II-15 EFFECT OF CARBON ON DISLOCATION STRUCTURE OF IRON MICROCRYSTALS

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By an etch-pit technique a change in the character of dislocation rosettes in iron microcrystals as a result of carburization was studied. The results obtained are attributed to the effect of carbon on the cross-slip of dislocations in alpha iron (α-Fe).

Studies$^{(1-8)}$ carried on various crystals (particularly alkali-halides with rocksalt structure, LiF, NaCl, MgO, as well as FeSi) have shown that dislocations appearing under the action of a localized load are distributed around the site of the indentation (or impact) as a characteristic group, or "rosette."

For rosette formation, nucleation of dislocations at the point of the load action, as well as their movement and multiplication are needed. Thus all the elementary processes taking place during the plastic deformation occur in the rosette formation. The appearance and size of a rosette are affected by peculiar aspects of dislocation properties and movement under the action of applied stress. That is why an investigation of dislocation configuration in a rosette permits us to explain -- at least qualitatively -- a number of peculiarities in development of plastic deformation and strengthening of crystals.

An analysis$^{(5,6)}$ of a space distribution of dislocations and of different dislocation loops form has shown that in NaCl crystals, a rosette is in reality a dislocation net containing glide as well as sessile prismatic dislocation loops. If it is assumed that prismatic loops in NaCl are formed by the cross-slip mechanism, one would conclude that there must be exceptionally great cross-slip in the dislocation structure formation process. It is the presence of prismatic loops, as well as prismatic and glide dislocation loops, whose interaction results in sessile dislocation formation,

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which in turn seems to secure strength and immobility of the dislocation net, and which prevents the extension of newly formed dislocations under the load, further into the crystal.

As far as iron is concerned, direct electron-microscope observations(9, 10) have shown that the great tendency of dislocations to cross slip is the characteristic peculiarity of plastic deformation. It is caused by the fact that in the bcc lattice of α-Fe, there are many possible slip planes, and dislocations themselves are narrow and can easily glide from one slip plane to another. Even in the most initial stages of plastic deformation, there are conditions for frequent intersection of dislocations belonging to different slip planes of various types -- {110}, {112} and, possibly, {123}. Observations show that dislocation intersections and interactions cause the formation of a very unhomogeneous structure, and the appearance of local "tangles" of dislocations which act as barriers for other dislocations movement, but at the same time can be active dislocation sources.

One can consider a dislocation net, appearing under a localized load acting as a tangle, so that all the peculiarities of dislocation movement and interaction, characteristic for iron, must affect the rosette formation in iron microcrystals.

Experimental technique: Iron microcrystals were grown(11) by hydrogen reduction of ferrous chloride at the temperature 730-750°C. Under examination, the microcrystals had clean, even brilliant, surfaces with cross dimension up to 1 mm and length up to several centimeters.

The orientations most frequently observed were: four faces of {100} type with growth axis <001> ; two faces of {100} type and two {110} with growth axis <110> ; six faces of {110} type with growth axis <111> .

Carburization of iron microcrystals was achieved by one of two techniques. Specimens were treated either in H_2-stream containing heptane vapor for 40 min at the temperature 550-600°C and flow rate up to 0.5 litre per min; or in CO-atmosphere at 800°C.(12) The carbon contents provided by the techniques used were respectively, 0.007 and 0.02% . For a localized loading of microcrystals, measurement apparatus PMT-3 was used.

Dislocation structures of the microcrystals were observed by an etch-pit technique.(13) The etching was carried in an equal volumetric mixture of dilute alcohol solutions of picric (4% picral) and nitric (2% nital)

acids for 7-10 min at room temperature. It has been shown that the technique used gives etch-pits on {100} faces corresponding to dislocation intersections with the crystal surface. "Aged" dislocations as well as deformation-induced "fresh" ones can be made visible without previous decoration by impurities.

A further study has shown that to observe the dislocation sites on faces other than cubic, one must decorate dislocations with carbon (30-40 min at 160°C). That is why the rosettes on {110} faces were observed by the etch-pit technique only in carburized specimens.

Results: In iron microcrystals, grown by the technique mentioned, that are relatively pure and perfect, one can observe after a localized loading net dislocation rosettes, beams of which are always stretched along certain crystallographic directions. These directions are traces of intersections of active slip planes with the crystal surfaces.

By means of the etch pit technique, individual dislocation movement during the deformation of iron microcrystals has already been observed.(13) The dislocation movement occurred along <110> directions which are the traces of possible slip planes {110} and {112} on the crystal surface corresponding to a {100} plane.

When the indentor is pressed into crystal dislocations in the iron, microcrystals can move simultaneously at least in 18 slip systems -- six planes {110} and twelve {112} taking no account of {123} planes. It is difficult to expect the form of the rosette, formed by that process, to be as obviously and clearly related to the anisotropy of glide as in the case of alkali halide crystals having only six possible glide planes {110} .

In Figure 1 an etched four-petaled dislocation rosette is shown formed around an indentation site on a {001} face in iron microcrystals. The rosette beams which are observed on {001} surfaces of iron microcrystals are always stretched along <110> directions that correspond to possible slip planes {110} and {112}.

In the presence of carbon one can observe rosettes not only on cubic, but also on {110} faces of iron microcrystals. On {110} faces of carburized microcrystals the rosette beams are preferably along <111> directions (slip planes {110}, {112} and {123}) - Figure 2. On {001} faces frequently, but not always, the rosette beams are stretched along <210> directions, corresponding to slip planes {211} and {123} - Figure 3 - that prevent {110} planes from being possible slip planes in these crystals.

Finally one can see from the figures that in the presence of the impurity the rosette character itself is changed. As a rule, in carburized crystals they become more clear and sharp. So the introduction of small amounts of carbon essentially changes the dislocation rosette's orientation and character.

In addition, the following peculiarity was noted: in carburized microcrystals rosettes with very straight sharp beams are observed, i. e. in the presence of an impurity, plastic deformation easily extends from the rosette localized around the pin-hole, to very large parts of the crystal - Figure 4. The appearance of such long sharp beams throughout the crystal practically makes impossible any quantitative measurements of rosette dimensions in order to compare them with other characteristics of material properties, as is frequently the case for alkali-halide crystals. (4, 7)

The extension of narrow localized slip bands from a rosette is observed not only with localized loading, but simply on precipitated particles of carbides on over-carburized specimens - Figure 5.

Thus one can observe on carburized specimens the appearance of rosettes with very long (as compared with as-grown pure crystals) beams, i. e. iron is "softened" as a result of introducing carbon.

Discussion: Impurities introduced in crystals, precipitating on dislocation lines, can pin them and stop their movement, which causes hardening of material. On the other hand, breakaway of dislocations from impurity atmospheres (under the action of temperature or stress) causes its softening.

However, the effect of impurity introduction can have quite a different effect.

Being absorbed on dislocation lines, impurities can essentially influence such microscopic characteristics of dislocations as their width, their tendency to cross-slip, and others and correspondingly can appreciably change distribution, character of movement, and interaction of dislocations during plastic deformation.

In accordance with available data, (14) the presence of a small amount of impurities in bcc metals causes dislocation extension accompanied by formation of stacking faults lying preferably in twinning planes {112}. Such dislocation extension must effectively diminish their tendency to cross-slip.

If in fact formation of a strong and immobile dislocation net under the pin-hole is caused by interaction of glide dislocations lying in various slip planes with sessile prismatic loops which had resulted from cross-slip, then, as a result of its impediment after carburization, dislocation rosettes under the indentor must become less strong.

Also, the localization of the slip process in several certain slip planes makes the material more rigid, impeding stress relaxation in these planes. Dislocations and piling-up in these slip-planes easily break weak dislocation net, surrounding the deformed field under the indentor and, meeting no obstacles, go far into the crystal by narrow slip bands ("beams").

Thus in each separate case (depending on the plastic deformation conditions and the state of material) the impurity may cause hardening or softening of an as-grown crystal.

CONCLUSIONS

1. By means of a selective etch-pit technique, dislocation structures -- "rosettes" -- resulting from local loading (pinning) in iron microcrystals were observed. The form and orientation of a rosette are shown to depend on slip geometry in the crystal and therefore on its orientation and properties.

2. The effect of small amounts of carbon on rosette character and orientation has been observed. The data obtained suggest that cross-slip plays a great role in rosette formation in iron microcrystals. It is the decrease of a dislocation's tendency to cross slip in the presence of carbon that causes the change of rosette appearance and orientation in carburized microcrystals.

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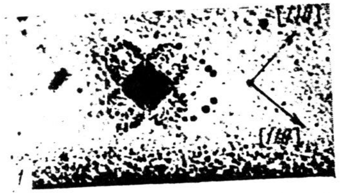

Fig. 1. Rosette around indentation on {001} face of iron microcrystal. x 315. Load 50 g, loading temperature 196°C.

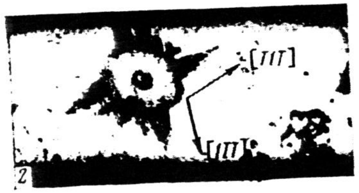

Fig. 2. Rosette around indentation on {110} face of carburized iron microcrystal. x 135 (0.007% C, load 50 g, room temperature).

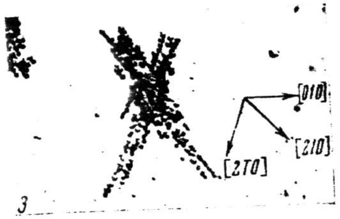

Fig. 3. Rosette around indentation on {001} face of carburized iron microcrystal. x 135 (0.007% C, load 2 g, room temperature).

Fig. 4. Rosettes around indentations on carburized iron microcrystals (room temperature). a) On {001} face, 0.02% C, load 50 g. x 135; b) on {110} face, 0.007% C, load 20 g. x 315.

Fig. 5. Rosettes around precipitations of carbide particles on {110} face of carburized iron microcrystal. x 315.