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Author

Narayan, J.

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ON THE STABILITY OF DISLOCATION LOOPS NEAR A FREE SURFACE

J. Narayan and J. Washburn

Inorganic Materials Research Division, Lawrence Berkeley Laboratory and
Department of Materials Science and Engineering, College of Engineering;
University of California, Berkeley, California

ABSTRACT

The effect of surface forces on the stability of edge dislocation loops, $b = \frac{1}{2} [101]$, in MgO thin foils has been studied. The circular loops lying within a critical distance from the surfaces slip out without appreciable rotation. The critical distance in $\{100\}$ foils, where the glide cylinders are at 45° to the foil surfaces, was found to increase with increasing loop size.

INTRODUCTION

When dislocation loops are near free surfaces, they experience a force which can remove them from the crystal or change their shape and orientation. Theoretical estimates of these forces, for prismatic dislocation loops on a glide prism of square cross section using linear elasticity theory, were obtained by Groves and Bacon^{1,2} for a semi-infinite isotropic elastic medium. The purpose of this paper is to present experimental observations of the effect of surface forces on circular prismatic dislocation loops. A technique for annealing thin foils of MgO at high temperatures ($> 1000^{\circ}\text{C}$) and repeatedly photographing the same area under identical diffraction conditions, was developed. Stereo microscopy was done at each step to permit measurement of the depth below the surface of each loop. The stability of dislocation loops of various sizes was studied as a function of their initial distance from the surface. Dirt particles on the surfaces were used as reference for the measurements. MgO is an ideal material for this kind of study because even at high temperatures ($> 1000^{\circ}\text{C}$), the friction stress opposing dislocation motion remains quite high. Therefore slow displacements of many loops can be observed.

EXPERIMENTAL

Large grained polycrystalline MgO was purchased from Muscle Shoals Electro-Chemical Corp., Tuscumbia, Alabama. Semi-quantitative analysis revealed the following impurities (in PPM):

Al-200, Si-200, Fe-30

all others elements were undetected.

Single crystal specimens in the form of thin sheets ($\sim 0.50 - 0.25$ mm thick) were obtained by cleaving along {100} planes. The dislocation loops were introduced by plastic deformation and subsequent annealing.³ Electron microscope foils were obtained by a jet polishing technique.⁴ All the foils were examined in a Siemens 100 kV electron microscope. The same diffraction conditions were used during a series of observations. Most of the stereomicroscopy was done along the 200 Kikuchi band and around the 001 pole. Objective lens current was measured at each step of the picture making, keeping intermediate and projector lens currents fixed. The microscope was calibrated in small intervals of objective lens currents for the same projector and intermediate lens currents.

All the quantitative measurements were made directly from the electron micrograph plates. Electron micrographs were observed in a Nikon enlarger at $20\times$ to make measurements. The accuracy of depth measurements from stereo pairs was estimated to be $\pm 15\text{\AA}$.

RESULTS AND DISCUSSION

Figs. 1, 2 and 3 are sets of pictures of the same areas of thin foils after different annealing treatments done outside the electron microscope. The depths of dislocation loops of various sizes was determined from stereo pairs (see Fig. 4) and their stability studied during the series of annealing treatments in the temperature range 1100 to 1250°C. The circular edge dislocation loops (1, 2, 3, 4, 5 and 7 in Fig. 1; 1 and 2 in Fig. 2) with $b = \frac{1}{2} [101]$ which are near one of the surfaces slip out during the annealing series without appreciable rotation. Figure 4 is the plot of initial depth of dislocation loops in the foil against their diameters. The dislocation loops a, b, c, d, e, f, g, h, i, j, k, l, m and p in Fig. 1 and loops 2, 3 and 4 in Fig. 3 with $b = \frac{1}{2} [101]$ do not move during the annealing sequence because of surface forces due to one of the surfaces of the foil.

In an infinite body, the circular edge dislocation loops have minimum self energy at the pure edge orientation⁵ unlike square and rhombus shaped loops where the energy minima are displaced from pure edge orientation.² In a semi-infinite case (near a free surface) square and rhombus shaped dislocation loops are better stabilized from pure edge orientation.¹ However for circular edge dislocation loops, the present observations suggest that loop self energy predominates over surface interaction forces and keeps the loops circular very close to free surfaces. However, there is some rotation during the motion of rectangular edge dislocation loops with $b = \frac{1}{2} [101]$ (see loop 1, Fig. 3).

To get an approximate depth of stability for circular dislocation loops of various sizes, the following Grove and Bacon's expression for prismatic dislocation loop on glide prism of square cross section is used. For a loop with Burgers vector perpendicular to the surface, the force per unit length acting towards the surface at the mid point of each side is given by:¹

$$F^T = \frac{\mu b^2 y}{4\pi(1-\nu)h} \left\{ \frac{(y^2 + 40y + 768)}{(y^2 + 16)^{5/2}} + \frac{16(105y^3 + 460y^2 - 544y - 3072)}{(y + 4)^3 (5y + 16)^{5/2}} \right\}, \quad (1)$$

where $y = h^2/a^2$. 'a' is the distance from the surface, and 'h' is the length of one side of the square loop and μ is the shear modulus. F^T is always positive and the dislocation loop is therefore only stable when F^T is less than frictional force opposing dislocation motion. For the dislocation loops which are away from the plane of the foil, the segment nearest the surface will experience a stress stronger than the friction stress and possibly drag the rest of the loop along with it. Therefore 'a' is the distance of the nearest segment of the loop from the surface. Equation (1) is true for the surface of a semi-infinite medium, i.e., for a thin foil, if the effect of the other surface is negligible. In thin foils, the effect of the other surface might be important depending upon the thickness of the foil. Groves and Bacon¹ estimated that a surface exerts a significant influence only if 'a' is less than about two times 'h'. Thus a loop which is farther than this distance from one or both surfaces of the foil, can reasonably be treated as being in a

semi-infinite or an infinite body respectively. If the thickness of the foil is less than about $5h$ then the effect of the second surface is not negligible. Due to attraction from the other surface, the calculated 'a' for the stability of the loop from Eq. (1) would be an overestimate.

The curve with squares in Fig. 4 represents the calculated depth for retention taking into account the variation of friction stress with the size of the loop.³ The curve with triangles is for an assumed constant value of friction stress $10^{-4} \mu$. The dislocation loops on the left side of the curve should be stable in the foil and those on the right side should be unstable and tend to slip out of the foil. The edge dislocation loops (1, 2, 3, 4, 5 and 7 in Fig. 1; 1 and 2 in Fig. 2) with $b = \frac{1}{2} [101]$ which slipped out of the foil after various annealing treatments are close to or on the right side of the curve (see Fig. 4). The dislocation loops 2, 4 and 5 lie close to the right side of the curve but still slip out of the foil. This might be because of uncertainty in friction stress and/or approximation of circular dislocation loops by squares. The dislocation loops a, b, c, d, e, f, g, h, i, j, k, l, m and p in Fig. 1 and loops 2, 3 and 4 in Fig. 3 with $b = \frac{1}{2} [101]$ lie on the left side of the stability curve (see Fig. 4) showing that loops are stable in the foil. From Fig. 4, the dislocation loop of diameter about $3500\text{\AA}$ or greater should be unstable in the foil of thickness about 0.6μ or less. This is true as the dislocation loop 1 in Fig. 3 (diameter $\sim 3500\text{\AA}$) is at the center of the foil but slips out of the foil ($\sim 0.6 \mu$ thick).

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FIGURE CAPTIONS

Fig. 1. Set of pictures of the same area after various annealing

treatments: A → B, 4502 secs at 1100°C
 B → C, 3257 secs at 1200°C
 C → D, 2925 secs at 1237°C
 D → E, 3880 secs at 1200°C
 E → F, 2720 secs at 1200°C

Dislocation loops 1, 2, 3, 4, 5 and 7 are unstable in the foil and slip out without appreciable rotation from pure edge orientation. The dislocation loops a, b, c, d, e, f, g, h, i, j, k, l, m and p are stable in the foil.

Fig. 2. The same area of another foil after various annealing

treatments: A → B, 44 min 50 secs at 1100°C;
 B → C, 54 min 17 secs at 1200°C;
 C → D, 38 min 45 secs at 1237°C;
 D → E, 64 min 40 secs at 1200°C.

Stereo pair of B is also given.

The dislocation loops 1 and 2 slip without appreciable rotation from edge orientation. Note both dislocation loops lie on right side of the stability curve. (Fig. 4).

Fig. 3. The same area of the foil after a series of annealing

treatments:

- A → B, 20 min 2 secs at 1250°C;
- B → C, 19 min 29 secs at 1250°C;
- C → D, 49 min 15 secs at 1100°C;
- D → E, 45 min 12 secs at 1100°C;
- E → F, 44 min 50 secs at 1100°C;

The dislocation loop 1 (diameter ~ 3500Å) is unstable anywhere in the foil. Note the rotation of the loop. The dislocation loops 2, 3 and 4 lie on the right side of the stability curve (see Fig. 4) and are stable.

Fig. 4. Plot of depth of stability against diameter of the dislocation loop. The loops lying on the left side of the curve are stable and those on the right side are unstable and slip out of the foil.

XBB 723-1587

Fig. 1.

XBB 711-5551

Fig. 2.

XBB 722-899

Fig. 3.

XBL723-6100

Fig. 4.

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