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THE EFFECT OF NEUTRON IRRADIATION
ON SLIPLINES IN MOLYBDENUM

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THE EFFECT OF NEUTRON IRRADIATION
ON SLIPLINES IN MOLYBDENUM

by

J. L. Brimhall

Metallurgy Research Section
Reactor and Materials Technology Department

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PACIFIC NORTHWEST LABORATORY
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INTRODUCTION

Although the mechanical behavior of irradiated materials has been studied extensively, the precise mechanism of irradiation hardening has not been established. Some information on the microstructural species responsible for hardening of irradiated materials has been derived from direct and indirect observations. The direct observation of the structure of deformed irradiated metals, particularly by transmission electron microscopy, is a relatively new and unexplored area.

Slip line studies in face centered cubic metals (fcc) have been reduced primarily to copper. ⁽¹⁻³⁾ Very coarse slip lines, representing slip on many closely spaced planes, have been observed in deformed, irradiated copper. ^(1, 2) Coarse slip lines have also been observed in irradiated aluminum. ⁽⁴⁾ Clear channels in irradiated copper caused by dislocation motion through the defect structure have been seen by transmission electron microscopy and correlated with slip lines. ^(5, 6)

Little work has been reported on the irradiated body centered cubic (bcc) metals. Eyre described the slip lines around hardness indentations on irradiated iron and found them to be more distinct than lines in unirradiated material. ⁽⁷⁾ Mastel, et al., ⁽⁸⁾ have observed channels in deformed irradiated molybdenum similar to those reported in copper.

As part of a program concerned with the effect of neutron irradiation on metals, the slip line structure was studied on irradiated molybdenum single crystals deformed by bending. The purpose of the study was to determine the nature and homogeneity of the deformation process in an irradiated bcc metal single crystal known to contain defects and to compare this behavior with a similarly deformed but unirradiated crystal.

The slip line formation was studied primarily as a function of strain, carbon content of the specimen and orientation. Correlations were made

between the surface slip line structure and bulk structure as observed by transmission electron microscopy. Correlations were also made between the irradiated and unirradiated material.

SUMMARY

Slip line formation during bending has been studied on neutron irradiated molybdenum single crystals. For equivalent strains, the slip lines are coarse and distinct in the irradiated molybdenum and not resolvable in the unirradiated molybdenum. Defect-free channels form in the irradiated material during deformation and the slip lines have been directly correlated with these dislocation channels. The heterogeneous distribution of the dislocations during deformation in the irradiated material results from dislocation interaction with the defects produced during irradiation. Impurities, in the form of carbides, affect the dislocation channeling by acting as dislocation sources.

EXPERIMENTAL PROCEDURE

The molybdenum was supplied by the Materials Research Corporation. The material was of two different compositions listed by the supplier as 10 ppm carbon and 450 to 500 ppm carbon. Single crystals were grown in an electron beam zone refining unit. Crystals with the proper orientations were subsequently cut and polished to rectangular bars approximately 2 by 2 by 35 mm. The orientation of the longitudinal axis and the crystal faces is shown in Figure 1. Future reference will be made to Crystals A and B as designated in Figure 1.

FIGURE 1

Orientation of Single Crystals of Molybdenum
for Irradiation and Subsequent Bending

Both crystals were irradiated to 10^{19} nvt ($E > 1$ MeV) at approximately 40 C. A crystal, identical to Crystal A, was polished and served as an unirradiated standard.

Deformation was performed by four-point bending in the jig illustrated in Figure 2. The deflection of the bar at the midpoint and the maximum radius of curvature were used as a measure of the total strain.

FIGURE 2
Four-Point Bending Jig

All measurements and observations, including replication, were made while the sample was under load.

Observations by both optical and replica electron microscopy were made at various stages of bending. Carbon negative replicas shadowed with uranium were taken from plastic intermediate replicas of the two perpendicular polished surfaces. The operative slip systems and slip line density were determined by optical metallography. The depth and width of the slip lines were obtained from replicas that had been sprayed with latex balls of known size before shadowing. After the final deformation

sections for observation by transmission in the electron microscope were cut from the center portion of the bent crystals and thinned by a technique described previously.⁽⁸⁾

RESULTS

Optical Microscopy

Crystal A was bent in the orientation shown in Figure 3. The appearance of the slip lines on the two faces is shown in the optical micrographs in Figures 4a and 4b.

FIGURE 3

Schematic Representation of Crystallographic Directions
of the Slip Traces on Molybdenum Crystal A

There are two important features of the surface markings:

- The lines on both faces are distinct and appear after very low deformations
- The lines on the $(1\bar{1}0)$ surface are straight; whereas, those on (001) are branchy and irregular.

If a straight line path is constructed for the major segments of the slip lines on the (001) face of the crystal, then these slip traces correspond to $(011)[111]$ and $(011)[11\bar{1}]$. Slip on some other system must be responsible for the very short, branching segments, but its identity could not be determined by optical metallography. The resolved shear stress is slightly greater on the $\{112\}$, $0.46\sigma_0$, than on the $\{110\}$, $0.416\sigma_0$; however, only slip on $\{110\}$ was identified. The results are in agreement with other

a. (001) Face

b. ($1\bar{1}0$) Face

FIGURE 4

Slip Lines on Irradiated and Bent Molybdenum
Single Crystal A 120 X

observations in which the $\{110\} \langle 111 \rangle$ is found to be the predominant slip system in the bcc metals. (9)

The density of slip lines as measured optically on the $(1\bar{1}0)$ face increases with increasing deformation. Figure 5 is a plot of the density of lines versus deflection. At higher deformation where clustering of lines is most prevalent, lines separated by less than 0.040 mm were considered as one line.

FIGURE 5

Increase in Slip Line Density as a Function of Strain

Both the density and the width of the lines decrease markedly near the neutral axis. The slip lines appear to bend so as not to cross the neutral axis. The general appearance is one of slip lines originating at the surface, splitting, and tapering off near the neutral axis, as revealed by the micrograph of the (001) surface of Crystal A.

In the unirradiated crystal of the same orientation as Crystal A, no slip lines were observed by optical metallography. A very diffuse waviness of the surface was present. The development of slip lines on molybdenum has been reported.^(9, 10) Generally, the lines are fine and diffuse and apparently only form at higher values of strain than used in these experiments.

Crystal B, containing carbon and of a different orientation than Crystal A, revealed similar behavior when bent in an identical manner as Crystal A. A few distinctions can be pointed out, however.

Because straight, fine lines and coarse, irregular bands both formed on the same surface, it was concluded that at least two principal slip directions were operating initially on Crystal B as compared to only one on Crystal A, and it was evident that both $\{112\}$ and $\{110\}$ slip planes were operative during the initial deformation. The gross irregularity of the slip bands did not allow determination of the slip system with complete certainty. The slip lines best fit the systems $(10\bar{1})(011)[11\bar{1}]$, $(11\bar{2})[111]$, and $(112)[11\bar{1}]$.

On the average, the slip lines were narrower and their density higher after equivalent strains in Crystal B (Figure 5).

Replica Electron Microscopy

On the replicas, the slip lines on the (001) surface appear as broad bands with many side branches, and on the $(11\bar{0})$ surface they appear as narrow straight bands (Figures 6a and 6b). Fine lines within the broad bands were not resolvable; in some places, islands of undeformed material were present within the bands. Clustering of the lines increased with increasing deformation. The new slip lines formed in the region where slip had already occurred (Figure 6b).

The width of slip bands as revealed on replicas prepared from identical regions of the specimen increased with increasing deformation. The maximum width of bands present in Crystal A after the final strain ($\sim 5\%$) was 4.0μ as measured directly on the replica.

a. Broad Bands;
(001) Face
3200 X

b. Fine, Straight
Line; (110)
Face
7000 X

FIGURE 6

Replica of Irradiated and Bent Crystal A

The replicas of Crystal B containing carbides revealed coarse, irregular slip bands "flowing" around carbides (Figure 7 a). The straight slip lines disappear or fan out in the region near the carbides (Figure 7 b). Short slip line segments which appear to emanate from the carbides are also revealed in Figure 7 b.

The average angle between the surface of the slip line and the surface of the crystal was determined from the change in shadow length of latex balls located near the slip bands on the replicas. With reference to Figure 8, the angle, θ , made by the slip band is calculated from the equation,

$$\tan \theta = \frac{(\ell_1 - \ell_o) d}{\ell_o \sqrt{\ell_1^2 - d^2}}$$

All values of θ for the (001) surface ranged from 20° to 30° . The values of θ were not a function of the width of the line nor of the strain. From these measurements it must be concluded that slip occurs on very closely spaced parallel planes. If slip were occurring predominantly on one single plane, θ would be 45° . Similar measurements on $(1\bar{1}0)$ could not be made because θ was too small.

Transmission Electron Microscopy

Thin sections, perpendicular to the principal slip direction $(11\bar{1})$, were cut from the bent crystal and observed by transmission electron microscopy. Clear channels, caused by the passage of dislocations through the defect structure, are shown in Figure 9.

Crystallographic analysis showed the large, main channels to be parallel to the (011) and (101) planes. This is in agreement with the results deduced from the optical observation of slip bands on the surface. Some short segments were parallel to $\{112\}$ type planes as indicated in Figure 9. The similarities in the spacing, width, and general appearance of the channels and slip bands are evidence that the dislocation motion responsible for the channels is also responsible for the slip bands.

a. Effect of
Carbides on
Slip Lines;
(110) Face
3200 X

b. Note Dark
Lines
Emanating
from
Carbide;
(110) Face
3200 X

FIGURE 7

Replica of Irradiated and Bent Crystal B

- d = diameter of ball
 l_0 = shadow length on slip line
 l_1 = shadow length without line
 θ = angle of inclination of slip step to surface

FIGURE 8

Cross Section of Replica of Slip Step Illustrating the Change in Shadow Length of Latex Balls

FIGURE 9

Electron Micrograph of Section Cut from Bent Crystal A Illustrating Channel Formation Through Irradiation-Produced Defects (The traces of the slip planes are noted.) 30,000 X

The density of dislocations was generally highest at the edges of the channels and at the intersection of channels. No direct evidence was obtained on the precise source of the channels. Tapering off of the channels to a point would be expected near the neutral axis; this was confirmed. Many dislocations were also located at the taper point.

Thin foils prepared from Crystal B showed channels similar to Crystal A. The channels corresponded to $\{110\}$ and $\{112\}$ planes. Only occasional small carbides were seen; the larger ones were presumably lost during thinning. No evidence of source operation from a carbide was seen in the thin foils.

Sections were also cut from the bent unirradiated crystal, thinned, and examined in the electron microscope. Isolated dislocation tangles were observed (Figure 10) but no well-defined cell structure had formed. From changes in contrast during tilting of the foils, it was concluded that each tangle is composed primarily of dislocations with the same Burgers vector. Because tangles were more closely spaced than channels in the irradiated crystals, it was also concluded that no relationship exists between tangles and channels. Nothing suggestive of restricted dislocation motion leading to formation of a channel was noted in the deformed, unirradiated crystal.

DISCUSSION

The development of channels in unirradiated materials can be explained in the following manner. The initial dislocations interact with a certain number of the defect clusters encountered during glide. Some of the defects are absorbed or annihilated. Subsequent dislocations will tend to follow in the same regions as the first dislocations because they prefer to move in these regions where the defect concentration has been reduced. The channels then represent the least restrictive path for future dislocation motion. The formation of channels can explain the high initial yield point and long easy glide region observed in some irradiated single crystals. (3, 5)

The formation of deep, well defined slip lines results naturally from channeling. A large number of dislocations moving in the restricted volume

FIGURE 10

Electron Micrograph of Section Cut from Bent Unirradiated Crystal
Illustrating Isolated Dislocation Tangles 30,000 X

of the channel produce considerable offset at the surface at low values of strain. The correlation between slip lines and the channels demonstrates that for this particular case, bulk behavior can be deduced from surface observations of slip bands. Similar conclusions have also been reached by Seeger, et al. ⁽⁶⁾

It is not possible to reconstruct the paths of dislocations in the unirradiated material. Therefore, it is not possible to make a conclusive comparison between the result of dislocation motion observed in unirradiated molybdenum and irradiated molybdenum containing spot defects. The very tangled nature of the dislocation array in the unirradiated crystal suggests much cross slip and/or intersection of the dislocations. At low values of strain the dislocation resistance is uniform in the unirradiated crystal; hence the dislocation distribution is homogeneous and slip bands are faint and diffuse.

The lateral growth of the slip bands and channels in the irradiated molybdenum occurs through dislocation motion near the edge of the channel where probability of "sweeping up" defect clusters is high. Makin⁽¹¹⁾ recently has analyzed the interaction between moving dislocations and loops and has found that loops and dislocations need not be in direct contact for sweeping to occur; a dislocation can attract nearby loops. Channels may also widen by the merging of two parallel channels. At some stage during the merging, islands of undeformed structure defects should be present, both within channels and in slip bands. This has been verified.

Cross-slip of the dislocation onto another major slip plane results in branching of the channels. It is not known precisely what induces the dislocation to cross-slip at a particular place in the matrix. Because the resolved shear stresses on the $\{110\}$ and $\{112\}$ are nearly equal, small barriers would be sufficient to induce cross-slip. In Crystal B, the carbides are an effective barrier, as shown by the curved slip lines. The dislocations which cross-slip to form a new channel may eventually, through double or triple cross-slip, rejoin the original channel. Analogous observations have been made in iron where the splitting up and rejoining of the slip bands, with no change in slip direction, was observed.⁽¹²⁾ Any configuration of line or channel can be constructed by allowing the dislocation to cross-slip on the appropriate planes. It is generally found, however, that the channels or lines which appear curved can be separated into segments which are parallel to $\{110\}$ or $\{112\}$ planes.

The formation of straight lines and wavy lines on different surfaces of deformed crystals has been observed in other bcc metals.⁽¹³⁾ When the Burgers vector of the gliding dislocation makes a high angle with the surface, the jogs produced through cross-slip are reproduced on that surface, as on the (001) face of Crystal A. When the Burgers vector is near parallel to the surface, as for the $(1\bar{1}0)$ face of Crystal A, the lines are straight.

Both the width and density of the slip bands decrease on the face intersecting the neutral plane of the bent crystal as the neutral axis is approached. This fact supports the conclusion that the channels originate at or near the

outer surface, e. g., the $(1\bar{1}0)$ face of Crystal A. This is in agreement with etch pit work on bent crystals on which the etch pit density decreases from the outer surface toward the neutral axis.⁽¹⁴⁾

In the sample containing carbon, the carbides also act as dislocation sources. This is shown both by the increased slip line density in this material and the slip line segments which appear to emanate from the carbides. Friedel has proposed that particles can act as points of concentrated stress, and that dislocation loops can be emitted from particles under the application of external stress.⁽¹⁵⁾ Accordingly, dislocations originating at a particle located at or just below the surface could produce some of the slip line segments observed.

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