DISCUSSION

Yoshida, M.: 1) By which method did you make the specimen, by the floating zone or the pull method? 2) By which method did you introduce Ni and Cu, by doping or diffusion?

Levi F.A.: Specimens were received as single crystal slices 100 μ thick, already doped by Ni or Cu.

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON CRYSTAL LATTICE DEFECTS, 1962, CONFERENCE
JOURNAL OF THE PHYSICAL SOCIETY OF JAPAN VOL. 18, SUPPLEMENT II, 1963

Intensity Variations of X-ray Transmitted Diffraction Depending on the Direction of Applied Stress

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Measurements are made, by means of a photographic film and G. M. counter, on the intensity of diffraction of X-ray beams (MoKα) transmitted through a silicon plate, on one edge of which a mechanical force is applied. The results of the measurements show that the intensity of diffraction depends on the sign of the strain gradient generated in a crystal, as well as the magnitude of the strain gradient; the intensity of diffraction becomes stronger or weaker than that in the case where the strain gradient is absent in the crystal, according to whether the diffraction vector is parallel or antiparallel to the strain gradient.

These observations seem, macroscopic ones as they are, to have some correspondence to the asymmetry observed in the X-ray topographic images of crystal lattice defects.

1. Introduction

It is well known that the intensity of X-ray diffraction suffers a remarkable change with a crystal lattice distortions[1)-4)]. Previously, one of the present authors (E.F.[5)]) measured the intensity of diffraction of X-ray beam transmitted through a quartz plate on one edge of which mechanical force was applied, and found that the intensity was dependent upon the gradient of lattice strain, not upon the strain itself. In the present work reported here, the similar experiments were carried out on single crystals of silicon, and it was found that the intensity of diffraction depends not only upon the strain gradient as observed in the case of quartz[5)], but also upon the sign of the strain gradient relative to the diffraction vector concerned[6)].

It has been noticed that the X-ray microscopic images of individual dislocation lines[7)] and of oxygen layer segregations[8)] in single crystals of silicon had asymmetric intensity distributions and their intensity distributions changed when the sign of the diffraction vector was changed.

The present observations seem, macroscopic ones as they are, to have some correspondence to the asymmetry of X-ray topographic images of crystal lattice defects.

2. Experimental Methods and Results

2. 1 *Photographic method*

Single crystal of silicon was cut in a form of plate with surface parallel to (111), one side parallel to $(1\bar{1}0)$ and another side to $(11\bar{2})$, the size being $10\times10\times1.6$ mm. Local stress was applied elastically on one edge of a specimen plate by means of a blunt knife edge, whose radius of curvature was about 0.5 mm, as shown in Fig. 1. Strain field thus

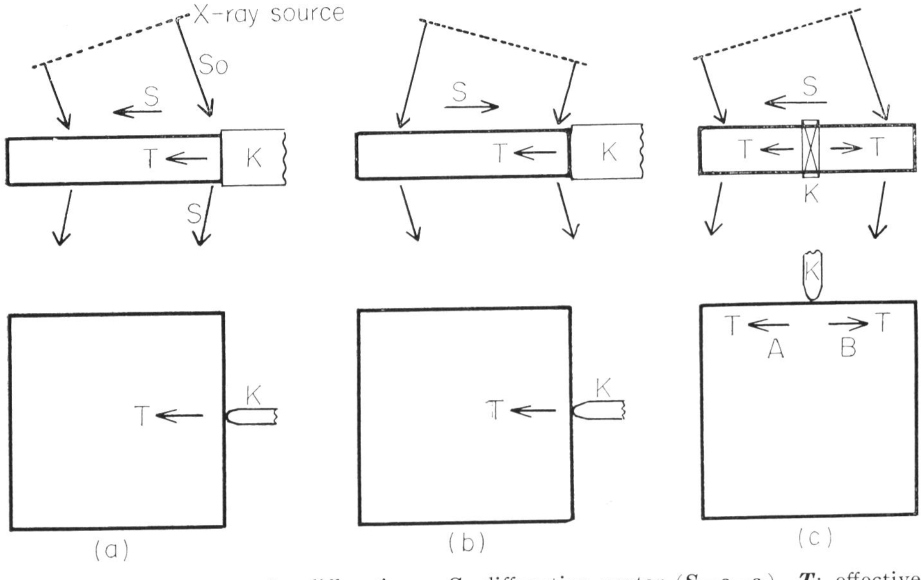

Fig. 1 Three arrangements for diffraction. $\boldsymbol{S}$: diffraction vector ($\boldsymbol{S}=\boldsymbol{s}-\boldsymbol{s}_0$) $\boldsymbol{T}$: effective strain gradient. K: blunt knife edge.
(a) $\boldsymbol{S}$ is parallel to $\boldsymbol{T}$. (b) $\boldsymbol{S}$ is antiparallel to $\boldsymbol{T}$. (c) $\boldsymbol{S}$ is parallel to $\boldsymbol{T}$ in one side A and antiparallel in the other side B.

generated in the specimen is very complicated. But it may be thought that the component of the strain gradient which is normal to the reflecting net planes is effective for diffraction.

Diffraction photographs were taken under three arrangements as shown in Fig. 1. In Fig. 1(a), the diffraction vector $\boldsymbol{S}_{(2\bar{2}0)}$ is parallel to the effective strain gradient $\boldsymbol{T}$ which is normal to the reflecting plane (parallel arrangement), and in (b) $\boldsymbol{S}_{(\bar{2}20)}$ is antiparallel to $\boldsymbol{T}$ (antiparallel arrangement). In Fig. 1(c), $\boldsymbol{S}_{(2\bar{2}0)}$ is parallel to $\boldsymbol{T}$ in one side A and antiparallel in the other side B (mixed arrangement).

MoKα radiation was used. X-ray beams from a line source fell on a specimen plate under the Bragg condition. Diffraction pattern over relatively large area of specimen was recorded on a photographic plate.

The results are shown in Fig. 2. A diffraction photograph in Fig. 2(a) was obtained under the condition in Fig. 1(a). The dark part in the photograph corresponds to more intense reflection from a distorted region in the specimen plate. A diffraction photograph in Fig. 2(b) was taken under the condition in Fig. 1(b). The light part in the photograph corresponds to less intense reflection.

In the former case, the effective internal strain gradient $\boldsymbol{T}$ is parallel to the diffraction

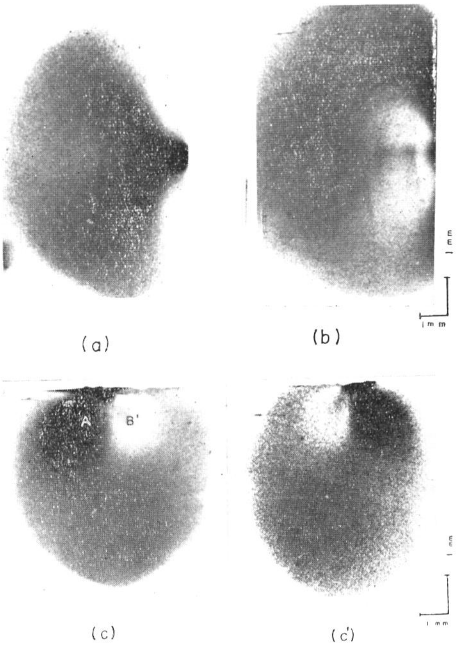

Fig. 2. (a) and (b) diffraction photographs $(2\bar{2}0)$ and $(\bar{2}20)$ taken with the arrangements (a) and (b) in Fig. 1 respectively.
(c) Diffraction photograph $(2\bar{2}0)$ with the arrangement (c). (c′) Diffraction photograph $(\bar{2}20)$, where the direction of diffraction vector $\boldsymbol{S}$ is in the opposite direction against that in (c).

vector $\boldsymbol{S}$. The diffraction intensity from a distorted crystal region is larger than that from undistorted. In the latter case, the diffraction vector was taken in the opposite direction against in Fig. 1(a). The diffraction intensity from distorted crystal region is smaller than that from undistorted. In both cases mentioned here, only the difference of the sign of effective internal strain gradient $\boldsymbol{T}$ relative to the diffraction vector $\boldsymbol{S}$ is essential for intensity variations.

Fig. 2(c) shows a diffraction photograph taken under the condition in Fig. 1(c). The dark part (A′) of the left hand side in the photograph, and the light part (B′) in the right hand side correspond respectively to the intensity enhancement and reduction of diffraction from distorted regions of a specimen plate. In the dark part, the internal strain gradient $\boldsymbol{T}$ is parallel to the diffraction vector $\boldsymbol{S}$, and in the light part, $\boldsymbol{T}$ is antiparallel to $\boldsymbol{S}$. Next, a diffraction photograph in which the diffraction vector was turned in the opposite direction against that in (c) was taken and is shown in Fig. 2(c′). Comparing the photograph Fig. 2(c) with Fig. 2(c′), intensity contrast at the corresponding sides is reversed clearly.

2. 2 *Measurements of diffraction intensity*

The local changes of peak intensity of diffraction in the mixed arrangement Fig. 1(c) were measured by a G. M. counter. Incident X-ray beams were limited by a circular slit 0.5 mm in diameter. Measurements were carried out on each position on a line 0.5 mm inside from the plate edge and the results are shown in Fig. 3. The abscissa is a position on a crystal plate and the ordinate the relative intensity.

Firstly, measurements were made on $(2\bar{2}0)$ diffraction and the result is shown by a solid line in Fig. 3. Secondly, the measurements were made on $(\bar{2}20)$ diffraction and the result is shown by the dashed line. Thirdly, the external force was removed. The result is shown by the chain line. The intensity curves in the former two cases, in which the diffraction vectors were in the opposite direction, show symmetrical distribution relative to the line of force application F.

The variations of the intensity of diffraction $(2\bar{2}0)$ with the total applied force to 3 kg were measured at the points indicated by

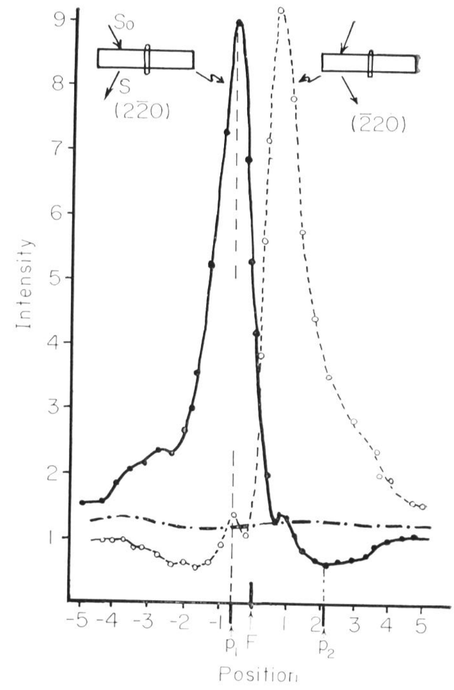

Fig. 3. Curve of diffraction intensities at each position on a line 0.5mm inside from the plate of a specimen.

P_1 and P_2 in Fig. 3. The results are shown in Fig. 4. The abscissais the total applied force and the ordinate is the relative intensity. At the position P_2, the diffraction intensity decreases with the force, as shown by the curve ($\boldsymbol{S}//-\boldsymbol{T}$), but almost constant for the value of total applied force more than 1 kg. At the position P_1, the intensity increases with the force, as shown by the curve ($\boldsymbol{S}//-\boldsymbol{T}$), and for the force of 3 kg the intensity amounts to about five times of the initial value.

In the present experiments, the value of μt (μ: linear absorption coefficient, t: thickness) is not large ($\sim$2.3). For the case of low absorption, it is usually considered that intensity of diffraction increases always by the reduction of extinction effect, when any lattice distortions exist in crystals. The present results show, however, that the intensity does become weaker when the diffraction vector is antiparallel to the strain gradient in crystals, although the rate of intensity reduction is rather small in comparison with the rate of intensity enhancement for larger value of force as shown in Fig. 4.

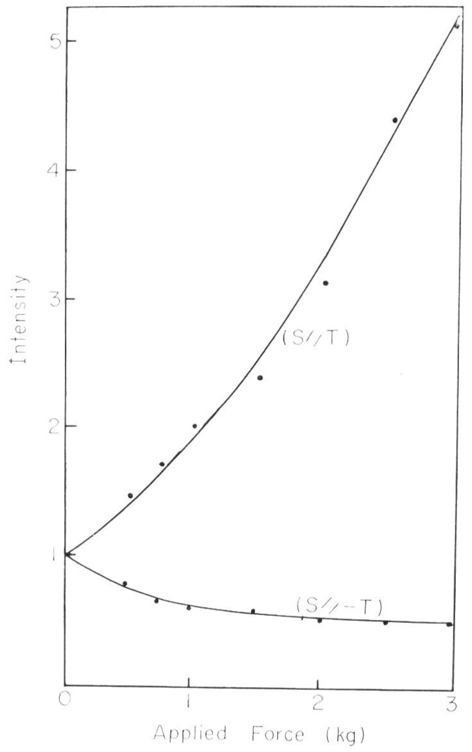

Fig. 4. Force *vs* intensity of diffraction $(2\bar{2}0)$ at the positions marked by P_1 and P_2 in Fig. 3. At P_1, $\boldsymbol{S}$ is parallel to $\boldsymbol{T}$ and at P_2, antiparallel.

3. Discussion

In the X-ray diffraction topographs by Lang's method, a dislocation line is observed as enhanced, black line on a photographic plate. It has often been noticed, however, that the one side of the black line is anomalously white. This phenomenon would be explained from the results in the present experiments, if it can be assumed that, at a dislocation, the strain gradient is parallel to the diffraction vector in one side of the dislocation line and antiparallel in the other side of the line.

Kohra and his co-workers[8] measured the periodic intensity variations from a series of sheets of oxygen segregation in silicon single crystals and found that the phase of periodicity changes when the direction of the diffraction vector is reversed. The change of the phase of the periodic intensity variation by reversing the direction of diffraction vector is expected also from our results although more serious consideration is needed on the nature of the oxygen bands in the present stage.

When E. Fukushima[5] studied the relation between the mechanical stress and extinction effects, using a quartz plate, he reported that the increase of diffraction intensities by the strain gradient is independent of the sign. Recently his results has been re-examined by means of the same equipment as in this experiment. The result has been the same as his, that is, the variation of diffraction intensities dependent of the sign of the strain gradient has not been observed in a quartz plate. This would be probably due to the more complicated structure of a quartz than a silicon crystal.

A possible interpretation of these phenomena is now being considered and will be discussed in detail elsewhere.

References

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DISCUSSION

Miyake, S.: Have you measured the change in angular width of reflection curves which may accompany the change in the reflection intensity?

Hayakawa, H.: In our experiments we used the divergent beam of X-rays as incident beam, so that we could not measure the change in angular width of reflection curves.