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Crystallography

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Abstract

Full Text

Crystallography

V. L. Indenbom, V. I. Nikitenko, and L. S. Milevskii

Observation of Internal Stresses around Dislocations in Silicon

(Presented by Academician A. V. Shubnikov, 5 VI 1961)

According to dislocation concepts, the internal stresses existing in crystals that are not subjected to external forces are caused by defects of the crystal lattice. Around each dislocation line there exists a stress field determined by the shape of the line and by the Burgers vector of the dislocation. Depending on the mutual arrangement of the dislocations, the stresses they produce may either compensate one another and decrease over distances of the order of the distance between dislocations (microstresses), or add together, forming long-range stresses (macrostresses) that decrease over distances of the order of the dimensions of the crystal.

The most promising objects for a direct test of the dislocation theory of internal stresses are transparent crystals, which permit the use of the polarization-optical method for investigating stresses. In a series of studies carried out on ionic crystals, a correspondence had already been noted between the stress fields and the dislocation structure of the crystal as revealed by the method of selective etching. Measurement of the macroscopic stresses bordering slip lines in corundum made it possible to establish that for each etch figure, presumably corresponding to the end of an atomic dislocation, there is indeed a displacement approximately equal to the lattice parameter in the direction of slip ⁽¹⁾. A qualitative correspondence between stress bands and slip bands was also observed by a number of authors ^(2, 3). In work ⁽⁴⁾, the microstructure of stresses in slip bands was revealed, corresponding to the density of dislocations and apparently connected with those very microstresses that figure in the dislocation scheme of a slip band.

The high rigidity and large photoelastic constant required for revealing atomic dislocations ⁽⁴⁾ are fortunately combined in semiconductor crystals transparent in infrared light. Photographing silicon between crossed nicols on plates sensitized to infrared radiation, Bond and Andrus ⁽⁵⁾ obtained—apparently by chance—a photograph which, in their opinion, corresponded to stresses around an atomic dislocation. This assertion, however, was called into question by a number of authors ^(6, 7), who believed that the photograph ⁽⁵⁾ more likely corresponded to a macroscopic dislocation. In particular, Bullough ⁽⁷⁾ noted a discrepancy between the calculated and actual intensity of the birefringence. It should also be noted that the rosette form of birefringence around an edge

Fig. 1

Figure 1: Fig. 1

Fig. 2

Figure 2: Fig. 2

dislocation calculated by Bond and Andrus does not agree with the results of works (7, 8).

Over the past several years, despite the progress of various methods for revealing dislocations in silicon, the question of the possibility of observing the stress field around dislocations has remained open. In the present article the possibilities of a direct polarization-optical field...

Fig. 1. Longitudinal section of an ingot. Dislocations (decorated with copper) are parallel to the growth axis [110]. 80×

Fig. 2. Revealing dislocations by various methods: *a*—etch pits, *b*—stresses around dislocations, *c*—decorated dislocations, *d*—stresses around decorated dislocations. 45×

Fig. 3. Birefringence rosette at $\alpha = 45^\circ$. The slip plane of the dislocation is horizontal. *a*—calculated rosette, *b*—one of the rosettes of Fig. 2*b*. 220×

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...microscopic investigation of undecorated and decorated dislocations in silicon by means of an electro-optical transducer.

The setup was an ordinary polarization microscope (with nicols), supplemented by an electro-optical transducer of the VEI-3 type. An OI-24 illuminator with an infrared filter served as the light source. In order to ensure the required orientation of the dislocations (the dislocations must be strictly parallel to the direction of observation), special specimens were grown. The growth direction was the close-packed direction [110]. As a result of geometrical selection, the seed dislocations were inherited mainly as dislocations lying along the growth direction [110] (9). The rectilinearity of the dislocations is clearly visible in a control specimen cut parallel to the growth axis and decorated with copper (Fig. 1).

The specimens studied were plates 2-3 mm thick, cut perpendicular to the growth axis (i.e., perpendicular to the dislocations) and carefully polished. In all sections practically the same picture was observed (minor variations, as was

Fig. 3

Figure 3: Fig. 3

established, proved to be associated with dislocations located at an angle to the growth axis).

As was shown in Ref. (8), the calculated birefringence field around a dislocation with edge or mixed orientation, in the approximation of an isotropic crystal, can be described by a rosette of equal intensity

$$r = C \cos \theta \cos 2(\theta - \alpha), \quad (1)$$

where θ is the azimuth measured from the slip plane; α is the angle between this plane and the plane of the polarizer (or analyzer); C is a constant proportional to the edge component of the Burgers vector of the dislocation, the rigidity of the crystal, and the photoelastic constant. The neutral line ($\theta = \pi/2$) is perpendicular to the slip plane of the dislocation; the isoclines ($\theta = \alpha \pm \pi/4$) form a cross diagonal with respect to the polarizer and analyzer.

The distribution of dislocations in the ingot can be judged from Fig. 2a, which shows a specimen in which the dislocations were revealed by an acid etchant (a mixture of HNO_3 , HF, and CH_3COOH). In Fig. 2b a neighboring specimen is photographed between crossed nicols. The planes of polarization make an angle $\alpha = 45^\circ$ with the slip plane of the dislocations. According to equation (1), the birefringence rosettes should be described by the formula $r = C \cos \theta \sin 2\theta$, illustrated in Fig. 3a (black and white regions correspond to birefringence of opposite signs). As is seen from a comparison of Figs. 2a and 2b, around each dislocation there is indeed observed a birefringence rosette similar to Fig. 3a (see Fig. 3b). The black-and-white "coloration" of the rosette petals is associated with the superposition of the microstresses caused by the given dislocation and the total stress field caused by the other dislocations. In those parts of the specimen where the macrostresses were canceled, four equally brightened petals were observed around each dislocation.

Figure 2c shows dislocations in the neighboring (on the other side) section, revealed by copper decoration. The dislocations (visible as dark spots) are arranged exactly as in Figs. 2a and 2b. Around the dislocations light regions are visible, corresponding to a reduced copper concentration (from these regions the copper has been drawn to the dislocations). The volume concentration of copper is especially large in dislocation-free regions of the crystal (see the upper left corner of the photograph).

After decoration, the picture of microstresses around the dislocations is completely distorted (Fig. 2d). The cross of isoclines rotates by 45° and is arranged parallel to the vibration planes of the analyzer and polarizer; the neutral band disappears; the signs of birefringence in the rosette petals symmetric (with respect to the dislocation) are no longer...

opposite, but the same (the sign of birefringence, as in Fig. 2b, is determined from the distortion of the rosette upon the application of macroscopic stresses). The signs of the microstresses around decorated dislocations correspond to radial

compression and tangential tension. The particles of the new phase precipitated on the dislocations seem to wedge the crystal apart. It is not clear to what extent the observed effect should be attributed to the difference between the expansion coefficients of silicon and of the decorating phase, and to what extent to the change in volume upon precipitation of the new phase.

With ordinary decoration, the intensity of the microstresses around dislocations increases so much that not only rectilinear but also curvilinear dislocations are readily detected. At the same time, the original microstresses caused by the dislocations themselves are completely lost. Only the macrostresses associated with the collective action of many dislocations are preserved (the long-range field of the dislocations cannot be removed by decoration). As the amount of impurity introduced into the crystal is decreased, the redistribution of stresses around the dislocations naturally diminishes. It is of interest, however, to attempt (at least by extrapolation) to investigate how the change in the elastic fields under the action of the impurity deposited on a dislocation affects the deformation of the energy bands of the semiconductor and reduces the effectiveness of dislocations in the recombination of current carriers.

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