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ETCH PITS AND SLIP BANDS IN SILICON

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A metallographic examination was made of the chemical etch pit distribution in a silicon single crystal, which had been plastically deformed by bending. The relationship of this distribution to the stress pattern in the crystal clearly shows that etch pits can be useful for examining the development of slip bands as well as other deformation markings.

Plastic deformation by slip is evidenced by the appearance on the surface of the crystal of slip bands, which represent the intersection of a group of optically unresolvable slip lamellae with the boundary surface. For this reason, microscopic studies of slip band development are usually carried out on highly polished, essentially stress-free surfaces. It was recently shown,[1] however, that the effect of slip could still be observed in bent germanium crystals, which were lightly ground and etched after deformation, by the alignment of etch pits along the slip traces. It is the purpose of this bulletin to show that similar effects can be obtained in silicon, and to illustrate in some detail how chemical etch pits can be used to examine the appearance and development of slip bands, as well as other deformation markings.

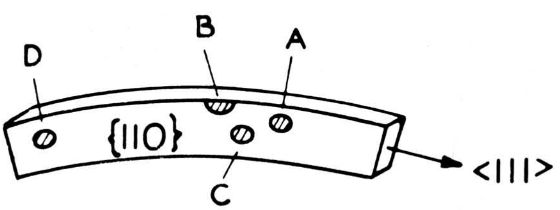

Fig. 1 – Schemactic representation of bent crystal showing areas where photomicrographs of slip bands were taken.

To illustrate this technique a 30 ohm-cm, p-type single-crystal bar, 2 x 1/4 x 1/8 inches, was plastically deformed by bending at approximately 1200 degrees C in a vacuum of 1×10^{-5} mm Hg. The orientation of the crystal was such that its axis was coincident with a <111> direction, and the surface normal to the axis of bending was within four degrees of a {110} plane. Prior to deformation, the {110} surface was mechanically polished with No. 305 American Optical Compound, and then chemically polished with a dilute solution of hydrofluoric acid in concentrated nitric acid to remove surface stresses resulting from the mechanical working. The bending was accomplished by simply supporting the ends of the bar on a thick quartz tube, and applying the load at the middle in the form of weights placed on a water-cooled stainless steel rod, tapered at one end to a knife edge. A Wilson-type seal was used to permit vertical movement of the steel rod in the vacuum. Fig. 1 shows schemetically the bent crystal, as well as the areas on the {110} surface where photomicrographs were taken.

After deformation the crystal was ground to one-half its original thickness, and then etched for 20 hours in a solution of three parts standard CP-4 etch and five parts No. 1 etch.[2,3] The photomicrographs in Figs. 2 through 6 illustrate the appearance of slip bands, as outlined by the etch pits, in regions of the bent crystals designated A to D in Fig. 1. In examining the micrographs, one should bear in mind that the usual practice in metals of grinding and etching an as-deformed crystal surface results in the disappearance of slip bands when viewed by ordinary light microscopy.

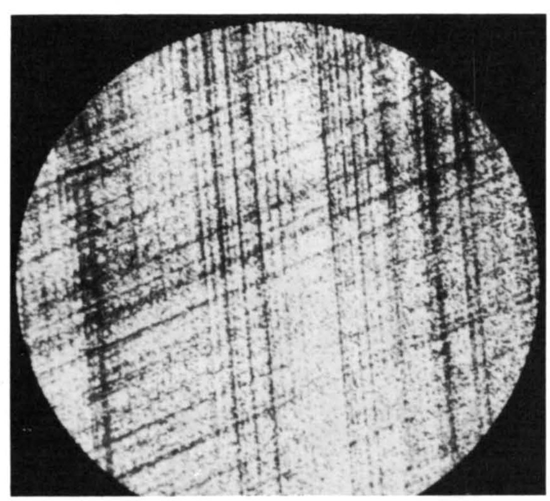

Fig. 2 – Etch pit technique showing multiple slip on {111} plane. x 30.

[1] F. L. Vogel, Jr., *Acta Metallurgica,* 3, 95 January 1955.

[2] S. G. Ellis, *Jour. Appl. Phys.* (1955) 26, 1140.

[3] LB-1023, *Etch Pits and Dislocation Studies in Silicon Crystals,* by S. M. Christian and R. V. Jensen.

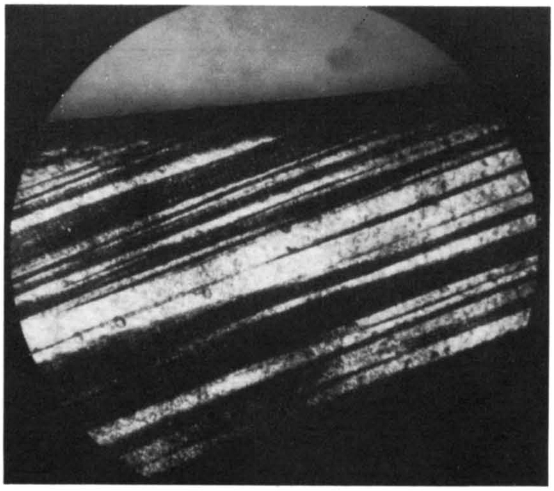

Fig. 3 – Etch pit technique showing appearance of coarse widely-spaced slip bands. x 30.

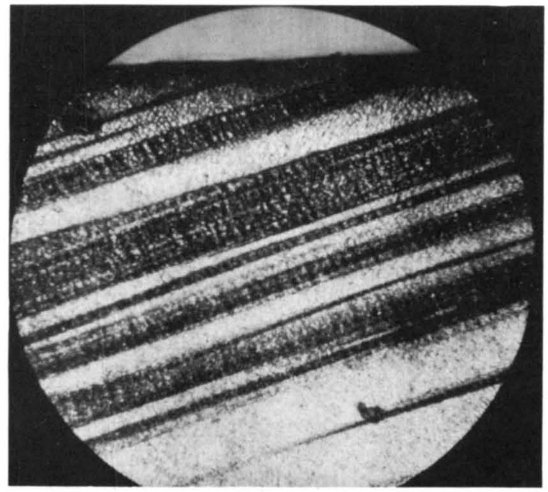

Fig. 4 – Appearance of {110} markings in coarse slip bands of Fig. 3. x 100.

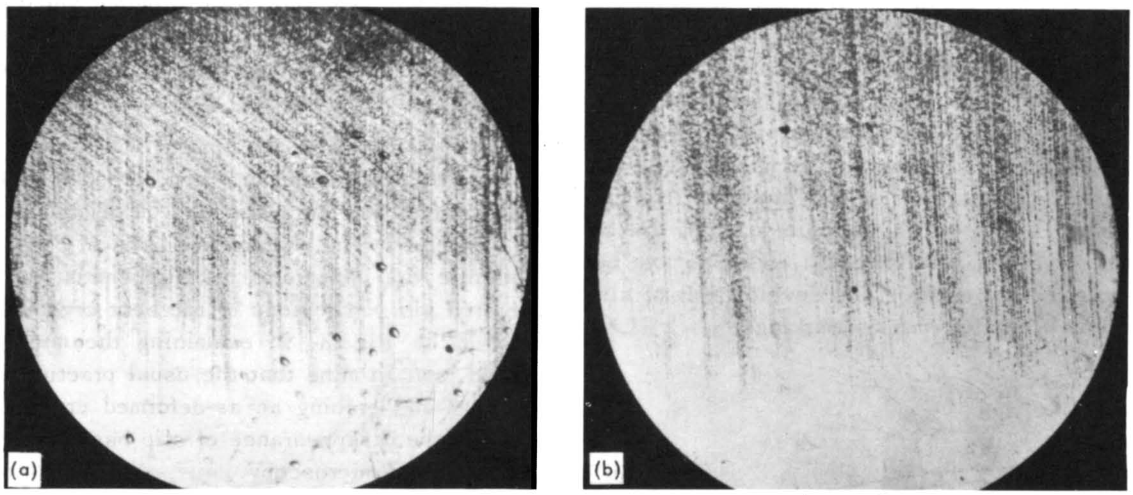

Fig. 5 – Etch pit technique showing gradual disappearance of slip bands in neutral zone of bent crystal. a, x 30; b, x 100.

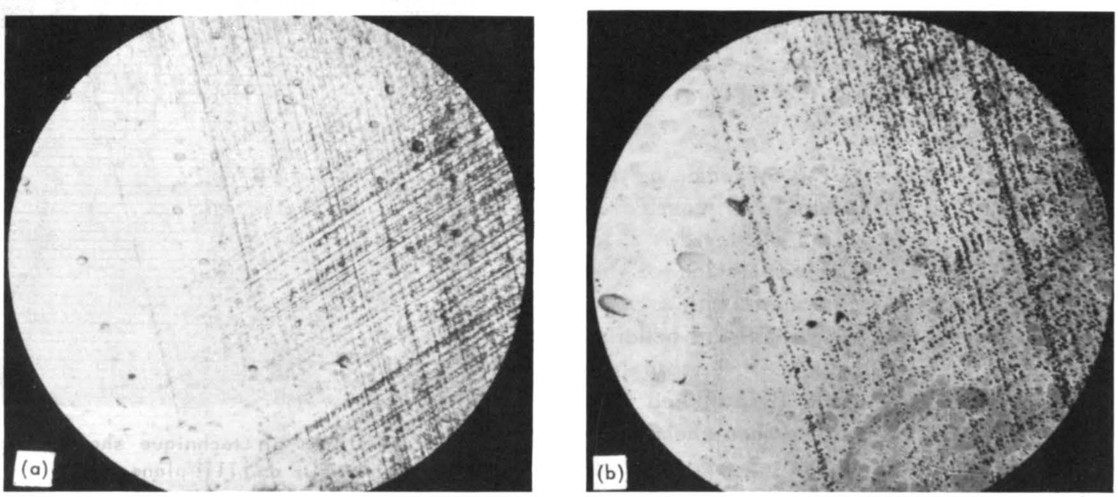

Fig. 6 – Etch pit technique showing gradual disappearance of slip bands in vicinity of supported end of crystal. a, x 30; b, x 100.

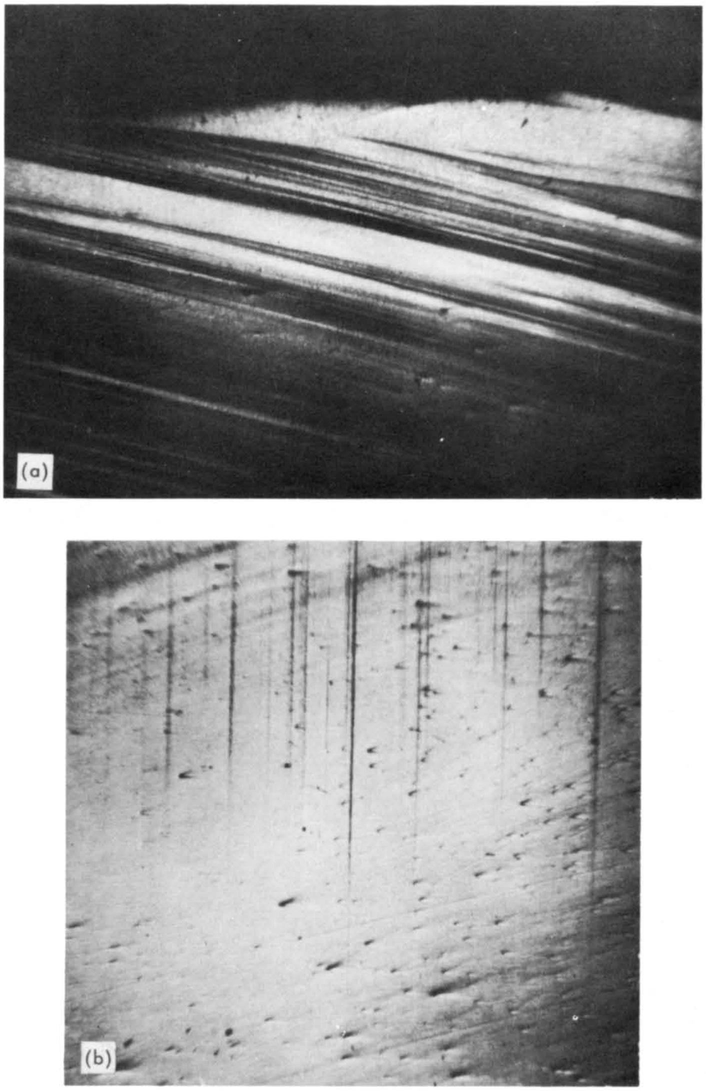

Fig. 7 – Appearance of slip bands on as-bent crystal prior to grinding and etching. x 100.

The micrograph in Fig. 2, taken at region A in Fig. 1, shows the appearance of multiple slip on two {111} planes, as might be expected from the crystal orientation. The fine, closely-spaced slip bands are well-defined by the etch pit alignment; and in some regions of slip band intersection there is an increase in the density of etch pits, as evidenced by increased blackening.

In Fig. 3 the etch pits reveal a pattern of coarse, widely-spaced slip bands in the vicinity of the convex surface (tension side) of the crystal, region B in Fig. 1. In the micrograph taken at higher magnification, Fig. 4, one can see within the coarse slip bands a high density of dark, parallel segments which make an angle of approximately 90 degrees to the slip bands. X-ray analysis showed these lines to be parallel to a {110} plane, whose normal would be the expected slip direction from the criterion of a maximum shear stress law. This crystallographic geometry suggests that the segments are fine kink bands, which might be expected to occur because of the stress inhomogeneties in a bend test.

The etch pits in the micrographs of Fig. 5 show the gradual disappearance of slip bands in region C of Fig. 1 at different magnifications. This slip behavior is to be expected, since the central region of the crystal in a bend test is under minimum stress due to the neutralization of compressive and tensile stresses. The etch pits in Fig. 6 reveal a similar slip band pattern at one end of the specimen (region D in Fig. 1), where again a sharp stress gradient can be expected.

For purposes of comparison, the micrographs in Fig. 7 show the appearance of slip bands on the same surface prior to the grinding and chemical etching operations. Fig. 7a should be compared with Figs. 3 and 4; and Fig. 7b with Fig. 5a, b. It would appear from the gross similarities in the slip patterns that the etch pit technique could be useful in the study of deformation markings.

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