Microdeformation of [111] Iron Whiskers

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(Received 19 January 1982; accepted 7 May 1982)

Abstract
Iron whiskers with [111] growth direction were subjected to a tensile stress and simultaneously observed by X-ray transmission topography. The initiation of the deformation occurred in the part of the whisker which revealed grown-in lattice defects. The distorted area propagated not only as a result of increasing the tensile stress but also during its relaxation at constant elongation. The dislocations introduced during the deformation were of the same type as grown-in dislocations. Increasing the stress led to a non-uniform rotation of the crystal lattice during the deformation. The axis of rotation was found to be [332] for one whisker and [111] for another one.

I. Introduction
In the case of b.c.c. metals the slip system depends on the orientation of the specimen, deformation rate, temperature, impurities and nature of the metal. The slip planes are generally considered as planes of the (111) zone (Sesták, 1966; Kroupa, 1968). At low temperatures the {112} slip planes predominated while at intermediate temperatures the {110} planes operated and at high temperatures the {123} planes were found to participate in the deformation (Honeycombe, 1971).

Iron whiskers are very useful specimens for the investigation of the deformation process in b.c.c. metals by means of X-ray transmission topography. They have a high degree of purity, low density of dislocations and they are bounded by low-index faces. The faces are in general flat and smooth. The morphology and the grown-in dislocation structure of iron whiskers have been described earlier (Surowiec & Polcarova, 1976; Bojarski & Surowiec, 1979). There is little information available on the dislocation structure of the deformed iron whiskers. The dislocations generated on a face of the whisker by an indenter were investigated using the etch pit method (Coleman, 1958; Kushnir, Mihaylova & Osipyan, 1965). Iron whiskers deformed during a tensile test were studied using the X-ray precession method (Gardner & Hanscom, 1976). X-ray transmission topography was applied for the in situ observation of dislocations produced by a tensile deformation of iron whiskers with [100] growth direction (Mende & Hagen, 1977).

The aim of this work is to obtain more detailed information on the multiplication of dislocations in deformed [111] iron whiskers showing a defined initial dislocation structure. X-ray transmission topography was used for the in situ observations.

II. Experimental technique
Iron whiskers were produced by the hydrogen reduction of commercial ferrous chloride at temperatures between 993 and 1023 K using the method developed by Brenner (1956). Whiskers 10-20 mm long and about 100-200 µm thick with [111] growth axis and {110} faces were selected for the investigations. The tensile machine described by G’sell & Champier (1980) was used in order to observe the evolution in situ of the dislocation configuration in the stressed whisker under stress. The specimen ends were attached to the machine by Araldite polymeric cement. It was possible to apply a given stress to the crystal with an accuracy within 5%. Owing to the error in the crystal cross-section determination, the final error on the resolved shear stress was about 10%. Before the microplastic deformation of the specimens the dislocation structure of each whisker was fully characterized. The stress was then increased step by step and a 10T topogram was made for each value. Silver Kα radiation was used with an exposure time about 10–20 min. Topograms were recorded on fine-grain Ilford L4 plates.

III. Results
III.1. General survey
The first whisker, sample A, was 9 mm long and its thickness varied from 100 to 140 µm; in the vicinity of both ends of the whisker there were grown-in dislocations; near the thicker end the whisker contained a small region giving a high contrast on the X-ray topogram; the middle part of the whisker had a high degree of perfection (Fig. 1).
The second whisker, sample B, 5 mm long and 121 µm thick had a uniform lattice twist equal to 3 mm\(^{-1}\) caused by a set of axial dislocations with screw component. Except for the previous set the initial density was almost equal to zero.

During the tensile test experiments we have observed several stages in the evolution of the dislocation structure:

1. Effect of the cement on the dislocation structure: near the cemented ends multiplication of dislocations took place.
2. When the stress was applied the multiplication and the propagation of dislocations started in the parts containing grown-in defects.
3. The propagation of disturbances was observed during the time the elongation was maintained constant.
4. Beyond a certain stress limit we have found non-continuous as well as continuous changes in the orientation of the crystal lattice of the whisker. The rotation axis responsible for such a deformation was determined.

The dislocations in as-grown iron whiskers are of the screw type and lie in \{110\} or more rarely in \{211\} planes (Surowiec & Polcarova, 1976; Bojarski & Surowiec, 1979). In the 10T topogram the dislocation lines were visible as straight lines running in two directions (Fig. 2). These directions are projections of [\(\overline{1}11\)] and [111] directions. The dislocations having the third direction [111] are invisible (g.b = 0). The fourth direction [111] is the direction of the whisker axis; the dislocations with this direction are invisible too. The dislocation density in the as-grown iron whisker is non-uniform; some zones of high density and other ones almost defect free are observed.

III.2. Tensile test results for sample A

During the cementing of the whisker fresh dislocations were produced in the embedded region and its near vicinity. The X-ray topogram in Fig. 3(a) was made directly after setting the whisker end into the cement. The same topogram repeated after 24 h, when the cement was hard, showed changes in the contrast (Fig. 3b). Simultaneously an elastic twist and/or bending of the specimen occurred. The initiation of the distorted region was observed after the first step when the applied tensile stress was equal to 8.7 MPa. It was visible that the multiplied dislocations appeared in the place where grown-in dislocations were present (Fig. 3c). Unfortunately it was not possible to distinguish single dislocation lines in the new deformed area.

Between the disturbed region and the end of the whisker there was a perfect fragment of the whisker.

It was characteristic in our experiments that, when the elongation was kept constant for several hours, the disturbed zone extended and the applied stress relaxed. Such an example of the change in the internal structure during the relaxation is shown in Fig. 4. The stress decreased from the value \(\sigma = 8.7\) to \(\sigma = 3.5\) MPa during a period of about 5 h.

It was noteworthy that there was a relatively sharp limit on topograms in Figs. 4(b) and (c) between the disturbed and the non-disturbed areas; this limit

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**Fig. 2.** Dislocations in as-grown iron whisker with [111] growth direction. Ag K\(_\alpha\) \(_1\) radiation, 110 reflection.

**Fig. 3.** X-ray topograms of the same part of the [111] whisker. (a) Before drying the cement; (b) after cement was hard; (c) after applying the stress \(\sigma = 8.7\) MPa.
looked like a Lüders band. The deformation shown in Figs. 3 and 4 propagated from the thinner end of the whisker. Near the other end there was a grown-in defect area from which the propagation of the deformation occurred in both directions while a rotation of the crystal lattice took place. Two topograms were necessary to obtain the image of the whole length of the whisker: the right part of the whisker was seen on one topogram (Fig. 5b), whereas after a small change in the specimen orientation the left part was seen on the topogram in Fig. 5(c). In both cases the applied stress was equal to 7.8 MPa.

The middle part of the whisker, the relatively most perfect one, remained unchanged for a long time (Fig. 6a). It was disturbed after about 24 h only during the relaxation of the stress (Fig. 6b).

The increase of the stress to \( \sigma = 17.2 \, \text{MPa} \) led to changes not only in the contrast but also in the geometry of the X-ray image. The image was composed of two segments denoted as I and II in Fig. 7. The disorientation angle was equal to 3°46'. The thinner part of the whisker was not observed on this topogram because of the rotation of the crystal lattice. Only after a long exposure time was the image of the thinner part obtained from the white radiation part of the incident beam. Three such topograms for different reflections \( 110, 101 \) and \( 011 \) of the same part of the whisker are shown in Fig. 8. It corresponds to \( \sigma = 35.1 \, \text{MPa} \). The topograms show a similar, continuous but non-uniform change in the orientation of the diffracting planes. While its image obtained on the topogram is very disturbed the whisker remained straight and its external morphology is unchanged.

The position of the rotation axis was deduced from the reflections by the distorted and undistorted parts of the whisker using the Lang camera with a micro X-ray beam (120 x 60 µm). The diffracted beams coming from both fragments were recorded on the same photographic film which was placed at the distance of 150 mm from the sample.

The undeformed part of the whisker was taken as the reference; to obtain the Bragg diffraction from the disoriented part of the whisker it was necessary to rotate the whisker around the vertical axis by an angle \( z \) so that the reflected beam was shifted in vertical direction by a distance \( Z \) on the photographic plates. Measurements of \( z \) and \( Z \) for three different reflections allowed the determination of the position of the rotation axis and it was found to be [332]. It is noteworthy that the value of the angle \( z \) did not vary uniformly along the whisker length (Fig. 9).

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![Fig. 4](image-url)  
**Fig. 4.** Propagation of the disturbed area as a result of relaxation of the stress, the elongation being kept constant. 110 reflection, Ag K\text{x}\text{I} radiation. (a) \( \sigma = 8.7 \, \text{MPa}, \ t = 0 \text{h}; \) (b) \( \sigma = 5.2 \, \text{MPa}, \ t = 4 \text{h}; \) (c) \( \sigma = 3.5 \, \text{MPa}, \ t = 5 \text{h}. \)

![Fig. 5](image-url)  
**Fig. 5.** Initiation of the deformation in the defect region of the whisker. 110 reflection, Ag K\text{x}\text{I} radiation. (a) As-grown state; (b) and (c) propagation of the deformation in both directions.

![Fig. 6](image-url)  
**Fig. 6.** Topograms of the middle part of the whisker. 110 reflection, Ag K\text{x}\text{I} radiation. (a) A small region which is still perfect; (b) the same area after deformation.

![Fig. 7](image-url)  
**Fig. 7.** X-ray topogram showing a change in orientation of the whisker crystal lattice. Ag K\text{x} radiation, 110 reflection.
III.3. Tensile test results for sample B

The whisker B has shown a uniform lattice twist around its growth axis. The 110 topogram consisted of two bands which corresponded to the parts of the sample reflecting the Kα1 and Kα2 radiations respectively (Fig. 10a). In the 222 topogram, the reflecting plane being perpendicular to the growth axis of the whisker, a visible white–black image could be connected to a set of axial screw dislocations (Fig. 10b). The observation of all the length of the whisker in the 110 reflection was difficult during the tensile test investigation because of the lattice twist, so several exposures were made varying slightly the orientation of the whisker on the Lang camera to obtain the images of all the parts successively. In the as-grown state the lattice twist was uniform along the length but a change in the value and the character of the twist took place after the cementing and the application of the stress. The topogram recorded when the cement was hard showed an enhancement of the contrast near both ends of the whisker (Fig. 11a). The disturbance began to propagate when the stress was increased to 17 MPa (Fig. 11b). The whole length of the whisker was disturbed when the stress was equal to 23 MPa (Fig. 11d). The increasing of the stress to the value 32 MPa led to a change in the crystal-lattice orientation as in the case of sample A (Fig. 11e).

The exact measurement of the change in the lattice orientation around the whisker axis after the last step of elongation showed that the lattice rotation was not uniform (Fig. 12). Using the same method as for sample A, we found that the rotation axis connected to the image on Fig. 11 was parallel to the growth axis of the whisker.

It is generally difficult to distinguish on the topograms single dislocation lines in the deformed areas of the whisker. However, for other deformed whiskers, some single dislocation lines emitted during the elongation were observed (Fig. 13). These dislocations had the same orientation as the as-grown dislocations and they were pure screw dislocations. We assumed that the observed bands on topograms (Figs. 6 and 7) consisted of a large number of multiplied pure screw dislocations.

IV. Discussion

It should be remembered that in our experiments the elongation increased step by step over a few days. The mechanism of such a deformation was certainly different from the usual tensile test with iron whiskers (Brenner, 1959; Yoshida, Onazuka & Yamamoto, 1971). For example, the deformation went on during the relaxation of the applied stress. However, our results show that the dislocations moved and multiplied in the whisker in the so-called elastic part of the stress–strain curve.

We now have to determine if the rotation observed in each whisker, around axes [332] and [111] respectively, corresponds to the rotation of the crystal being deformed through stressing while the direction of the applied stress is kept constant. Because of the symmetry one may expect the slip to occur on six slip systems...
Fig. 11. Propagation of the disturbance as a result of the increasing of the stress. (a) Whisker after cementing; (b) $\sigma = 17$ MPa; (c) $\sigma = 15$ MPa, after some relaxation; (d) $\sigma = 23$ MPa; (e) $\sigma = 32$ MPa.

Fig. 12. Change in the angle $\alpha$ for the sample B (a) before and (b) after the tensile experiment.

Fig. 13. Comparison of the same parts of the whiskers. (a) and (c) Before deformation; (b) and (d) after deformation; arrows show fresh dislocations.

with slip directions [111], [1T1] and [T1T]. In fact the experiment shows that only one slip system is activated, which certainly is true in the stage of the microdeformation we have studied. However, we cannot assume which slip system will be effectively active and we have to study each of the six possible cases. During the rotation of the crystalline lattice, the slip direction tends to draw nearer to the direction of the applied stress by moving in the plane containing both these directions; the rotation axes are then normal to the previous planes, that is in our case [101], [110] and [011] (Fig. 14). These axes do not agree with our experimental observation; even a combination of rotations around two or three of the axes will not account for the experiment. We must therefore conclude that the microdeformation of iron whiskers proceeds from more complex slip systems than those we have considered. Unfortunately, the resolution limit of X-ray topography does not permit the identification of those slip systems. The fact that we did not find the same rotation axis for the two whiskers examined could be due to the presence of a set of axial screw dislocations in whisker B whereas whisker A was perfect except for well-localized places and even there the density of dislocations was very low.
V. Conclusion

We have studied the microdeformation of iron whiskers subjected to a tensile stress increasing by steps and we have thus observed through X-ray topography the changes in the configurations of dislocations after applying the stress and during stress relaxation while the elongation was kept constant.

We observed that the deformation does develop essentially in the areas originally containing defects and dislocations. When possible we showed that fresh dislocations were of the same type as the grown-in dislocations, that is pure screw dislocations. The resolution limit of X-ray topography has not permitted the same precision in the case of areas with a high dislocation density. As the deformation proceeded the defect areas propagated along the whisker.

In the final state of the microdeformation we noted a non-uniform rotation of the lattice in certain areas and found directions [332] and [111] as rotation axes. However, it has not been possible for us to account for this result starting from the rotations of the lattice that can be expected when the deformation proceeds through slip with the direction of the applied stress remaining constant.

References