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# EFFECT OF SOLUTE ATOMS ON THE MOTION OF A LOW ANGLE TILT BOUNDARY

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## Introduction

The stress-induced movement of low-angle tilt boundaries has been observed in single crystals of zinc by several investigators (1-6). The motion of the boundary under the action of a sheer stress in the direction of the Burgers vector of the dislocations comprising the boundary is well established. However the reported modes of the boundary motion are different from one investigation to another. Bainbridge et al. (3) have observed that the movement at room temperature was jerky and during each jump the boundary moved through an appreciable volume of the material while Vreeland (4) reports a smooth and continuous motion at the same temperature. One would probably attribute this discrepancy to the different levels of impurity content. Zinc crystals of 99.99 wt% purity were used in the former investigation while crystals of higher purity (99.999 wt%) and zone refined zinc were used in the latter. Thus we decided to examine this point further by introducing much higher doses of substitutional solute atoms into the crystals. We used alloy crystals containing controlled amounts of silver as an alloying element in addition to high purity zinc crystals.

## Experimental

Single crystals of high purity zinc (99.999+ wt%) and alloys containing 0.05, 0.10 and 0.21 wt% silver were grown in a graphite mold under helium atmosphere by a modified Bridgman technique at a rate of 0.4 cm/hr. The crystals were rectangular 1.3 cm × 0.7 cm and 8 cm long and were oriented in the direction $[1\bar{1}20]$ with their basal planes parallel to the wider faces. The side faces were parallel to the prismatic planes. In the case of alloy crystals, a reservoir was designed in the upper portion of the mold (7) to reduce the concentration gradient along the 8 cm length to about ±3%. The top parts were removed by spark cutting and the samples were cut to 2.5 cm length by an acid sawing technique. Then the following sequence of preparation and annealing under He atmosphere was applied: i) 1-hr. anneal at 400°C and furnace cool; ii) side surfaces were acid polished; iii) cleaved along the basal plane to reveal the natural surface of this plane; iv) 1-hr. anneal at 400°C and furnace cool; v) ∿2° angle tilt boundary (FIG. 1) introduced by bending at room temperature; vi) 1-hr. anneal at 400°C and furnace cooled to 200°C;

vii) 48-hr. aging at 200°C followed by air quench and then stored in liquid nitrogen for testing. The cleaved specimens were 2.5 cm long, ~0.8 cm wide and 0.2 to 0.4 cm thick. The width and thickness could not be controlled very closely due to the difficulty of cleaving the acid polishing.

The observation of the boundary motion was performed by using essentially the same technique and stressing apparatus as used by Bainbridge et al. (3). The displacements of the boundary were measured by reading the distances moved by the boundary line on the basal plane of the crystal surface. The boundary in each crystal was allowed to move by a certain distance and then moved backward until it just passed the original position by changing the direction of the external stress. This reversal was repeated once or twice. The motion of the boundary was followed on both sides of the crystal. In carefully prepared specimens the boundary seemed to move as a relatively flat interface, though occasional bowings were noticed.

## Results

Representative data at room and liquid nitrogen temperatures are shown in FIGS. 2(a) and 2(b), respectively. The stress for the motion in the reverse direction is plotted below the horizontal axis and the data on the left side of the vertical axis of the coordinates show the motion past the original position. Solid lines show the behavior of pure and dashed lines those of alloy crystals containing 0.21 wt% silver. It should be noted that a displacement at a constant stress level represents an instantaneous jumping distance, not a steady motion of the boundary.

The following interesting points are revealed by the data shown in FIG. 2. i) The initial movement is a rapid and long-distance jump in all four cases. ii) The boundary motion in the reverse direction becomes smooth and continuous in the pure crystals at both temperatures but the alloy crystals show this behavior at liquid nitrogen temperature only. The reverse motion in the alloy crystals is discontinuous at room temperature. In pure crystals the room temperature results of Vreeland and the liquid nitrogen temperature results of Bainbridge et al. are confirmed. iii) The boundary apparently remembers its original position in both pure and alloy crystals at liquid nitrogen temperature and becomes immobilized when it returns to this position. Further movement starts with a jumping motion at a discontinuously increased stress level. However the motion at room temperature is different and the original position does not impose an increased stress. This phenomenon was not observed in the earlier works. iv) The stress level on the reverse motion is initially sharply reduced when compared with the level before the reversal. This phenomenon which is similar to Bauschinger effect (8) was also observed in the earlier report (2). v) The stress levels for the initial jump are higher in alloy crystals than in pure crystals but a quantitative trend could not be established as a function of solute content due to the wide scatter in the data. In addition to these observations, a gradual decrease in the boundary angle was noticed in all crystals during the movement at both 77° and 300°K as reported by Bainbridge et al. (3).

## Discussion

The initial jump seems to be a breakaway motion of the boundary from impurities and solute atoms which have segregated to the boundary and have pinned the dislocations there during the prolonged annealing at 200°C. Such a breakaway motion will appear as a sharp yield point phenomenon in tensile tests.

The mobility of vacancies is strongly suppressed at liquid nitrogen temperature. Thus the impurity and solute atoms cannot diffuse away after the breakaway. When the boundary returns to its original position, it will be recaptured by the same impurity and solute atmosphere which is responsible for the initial breakaway, thus causing a breakaway jump again with a discontinuous increase in stress as shown in FIG. 2(a). In pure crystals the stress increase in such an event is much smaller than in alloy crystals. At room temperature (FIG. 2(b)) the situation is somewhat different. Immediately after the initial breakaway, most of the impurity and solute atoms will diffuse away with the aid of the excess vacancies preserved when the crystal was quenched from 200°C. Thus when the boundary returns to its original position, it will not be recaptured because the initial segregation of impurity and solute atoms has been eliminated.

The discontinuous motion in the alloy crystals at room temperature could be attributed to the diffusivity of solute atoms since such behavior is not observed in the alloy crystals at liquid nitrogen temperature and in the pure crystals at room temperature. A situation similar to the "dynamic strain aging" (9) seems to have been created around the moving boundary. A further study is necessary to determine the diffusion mechanism. The absence of such discontinuous motion in the pure crystal seems simply due to the lack of sufficient impurity atoms. Thus from the present study it seems quite clear that the jerky motion in pure crystals at room temperature observed by Bainbridge et al. is due to the higher impurity content of $\sim$100 ppm compared with that of <10 ppm in the present and Vreeland's works.

## Acknowledgement

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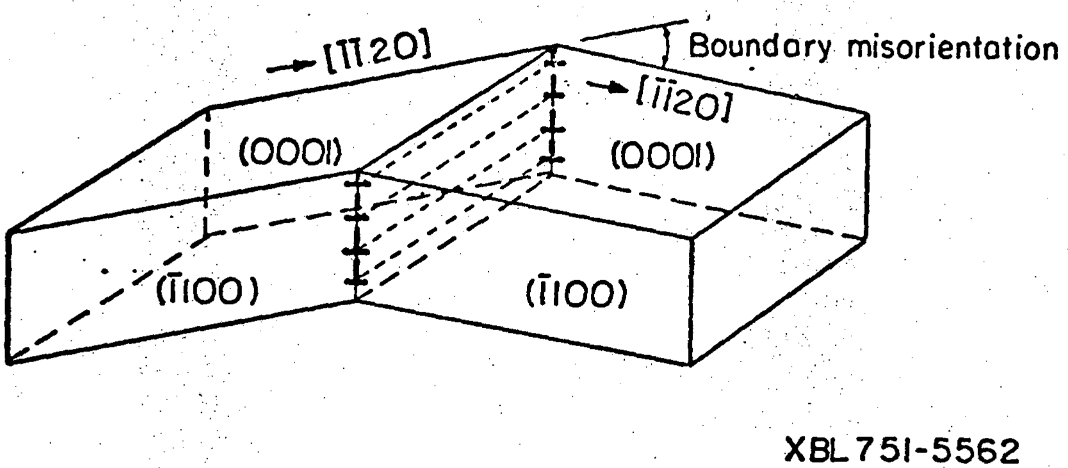


FIG. 1. Geometry of low angle tilt boundary in a hexagonal crystal.

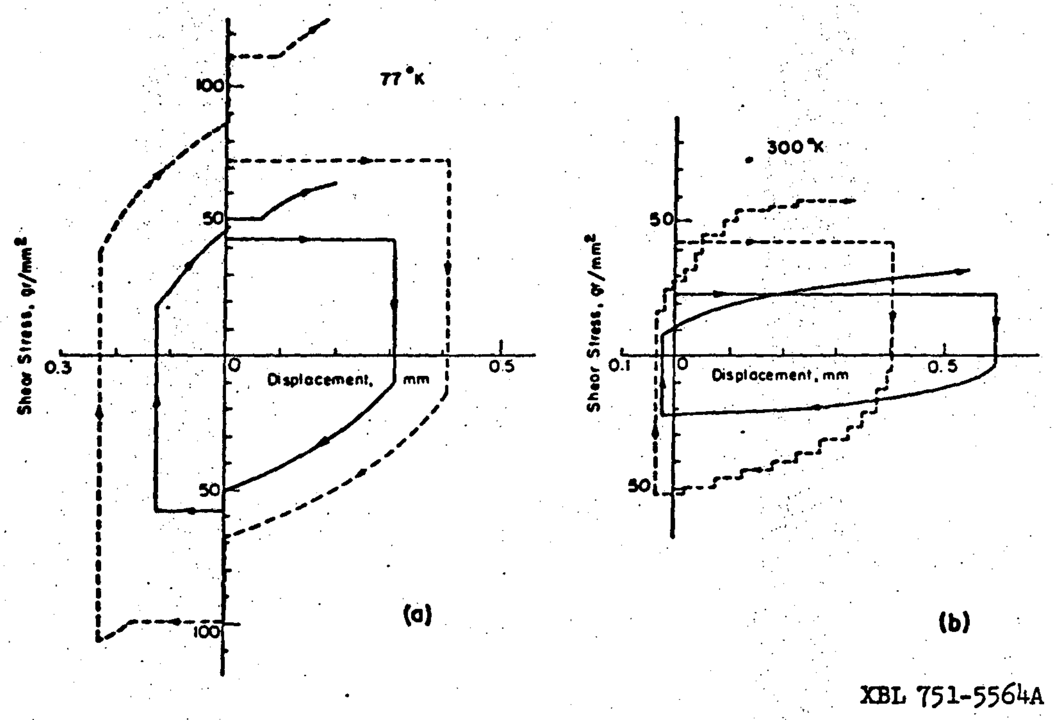


FIG. 2. Shear stress vs. displacement in pure (solid line) and alloy (dashed line) crystals at (a) liquid nitrogen and (b) room temperature.

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