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Publication Date

1972

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January 1972

AEC Contract No. W-7405-eng-48

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DIRECT OBSERVATION OF PIPE DIFFUSION IN MAGNESIUM OXIDE

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Direct observations have been made in MgO thin foils on the self climb of prismatic dislocation loops, $b = \frac{1}{2}\langle 101 \rangle$, due to pipe diffusion along the edge dislocations comprising the loops. Pipe diffusion along a screw dislocation has been observed in a configuration, where a prismatic edge dislocation loop was connected to both surfaces of a thin foil by a screw dislocation of the same b-vector, $\frac{1}{2}\langle 101 \rangle$.

Thin foils of MgO (impurities in PPM, Al-200, Si-200, Fe-30) for transmission electron microscopy were prepared by a method similar to that of Washburn et al. (1). An atmosphere controlled furnace was used to anneal the thin foils outside the electron microscope without contamination. The same area was photographed repeatedly under identical diffraction conditions in a Siemens 100 kV electron microscope. Stereomicroscopy was done at each step of picture-making to locate the dislocation loops precisely in the foil.

In Fig. 1, from 1A - 1B the annealing treatment was 20 minutes at 1126°C. Figure 2 shows a set of pictures with the following annealing treatments: 2A → 2B, 32 min. at 1194.5°C; 2B → 2C, 40 min. at 1194.5°C; 2C → 2D, 45 min. at 1194.5°C. Coplanar pairs of loops ($b = \frac{a}{2}[101]$) at 1 (Fig. 1) and at 1 and 2 (Fig. 2) gradually moved closer together and finally formed single loops of approximately conserved total area. Similar coplanar pairs at 2 (Fig. 1) and at 3 and 4 (Fig. 2) also move closer toward each other. This movement is due to mutual interaction between the two dislocation loops in a pair. The climb force varies around the periphery of the loops being maximum attraction at the closest points and repulsion at the farthest points. This variation creates a driving force for vacancies to move along the edge of the extra half planes from the far-half to the near-half in each loop of a pair. This vacancy migration results in a movement of dislocation loops toward each other. This has been called conservative climb or more appropriately self climb; the total area of the loop projected perpendicular to its Burgers vector remains unchanged. The rate of self climb was measured at a series of temperatures and the activation energy for pipe diffusion temperatures and the activation energy for pipe diffusion along edge dislocations was determined to be $60,300 \pm 3500$ cal/mole. (2). The pipe diffusion probably occurs by a vacancy exchange mechanism.

All pairs on which measurements were made were near the center of the foil as confirmed by observation of stereo-pairs. For loops which were near the center of the foil change in loop size due to loss of vacancies to the foil surface by bulk diffusion in the temperature

range of our experiments was negligible. For loops that were near the surface, shrinkage did occur. For comparison note, loops at 4 in Fig. 1 and loop 5 in Fig. 2 (both have $b = \frac{a}{2}[101]$). These loops did not shrink appreciably in spite of being some of the smallest loops in the field of view and therefore having had a large driving force for shrinkage. In contrast, loop 7 in Fig. 2 which was near one of the surfaces of the foil shrank at a much faster rate. Loops which were within about one loop diameter of a surface often slipped out of the foil by movement along the glide cylinder, see loop 8, Fig. 2. Surface interaction is of a short range nature and is important only within about one loop diameter.

Configurations such as at 2 in Fig. 1 show the shrinkage of a dislocation loop, $b = \frac{a}{2}[101]$, due to pipe diffusion along a screw dislocation, $b = \frac{a}{2}[101]$. The rate of shrinkage of dislocation loops in this configuration has been measured at three temperatures and the activation energy for pipe diffusion along screw dislocations was found to be $62,700 \pm 2000$ cal/mole (4). The shear stress for prismatic slip in LMgO at $\sim 1150^\circ\text{C}$ is of the order of 10^8 dynes/cm² (2). This relatively high value of the critical shear stress prevents glide of the dislocation loop along its glide cylinder when cut by the screw dislocation.

Acknowledgement

We acknowledge the continued support of the U.S. Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory, Berkeley, California 94720.

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Fig. 1.

Fig. 2.

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