Title
ON THE NATURE OF THE DISLOCATION SUB-STRUCTURE IN DEFORMED COPPER

Permalink
https://escholarship.org/uc/item/83p694w8

Authors
Price, W.L.
Washburn, J.

Publication Date
1962-10-01
ON THE NATURE OF THE DISSLOCATION SUBSTRUCTURE IN DEFORMED COPPER

TWO-WEEK LOAN COPY
This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

Berkeley, California
DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.
ON THE NATURE OF THE DISLOCATION SUBSTRUCTURE IN DEFORMED COPPER

W. L. Price and J. Washburn

October 1962
ON THE NATURE OF THE DISLOCATION
SUBSTRUCTURE IN DEFORMED COPPER

by
W. L. Price* and J. Washburn**

Introduction

Previous transmission electron microscope observations on the arrangement of dislocations in deformed face-centered cubic metals have shown that when the stacking fault energy is not too low, (i.e., above about 10 ergs/cm²) plastic deformation produces dislocation tangles. (1-8) As the strain increases the tangles become the boundaries of a three-dimensional cell structure.

A realistic model for the strain hardening of pure face-centered cubic metals depends on a detailed knowledge of the damage that accumulates in the crystal as the strain increases; it is this damage that makes necessary an increase in stress to continue the nucleation and propagation of slip bands through the crystal. (9) However, the complexity of the dislocation arrangement in deformed metals, combined with the fact that only a projection can be observed of what is really a three-dimensional structure, have made it impossible to clearly answer a number of elementary questions about the dislocation tangles:

* Lockheed Missiles and Space Corporation, Palo Alto, California, U.S.A.
** Department of Mineral Technology and Inorganic Materials Research Division, Lawrence Radiation Laboratory, University of California, Berkeley, California, U.S.A.
(1) Do the cell boundaries tend to form along glide planes, and how are they related to the slip bands that can be seen at external surfaces?

(2) Are Lomer Cottrell dislocations frequently found in the tangles, and do they play an important role in their growth?

(3) Do individual segments of dislocation line within the tangles lie accurately on a (111) plane or do they usually contain a high density of jogs?

(4) Are edge dislocation dipoles (positive-negative pairs) or prismatic loops often formed, and do they play an important role in the nucleation of tangles?

Answers to some of these questions concerning the complex arrangements of dislocations in deformed crystals can be obtained by transmission electron microscopy only by choosing a particularly advantageous orientation of crystallographic axes relative to the plane of a thin foil. Until now this has not been done; observations were made on polycrystalline specimens or on single crystals that did not have the most favorable orientation.

In the present work, copper single crystals were deformed in tension along the (111) axis and the dislocation substructure observed as projected onto the (110) plane. The (111) tensile axis was chosen so as to have six slip systems operating from the start of plastic deformation and therefore have the greatest probability of forming Lomer Cottrell dislocations and jogs due to intersections. The (110) foil plane was chosen so that one active glide plane, (111), and one inactive glide
plane, (111), would lie exactly at right angles to the plane of the foil. Any dislocation segments, regardless of their orientation or whether they were straight or curved, if they lay on these glide planes would project as straight lines along [112] or [112] in the (110) projection plane. The other two active glide planes, (111) and (111), were equally inclined at 35° to the plane of the foil. Their line of intersection, [110], lay in the plane of the foil so that if long Lomer Cottrell dislocations were formed at these intersections, long segments would be preserved in the thin foils.

Experimental Procedure

High purity copper (99.999) was used to grow flat crystals (.020" x .75" x 8"). They were grown from the melt in a graphite mold in an argon atmosphere, and were seeded to give [111] parallel to the long dimension and (110) parallel to the flat face within about 3°.

The crystals were strained at 20°C in an Instron testing machine at a strain rate of .005 per minute. A special fixture was used during mounting and removal from the testing machine to avoid bending.

Thin foils were prepared by chemical polishing in a solution of 50% nitric acid, 25% acetic acid, and 25% phosphoric acid at 20°C followed by electrolytic polishing in a bath of 33% nitric acid, 67% methanol at -30°C. Specimens were observed in the electron microscope immediately after polishing to minimize oxidation. All observations were made using a Hitachi HU-10 microscope operated at 100kV and a magnification of 10,000.
Observations and Discussion

A typical resolved shear stress vs resolved shear strain curve is shown as Fig. 1. The strain was plotted as the sum of the shear strains on the six systems, assuming that they were all equal. Points on this curve represent amounts of deformation at which thin foil observations of the substructure were made on different specimens.

Figures 2 to 9 are representative transmission electron micrographs that show the development of substructure with increasing plastic strain. All were taken with the foil and therefore the (11̅0) plane perpendicular to the axis of the electron microscope within a few degrees. The isosceles triangle which appears on each photograph is a projection onto (11̅0) of the edges of the tetrahedron formed by the four (111) planes. Its orientation was determined from selected area diffraction patterns. The solid line is the [110] direction; it is the trace of the two active glide planes (111) and (1̅1̅1) that are inclined at 35° to the plane of the foil. The dashed lines are traces of the (11̅1) and (1̅11) planes that lie at right angles to the plane of the foil. It was impossible to tell which was the active and which was the inactive glide plane from the diffraction patterns because it was not known whether specimens were being viewed along [11̅0] or [1̅10]. The solid base line of the triangle is always one micron in length on the photographs.

A detailed study of the electron micrographs leads to the following conclusions:

(a) The dislocation tangles in these foils, made from specially oriented single crystal specimens, were usually indistinguishable
from those observed previously in crystals of various orientations or even in polycrystalline specimens. Only rarely were areas found in which dislocation lines followed the [112] or [11\(\bar{2}\)] directions in the (110) projection plane. Assuming that at least the three Burgers vectors corresponding to the active slip systems were present in the tangles and that for a given Burgers vector there was an equal probability that a line should lie in either of its two possible glide planes, then one third of the total length of dislocation line in the tangles should have been on the (111) plane which is at right angles to the plane of the foil. This should have been noticeable as many short segments of line lying parallel to the trace of that plane. A few of the observed tangles did have the expected appearance, (see Figs. 7 and 10). Since this was not generally observed, and because the (111) plane could usually not even be distinguished from the inactive (111) plane by observation of the foils, it was concluded that most segments of line in the tangles were heavily jogged; they did not lie exactly on (111) planes.

(b) Short straight segments of dislocation lying along [110] that were probably of the Lomer Cottrell type were sometimes seen. (See, for example, the region near "A" in Fig. 2.) However, they were not a prominent feature of the tangles and no long straight dislocations lying along [110] were ever seen even though this direction lay in the plane of the foil. Therefore, sessile dislocations of this type do not have any special importance in the work hardening of pure copper even when deformed in tension along the [111] direction.
(c) For the multiple slip orientation studied (tensile axis along [111]) the tangles which gradually developed into subgrain boundary walls did not grow along (111) planes. There was no special relationship between the general directions of observed tangles and the traces of any of the (111) planes. Tangles often appeared to develop around dislocations that were probably present in the crystal prior to the start of deformation. During the first few percent plastic strain dislocation dipoles were often found starting at these dislocations, sometimes forming dense clusters like those in Fig. 11.

(d) Slip bands that are observed at the external surfaces of deformed crystals show that plastic strain is nearly always nonuniformly distributed. Within a slip band the local shear strain may be very large compared to the measured average strain. In these specimens one of the active slip planes was at right angles to the plane of the foil. Therefore, it was expected that some indication of nonuniform distribution of strain on this plane might be found. However, as with previous thin foil observations, no indication of slip bands was observed. There was not even any alignment of particularly dense parts of neighboring tangles that might indicate a heavy local shear strain along a particular group of glide planes. This suggests that the local damage that causes shear displacement to stop on a given glide plane and to spread to near-by parallel planes during growth of a slip band can not be easily detected in thin foils. One type of damage that may not be seen is small clusters of point defects, either in the form of prismatic dislocation loops less than 50 Å.
in diameter, or dislocation pairs having a spacing of only a few interatomic distances. Small closed loops and smaller black spots that could have been unresolved loops or some other type of point defect cluster were most numerous in the vicinity of dislocation tangles. (See Figs. 3, 5 and 7.) Since the observed dislocation substructures gave no indication of the location of slip bands, it suggests that the internal structure of the tangles depended primarily on the maximum stress that was reached rather than on the local shear strain. It is to be expected that any mobile length of dislocation line with length between nodes or large jogs greater than $\frac{G b}{T}$ should bow out and form new nodes so as to reduce its free length.

(e) After a system of subgrains had been formed, their average size remained almost constant with further increase in strain. However, there was a large spread in the sizes of the subgrains from about 0.3 $\mu$ to 3.0 $\mu$.

(f) It was difficult not to introduce new substructure into thin foil specimens during handling. Figures 12 to 15 are examples of dislocation substructures that were almost certainly produced by accidental bending of a thin foil during polishing or mounting in the microscope holder. The arrays of long dislocations that superficially resembled pile-ups always lay approximately parallel to [110] which was the common trace of the two inclined glide planes. Damage due to bending was most easily detected in undeformed or lightly
deformed specimens because long straight dislocations were introduced. Bending of a foil that contained a network of dislocation tangles resulted in less noticeable changes in the substructure.

Clear examples of cross-slip leaving short segments of dislocation line in the cross-slip plane can be seen in Fig. 12 at "A". Cusps where motion was held up by invisible barriers are shown in Fig. 14. These cusps are not likely to be due to jogs because the dislocation lines are primarily edge in character; the Burgers vector lies at about 60° to the line and jogs should be able to glide with little difficulty. Figure 15 shows a severely bent region where only a narrow strip is in good diffraction contrast.

These transmission micrographs of bent foils are included primarily to emphasize the fact that extreme care must be taken to avoid accidental damage to thin foils particularly if substructures characteristic of the early stages of plastic deformation are being studied. Many of the transmission electron micrographs that have been represented in the past as being characteristic of the dislocation substructure existing during stage I (easy glide) have probably actually shown long parallel dislocation lines introduced by slight bending of the foil.
References


Fig. 1. Typical stress-strain curve showing deformations at which thin foil observations were made.
Fig. 2. 1.3 kg/mm$^2$. 
Fig. 3. 1.3 kg/mm².
Fig. 4. 2.3 kg/mm$^2$. 

ZN-3319
Fig. 5. 2.3 kg/mm$^2$. 

ZN-3320
Fig. 6. 3.4 kg/mm².
Fig. 7. 3.4 kg/mm$^2$. 
Fig. 8. 6.6 kg/mm$^2$. 
Fig. 9. 6.6 kg/mm$^2$. 
Fig. 10. One of the few dislocation tangles that clearly showed dislocation segments lying on the (111) plane.
Fig. 11. Elongated closed loops and edge dislocation pairs that were formed near grown-in dislocations.
Fig. 12. Long dislocations in a bent foil, showing cross-slip at "A".
Fig. 13. Bent foil showing interactions between edge dislocations on close parallel glide planes at "A".
Fig. 14. Interaction between dislocations of primarily edge character and invisible obstacles to produce numerous cusps.
Fig. 15. Foil that has been severely deformed in bending after thinning.
This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.