UCRL-16713

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Berkeley, California

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Submitted to the
Journal of the American Ceramic Society

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AEC Contract No. W-7505-eng-48

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February 1966

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ABSTRACT

Observations on the formation of dislocation etch pits on {110} and {112} faces of Magnesium Oxide crystals suggested that two requirements must be met for formation of observable pits: (1) the line must not lie too closely parallel to the surface of observation (2) the Burgers vector must have a large component parallel to the surface of observation.

It is now a well established fact that etch pits can be produced at the points of emergence of dislocations at a crystal surface.(1) The classic experiments of Gilman and Johnston(2) on Li F are evidence of the power of the method when applied to the study of the properties of dislocations. Unfortunately, the mechanism of etch pit formation at dislocations is not completely understood: It may be influenced by the atomic structure of the dislocation core which is not well known. For this reason etching reagents usually are developed empirically.

In this note the results obtained with two etchants on the {110} and {112} faces of Magnesium Oxide are described. Dislocation etch pitting was of interest on these faces to facilitate observation of plastic deformation and slip band growth in specimens cut for loading along <111> axis.

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Compression specimens with <111> longitudinal axes were diamond sawed out of large single crystals, using the technique of Hulse et al.,[3] such that their longitudinal faces were {110} and {112} planes. The specimens were chemically polished in H_3PO_4 at 160°C and were subsequently deformed at temperatures greater than 500°C. Also, thin plates, having {110} faces, were diamond sawed out of previously deformed <001> loading axis specimens. The surface damage caused by the sawing was removed by chemical polishing. Two etchants were found to produce dislocation etch-pits on {110} and {112} surfaces: Etch A, 2 parts H_3PO_4, 1 part H_2SO_4; Etch B, H_3PO_4 at 105°C. Etch A also produced dislocation etch pits on {001} surfaces,[4] whereas Etch B acted as a polish for these surfaces. Etch B was very rapid, requiring only a few seconds to produce 5 micron pits; it was conveneint for quick determinations of the surface damage remaining after an increment of chemical polishing. Etch A was slow, requiring 3-5 minutes. It was preferred for controlled studies of dislocation distribution after deformation.

Etch-pit formation on {110} was found to be highly selective as to Burgers vector of the dislocation. There are six {110} <110> slip systems for the soldium chloride structure, but only three distinct orientations of the Burgers vectors relative to a {110} surface. For four of the six systems the Burgers vector makes an angle of 30° with the surface and the glide plane lies at 60° to the surface. Dislocations on these systems always produced etch-pits (see Fig. 1).

The second orientation is represented by only one system. For this case the plane lies at 90° to the {110} face and the Burgers vector is exactly normal to the surface; dislocations on this system did not produce visible

etch-pits. Proof that slip had occurred on this 90° system and was not revealed by etching is shown in Fig. 2. The unetched 90° slip bands have caused numerous small pile-ups on a 60° system. The presence of unetched 90° slip bands is revealed by birefringence.[5,6] Also, examination of the adjacent perpendicular {001} face showed rows of etch pits, belonging to the 90° system, lying in slip planes that, matched perfectly with the ends of the blocked 60° slip bands.

The third possible slip plane is closely parallel to the surface of observation. Dislocations lying in this plane also did not produce observable etch pits. Only grown-in substructure and a few 60° glide bands were seen in a deformed specimen cut parallel to the most active slip plane. Transmission electron microscopy observations on foils subsequently prepared from the same specimen, showed that the density of dislocations lying in the slip plane parallel to the faces was about 10^9 lines $/cm^2$. Because the {110} faces were obtained by diamond sawing and were only accurate to ± 1°, there should have been sufficient intersections of dislocations with the plane of observation to be noticed if effective etch-pitting had occurred.

The absence of etch pit formation at dislocations that were approximately parallel to the plane of observation may be understood from purely geometrical considerations. If the position of the dislocation surface intersection changes rapidly during etching as would be the case for a dislocation lying almost parallel to the surface, only a shallow groove might be formed along the projection of the dislocation. If the depth of the groove is small compared to the overall amount of material removed during etching, no etch pit or groove would be observed.

This geometrical effect appears to a less marked degree for etchant A when the surface of observation is {001}. Electron microscope replicas of the etch pits produced at dislocations belonging to the slip planes perpendicular to a {001} surface are sharper and more distinct than those belonging to the slip planes that lie at 45° to the surface. In the latter case, the intersection of the dislocation with the surface shifts by an amount equal to the thickness of material removed during etching, causing the pit to be elongated in the direction of the dislocation projection. These results suggest that even with other etching reagents and for other materials dislocations that lie too close to being parallel to the plane of observation may not be revealed.

The lack of etch pitting at dislocations on the 90° slip plane is less easily understood. A dislocation lying perpendicular to a {110} face and belonging to the 90° system is in pure screw orientation, and yet does not produce a visible etch pit. It appears that a second condition for etch pit formation, at least with these etchants, is that the Burgers vector have a large component in the plane of observation. A few etching experiments on {112} surfaces tended to confirm this second condition. Slip systems having only a small Burgers vector component in the plane of the surface that were known to have been active because of the etch pits observed on adjacent {110} faces, did not etch. For example, no etch pits were formed for an active {110} system for which the Burgers vector made an angle of 60° to a {112} face.

Because of the large number of variables affecting etch pit formation, the generality of the orientation requirements deduced from the present work is not known. It is clear, however, that the reliability of any etchant can only be determined by examination of several different surface orientations in the same specimen.

ACKNOWLEDGEMENT

This work was supported by the U.S. Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Radiation Laboratory.

REFERENCES

1. W. G. Johnston, "Dislocation Etch Pits in Non-Metallic Crystals", Progress in Ceramic Science, Vol. 2, pp. 1-75, edited by J. E. Burke. Pergamon Press, Inc., New York, 1962. Ceram. Abstr., 1963, August, p. 232b.

2. J. J. Gilman and W. G. Johnston, "Dislocations in Lithium Fluorids", Solid State Physics, Advances in Research and Applications, Vol. 13, pp. 147-222, edited by F. Seitz and D. Turnbull. Academic Press, New York 1962.

3. C. O. Hulse, S.M. Copley, and J. A. Pask, "Effect of Crystal Orientation on Plastic Deformation of Magnesium Oxide", J. Am. Ceram. Soc., 46 (7) 317-323 (1963).

4. S. M. Copley; private communication.

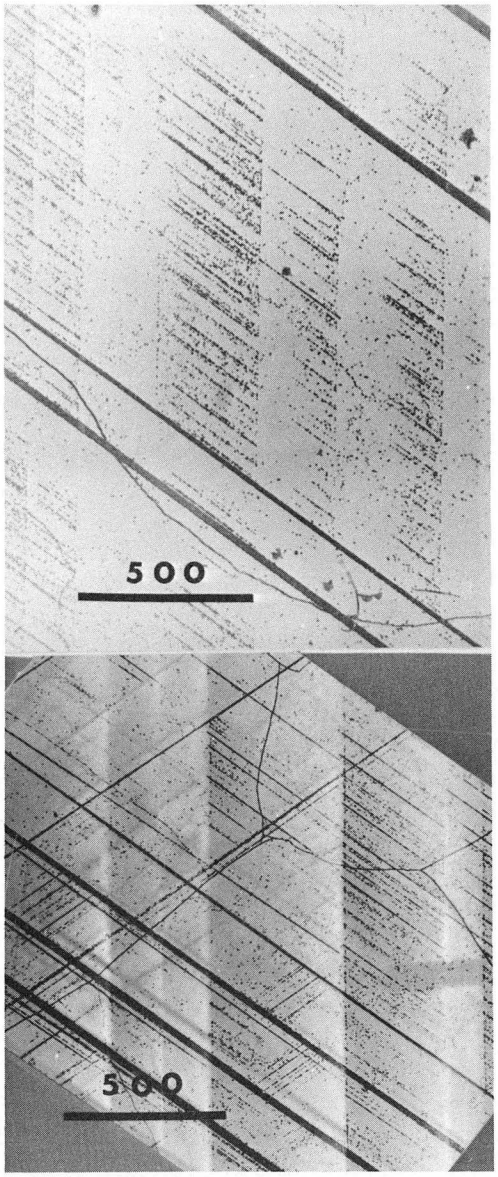
5. J. F. Nye, "Plastic Deformation of Silver Chloride", Proc. Roy. Soc., A 198, 190-204 (1949).

6. B. K. Kear and P. L. Pratt, "Quenching Stresses and Strains in Ionic Crystals", Acta Met., 6 (7) 457-461 (1958).

FIGURE CAPTIONS

Figure 1. Etch pit configuration on a {110} plane which was cut from a crystal deformed along a <001> loading axis. The vertical direction is parallel to the trace of the 90° system (the scale marker is 500 microns).

Figure 2. A thin {110} plate cut from a crystal deformed along a <001> loading axis. The birefringence shows that slip has occurred on the 90° system. The 60° systems are unfavorably oriented to produce birefringence.



ZN-5426

Figs. 1 and 2

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