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Citation	Memoirs of the Faculty of Engineering, Hokkaido University, 13(Suppl), 137-145
Issue Date	1972-05
Doc URL	http://hdl.handle.net/2115/37906
Type	bulletin (article)
File Information	13Suppl_137-146.pdf

Pseudo-Kossel line studies of lattice strain around deformation twin in bcc crystal

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An attempt has been made to investigate the accommodation effect on the formation of deformation twin in a 3.25% silicon iron alloy by divergent X-ray beam technique. The lattice rotations of matrix crystal near the twin are obtained by using the shift of deficient lines on Pseudo-Kossel photographs. Comparing with the bending of surface, obtained by the optical interferometry, it was concluded that the relaxation of stress at the front of twin was made without lattice rotation, and also that the observed lattice rotation would be produced by residual slip dislocations around the twin.

§ 1 Introduction

It might be important to investigate the mechanism of stress relaxation at the tip of the twin for understanding the formation of deformation twin, especially for that having large twinning shear as produced in b.c.c. crystals.(1) Since this relaxation may not be made perfectly, the accommodation strain will be retained around or/and in the twin lamellae. The residual strain inside the deformation twin in iron-silicon alloy was previously measured by using micro-beam X-ray technique. Special strains of the order of 10^{-3} have been obtained with respect to the direction of twinning shear and the axis of deformation.(2)

In this experiment, following to the previous one, the lattice rotation around the twin of the same material was measured by divergent beam X-ray technique. The special attention has been paid to set the specimen close to the focus on the target, since Pseudo-Kossel photograph must be magnified, corresponding to the specimen, to get the information around the thin twin. Interference microphotographs were also taken to measure the surface relief around the twin in comparison with the lattice rotation of matrix crystal.

§ 2 Experimental details

Coarse-grained polycrystalline sheets of iron-silicon alloy containing 3.25 wt%Si, 0.2 mm thick, were deformed at room temperature in tension with a strain rate of 0.1/min. The plane of the sheet was approximately parallel to (110), direction of tension being nearly parallel to [001]. The formation of twin was easily recognized by audible click. The specimens, approximately 100 mm long and 7 mm wide, were electro-polished in a solution of chromium trioxide in phosphoric acid. The surface relief produced by the twin, with (112) or (11$\bar{2}$) composition plane, on the top surface of the specimen, was observed under an optical microscope and also by an interference microscope. After taking photographs of the relief, the specimens were again thinned electrolytically to 100 μm thick, followed by etching finally to about 80 μm thick in nital. A series of Pseudo-Kossel photographs for the same area as the interference photographs were taken for the thinned specimen.

A capillary X-ray tube with pencil-shaped target was operated at 38 kv. with emission current of 3 μA. On the tip of the target made by Co-Fe alloy, a very fine X-ray source of about 7 μm in diameter, was obtained by a magnetic lens. The setting of the specimen was performed by using a simple optical system and a small goniometer stage with fine X-Y adjustment. The distance between the specimen and the X-ray focus was choosen as small as possible, say o.2 mm, in order to obtain the diagram which represents Pseudo-Kossel lines corresponding to the fine region around the twin in the specimen. Therefore, only the transmission Pseudo-Kossel patterns were obtained in this experiment. Moreover, the deficent line of the excess-deficient pair in the diagram was used for analysing the lattice rotation, since the "one to one correspondence" between the positions on the X-ray diagram and on the specimen was able to be made straight forward. The X-ray film with a dimension of 250X300 mm was placed 120 mm ahead of the focus. The magnification of the correspondence between specimen and film was about 600. A series of Pseudo-Kossel photographs were taken for the tip of twin, successively by displacing the specimen about 50 μm for each photograph in the direction parallel to the trace of the twin on the surface.

The patterns of the interference fringe were photographed under the magnification of 250. The specimen was placed so as to give the fringes perpendicular to the trace of the twin. A red filter, corresponding to the wavelength of 0.66 μm, was used to make the fringe pattern clear.

§3 Results and discussion

Fig.1(a) shows the interference micrograph near the tip of a twin for which a series of X-ray photographs were taken. The plane of the micrograph is nearly (110). The gradients of surface in the directions parallel and perpendicular to the trace of twin were obtained from the height on the two lines in this figure. The surface pattern obtained is schematically represented in Fig.1(b). The surface rotations, as indicated by dotted line in Fig.1(b), about the two axes, [001] and $[1\bar{1}0]$ were calculated along the trace of twin.

The variations of the angles of these rotations with respect to the distance from the tip are plotted by empty circles in Fig.3(a) and (b). As seen in these figures, the tilting angle increases up to the tip and maximum value is reached at about the position of tip. In front of the tip, the tilting continues for 200 um or more with the angle decreasing gradually. It is worth noting that the maximum rotation of the surface around $[1\bar{1}0]$ is about 3° and is much greater than that around [001]. The tilting angles about $[1\bar{1}0]$ plotted on the figure are the average values of those obtained on the two lines in Fig.1(a). The values on the extension line from the trace of the twin are about twice as much as the plotted values.

One of the X-ray Pseudo-Kossel photographs taken for the same area as in Fig.1(a) is represented in Fig.2. The dark pattern on the left hand side of the photograph is the shadow of a lead sheet which was placed for the purpose of the accurate positioning of twin. The broad dark bands running vertically at the upper right part of the photograph correspond to the selective etching pattern due to the twin. One is the shadow of the less etched " twin-hillock " on the top surface and the other on the back surface.

The dark lines, hyperbolae, ellipses and circles, are the deficient lines due to the Bragg reflection occurring on the specimen corresponding to those positions on the film. Main lines are being indexed on the pattern. The short lines marked by ↙ are due to the reflection from the twin, which can be distinguighed from those due to matrix not only by the length but also by the position, that is, they always appear between the two broad hillock bands described above. The lines due to matrix are usually very sharp except near the twin where they are broad or shifted as marked by ⇙ . The regions A and B of the photograph are enlarged for comparison, the former corresponds to the region distant from the twin, the latter being close to the twin. It is usual to understand that the shifts or the broadenings of the line are due both to the lattice strain and the lattice rotation of matrix crystal near the twin. In this survey, however, only the lattice rotation is treated as the origin of these irregularities.* The shifts of line due to matrix crystal near the twin were measured for a series of photographs with different positions. The rotation of lattice plane is

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* The observed directions of the shift from (hkl) and $(\bar{h}\bar{k}\bar{l})$ support that the origin is almost the lattice rotation. The previous results by micro-beam X-ray techbique are also in agreement with the present result.

calculated from the shift on the photograph by a simple geometrical relation. It should be noted that the direction of lattice rotation was the same as that of the surface tilting measured by interferometry as indicated in Fig.1(b).

The values of the rotation angle of lattice plane obtained along the twin are represented in Fig.3(a) and (b) by solid marks. The variation of the lattice rotation about [001] axis along the twin has a noticiable feature as seen in Fig.3(a). The lattice rotation of about 20' was retained at the boundary in the matrix. Near the tip this value increases up to about 1° and passing over the tip, the value decreases rapidly to zero. The similar variation is also seen in Fig.3(b) except less clear maximum at the tip.

If one compares the lattice rotation (●■▲ etc.) with the tilting of the surface (○), the important differences can be seen as follows:

(1) The lattice rotation diminishes rapidly at the tip, but the tilting of the surface continues for a long distance over the tip.

(2) The lattice rotation is less than 1°, but the tilting of the surface is usually more than that.

(3) At the tip, the amount of the lattice rotation about [001] is almost the same as the tilting angle of the surface, but this is not true for the rotation about $[1\bar{1}0]$.

From these experimental results, the following behavior of the accomodation of matrix crystal will be considered. That is, due to the large twinning shear in b.c.c. crystal, remarkable accomodation slips on (112), (011) and (101) must occur. The slips were frequently observed under the optical microscope. The situation around the twin will be like Fig.4 where one sees the slip dislocations with a/2 $[11\bar{1}]$ Burgers vector move to relax the accomodation stress. If there is some reason preventing the dislocation movement to the surface, the edge component of the dislocation, distributed like A in Fig.4 will produce the lattice rotation as shown in Fig.3(b). On the other hand, the screw dislocation, indicated by B in Fig.4, may produce the lattice rotation in Fig.3(a). Even though the remarkable relaxation must occur by the movement of screw dislocations at the incoherent twin boundary, the density of the residual screw dislocation will be larger than that on the coherent boundary as shown in Fig.4. This corresponds to the occurrence of the maximum rotation near the tip in Fig.3(a).

The above statement in qualitative nature must be reexamined quantitatively by adding the experimental data for another orientations.

References

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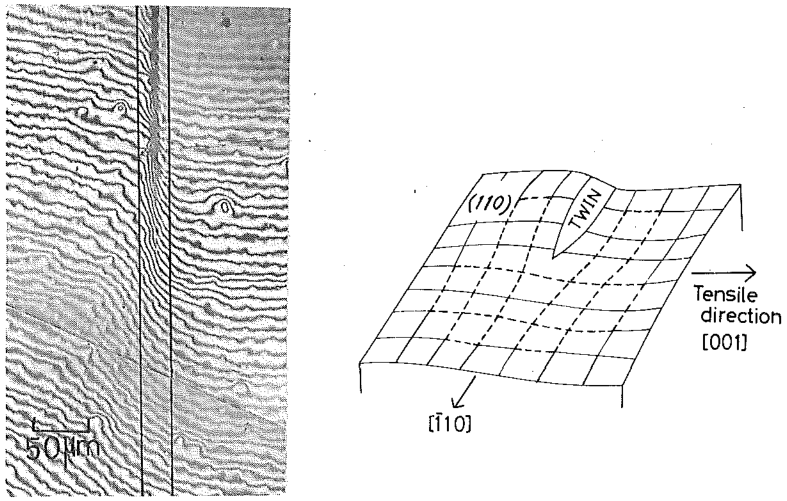
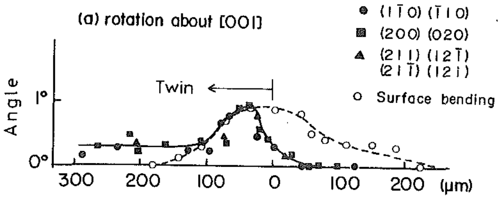


Fig.1 (a) Interference micrograph showing gradient of surface around a twin. The surface bending was measured on the two lines parallel to the twin.
(b) Schematic diagram showing surface bendings around a twin.



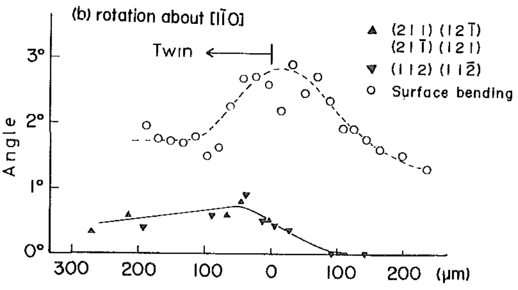


Fig.3 Surface bending and lattice rotation around the twin in Fig.2(a). The abscissa is the distance from the tip of twin measured parallel to the trace of twin, [1̄10]

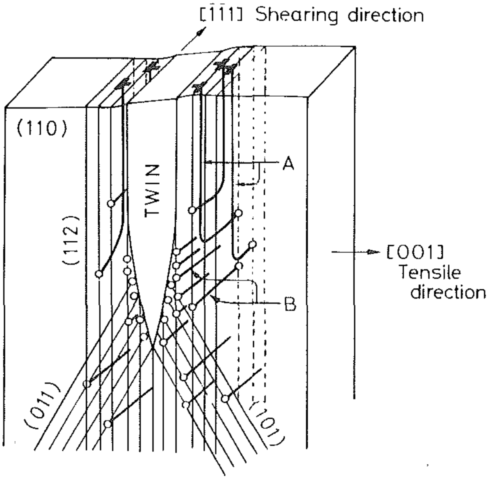


Fig.4 Schematic representation of accommodation around a twin

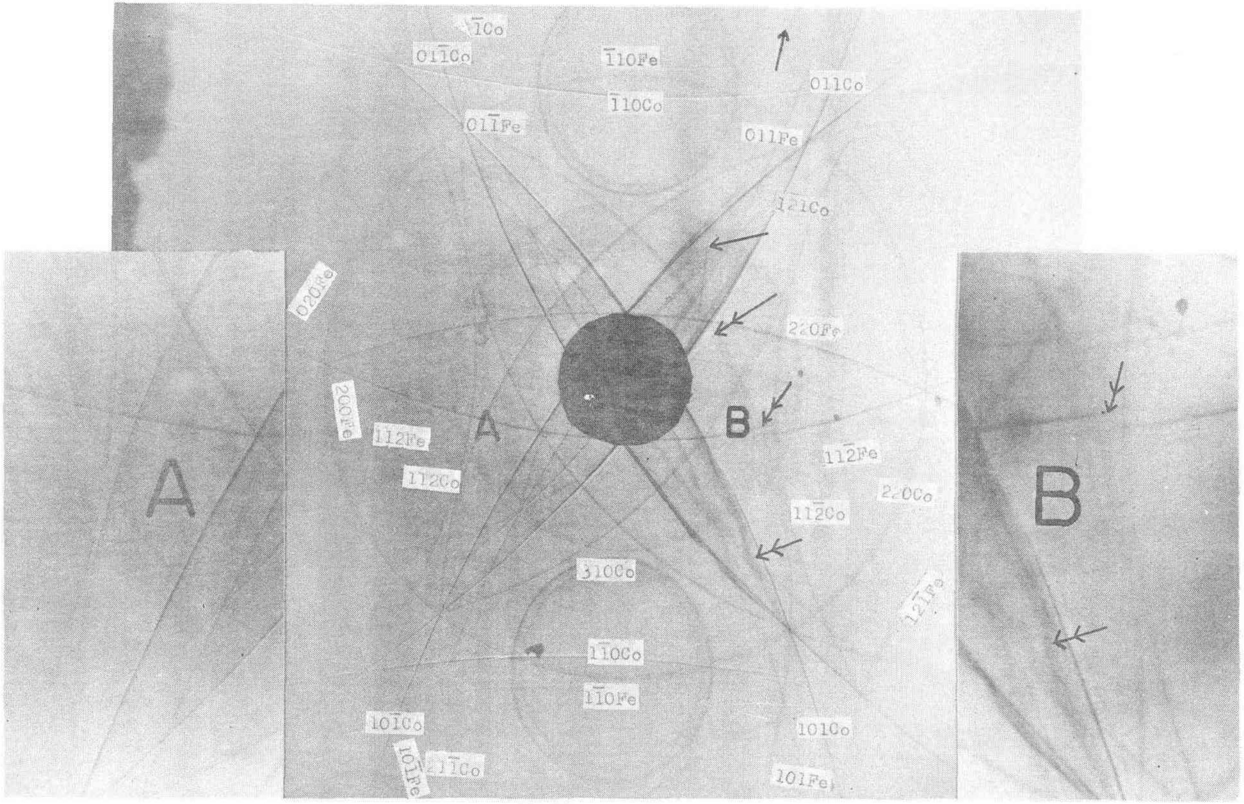


Fig.2 A Pseudo-Kossel photograph for the twin and its neighborhood in Fig.1(a). Regions A and B are enlarged for comparison.

↙: deficient line due to reflection from twin

⇙: broad or shifted line due to lattice rotation