

sults indicate that during annealing the larger black spot defects coarsen at the expense of the smaller ones until tetrahedra are formed. The fact that some of these are paired is thought to be an indication that the stress field in the neighbourhood of a tetrahedron modifies the local distribution of vacancies. If a black spot defect is favourably positioned with respect to a tetrahedron this local redistribution might favour its growth so that it too would become a tetrahedron. This interpretation is quite tentative however.

Both loops and black spot defects survive the first annealing stage, at about 200°C, in the irradiated gold, (Fig. 4). This stage is thought to be associated with the breaking away of vacancies from impurity atoms. The second stage centered at about 425°C, is accompanied by the simultaneous annealing out of both types of

defect. The fact that the final stages occur at different temperatures, and that no tetrahedra are observed in the annealed neutron-irradiated gold, in spite of the similarity in the initial concentrations of point defects (compare the heights of the two initial plateaux), has been taken to indicate that the loops in the irradiated gold are of the interstitial type<sup>3</sup>.

The author is indebted to Dr. D. G. Brandon for communicating this paper.

### References

- 1 R. M. J. Cotterill: *Phil. Mag.* **6** (1961) 1351.
- 2 J. Silcox: *Proc. European Regional Conf. of Electron Microscopy, Delft* (1960).
- 3 R. M. J. Cotterill: Thesis, University of Cambridge (1962).

**Saada, G.:** The model I developed can indeed be used to describe the stability of Frank sessile loops in irradiated metals. It should be very interesting to know if the 75 Å diameter loops are of Frank sessile or perfect type. The model suggests they should be of Frank sessile type.

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## The Observation of a Dislocation "Climb" Source

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It has been found, using thin film electron microscopy, that aluminium 3.5 and 7.3 atomic per cent magnesium alloys quenched into silicone oil contain concentric diamond-shaped dislocation loops lying upon the cube planes. An examination of the various dislocation interactions which occur, together with the electron diffraction contrast behaviour, show that these loops have alternate Burgers vectors of the  $a/2 \langle 110 \rangle$ -type lying in the cube face perpendicular to that in which the loops lie. The source is believed to be a dislocation of the  $a \langle 001 \rangle$ -type which accepts vacancies during the quench, but dissociates during climb into two component dislocations. This dissociation can occur with the new Burgers vectors lying in either of two possible cube planes and evidence of both types are frequently seen. The origin of the source is discussed.\*

Aluminium 3.5 and 7.3 atomic percent magnesium alloys quenched into silicone oil and examined in transmission in an electron

\* A full account will be published in the *Philosophical Magazine*.

microscope contain concentric diamond-shaped dislocation loops, (Fig. 1). The plane of the foil is approximately parallel to the (001) plane. It therefore follows, because the loops in this and many other foils extend

over a distance of several microns without emerging from the surface of the foil, that the loops are probably lying in the  $\{001\}$  planes. The electron diffraction contrast behaviour and dislocation interactions suggest that successive loops have alternate Burgers vectors of the  $a/2\langle 110 \rangle$  type lying in a cube face perpendicular to that in which the loops lie. For example in Fig. 2 a set of loops is shown, in (a) the 020 reflecting planes are giving rise to the diffraction contrast, and in (b) the 200 reflecting planes. Applying the criterion that a dislocation is "invisible" when its Burgers vector lies in the reflecting

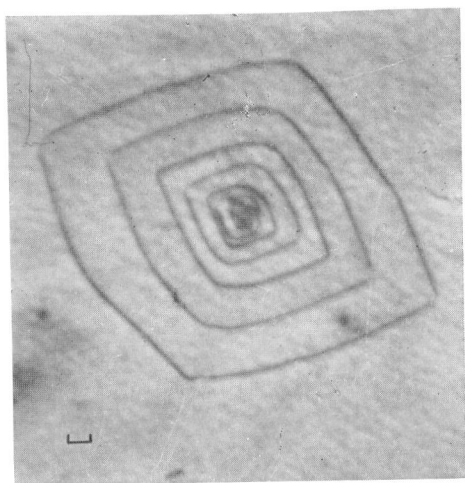


Fig. 1. Micrograph of concentric dislocation loops in quenched aluminium 3.5 at. % Magnesium. Magnification mark  $0.1\mu$ .

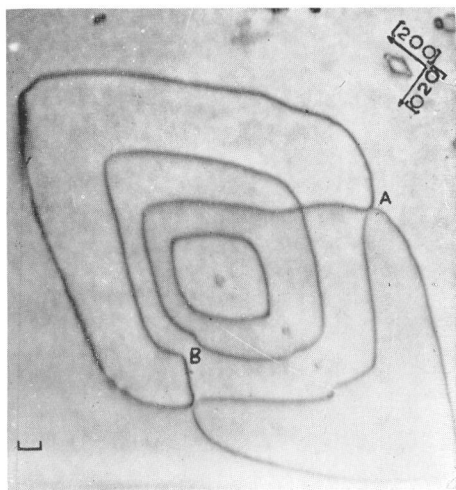


Fig. 2(a). Set of dislocation loops with the diffraction contrast arising from the 020 reflecting planes. Magnification mark  $0.1\mu$ .

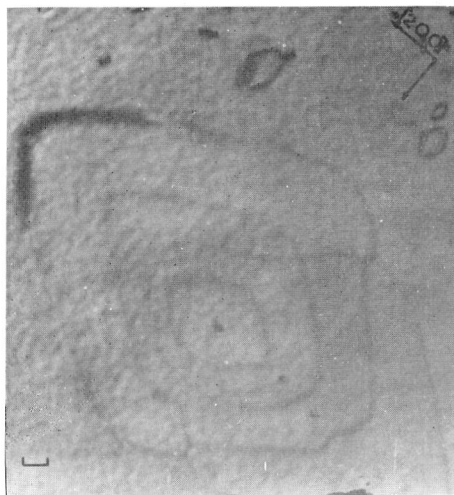


Fig. 2(b). The same loops with the 200 reflecting planes operating. The contrast behaviour in this photograph shows that the Burgers vectors of the loops are in the  $(100)$  plane. Magnification mark  $0.1\mu$ .

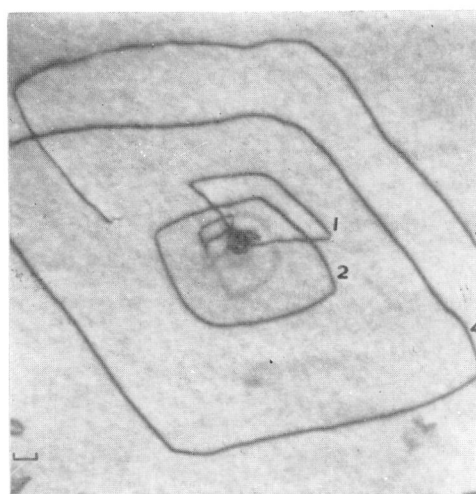


Fig. 3. The displacement of loops 1 and 3 by slip indicates that successive loops have different Burgers vectors. Magnification mark  $0.1\mu$ .

plane, it follows that the dislocations in Fig. 2(b) are invisible because the Burgers vector of the loops lie in the  $(100)$  plane. Fig. 2 also shows an independent loop from another source interacting with the set of concentric loops. The interaction with one of the loops at A differs from that with a neighbouring loop at B. In the latter the dislocations have amalgamated and thus have the same Burgers vector. Further evidence that alter-

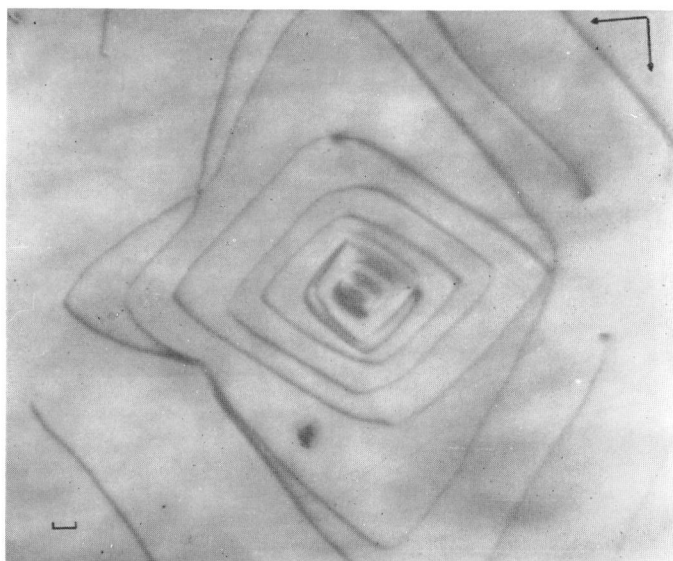
nate loops have different Burgers vectors is seen in Fig. 3 where loops 1 and 3 appear to have moved from their originally concentric position, presumably by slip, while loops 2 and 4 have not.

We believe the dislocation loops emanate from a source consisting of a dislocation of the  $a\langle 100 \rangle$  type which accepts vacancies during the quench but dissociates during climb

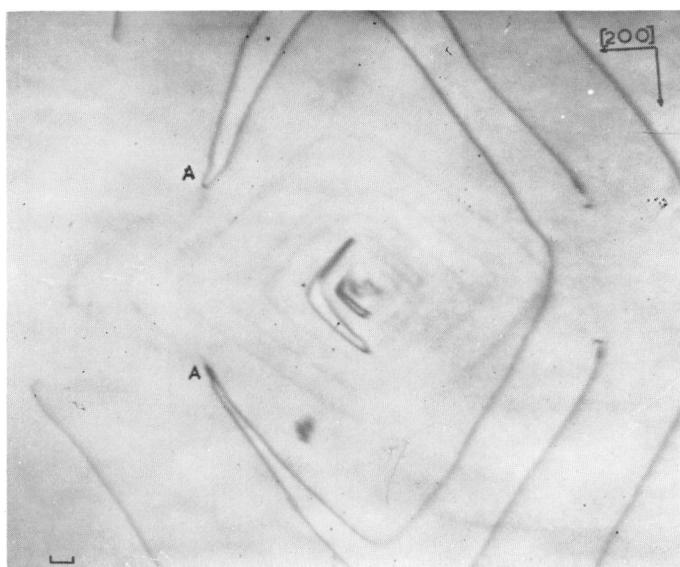
into its component dislocations. Such dissociation can occur in either of the two possible cube planes, *i.e.*

$$a[001] \begin{cases} a/2[011] + a/2[0\bar{1}1] & \text{which both lie in the (100) plane} \\ a/2[101] + a/2[\bar{1}01] & \text{which both lie in the (010) plane} \end{cases}$$

Evidence of both types are frequently seen



(a)



(b)

Fig. 4. Source generating concentric dislocation loops with all four possible Burgers vectors where the reflecting planes giving rise to the contrast are 220 and 200 in (a) and (b) respectively. Magnification mark  $0.1 \mu$ .

and the diffraction contrast behaviour shows the Burgers vectors to lie in the appropriate planes. For example, Fig. 4 shows a source generating dislocation loops with all four possible Burgers vectors. The contrast behaviour in (a) and (b), where the reflecting plane giving rise to the contrast are 220 and 200 respectively, shows that the outermost loops have Burgers vectors in the (010) plane and the innermost in the (100) plane. At AA two of the outermost loops have apparently combined and redissociated in the

alternative manner to give dislocations with Burgers vectors lying in the other vertical cube plane, corresponding to those of the inner loops. Such interactions show that the composite Burgers vector is a  $a[001]$  i.e. perpendicular to the plane of the loops and so give confidence that the loops are formed by climb and not slip. The origin of the source is thought to be a dislocation loop with an  $a[001]$  Burgers vector formed at a precipitate.

## DISCUSSION

**Kuhlmann-Wilsdorf, D.:** The loops shown in the micrographs have grown on the cube planes, even though their mechanical energy would be lowered if they turned into the  $\{110\}$  plane normal to their Burgers vector. I should like to suggest that this may be regarded as confirmation of the theory presented in lecture IF 12\* by Dr. L. A. Girifalco and myself. According to this theory, pure edge dislocations climb rather more sluggishly than mixed dislocations, so that large loops should be expected to consist of mixed dislocations rather than pure edge dislocations. I might add that the profuse presence of  $\{111\}$  dislocation loops in quenched aluminum, instead of loops on  $\{110\}$  with Burgers vector  $\frac{1}{2}\langle 110 \rangle$  which are mechanically the most stable is similarly surprising and may have the same explanation.

\* *Proc. Int. Conf. Cryst. Latt. Def.* (1962): J. Phys. Soc. Japan **18** Suppl. II (1963) 230.

**Barnes, R. S.:** The presence of loops with Burgers vectors  $\frac{1}{2}a\langle 011 \rangle$  on the cube planes is perhaps surprising. We have suggested that this is because the  $a[001]$  dislocation only dissociates after climb has begun and subsequent slip of the loops onto planes normal to their Burgers vector would introduce extra shear stress. This is because successive co-planar loops with alternate  $\frac{1}{2}a[011]$  and  $\frac{1}{2}a[0\bar{1}1]$  vectors reduce the shear stress on the (001) plane. However the diamond shape of the loops, with the Burgers vector lying at  $45^\circ$  to the minor axis and the plane of the loop, may well be explained by the dislocation climb rates.

**Bullough, R.:** I don't see why one should rely on simple elasticity theory to say that loops should lie on  $\{110\}$  planes. Such simple elasticity theory predicts that line dislocations should slip on  $\{110\}$  planes. The actual slip plane in the cubic metals is however  $\{111\}$  and not  $\{110\}$ .

**Barnes, R. S.:** A prismatic dislocation loop formed from a given number of vacancies will have a minimum energy when its plane is normal to the Burgers vector. Loops with  $\frac{1}{2}a\langle 011 \rangle$  Burgers vectors have frequently been seen to rotate into the  $\{011\}$  planes by slipping on their slip cylinders.

**Thomas, G.:** In your second figure on the left hand side (loops out of contrast) the outside loop shows very strong contrast at the bottom left, extending for some way from the corner. Does this mean that there is a change of Burgers vector or climb plane at this point or is there a possibility of segregation of Mg atoms there. If the latter is true, this might provide a means of determining whether solute atom segregation to dislocations occurs, simply by tilting the dislocation out of contrast and looking for residual contrast effects.

**Barnes, R. S.:** The Burgers vector can not change along a given dislocation line and a change in the climb plane is not likely to alter the diffraction contrast from a dislocation loop. I suggest that the changed contrast is probably due to this part of the dislocation loop being near the foil surface where some modification of the dislocation stress field occurs. Although there is little reason to believe that there

is localized precipitation on the dislocation loop, the standard test is to "extinguish" the dislocation contrast; the dislocation will remain visible if it has fine precipitates because of their extra contrast.

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## Precision Density Measurements as a Tool for the Investigation of Point Defects

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The technique of measuring smallest density changes of macroscopic metal specimens, based on Archimedes principle, has been improved to the extent that relative density changes as small as  $3.5 \times 10^{-7}$  can be detected. At the same time the experimental effort involved has been reduced. A further improvement, to a sensitivity of about  $2 \times 10^{-7}$ , is believed possible with only little additional effort. The method shall be employed for an investigation into the production or absorption of point defects and their aggregates by moving dislocations, and also into the annealing behavior of these defects. A preliminary result is in accord with a previous prediction, namely that annealed f.c.c. metals contain vacancies condensed into submicroscopic voids, and that these are eliminated by moving dislocations, leading to an initial increase of density in specimens deformed after annealing. The density increase in coarse grained aluminum specimens, deformed in tension by about 2%, is tentatively given as  $\Delta\rho/\rho = (1.32 \pm 0.09) \times 10^{-5}$ .

### 1. Introduction

The most direct measuring technique by which the behavior of point defects in general and of lattice vacancies in particular may be investigated, presumably lies in sensitive determination of density changes<sup>(1-5)</sup>. Unfortunately, with previously available methods the best resolution obtainable lay in the order of  $\Delta\rho/\rho = 10^{-5}$  or at most  $5 \times 10^{-6}$  for the atomic fraction of vacancies. This sensitivity is unsatisfactory since the total fraction of thermal vacancies in a solid usually is below  $10^{-4}$ , and since even a fraction of  $10^{-6}$  vacancies is believed to give rise to significant effects. For this reason an improved technique for the measurement of small density changes in macroscopic solid specimens has been devised. It is a further development of the method described by Bell<sup>(6)</sup>, in which the weight difference between a specimen and a dummy of the same material and of closely similar mass and dimensions

is determined, first in air and then in a suitable liquid.

### 2. Experimental Technique

For the case that specimen and dummy have closely similar mass, that the specimen is subject to a treatment which changes its density but not its mass, and that the density of the air is neglected compared with the density of the weighing liquid, the relative density change of the specimen is given by  $\Delta\rho/\rho = \Delta W_L / V\rho_L$ , where  $V$  is the specimen volume,  $\rho_L$  is the density of the liquid.  $\Delta W_L$  is the change of the measured weight difference between specimen and dummy, when both are in the weighing liquid, before and after the treatment. Consequently, the smallest relative density change which can be measured in a specimen of constant mass is limited by the smallest detectable weight difference  $\delta(\Delta W_L)$  when specimen and dummy are compared while immersed in the weighing