Acta Cryst. (1954). **7**, 459

A boundary effect on the intensity of X-rays reflected from a quartz plate.

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(*Received* 25 *March* 1953 *and in revised form* 8 *February* 1954)

It is well known that the intensity of X-rays reflected from a crystal varies greatly with surface treatment (e.g. Sakisaka, 1927; Evans, Hirsch & Kellar, 1948; Gay, Hirsch & Kellar, 1952). The present note contains observations of an anomalous enhancement of the intensity of reflexion, in transmission, at the boundary of the regions of different surface treatments.

Thin X-, Y- and Z-cut plates of various thicknesses were prepared from Brazilian quartz free from twins and faults. The uniformity of the plates was very carefully examined by measuring the intensity of reflexion in transmission at every part with a Bragg spectrometer. Using the plates of different cut and different thickness, the intensity of transmitted reflexions from various net planes was measured. As the effect was found to be almost the same in every case, only the results obtained from a Z-cut plate are reported here.

1. A quartz (0001) plate 0·98 mm. thick was carefully ground on both surfaces with carborundum FFF. One half of one surface (CG of Fig. 1) was covered with paraffin wax, the boundary being parallel to $(10\bar{1}0)$. The specimen was then immersed in 45% hydrofluoric acid for 15 min. to etch the uncoated surfaces. The change in the thickness of the plate caused by the etching was very small, averaging 0·012 mm., which was within the average fluctuation of the thickness of the plate.

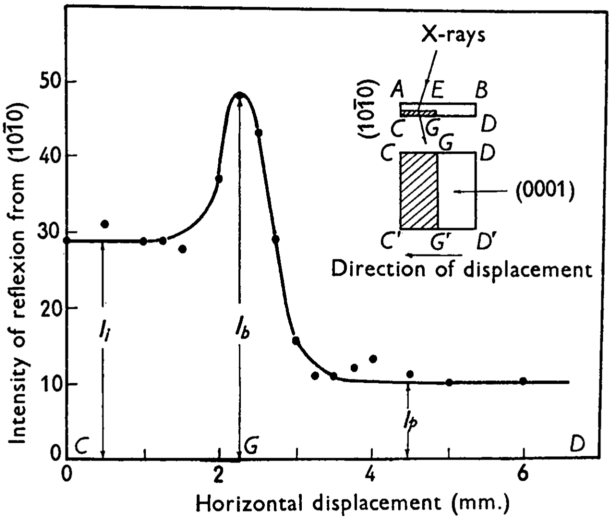

Fig. 1. The intensity of reflexion from net planes parallel to a boundary between etched and ground parts, showing the anomaly at the boundary. I_i: the intensity from the ground part; I_p: that from the etched part; I_b: that from the boundary.

A beam of Mo $K\alpha$ radiation was made to fall upon the plate as shown in Fig. 1, the collimation slits being 0·6 mm. wide and 10 mm. high. The plate was moved horizontally in small increments and at every position the intensity of reflexion from the $(10\bar{1}0)$ plane was measured. In Fig. 1 the observed intensities are plotted against the positions of etched and unetched areas. As was expected, the reflexion I_i from the unetched, i.e. ground portion, was always about three times as strong as I_p from the etched area. However, there is an extraordinarily great enhancement of the intensity I_b of reflexion from the neighbourhood of the boundary. The maximum occurs at the position of the boundary.

2. Similar measurements were carried out with the three other settings of the ground surfaces of the specimen relative to the incident beam, i.e. in positions of GD, AE and EB of Fig. 1 respectively, and similar results were obtained.

3. The variation of such boundary effect with different orders of reflexion was next examined. This is shown in Fig. 2. The curves for the first and the second orders show clearly the boundary effect. For the third order the difference between I_i and I_p was not found, while the existence of an enhanced maximum was hardly discernible.

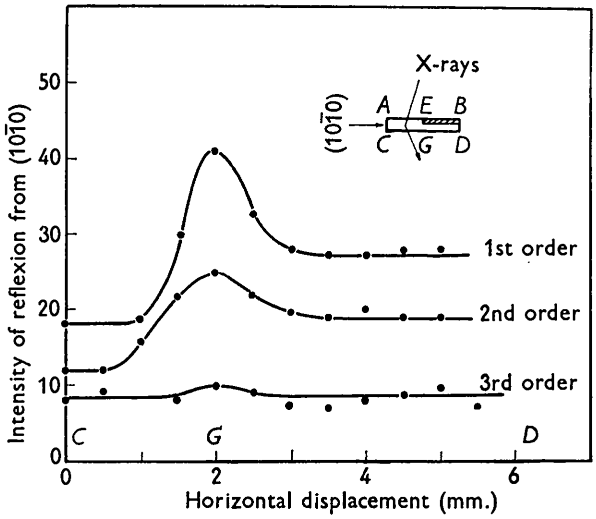

Fig. 2. The anomalous intensity from the boundary is shown in relation to the order of reflexion. The anomaly was clearly observed in the first- and second-order reflexions, but was scarcely detectable in the third order.

4. Using a quartz plate already ground uniformly on both surfaces with carborundum FFF and then reground on one half surface with rougher carborundum 80, an experiment was made in the same manner as above. The intensity of the $(10\bar{1}0)$ reflexion through the reground portion was 38 in an arbitrary scale, while that through the other half was 30. At the boundary, however, the anomaly of intensity was not observed.

5. With the specimen used in the experiment (1), but set in this case so that the boundary was horizontal, the intensity of the Laue reflexion from $(1\bar{2}10)$ was measured

at every position of the plate displaced vertically, the collimator slits being 1 mm. wide and 3 mm. high. The result is given in Fig. 3. While the difference in

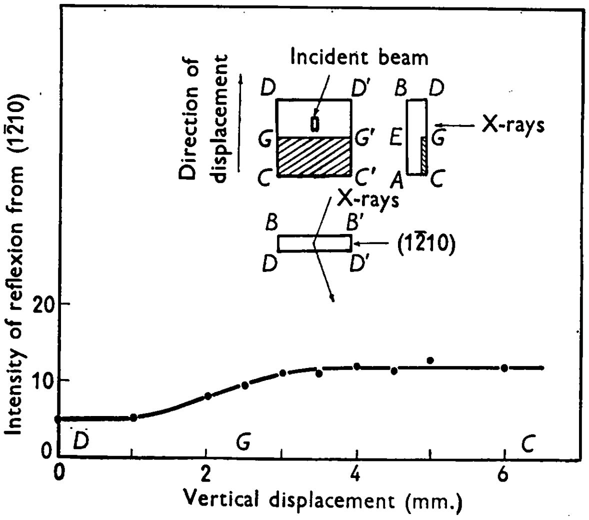

Fig. 3. The intensity of reflexion from net planes perpendicular to the boundary, showing no intensity anomaly at the boundary.

intensity of reflexions from the etched and unetched areas is shown clearly, the boundary effect was not observed.

6. In the case of Bragg surface reflexion, using the various specimens mentioned at the beginning, no anomaly was found at the boundary.

The present author (Fukushima, 1935*a*, *b*, 1936) has measured the intensity of transmitted reflexion from different parts of a quartz plate to which a mechanical stress was applied. In seeking a relationship between the extent of the reduction in the extinction and the distribution of the strain generated in the plate, the author came to the conclusion that the reduction in the extinction effect is proportional to the gradient of the strain and not to the strain itself. In view of these investigations and the inadequacy of supposing, in the present case, a zone of especially strong mechanical stress (refer to (5)) or a step structure produced by etching at the boundary, the following seems to be a reasonable explanation of the present experiments. At the boundary between the etched and unetched areas there exists a zone of fairly large strain gradient in a direction parallel to the surface and perpendicular to the boundary. The existence of such a zone is the cause (through a reduction of the extinction effect) of the anomalous increase of the intensity of X-ray reflexion from the net planes at the boundary and perpendicular to the above-mentioned gradient. From this point of view the results of the apparent non-existence of the enhancement in the experiments (5) and (6) is explained by assuming that the strain gradient does not exist, or is small, in the directions perpendicular to the above-mentioned direction of large gradient.

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Acta Cryst. (1954). **7**, 460

An analytic method for the determination of shape and location of Fourier peaks.

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(*Received* 7 *October* 1953 *and in revised form* 10 *February* 1954)

In this note a rapid and efficient method of locating maxima of Patterson and electron-density maps is described. It is assumed (as is implicit in Booth's treatment (Booth, 1948)) that a peak resembles an elliptic paraboloid near the maximum. In addition to locating the maximum, the procedure outlined below gives information concerning the shape of the contours near the maximum and the directions of steepest and most shallow descents. The method avoids graphical procedures (e.g. Carpenter & Donohue, 1950) or extensive least-square methods (Shoemaker, Donohue, Schomaker & Corey, 1950).

It is assumed that the Fourier function (i.e. the Patterson or electron-density distribution) has been evaluated at the points of a net having grid lines parallel to crystallographic directions X' and Y'. The grid lines are separated by some convenient interval (usually 1/60 or 1/120 of the unit cell dimensions). Part of this net is shown in Fig. 1. The value of the Fourier function at each point (x', y') of the net is designated by $Z(x', y')$. Let the highest value of $Z(x', y')$ be called $Z(0, 0)$. The true maximum of the Fourier function will lie close to $(0, 0)$. A good approximation of its true location can be determined from the value of $Z(0, 0)$ and the values of the eight surrounding points indicated in Fig. 1.

For convenience in studying the shape of the elliptic paraboloid that will be fitted to these nine points, the location of the maximum will be determined with respect to an orthogonal Cartesian coordinate system X, Y which is defined as follows: The X axis is collinear with the X,