frac{1}{2}$  years.