servo nulling torque magnetometers.[15] Finite magnetometer compliance has recently[16] been shown to be the major contributor to nonlinear superposition of dHvA frequencies[17–20] when data is taken by the torque method, despite the major improvement of servo nulling. Shoenberg's original objections to amplitude measurements can be dealt with. The nonlinear superposition of frequencies is a more basic problem.

Isothermal magnetocaloric measurements in slowly varying fields seem particularly suited to minimizing the nonlinear effects. As the crystal is rigidly mounted for these measurements, nonlinear effects due to finite compliance through the dependence $(\partial\Gamma/\partial\theta)^{-1}$ of field orientation on torque should not be expected. Such effects would arise instead through the dependence $(\partial S/\partial T)^{-1}$ of the temperature on the entropy. The problem is thus not one of finite compliance, but of satisfying the isothermal condition. In field modulated dHvA the value of $\partial\mathbf{M}/\partial\mathbf{H}$ is the crucial quantity and should carry over into the field-modulated magnetothermal effect[10,11] through $\partial\mathbf{M}/\partial T$. Isothermal magnetocaloric oscillations in slowly varying dc fields thus appear a most likely candidate for success in absolute amplitude measurements of dHvA phenomena, though it is not clear from the theory of the nonlinear effects that any method is secure. The data reported here support an affirmative answer, but are insufficient for a conclusion.

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Twinning and Slip in Zinc by Indentation*

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Observations of twinning and slip deformation caused by indentation of zinc reveal that extensive slip on the basal and second-order pyramidal systems takes place at loads up to 5 kg. Prismatic punching through 1-cm crystals is observed at indentation loads in excess of about 2.5 kg. It is concluded that the stress at the tip of the twins cannot be obtained by use of an elastic stress analysis.

INTRODUCTION

Yoo and Wei[1] have recently reported upon an experiment in which twins are nucleated on (0001) surfaces in zinc by a point load. A stress analysis in anisotropic elasticity is used to calculate the twinning stress from the experimentally determined length of the twins and the applied load. The critical point in the interpretation of their data is the assumption that an elastic stress solution is justified. It was also assumed that the twins grow into plastically undeformed material.

This note presents a critical examination of the above-mentioned assumptions made by Yoo and Wei. The most likely forms of plastic deformation in these experiments (in addition to the twinning) are basal slip and second-order pyramidal slip which can be observed by means of Berg–Barrett x-ray diffraction topographs.[2] Second-order pyramidal slip can also be detected by chemical etch pitting.[3,4] Prismatic slip[5] can be detected by the same means, but it is more likely that pyramidal slip will occur in preference to prismatic slip.[4] Finally, prismatic punching in a soft material such as zinc might be detected by optical examination of the surfaces.

EXPERIMENTAL

We have performed indentation experiments using the same loading conditions as Yoo and Wei, but the crystal preparation was somewhat different. Zinc of 99.999% purity was grown by a modified Bridgeman

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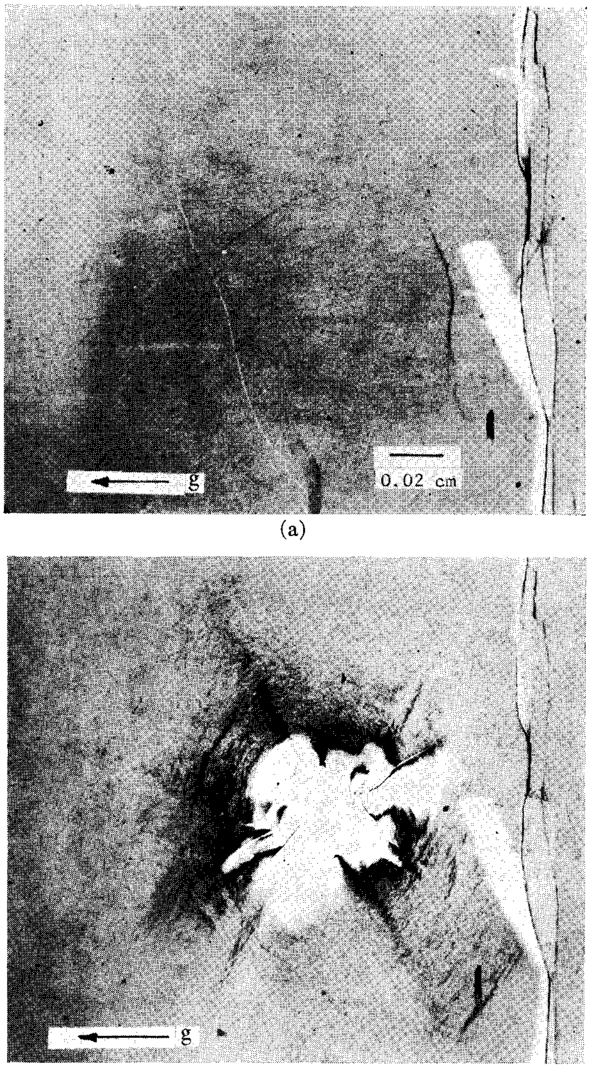
technique and the unoriented crystals were converted into oriented test specimens in the form of 1-cm cubes by acid machining operations. The test surface was prepared by cleavage in liquid nitrogen. The final step in the crystal preparation consisted of annealing in purified hydrogen at 340°C for at least 2 h. Cooling and heating rates in the cleaving and annealing processes did not exceed 5°C/min.



(a)

(b)

FIG. 1. Topograph of an (0001) cleavage surface of zinc (a) before and (b) after a 1-kg indentation load was applied, (10$\bar{1}$3) reflection with CoK_α radiation.

Berg–Barrett x-ray topographs of the cleavage surfaces were taken using a (10$\bar{1}$3) reflection and CoK_α radiation. Dislocations which lie within about 5 μ of the surface are imaged in these topographs. A topograph taken prior to indenting is shown in Fig. 1(a). The trace of the normal to the diffracting plane **g** is indicated, and **g** is perpendicular to an **a** vector in the basal plane. The vertical feature is the image of a broken cleavage chip, and the white areas are its shadows. Figure 1(b) shows the same area as Fig. 1(a) after a point load of 1 kg was imposed. The irregularly shaped white area in the center is a region where extensive plastic deformation has rotated the lattice such that the diffraction conditions are no longer satisfied. The twin orientation does not satisfy the Bragg diffraction conditions so the long narrow twins are represented by white regions whose traces are parallel to a directions. The shorter black lines are second-order pyramidal slip dislocations, whereas the longer lines further out from the indentation are basal dislocations parallel to the cleavage surfaces. Three twins are barely visible beyond the nondiffracting

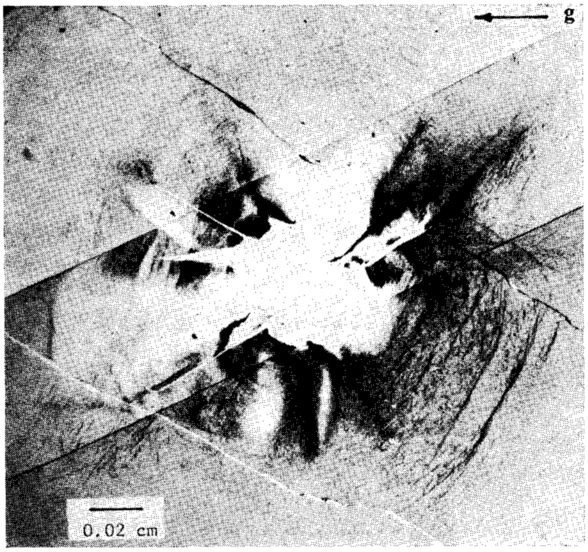


FIG. 2. Topograph of an (0001) cleavage surface of zinc after a 2-kg indentation load was applied. (10$\bar{1}$3) reflection with CoK_α radiation.

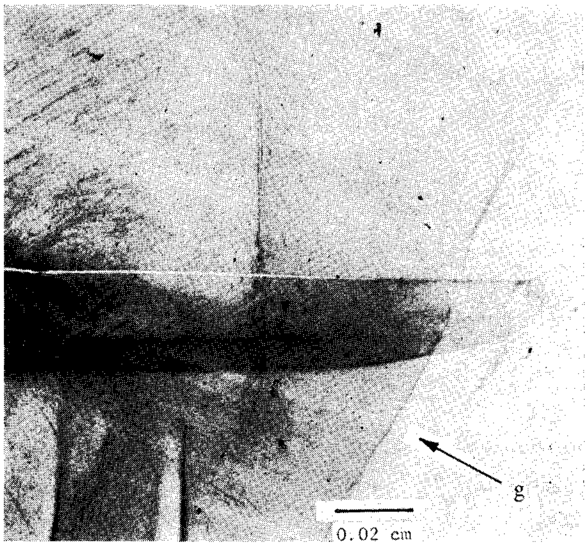


FIG. 3. Topograph of an (0001) cleavage surface of zinc showing a long twin and accommodation deformation formed by a 5-kg indentation load. (10$\bar{1}$3) reflection with CoK_α radiation.

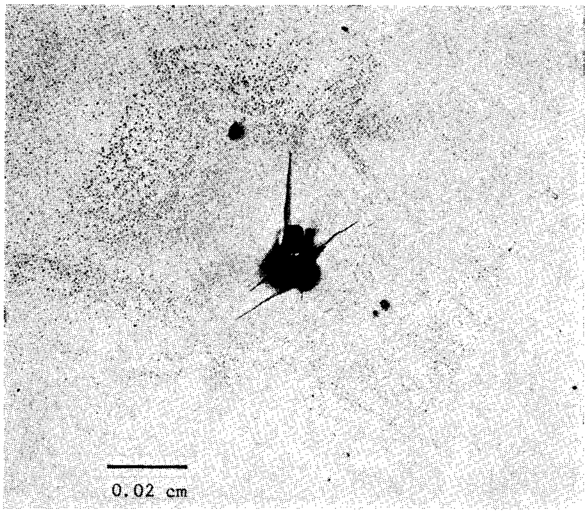


FIG. 4. Etched (10$\bar{1}$ 24) surface of zinc (5° off of the basal plane) showing slip-band activity on second-order pyramidal planes and twins produced by a 0.5-kg indentation load.

crater area, and the slip deformation clearly extends beyond the twin tips.

Figure 2 shows a topograph taken after an indentation load of 2 kg was applied. Two irregular cleavage steps (with shadows) can be seen, as well as two nearly straight small-angle tilt boundaries. Three twins extend beyond the nondiffracting crater, and again plastic deformation extends well beyond the twin tips.

Figure 3 shows a long twin produced by a 5-kg indentation load. Kink accommodation is evident on one side of the twin, and second-order pyramidal slip accommodation is evident at the twin tip. The twin growth was probably arrested at the point where its thickness changes slightly and accommodation deformation is particularly intense. The records of load vs time also indicate discontinuous twin growth, particularly at the higher loads.

A (10$\bar{1}$ 24) surface, which is only 5° from the basal plane, was indented in the [0001] with a 0.5-kg load. Figure 4 shows the surface after etching.[3] Etch pits aligned along traces of the second-order pyramidal slip systems are evident, again well beyond the twins which are revealed as narrow dark lamella.

Prismatic punching was observed on the bottom surface of the specimens directly under the indentation when the load exceeded about 2.5 kg. Twin lengths increased rapidly at the onset of prismatic punching, usually traversing the 1-cm specimen at a 5-kg load. Table I lists the radial extent of the deformation observed on x-ray topographs due to twinning, pyramidal slip, and basal slip at 1- and 2-kg indentation loads.

DISCUSSION

We find good agreement between our data on twin length vs load and that of Yoo and Wei for loads of 1 and 2 kg. The prismatic punching and rapidly increasing twin lengths we observed at loads of 3 to 5 kg may indicate that our crystals were softer than those used by Yoo and Wei.

TABLE I. Radial extent of deformation (mm).

Load (kg)	Twinning	Second-order pyramidal slip	Basal slip
1	0.6	1.0	1.6
2	0.7	1.6	2.8

Yoo and Wei did not check the assumption of negligible slip deformation in their experiments by use of etch pitting or x-ray topography. In view of the extensive plastic deformation observed in our experiments, we believe that their specimens, although somewhat harder, must have deformed by both basal and second-order pyramidal slip in the vicinity of the twins. Such deformation introduces an error in the twinning stresses that they reported.

Use of an elastic stress analysis underestimates the twinning stress when plastic deformation is present. This can be shown by consideration of a somewhat simpler problem for which a closed-form solution exists. The stress distribution in an *isotropically-elastic–perfectly-plastic* body of infinite extent containing a cylindrical, pressurized cavity was compared to that in a similar, *isotropic elastic* body with the same pressure.[6] It was assumed that the plastic zone extended to five times the radius of the cavity, corresponding roughly to the extent of plastic flow observed around the indenter at a 1-kg load. Resolved shear stresses differ by a factor of about 2.1 in the two cases at a radius equal to four times that of the cavity. The factor is 5.9 at the elastic–plastic boundary. These factors are upperbounds for the error since work hardening was ignored. The work hardening, which is small for basal slip and large for pyramidal slip, will reduce the error by an unknown amount.

CONCLUSIONS

It has been shown that indentation of zinc monocrystals produces extensive plastic deformation in addition to twinning. Therefore, an elastic stress analysis is not justified and a stress analysis involving anisotropy, plasticity and work hardening on several slip systems is intractable. Hence, it is not possible to draw any meaningful conclusions from an indentation experiment regarding the stress required for the growth of a twin into previously undeformed material.

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