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Abstract

Twinning deformation which appeared on the X-ray radiographs taken with the iron single crystal plates by the divergent X-ray method was investigated. The clearness of the band-like transmission image of the specimen representing the deformation twinning at the black center of the X-ray radiographs was observed clear only by the method of etching, irrespective of the thickness of the specimen. But, the image was made clear neither by thinning the specimen through electrolytic polishing nor by charging the exposure time of the X-ray. From these facts, the area only of deformation twinning bands, while from the slip deformation is proved to appear simultaneously with the deformation twinning produced in the specimen, was clearly identified. Furthermore, the X-ray diffraction images from the area of the deformation twinning were found.

1. Introduction.

Previously, it was reported that by taking the X-ray radiographs by the divergent X-ray method,\(^{(1)}\) of single crystal plates of pure iron bearing deformation twinning, the patterns of deformation bands due to twinning could be identified in the X-ray diffraction patterns within a broad Laue-spot appearing on the X-ray radiograph.\(^{(2)}\)(\(^{(3)}\))

The aspects of these X-ray diffraction patterns depend on the angle of twinning plane to the reflecting crystallographic plane and also on the direction of the twinning shears of the twinning. Furthermore, from the aspects, the outline of distribution of distortion of the crystal in both the deformation twinning crystals and the mother crystals caused by deformation twinning was made clear. In addition to this, the traces of deformation bands due to twinning could be identified in the black area representing the transmission image of the specimen at the center of this kind of X-ray radiograph.\(^{(4)}\) In
the present experiment, therefore, by observing the topographic details of the X-ray transmission patterns of the deformation bands in the black area representing the transmission image of the specimen at the center of an X-ray photograph, the causes of the X-ray transmission patterns due to deformation twinning bands and the aspects of the bodily structure of deformation twinning in the crystal have been investigated. The obtained results are to be reported in this paper.

2. Experimental Procedure.

Iron single crystal plates with dimensions of $0.4 \times 5 \times 80 \text{mm}^3$ were produced from electrolytic iron sheets of such purity as mentioned before, by the method traveling furnace. The crystallographic orientation of the specimen is shown in Fig. 1. In the figure, the signs $\bigcirc$ and $\bullet$ indicate the ones of the lengthwise direction and of the normal to the top surface of the plate respectively. The figures given in brackets indicate the number of the specimen.

First, the central part of the plate, in a length of about 20mm of each specimen, was reduced in its breadth to about 3mm by etching, and then in its surface by slightly electrolytic polishing. The single crystal plates so cut were then subjected to annealing for 4 hours at 850°C in vacuum.

Secondly, the specimens No.1, No.2, No.3 in those specimens were gradually stretched by a Shinkoh Communication Industry TOM-200 testing machine, so as to make the load-elongation curve in the liquid nitrogen, until the
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typical sharp click with twin formation was heard, and the simultaneously occurring load-drop was reduced. In this case, the distance between chucks was fixed about 5cm, the motion speed of chucks 0.5mm/min. Fig. 2 is the typical load-elongation curve taken with specimen No. 3. Of the specimens, No. 4 and No. 5 were gradually stretched in liquid nitrogen by a manually operated stretching machine until the typical sharp click with twin deformation was heard as above-mentioned specimens No. 1, No. 2, No. 3.

All of the five specimens so treated were examined by naked eyes and an optical microscope; next, the appearances of the deformation twinning bands were observed by using close-up and micro-photography. The above specimen No. 1, No. 2, No. 3 were reduced to about 0.2mm thick by electrolytic polishing and then by slightly etching in the 10% nitric acid solution. The X-ray radiographs of the specimen so treated were taken by the divergent X-ray method. Next, the specimen No. 4 was etched in 10% nitric acid solution for
15 minutes, and the X-ray photographs taken restrictively of the transmission image, represented at the black center, of the specimen so treated, were taken by the divergent X-ray method, with the X-ray exposure time at 1 sec, 3 sec, 5 sec, 7 sec and 10 sec, respectively. In this case, the films used were the Fuji Industrial X-ray films. X-ray photographs of the specimen No. 4 similarly treated, with the gradual changes of the time of etching for 30, 50, 70 minutes were also taken respectively. As for specimen No. 5 the thickness was gradually reduced to 0.38, 0.32, 0.28, 0.21mm by electrolytic polishing, and photographs of the specimen at each thickness were taken. In this, the specimen 0.21mm was etched in 10% nitric acid solution for 10 minutes, and photographs of the specimen so treated were likewise taken. In taking these photographs, the point X-ray source was 15×20μ², and, the distance between the specimen and the X-ray source and the one between the specimen and the film were 70mm and 80mm, respectively. Then, the films used were the Fuji Softex FG films. The X-ray exposure time was 6 hr for the diffraction image in the circumference, and was 90 sec for the transmission image in the black center. Furthermore, a replica of the surface of the specimen, the twinning area of which seems to be concaved by etching, was made. And the aspects of the concave were examined by the optical microscope.

3. Experimental Results and Discussion.

3.1 Deformation bands due to twinning.

The typical examples of the photographs of the whole shape and the black center of specimens No. 1 and No. 2, respectively, taken by the divergent X-ray method, are shown in Fig. 3(A), (B) and Fig. 4 (A), (B). In these transmission images and diffraction images of X-ray photographs there appear clearly the patterns representing the deformation twinning bands similar to those mentioned in the previous paper.(2)(3) Accordingly, these twinning bands were examined in the same method that was used in the previous paper.(2) The number 8, given in the photographs, is after the notation of the Schmid & Boas. In both specimens No. 1 and No. 2 only deformation twinning 8 was found.

Next, Laue-spots I, II and III in X-ray photographs Fig. 3 and Fig. 4 are the X-ray diffraction images diffracted respectively on crystal planes (310), (003) and (110). In Fig. 5 (A), (B), (C) which is a enlarged photograph of the image, appears the pattern representing the deformation twinning bands. Particularly, in Fig. 5 (A) and (B), if examined minutely, the X-ray diffraction images representing the deformations twinning can be observed and just above Laue-spot I ; (310) and sideways above Laue-spot II ; (003), the patterns of the diffraction images which seem to represent the deformation
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In Fig. 5 (C), in the part where the deformation twinning band intersects the rim of the Laue-spot, there appears the abnormal diffraction, the so-called "water-column" reported in the previous paper, and in the same Laue-spot the patterns representing slip systems (101) (111) and (011) (111) were clearly observed.

3.2 The transmission image representing deformation twinning at the black center of the X-ray radiographs.

The enlarged photographs of transmission image of the specimen No. 4 by the divergent X-ray method, are shown in Fig. 6 (a), (b) and (c). In (a) the specimen was etched for 30 minutes, in (b) 50 minutes, in (c) 70 minutes. The transmission image of the specimen No. 5, at the black center of the divergent X-ray photographs and the X-ray diffraction images, of the same specimen, diffracted on the (011) crystal plane are shown in Fig. 7 (A), (B) respectively. Photographs (a), (b), (c) and (d) in (A) and (B) respectively indicate the stages of the specimen at 0.38mm, 0.32mm, 0.28mm and 0.21mm thickness, reduced by electrolytic polishing. The last stage (e) shows the specimen 0.21mm thick (which is the thickness of (d)), after it has been etched in 10% nitric acid solution for 10 minutes. The numbers in the photographs are after the notation of the Schmid & Boas.

Observing these X-ray photographs Fig. 6 and Fig. 7 will show that the clearness of the line-like X-ray transmission images representing the deformations twinning and that of the line-like X-ray diffraction images increase more when the specimen was etched, irrespective of its thickness. It was found that the image did not become clearer when the specimen was reduced in thickness by electrolytic polishing alone or when the X-ray exposure time was charged. This fact is definitely distinct from the changes of the patterns of slip traces. This distinct feature can demonstrate the difference between the deformation twinning area and the slip area in the twinning deformation related to the slip occurring at the edge of the twinning, and, at the same time, clearly indicate the aspects of the edge of the twinning.

Next, in Fig. 6 (C), in the line-like transmission image representing the deformation twinning seen in the transmission image at the black center and in the part, for example, the band of deformation twinning, appears one line-like X-ray transmission image. Parallel to the image, there appears one obscure line broken intermittently. The breadth of this line measured on a optic-microscopical photograph was about 5 \( \mu \). The breadth on the line-like X-ray transmission image representing the deformation twinning at the black center of the X-ray photograph was 300\( \mu \). The breadths of this line measured...
on a X-ray photograph is remarkably much wider than that of the deformation twinning appearing on the surface of the specimen, but this fact may be explained by the crosswise occurrence of deformation twinning in the above specimen. In specimen No. 4, the thickness is approximately 380μ. The angle of the twinning plane (112) of deformation twinning to the lengthwise direction of the specimen is about 23°. Therefore, the thickness of this deformation twinning can be reflected on the surface of the specimen. The breadth was about 150μ. The breadth of the transmission image appearing in the black center of the divergent X-ray photograph was about 300μ, which is the expected breadth of the affection of the divergent incident X-ray, that is, the two breadths are approximately the same. From these, it was found that the deformation twinning corresponded to the line-like X-ray transmission image on the transmission image at the black center. Deformation twinning series 2 and 7 being considered, it was also found that the deformation twinning band occurring on the specimen corresponded to the line-like X-ray transmission image at the black center.

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References

Aspects of Central Part in Divergent X-ray Radiograph by Deformation Twinning.

(A) X-ray photograph taken by the divergent X-ray method.

(B) Central black part.

Fig. 3
(A) X-ray photograph taken by the divergent X-ray method.

(B) Central black part.

Fig. 4
Aspects of Central Part in Divergent X-ray Radiograph by Deformation Twinning.

(A) Laue-spot (310)

(B) Laue-dot (103)

(C) Laue-spot (110)

Fig. 5
Fig. 6

(a) Central black part

(b)

(c)
Aspects of Central Part in Divergent X-ray Radiograph by Deformation Twinning.
Fig. 7
(a) Lane-spot (011)
(b)