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AND RECOVERED MOLYBDENUM

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AND RECOVERED MOLYBDENUM

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ABSTRACT

Specimens suitable for transmission electron microscopy have been prepared from bulk polycrystalline molybdenum after tensile deformation and recovery. The resulting dislocation substructures are described. Some tentative conclusions concerning the mechanism of plastic deformation in molybdenum are discussed.

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INTRODUCTION

The critical stress for slip in a crystalline material is partly determined by the dislocation substructure that is present. Increase of the critical stress for slip accompanying plastic deformation is associated with changes in substructure. Therefore, a theory of strain hardening requires knowledge of the density and distribution of dislocations as a function of strain. In the past, theories have been based on assumptions concerning development of substructure with strain.

It is now possible to observe the arrangement of dislocations directly by transmission electron microscopy. Therefore, it should be possible to determine whether or not the current models used to explain strain hardening and the temperature dependence of flow stress are realistic. The shape of the stress strain curve varies greatly from one material to another. It depends on crystal structure, purity, thermal history, and even the characteristics of the testing machine. So far, transmission electron microscopy has not succeeded in shedding much light on the reasons for these differences. With the exception of those FCC metals that have very low stacking fault energies, the development of dislocation substructure with increasing strain is strikingly similar in a large number of face-centered and body-centered cubic metals. It had been thought that the greater hardening capacity of FCC metals compared to BCC materials was associated with the possibility of forming segments of Lomer Cottrell dislocation which are immobile and therefore might act as particularly effective barriers. Transmission electron microscopy has provided some evidence for this dislocation interaction,⁽¹⁾

but at the same time, has tended to minimize its importance. It is difficult to argue that this particular reaction is of supreme importance because the kind of deformation substructure that has been observed in iron (BCC)⁽²⁾ in which Lomer Cottrell dislocations can not be formed, is at least qualitatively similar to that in copper and silver (FCC)^(3, 4). In spite of these preliminary results, it seems likely that more carefully controlled quantitative observations on polycrystalline and single crystal specimens will eventually be able to reveal the important parameters that influence the propagation of slip from one grain to another and from one subgrain to another. The present results are some preliminary observations on the kinds of dislocation substructures that exist in polycrystalline molybdenum after deformation and recovery.

RESULTS

The material was .002" molybdenum sheet which was obtained from Fansteel Metallurgical Corporation in the cold rolled condition. The chemical and spectrographic analysis (%) of the material was as follows: C 0.022, H₂ 0.0005, N₂ 0.0014, O₂ 0.014, Cr 0.003, Ca < 0.002, Ba < 0.003, Mg < 0.0005%. The as-received sheet was annealed in vacuum to give a recovered or a recrystallized structure. Dislocation substructures were examined by transmission electron microscopy in a Hitachi HU-10 microscope operating at 100 kv. Final thinning of the foils was accomplished by electropolishing at 15 to 25 volts in 98% H₂SO₄. Annealing the cold rolled sheet for one hour at 1200° C produced a system of sharp subgrain boundaries within the elongated grains and a few recrystallized regions. Relatively few dislocations remained

within the subgrains which were approximately 10 μ in diameter. There was a sharp preferred orientation. Nearly all the grains had a $\{100\}$ plane parallel to the surface of the sheet within a few degrees. Fig. 1 shows an example of this recovered structure. Complete recrystallization occurred after heating to 1400°C. The recrystallized grain size was about 50 μ . The high recrystallization temperature suggested the presence of second phase particles even though they were not directly observed. The annealed strip was deformed 1-10% in tension using an Instron machine at a strain rate of 0.01 ins/min.

Small amounts of plastic deformation in tension, following recovery, first resulted in the appearance of long irregular dislocation lines particularly within the larger subgrains or recrystallized grains. It was clear that more than one slip system was operative from the very start of deformation. Three fold nodes which probably resulted from the dislocation reaction:

$$\frac{a}{2} [111] + \frac{a}{2} [\bar{1}\bar{1}\bar{1}] \rightarrow a [100]$$

were a prominent feature of the substructure after 2% plastic strain. Fig. 2 is an example of networks forming a twist boundary by dislocation reactions of this type. The plane of the foil is approximately (001) and the network A makes a trace along $[100]$. Similarly at B the trace of the network is along $[010]$. These traces are consistent with the traces of the (110) and (101) planes which lie at 45° to the foil surface. The features at C, D and E all point to the fact that dislocation intersections occur even after 1% strain and may indicate that the networks at A and B are formed during the initial deformation. Resultant $[100]$ segments

formed at intersections may also act as nuclei for growth of more tangled networks like those shown in Fig. 3(a) and (b).

Examination of many areas after a few percent strain showed that irregular patches of twist boundary were often formed in planes other than $\{110\}$. Fig. 3(b) is an example. The foil surface is (001) and the networks plane appears to have a trace parallel to $[110]$. This means they cannot lie in $\{110\}$ since the $\{110\}$ planes having a trace on (001) of $\langle 110 \rangle$ are at right angles to the foil surface. Networks on these planes would not be observed. These fragments of twist boundary could lie in $\{111\}$ or $\{112\}$ planes.

Another notable feature in deformed specimens was the sharp kinks and cusps present on many of the isolated dislocation lines. Their irregular shapes suggested that expansion of a loop of dislocation in molybdenum is accompanied by frequent cross-slip, and by intersections between dislocations of opposite Burgers vector. The result of such an intersection is represented schematically in Fig. 4. Both of these processes can cause adjacent segments of a dislocation line to lie on different glide planes. Cross-slip was directly observed when dislocations moved due to heating of the electron beam. However, no traces were left behind by moving dislocations so it was not possible to make a photographic record of the glide paths. Another indication of frequent cross-slip was the formation, during deformation, of a large number of irregularly shaped closed loops of dislocation line. Fig. 5 shows some particularly good examples. Although loops are occasionally seen in the deformation substructure of FCC metals, they

do not form in such large numbers at such small strains. The irregular shapes of the dislocation lines suggested that loops may have been formed by the mechanism recently proposed for similar closed loops in deformed magnesium oxide⁽⁵⁾. This mechanism is illustrated in Fig. 6. Local double cross-slip around an obstacle in the slip plane can produce long jogs on a moving screw dislocation. If the jogs are not able to glide off the screw part of the loop, cusps will be formed; the dislocation bowing out on either side of the jog. Subsequent double cross-slip may then leave behind a closed loop or further increase the separation between the glide planes of adjacent segments of the dislocation depending on the direction of the next cross-slip. If the spacing between the glide planes of adjacent segments of the same dislocation line becomes greater than a critical value, (approximately:

$$H = \frac{Gb}{8\pi\tau}$$

where τ is the resolved shear stress on the slip plane, G is the shear modulus and h is the separation normal to the slip plane between the two parts of the dislocation) then the applied stress can drive the two parts of the dislocation past each other, spreading slip across two surfaces instead of just one. It is likely that this is an important dislocation multiplication mechanism in molybdenum.

In a polycrystalline material, particularly at small strains, it is impossible to accurately relate the local strain in a region as small as that observed in transmission microscopy to the measured average macroscopic strain. The local stress field also varies from one

region to another, not only in magnitude but also in the directions of the principal stresses. As might be expected, the dislocation substructure differed widely from one point to another as shown in Fig. 7 (a,b). Both pictures correspond to 2% average plastic strain.

Even after the smallest plastic strains, there was a tendency toward clustering of dislocations. This can be seen at an early stage in Figs. 3a, 3b and 7a. Close pairs of edge dislocations and fragments of twist boundary begin to separate the larger subgrains of the recovered structure into a system of smaller subgrains having average linear dimensions of $1/2$ to 1μ . Fig. 8 (a) and (b) shows regions of low dislocation density separated by a rather complete network of high density boundaries after 10% macroscopic strain. Most boundaries had a dislocation structure similar to that shown at high magnification in Fig. 9: a two dimensional tangle of joggy dislocations. With increasing strain, many of the dislocation spacings within boundaries became too small to be resolved. Ten percent was the greatest uniform tensile strain that could be achieved before the start of necking in the thin sheet specimens. With heavier plastic strains by rolling the dislocation density continued to increase. Foils prepared from molybdenum sheet cold rolled to 50% reduction in thickness were completely opaque because of the high dislocation density. Therefore, even after a system of subgrains of the order of one or two microns in diameter had become established, new dislocation tangles continued to develop in the relatively clear areas until the entire volume became

too thickly populated with dislocations to permit further observations. The average dislocation density was observed to increase from about 1×10^9 to about 8×10^9 as the strain was raised from 1 - 10%.

The effect of reheating to 300°C, 600°C, 900°C and 1200°C after 5% plastic strain was investigated. Because of the extreme variation in substructure from one point to another in the deformed condition it was impossible to determine, with accuracy, the minimum temperature at which rearrangement of dislocations began. However, there was some indication that even the 300°C anneal may have resulted in changes. Figure 10 was obtained from a foil which was held at 300°C for three hours. None of the as-deformed specimens that were examined had a comparable density of closed loops. The low temperature anneal may have allowed some additional annihilation of close screw dislocation pairs leaving behind small loops where edge dislocation jogs existed. A schematic illustration of this process is shown in Fig. 11. Even after heating to 600°C some of the dislocation tangles appeared to be pretty much unchanged. Those in the lower right part of Fig. 12 still closely resemble the as-deformed clusters. However, this photograph also shows one of the most regular tilt boundaries that was observed. It is not entirely clear whether this boundary was formed as a result of the five percent strain and 600°C anneal or whether it was formed during the previous 1200°C recovery anneal. Many examples can be seen of individual dislocation lines leaving the plane of the boundary. There are also several nodes within the boundary where two adjacent dislocations join to form a single line of resultant Burgers vector. This indicates the presence of at least two different $\frac{a}{2} \langle 111 \rangle$

Burgers vectors in the boundary. The resultant segments where they join are presumably of the type $a \langle 100 \rangle$. Figure 13 shows another area in the same foil. Comparison with Fig. 12 illustrates again the great differences in substructure from one point to another. Here the annealing treatment appears to have produced a definite decrease in dislocation density and also to have caused straightening out of many of the kinks which were characteristic of the deformation structure.

Heating to 900°C definitely decreased dislocation density. Those dislocations that remained tended to run straight from one node to another, as in Fig. 14. The small segment of twist boundary is one of the most perfect examples that was observed. Patches of twist boundary that form during deformation might be expected to be relatively stable during annealing. Figure 14 at A also shows an elongated loop which may be breaking up into smaller loops as indicated in Fig. 6.

SUMMARY

The development of dislocation substructure in molybdenum is qualitatively similar to that which occurs during plastic deformation of iron and even copper, and silver. Dislocations become heavily jogged during motion. Local tangles begin to develop in which attractive intersections caused by the dislocation reaction $\frac{a}{2} [111] + \frac{a}{2} [\bar{1}\bar{1}\bar{1}] = a [100]$ appear to play an important role. The local tangles develop into a system of subgrain boundaries with increasing strain; the majority of the dislocations being concentrated at the boundaries at strains below about 10%. However, new tangles continue to develop within the relatively clear volumes so that no regions of low dislocation density remain after

50% reduction in thickness by rolling. The formation of large numbers of closed loops at small strains is evidence for frequent cross-slip and suggests that double cross-slip is an important mechanism for spread of slip from one glide plane to nearby parallel planes.

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FIGURE TITLES

- Fig. 1 Grain boundary structures produced by heating rolled Mo sheet to 1200°C for one hour.
- Fig. 2 Fragments of twist boundary produced by 1% plastic strain in tension after recrystallization at 1400°C. Foil surface approximately (001).
- Fig. 3 Dislocation substructure after 2% plastic strain, (a) tangled groups containing many a $\langle 100 \rangle$ resultant dislocation segments, (b) irregular twist boundary fragments.
- Fig. 4 Schematic illustration of the intersection between dislocations A, B, of opposite Burgers vector. This intersection leaves no resultant dislocation.
- Fig. 5 Dislocation loops left behind by moving dislocations. (5% strain after 1200°C anneal.)
- Fig. 6 Schematic illustration showing the formation of dislocation loops from a moving screw dislocation containing a long jog.
- Fig. 7 (a, b) Variation in substructure from one region to another after 2% average plastic strain.
- Fig. 8 (a, b) Substructure boundary tangles separating relatively perfect regions after 10% strain.
- Fig. 9 Typical tangled arrangement of dislocations in subgrain boundaries. (Annealed 3 hours at 600°C after 5% tensile strain.)
- Fig. 10 Large number of closed loops observed after 5% strain followed by heating to 300°C for one hour.

FIGURE TITLES (cont'd)

- Fig. 11 Schematic illustration showing the formation of dislocation loops from long dislocation pairs of opposite sign.
(I-III) dislocation pair close to screw orientation;
(IV-V) pair close to edge orientation.
- Fig. 12 Regular tilt boundary and irregular clusters after 5% strain followed by heating to 600°C for 3 hours.
- Fig. 13 Another area of the same specimen shown in Fig. 12.
- Fig. 14 Segment of twist boundary remaining after heating to 900°C for 3 hours after 5% plastic strain.