# Letters to the Editor

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## Method for the Detection of Dislocations in Silicon by X-Ray Extinction Contrast

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SEVERAL experimental methods have been found for observing individual dislocations in crystals.[1–3] Most of these methods involve special etching techniques and/or procedures for decorating the dislocations with foreign atoms which in some way make the dislocations detectable. A. R. Lang has recently described a method for the examination of crystal sections using characteristic short-wavelength x-radiation from a microfocus source.[4] The purpose of this note is to show that a simple x-ray diffraction method, sometimes called the Berg-Barrett (B-B) technique,[5] is capable of showing the positions of individual undecorated screw dislocations emerging from a polished surface of a silicon crystal. This method has the advantage of being nondestructive with regard to the specimen as a whole as well as to the dislocations themselves, since no decoration is necessary. The method also has the advantage that the positions of the dislocations are found with respect to the specific topography of the specimen surface. Furthermore, since the method is direction-sensitive, the direction of the Burgers vector of the observed dislocations can be determined by proper choices of the x-ray reflections which are used to form the B-B image.

A crystal of five-pass zone-refined silicon was grown by the Czochralski technique in a quartz crucible under an atmosphere of argon gas at atmospheric pressure. The crystal was grown with its long direction parallel with [111]. A specimen having the shape of a parallelepiped measuring about 3×3×25 mm was cut from the crystal with a diamond saw. The sides of the specimen were approximately parallel with (111), ($11\bar{2}$), and ($\bar{1}10$). The specimen was then chemically polished and twisted about its long [111] axis. During this operation the sample was held at about 900°C in air for less than 5 minutes. Subsequent etching in Dash's etch (1 HF, 3 $HNO_3$, 10 glacial acetic acid) developed several straight rows of etch pits on the ($1\bar{1}0$) and ($11\bar{2}$) surfaces. These rows extended in directions parallel with the (111) face. The specimen was then cut into four similar rods with axes parallel with the long [111] axis of the original specimen. The sketch in Fig. 1(a), showing one of these rods, indicates the etch pits and the associated dislocation loops subsequently found by decorating the dislocations and examining the specimen by infrared microscopy according to the method described by Dash.[1] The specimen was sectioned through the apparent source of one of the sets of loops as indicated by the dashed line in Fig. 1(a). The new ($11\bar{2}$) surface on piece *A* was polished mechanically [Fig. 1(b)] and immersed for a few minutes in Dash's etch. This treatment revealed rows of etch pits (see Fig. 2) which were associated with those on the ($1\bar{1}0$) surface as indicated in Fig. 1(b). After the etch pits were photographed, the ($11\bar{2}$) surface was ground well below the bottoms of the etch pits and was carefully finished on a metallographic lap. An x-ray diffraction micrograph (or B-B photograph) was then made from this surface. The B-B image of the surface was formed by the $02\bar{2}$ reflection of Cr $K_\alpha$ radiation. With the specimen 13 cm from the target of a standard CA-7 x-ray tube operated at 40 kv and 20 ma, the exposure times were of the order of 40 minutes. The image was recorded

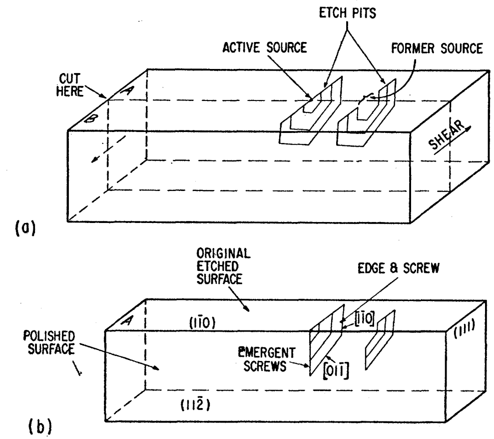


FIG. 1. This specimen was cut from a larger rod which had been twisted about the long [111] axis. Dislocation loops have spread from the surface sources as indicated.

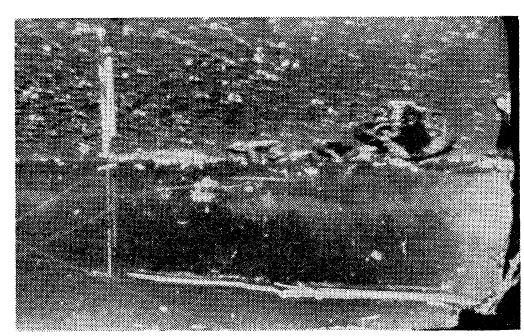

FIG. 2. Etching revealed two rows of pits on the previously polished ($11\bar{2}$) surface. The rows correspond with the two rows on the original ($1\bar{1}0$) surface. Magnification 20×.

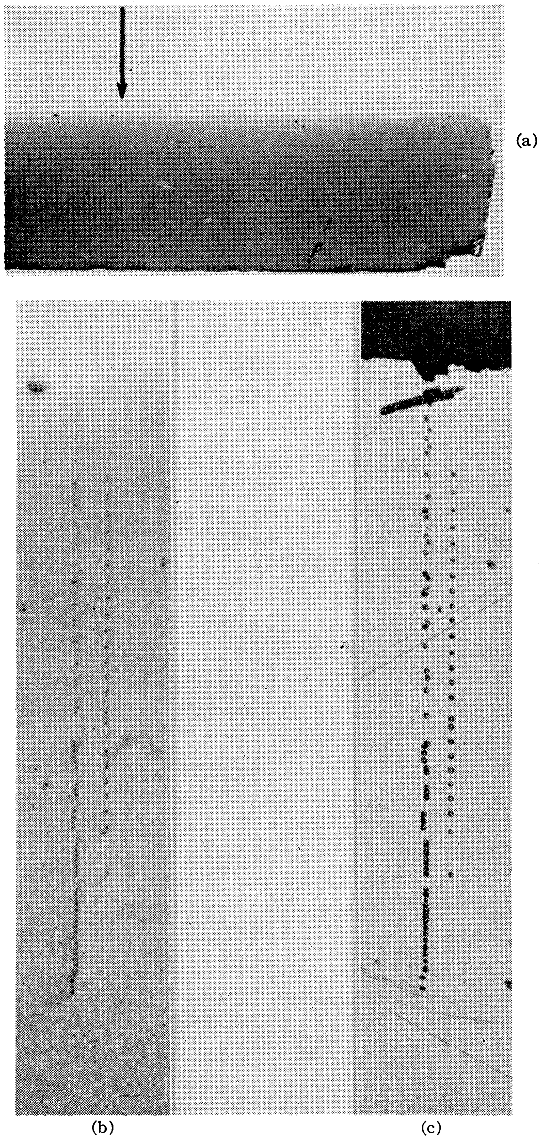

FIG. 3. A B-B photograph of the repolished $(11\bar{2})$ surface shows two rows of spots which correspond with pits on the formerly etched surface. (a) B-B image optically magnified twenty times; (b) B-B image optically magnified 115 times; (c) Light micrograph of the etched $(11\bar{2})$ surface corresponding to the area in Fig. 3(b). Magnification 100×.

on a high resolution spectrographic plate. The B-B photograph is shown at two magnifications in Fig. 3(a), (b). A light micrograph of the etched area corresponding to Fig. 3(b) is given in Fig. 3(c). The correspondence of B-B spots and etch pits is evident.

The extra diffracted intensity which renders visible the dislocations in the B-B image is probably due to a reduction in x-ray extinction induced by the perturbation of lattice periodicity normal to the reflection planes at dislocation sites.[6] Experimental conditions were chosen so as to maximize the effect of lattice distortion, due to the dislocations, upon the B-B image. Since the leg of the dislocation loop which emerges from the $(11\bar{2})$ surface is largely made up of screw dislocations with $[01\bar{1}]$ Burgers vectors, the loss of lattice periodicity at the dislocation sites would be expected to be high in the $[01\bar{1}]$ direction. The extinction effect is, therefore, expected to be large for the $(02\bar{2})$ reflection.

As a test for this hypothesis the specimen was mechanically polished on the $(1\bar{1}0)$ surface and a B-B photograph was made (from that surface) using the $(2\bar{2}0)$ reflection. It was found that spots, corresponding to surface pits which developed on subsequent etching, did appear on the B-B photograph but were much less distinct than those observed in the $(02\bar{2})$ reflection from the $(11\bar{2})$ surface. This result is consistent with the idea that the dislocations in the leg emerging from the $(1\bar{1}0)$ surface have a small $[2\bar{2}0]$ component of Burgers vector.

*Conclusion.*—By means of x-ray reflection microscopy, emergent screw dislocations can be seen on the polished surface of a single crystal of silicon. Since the method depends upon x-ray extinction, the best contrast is obtained when a reflection is used which has a large component of the dislocation Burgers vector normal to the reflecting planes.

The writer is grateful to W. C. Dash for supplying the specimen used in this experiment, for decorating and examining the dislocations contained in it, and for helpful discussions about dislocations in silicon. Valuable help in preparing the specimen was also given by R. F. Reihl, L. J. Keifer, and E. F. Koch.

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[5] C. S. Barrett, Trans. Am. Inst. Mining Met. Engrs. **161**, 15 (1945).
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## Variation Theorem for Excited States*

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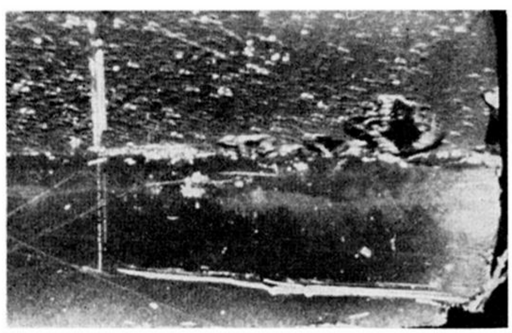
THE variation theorem[1] has proved of utmost practical importance in its application to the lowest state of each symmetry type in a given system. In addition, it has had limited applicability to excited states because of a theorem first proved by Hylleraas and Undheim[2] and later by MacDonald,[3] that if the roots of the secular equation, $J_i$, are ordered such that $J_0 \leqslant J_1 \leqslant \cdots \leqslant J_n$, then each such root is an upper bound to the corresponding exact eigenvalue: $J_i \geqslant E_i$. Eckart[1] has analyzed the mean square deviation of the lowest eigenvector $\phi_0$ from the true eigenfunction, $\psi_0$,



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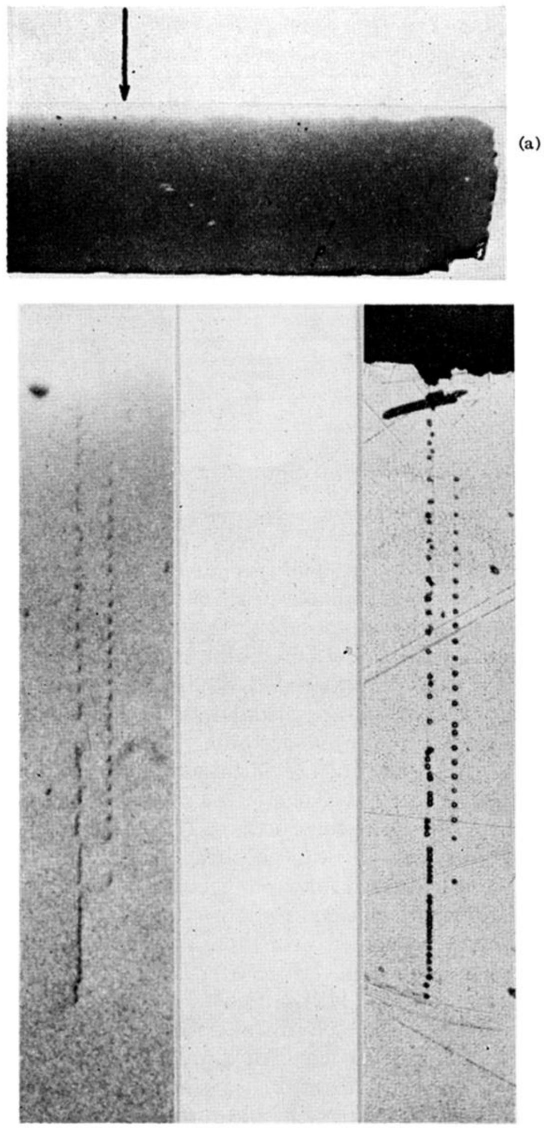


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