

# CLIMB OF PRISMATIC LOOPS IN Cu-Al ALLOYS DURING IRRADIATION IN THE HVEM\*

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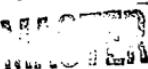
## ABSTRACT

Nucleation and growth of prismatic interstitial loops was observed during irradiation in the HVEM at different temperatures and damage rates. The loops remained rhombus shaped throughout growth, with sides lying on {111} planes, suggesting low jog densities. Weak-beam electron micrographs showed that the loops were dissociated on the {111} planes containing the loop sides. A minimum damage rate,  $\Phi_{min}$ , was found to be necessary to induce growth at measurable rates. This, together with the low jog densities observed, was taken to indicate that the climb rate was affected by the rate of jog nucleation, rather than controlled entirely by the rate of diffusion of point defects to the dislocations.  $\Phi_{min}$  was found to be smaller for alloys of higher stacking fault energies, probably due to the influence of stacking fault energy on the energy of jog formation on an extended dislocation. The growth rate was found to be constant for individual loops observed over a large size range, suggesting that jog nucleation occurred preferentially at loop corners.

## INTRODUCTION

It is generally accepted that the growth of voids in metals during irradiation requires the presence of dislocations, which absorb more interstitials than vacancies due to the stronger interaction of the larger strain fields of interstitials with dislocation strain fields. The dislocations climb at a rate determined by the difference between

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the absorption rates of interstitials and vacancies. If an energy barrier exists for the absorption of point defects at the dislocation core, the climb rate may be affected by the rate at which absorption occurs, rather than controlled only by the rate of diffusion of point defects to the dislocation.

The absorption or emission of point defects by a climbing dislocation requires the presence or nucleation of jogs, whose formation is more difficult in FCC metals of low stacking fault energy ( $\gamma$ ) because of the splitting of perfect dislocations into a pair of Shockley partials separated by a ribbon of stacking fault. The formation of jogs on extended dislocations has been considered by several authors.<sup>1,2,3</sup> Calculations of the energy of completely extended jog pairs in Cu-7.5 wt.% Al ( $\gamma = 5$  ergs per  $\text{cm}^2$ )<sup>4</sup> yield values of about 58 eV, depending on the estimate used for the energy of the closely spaced stair rod dipoles.<sup>5</sup> The energy of a double constriction on such a dislocation should be 107 eV. Thus, the formation of jogs on widely split dislocations is a difficult process; the exact mechanism is still unknown.

Theories of void formation<sup>6</sup> and of dislocation loop growth<sup>7</sup> in metals during irradiation generally assume diffusion controlled climb. In the present work evidence is presented which suggests that in FCC metals of very low stacking fault energy, climb may sometimes be affected by the rate of jog nucleation.

#### EXPERIMENTAL

Thin foils of Cu-Al alloys with 1.0, 3.5, and 7.5 wt.% Al were irradiated at 300°C and 450°C in an Hitachi Hu650 high voltage electron microscope operated at 650kV. The damage rate was varied by changing the excitation of the second condenser lens, and the beam current densities thus obtained were measured with a Faraday cup situated below the screen. Series of micrographs were taken during irradiation, and the loop growth rates were obtained from measurements of the loop sizes on contact prints.

## OBSERVATIONS

Nucleation and growth of interstitial dislocation loops were observed over a range of temperatures. Although the maximum damage rate obtainable (about  $1.5 \times 10^{-4}$  dpa/sec with the beam focussed) was such that loops could not be nucleated above about 400°C, it was observed that loops nucleated at lower temperatures would still grow at 450°C at rather low damage rates, indicating that loop growth requires a lower supersaturation of point defects than does loop nucleation.

The loops observed were always rhombus-shaped prismatic loops with sides lying on (111) planes. Fig. 1 shows a typical set of loops grown at 300°C in a 110 foil of the 3.5 wt.% Al material. The correctly oriented Thompson tetrahedron is shown with the projected shapes of rhombus-shaped pure edge prismatic loops of all six  $a/2 <110>$  Burgers vectors. Type A, B, and C loops are out of contrast, since  $\vec{g} \cdot \vec{b} = 0$ . Type D and type E

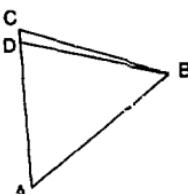
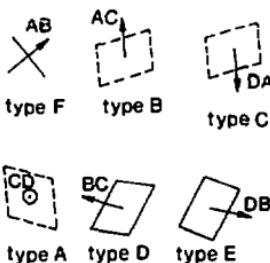


Fig. 1. Loops grown in Cu-3.5 wt.% Al at 300°C. The oriented Thompson tetrahedron and projected shapes of pure edge rhombus loops are shown.

loops each have a pair of sides, lying in the BCD  $\{111\}$  plane, which appear straight. Because the BCD plane is seen nearly edge on, this indicates that the dislocations have low jog densities, or at least equal densities of opposite-sign jogs. The curvature that appears on other sides is due to glide bowing of the side on the  $\{111\}$  plane.

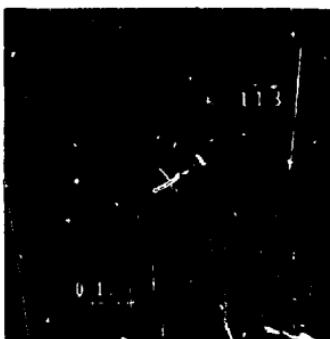
A typical growth series, obtained using  $0.081$  amps/cm $^2$  ( $1.26 \times 10^{-5}$  dpa/sec), is shown in Fig. 2. Notice that the loop sides which lie on inclined  $\{111\}$  planes are curved. Careful comparison of observed loop shapes with the projected shapes for pure edge loops indicates that the loops are generally rotated somewhat from the pure edge orientation, and that the curvature of the sides increases with increasing deviation from edge character. That the curvature of the sides is due to their being bowed out in their glide planes rather than to being heavily jogged is demonstrated in Fig. 3, which shows a loop with bowed sides which has slipped to the foil surface between micrographs. A slip trace is left which shows the intersection of the glide prism with the foil surface to be straight sided.



Fig. 2. Loops grown in Cu-3.5 wt.% Al at  $300^\circ\text{C}$ ,  $1.26 \times 10^{-5}$  dpa/sec.



(XBB 755-3978 C & D Only)  
Fig. 3. A loop which slips to the foil surface leaving a straight-sided slip trace.



(XBU 759-7195)  
Fig. 5. 650kV weak-beam image of a loop with a partially constricted superjog.

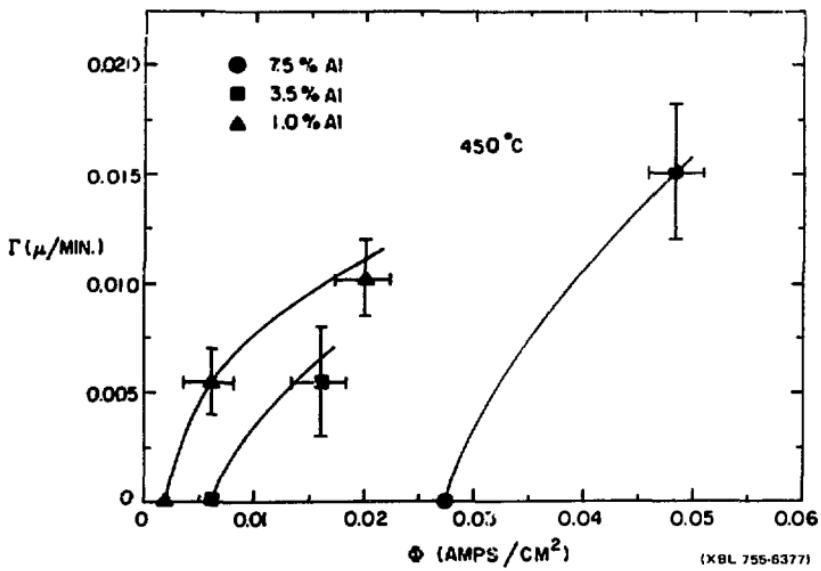


Fig. 4. Loop growth rate ( $\Gamma$ ) versus beam current density ( $\Phi$ ) at 450°C.

Loops were observed to grow at a constant rate. Fig. 4 shows the results of a preliminary determination of the dependence of the loop growth rate  $r$  on the beam current density  $\phi$ . It should be emphasized that the growth rates observed at the points at which the curves intersect the axis were negligibly small.

Fig. 5 shows a 650kV weak-beam image of a loop with both Shockley partials in contrast on the two sides lying in the inclined (111) planes. The splitting should not be visible on the other two sides since they lie in the nearly edge-on (111) planes. This is a 110 foil of Cu-7.0 wt.% Al;  $\vec{g} = \bar{1}\bar{1}3$ ;  $\vec{b} = a/2 [01\bar{1}] + a/6 [\bar{1}2\bar{1}] + a/6 [1\bar{1}2]$ . A partially constricted superjog, about 46 Å high, is seen on one side, and it is also seen that the corners are at least partially constricted.

Isolated dislocations initially present in the foil were often observed to climb into helices with straight sides also lying in {111} planes, as in Fig. 2. The helices increased their diameters at about the same rate as the loops.

#### DISCUSSION

The observation (Fig. 4) that loop growth ceases below a certain beam current density indicates that there is an activation barrier for the absorption of interstitials by the loops. The supersaturation of interstitials was calculated using the model of Foreman,<sup>8</sup> and the partial molar free energy was found to be about 2 eV per interstitial even at 450°C and 0.01 amps/cm<sup>2</sup>. Since this is much less than the energies calculated for extended jogs or for constrictions, jog nucleation must proceed via some intermediate configuration with an energy not much larger than 2 eV. Since weak-beam observations of the loops show that they do remain widely split while climbing, it is to be expected that the activation energy required will depend on the stacking fault energy through its effect on the width of splitting. The observation that lower supersaturations of interstitials are required to induce growth at 450°C of the less widely split loops in the lower Al content alloys implies that the activation barrier for growth is smaller in alloys of larger stacking

fault energy, as expected. Since the jog nucleation rate should depend on the number of nucleation sites on a loop, the observations of constant growth rate and very low jog density suggest that jogs may be preferentially nucleated at loop corners rather than along the loop sides. This might be expected if the corners are constricted, as they appear to be (Fig. 5).

The rotation of the loops was attributed to the presence of shear stresses in the foil. These stresses were estimated by measuring the radius of curvature of the loop sides. Calculations of the rotations to be expected due to such stresses, based on Bullough and Foreman's<sup>9</sup> expression for the total energy of a loop, predicted consistently larger rotations than those observed. However, in weak-beam micrographs the partial on the convex side was usually more curved than the other. When the partials are not resolved, this could lead to an overestimate of the curvature, and therefore of the stress acting on the loop.

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