ments and no physical contact with the specimen is necessary for such observations. A variety of domain patterns has been studied in Fe+3% Si alloy[5], an example is shown in Fig. 2. This is a topograph of part of a thin (112) plate, thickness about 50 $\mu$. The pattern, though complicated, is simpler than the colloid pattern. The X-ray pattern averages the strains of surface closure domains over a range of several microns, producing a stripe pattern with stripe repeat period in the range 10 to 15 microns. The stripes lie either along $[20\bar{1}]$ or along $[0\bar{2}1]$, these directions representing the outcrops of the two cube planes most steeply inclined to the (112) surface. The boundaries separating areas of differently directed stripes are 90° Bloch walls and they lie along $[11\bar{1}]$ or $[1\bar{1}0]$. Extra strong stripes can be seen leading from corners in the 90° walls: they show the location of 180° Bloch walls.

**Acknowledgment**

The work here reported has been sponsored in part by the Aeronautical Research Laboratory, Office of Aerospace Research, United States Air Force, through its European Office.

**References**

1 A. R. Lang: Acta Cryst. **12** (1959) 249.
2 A. R. Lang: Acta Met. **5** (1957) 358.
3 A. E. Jenkinson and A. R. Lang: *Direct Observation of Imperfections in Crystals*, ed. Newkirk and Wernick, Interscience, New York (1962) p. 471.
4 W. Kaiser and W. L. Bond: Phys. Rev. **115** (1959) 1546.
5 M. Polcarová and A. R. Lang: Submitted to J. Appl. Phys.

---

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

# Studies on Lattice Defects of a Pair of Rods and Platelets in Silicon Single Crystals Observed by X-ray Diffraction Micrography

Mitsuru YOSHIMATSU

*Rigaku Denki Co. Ltd., Haijima, Akishima, Tokyo, Japan*

Two kinds of characteristic images, a pair of rods (or dots) and platelets are observed by X-ray diffraction micrography. The former is observed for specimens grown by the pull method as well as the floating zone method and becomes weaker in intensity or disappears with a heat-treatment. The Burgers vector of this defect is parallel to a $\langle 111 \rangle$ direction. The latter is observed for specimens grown only by the floating method and found to be in a {111} plane, where the distortion is minimum in the [111] direction in contrast to the former case. The intensity contrast becomes stronger with a heat-treatment at 950°C for 30 minutes.

## § 1. Introduction

In some silicon single crystals, surfaces are unusually roughened by chemical polishing. Such specimens were studied by X-ray diffraction micrography of the Lang method and a preliminary report was already given[1]. In this paper some results of further studies are reported.

## § 2. Experimentals

Specimens of thin plates and a pillar were prepared from silicon single crystals made by the floating zone method and the pull method. Three kinds of etchant were used. One etchant is a solution of HF (1 part) and $HNO_3$ (2 parts), which is used for fast etching. Another is a solution of HF (1 part), $HNO_3$ (2 parts) and $CH_3COOH$ (1 part), by which a smooth surface was obtained for the present specimens. Dash's etchant is used for obtaining etch pits. The experimental technique of X-rays is the same as the one previously used by the author[2] and, in principle, the one of Lang. $AgK\alpha_1$ radiation from a source of effective size of 25 $\mu \times 40$ $\mu$ was used under the operating condition of 50 kv and 1 mA. As photographic plate Fuji nucleus plates with emulsion 50 $\mu$ thick were used.

## § 3. Unusually Etched Surfaces and Etch Pits

Optical micrographs of the etched (111) surface are reproduced in Fig. 1. Both crystals were made by the pull method and etched by the 1st etchant. In Fig. 1(a) the surface is smooth, while in Fig. 1(b) roughened in the central region. The latter characteristic phenomenon was also observed for the surfaces {110} and {211} cut parallel to the growth axis from the same mother crystal. Such abnormally roughened surfaces were often observed for crystals made by the floating zone method and some-times for crystals by the pull method.

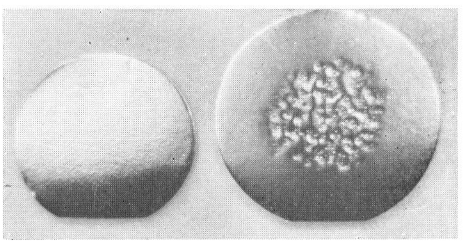

Fig. 1. Optical photographs showing a smooth surface and a roughened surface after etching.

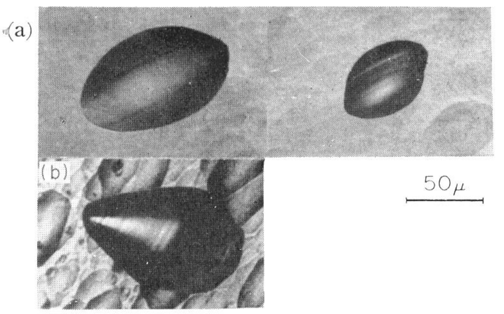


Fig. 2. Optical photographs of special etch pits and a dislocation etch pit on the $(0\bar{1}1)$ surface, respectively.

Fig. 2(a) shows patterns of etch pits revealed by Dash's etchant. The etching was done at 50°C for a few dozen minutes. In this stage of etching, etch pits due to dislocations did not appear. For comparison, the etch pit of dislocation obtained by deep etching (for about 24 hours) is reproduced in Fig. 2(b). These special pits were confirmed to correspond to a new type of defects which were found on a surface by stereographic observation of X-ray diffraction micrographs, as will be described in § 4 (iii).

## § 4. Observations by X-rays

i) *General features of lattice defects.* A typical X-ray micrograph taken from a specimen made by the pull method is reproduced in Fig. 3. In this photograph many images with a noticeable feature are observed besides dislocations. Most of them consist of a pair of rods. The length of rods varies with the individuals. They are sometimes as long as a half millimeter, and sometimes shorter and look rather like a pair of rods. On the other hand, it is to be noted that the separation of a pair is roughly the same and about 50 $\mu$ when the length of the rods is not shorter than the separation. In the case where the rods are shorter than the separation, there is a tendency that the separation becomes shorter with the rod length. As a general tendency, it is observed that the images are longer and the density is higher in the upper, half part of the photograph which corresponds to the inner part of the crystal, than in the lower

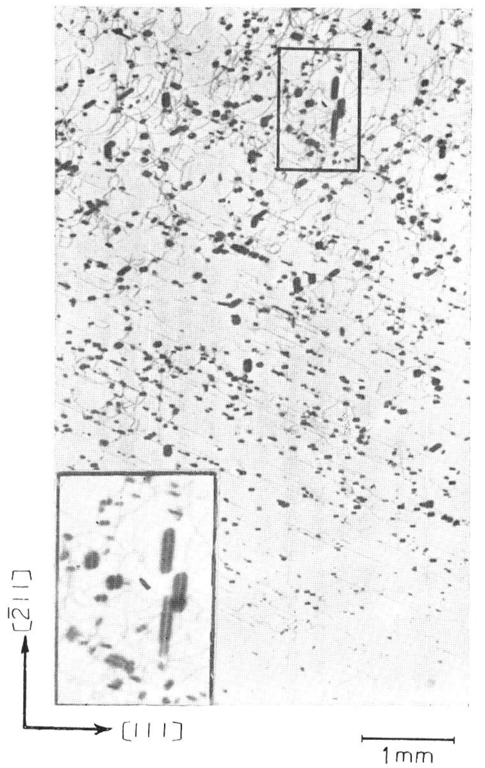


Fig. 3. X-ray photograph showing images of a pair of rods (or dots).

part. Near the crystal circumference any images of this kind were not observed. This tendency as to the distribution of defects is consistent with the feature of the etched surface mentioned above.

ii) *Shape and intensity contrast.* In order to study the variation in shape and intensity contrast of images with different reflections in detail, a pillar specimen was prepared from the same mother crystal. X-ray photographs are reproduced in Fig. 4(a). A top view of the specimen and a pole figure projected along the specimen axis [$\bar{2}$11] are given in Fig. 4(b) and (c), respectively. As seen from the pole figure, projecting directions for each reflection are illustrated in the picture of the specimen. Now let us notice the image indicated by an arrow. This image consists

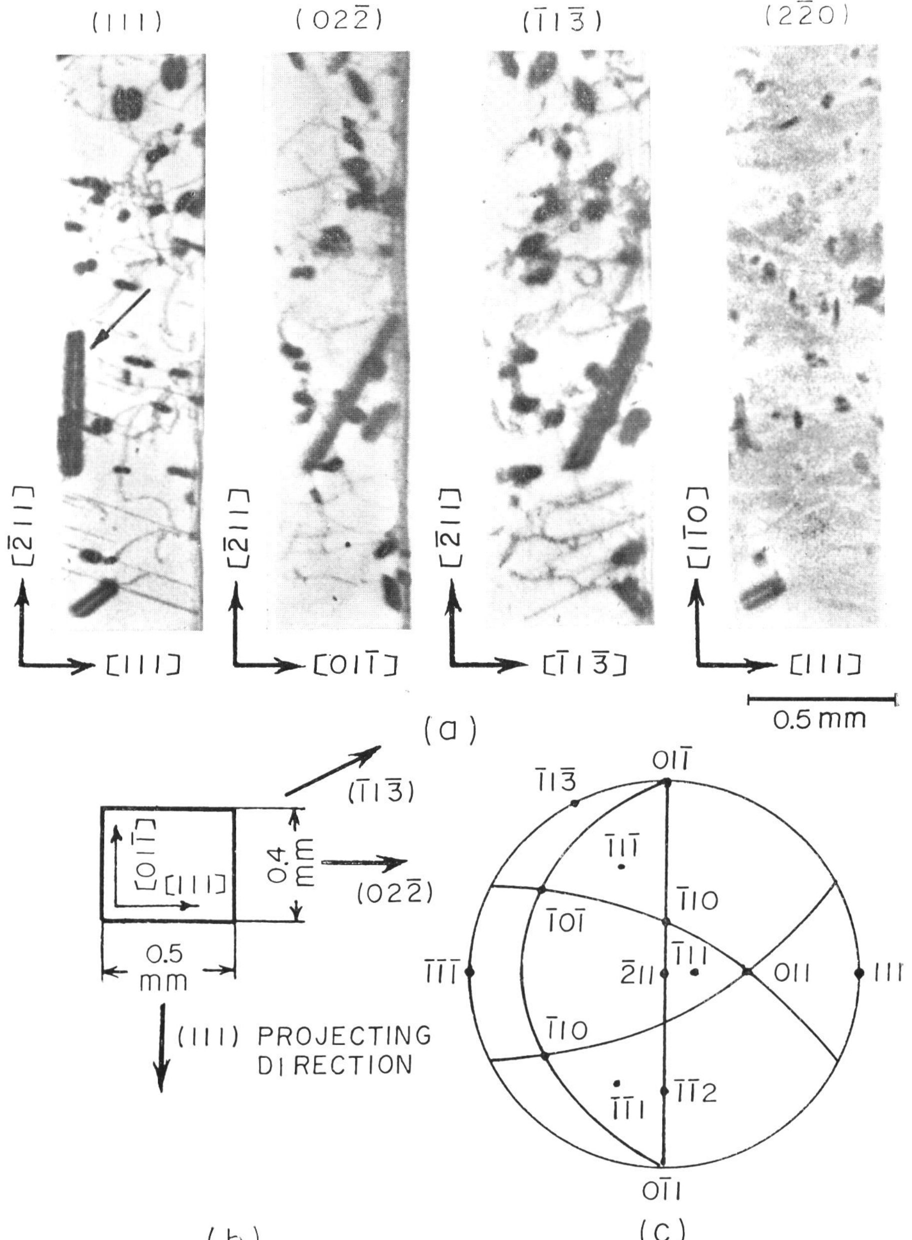


Fig. 4. X-ray photographs of a pillar specimen taken by different reflections (a), a top view of the specimen (b) and a pole figure projected along the specimen axis [$\bar{2}$11] (c).

of a pair of rods for the (111) reflection, while it appears as a rod with a diffuse boundary in one side for the $(02\bar{2})$ reflection whose reflecting plane is perpendicular to the (111) plane. In the $(\bar{1}1\bar{3})$ reflection it is observed as a pair of rods, in spite of the direction of projection making an angle of about 30° against that for the $(02\bar{2})$ reflection. From these variations in shape, it is concluded that the region responsible for the image should be as illustrated schematically in Fig. 5. The axis of the rods is along the $[\bar{1}10]$ direction. The pair of rods is found to be in the $(\bar{1}\bar{1}2)$ plane, in other words, the rods lie side by side in the [111] direction.

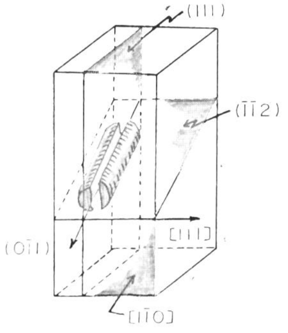


Fig. 5. Schematic drawing showing the regions responsible for the image of the pair of rods indicated by an arrow in Fig. 4 (a).

Next, let us consider the intensity variation. The intensity contrast is strongest for the (111) reflection among different reflections and the image disappears for the $(2\bar{2}0)$ reflection whose reflecting plane is normal to the rod axis, although the image itself is not observed in this case. Furthermore, the images does not disappear but has a considerable intensity for the $(02\bar{2})$ reflection whose reflecting plane is parallel to the [111] axis. From the above facts, it may be concluded that the maximum strain gradient contributing to the maximum intensity enhancement, in other words, the Burgers vector of this defect is parallel to the [111] direction.

iii) *Interaction between the dislocation and the pair of rods.* In Fig. 6, a pair of photographs for the stereographic observation is reproduced. They are taken by the (111) and $(\bar{1}\bar{1}\bar{1})$ reflections. It is observed that dislocations pass through pairs of rods. This situation seen in a region indicated by round circles is drawn schematically in Fig. 6(b). This means that the present defect has something to do with the dislocation line.

iv) *Plate-like images.* Besides the image of a pair of rods (or dots), plate-like images were observed. These image were found only in

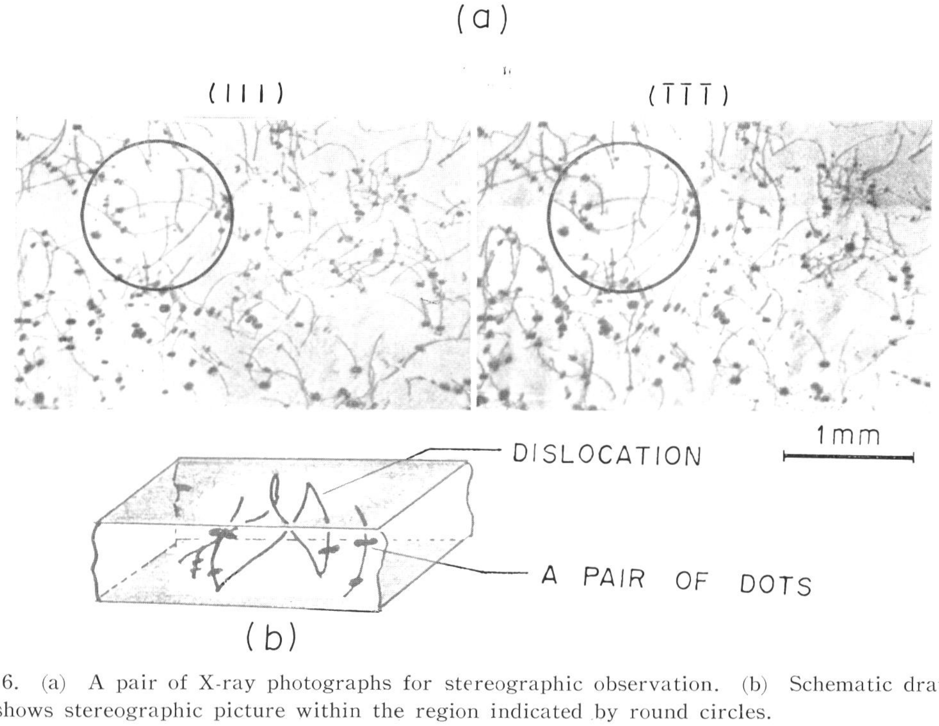


Fig. 6. (a) A pair of X-ray photographs for stereographic observation. (b) Schematic drawing shows stereographic picture within the region indicated by round circles.

crystals grown by the floating zone method as far as we examined, while the former defects were found in crystals grown by both floating zone and pull methods. An example of photographs is reproduced in Fig. 7.

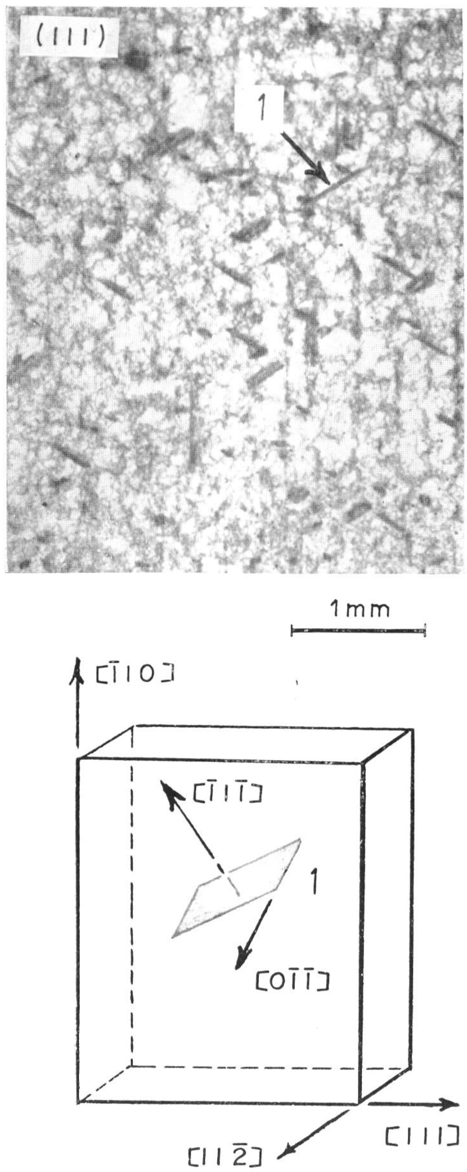


Fig. 7. X-ray photograph showing plate-like images.

The variation in intensity contrast with reflecting planes is also different from that for the rod-like defect. The intensity contrast is minimum or almost disappears for all other reflecting planes.

v) *Effect of heat-treatment.* In order to study the nature of these defects, a heat-treatment was tried. With a heat-treatment at 950°C for 30 min, it was observed that the defect of the former type disappeared, whereas the defect of the latter type increased in intensity contrast without growth of its size.

## § 5. Discussions

i) As was mentioned in § 4(ii), the intensity variation of the image of a pair of rods can be explained neither by the ordinary dislocation loop nor by the stacking fault. As one possibility, the nature of the intensity variation can be explained in terms of the sessile dislocation.* Let us consider the case where a vacancy cluster collapses to an elongated dislocation loop, and the plane of the cluster or the dislocation loop is parallel to the (111) plane. In this case, the Burgers vector is parallel to the [111] direction. The intensity contrast should be the strongest for the (111) reflection, and moreover it should disappear for the reflecting plane which contains the Burgers vector and is perpendicular to the long axis of the loop. Thus, the observed intensity variation of the image can be explained in terms of the sessile dislocation. However, it should be noticed that a pair of rods observed actually in the photograph does not correspond directly to the dislocation loop concerned. That is, the observed rods lie side by side in the [111] direction which is normal

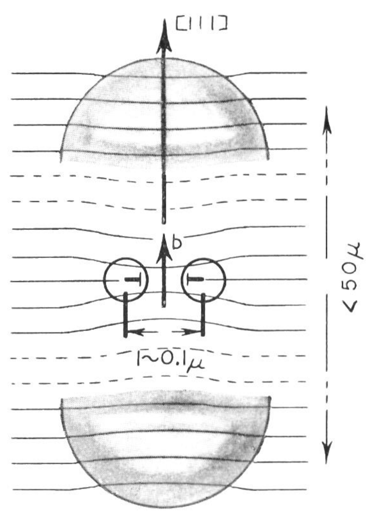


Fig. 8. Illustration of regions contributing to the formation of a pair of rods in terms of the sessile dislocation.

* According to a suggestion of Dr. C. Elbaum[3].

to the plane of the dislocation loop. This fact could be explained in the following way. Here, it is to be remembered that in the case of electron microscopy the image due to the dislocation loop lies in the (111) plane itself. In X-ray diffraction micrography, the image is thought to be formed mainly by the slightly distorted region far apart from the dislocation loop, namely misoriented with the order of second in arc, compared with the case of the electron microscopy, as shown in Fig. 8.

ii) According to Kobayashi,[4] the appearance of the rod-like image seems to depend only on the temperature condition during crystal growth, but not on the atmosphere of gas.

iii) Dr. Gilman[5] suggested another possible explanation in terms of dipole dislocations. The variation of intensity contrast as well as the position of the image can be also explained by this model.

iv) The origin of the plate-like image is being studied.

The author would like to express his hearty thankfulness to Prof. K. Kohra for his kind guidance and to Dr. Y. Shimura for his encouragement during the course of this study. The author is also grateful to Dr. C. Elbaum for his suggestion, and to Dr. T. Suzuki and Dr. A. Kobayashi for supplying silicon crystals used in this study and for their discussions, and to Mr. K. Sakayama for his technical assistance of taking photographs.

## References

1 M. Yoshimatsu, T. Suzuki, A. Kobayashi and K. Kohra: J. Phys. Soc. Japan **17** (1962) 583.
2 M. Yoshimatsu: J. Phys. Soc. Japan **16** (1961) 1465.
3 C. Elbaum: Private communication.
4 A. Kobayashi: Private communication.
5 J. J. Gilman: Private communication.

## DISCUSSION

**Elbaum, C.**: It seems to me that additional evidence for suggesting that the defects under discussion are prismatic dislocation may be based on the fact that their density decreased toward external surfaces.

**Kohra, K.**: The difficulty is that the observed image is displaced from the position of the defect. Therefore, the suggested explanation is only tentative.

**Auleytner, J.**: How much does the intensity of the diffracted beam increase in the region with the defect? In our study of the distorted region due to individual dislocations in Si, the increase was not more than 10% of the intensity from the perfect region.

**Kohra, K.**: We have not measured the intensity, but the intensity increase seems to be much more than 10% in our case. The magnitude of the intensity increase depends sensitively on the experimental condition such as the spreads of angle and wave length of the incident beam.

**Bragg, R. H.**: A few years ago Prof. Azaroff (Illinois Inst. of Tech.) and myself made reflected intensity measurements on silicon single crystals which contained no dislocations (as determined by etch pit counts). Even so the intensity was about a factor of two higher from the edges than from the center.

**Auleytner, J.**: I think we have obtained the different results because we used different methods. In some case the overlapping of curves takes place so that the intensity increases.

**Young, J. W., Jr.**: Have you measured rocking curves on the same specimens for which you have X-ray topographical pictures? I would be interested in comparing the two types of measurements.

**Kohra, K.**: We have not yet measured the rocking curve. But, as we shall report in the next paper, we have tried such a comparative study on a specimen of Si containing segregated oxygen. We should like to apply the comparative study to some other specimens as well as the one studied here.