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SUMMARY

Deformation substructures of polycrystalline tungsten, tungsten - 2, 9, and 24 weight percent rhenium, and tungsten - 3 weight percent tantalum were studied by transmission electron microscopy. The stress-strain curve for unalloyed tungsten shows gradual yielding followed by work hardening. Electron micrographs indicate a gradual increase in dislocation density with increase in strain up to 5.0 percent. Dislocations, although frequently jogged, are straight over moderate distances and are in a random array.

Stress-strain curves for alloy specimens of tungsten - 2 and 9 percent rhenium and tungsten - 3 percent tantalum exhibit a drop in stress at yielding followed by only slight work hardening. Electron micrographs of these specimens reveal the absence of dislocations at strains of 0.05, 0.1, and 0.5 percent. After 2.0 percent strain, the three alloys exhibit dense dislocation networks. Tungsten - 3 percent tantalum is characterized by straight, frequently jogged dislocations comparable with the dislocation structure in unalloyed tungsten after a similar amount of strain. In contrast, tungsten - 2 percent rhenium exhibits dislocations that contain widely spaced jogs, while tungsten - 9 percent rhenium developed a cell structure after the relatively small strain of 2.0 percent. The tungsten - 24 percent rhenium alloy contained a few dislocations after 0.1 percent strain, while after 0.5 percent strain, twins were evident. Dislocation slip bands apparently preceded the twins. The stress-strain curve for the alloy indicates that twinning commenced after approximately 0.25 percent strain.

These results indicate that the primary effect of low rhenium concentrations (2 and 9 percent) in tungsten is to increase dislocation multiplication after macroyielding by reducing the Peierls-Nabarro force (lattice resistance to dislocation motion).

The dislocation bands that precede twins in tungsten - 24 percent rhenium may be caused by localized internal stresses resulting from a metastable structure, for example, clustering of rhenium atoms.

INTRODUCTION

The effect of high rhenium additions (22 to 39 at. % Re) on the mechanical properties of Group VI A metals is well documented (refs. 1 to 3). A substantial reduction in the ductile to brittle transition temperature of tungsten, from 675° F (603° K) to room temperature, results from alloying with approximately 28 atomic percent rhenium (ref. 1). In addition, fabricability of the Group VI A metals improves upon alloying with 25 to 35 atomic percent rhenium (ref. 1). Group VI A metals containing rhenium concentrations near the maximum solubility in chromium, molybdenum, or tungsten deform, at low temperatures, primarily by twinning (ref. 3).

Several investigators have reported on the effects of low rhenium additions (2 to 9 percent) on mechanical properties of tungsten (refs. 4 to 7). Klopp, Witzke, and Raffo (ref. 5) reported bend transition temperatures as low as -100° F (200° K) for dilute electron-beam-melted tungsten-rhenium alloys tested in the worked condition. Recrystallization increased the bend transition temperature, but alloys with 2 to 4 percent rhenium were still markedly superior to unalloyed tungsten. Fractographic examinations of tungsten and tungsten - 3 percent rhenium and tungsten - 5 percent rhenium alloys by Gilbert (ref. 6) revealed that these low rhenium alloys showed a greater tendency toward cleavage failure than did tungsten. Garfinkle (ref. 7) showed that rhenium additions, up to 9 percent, to $\langle 100 \rangle$ oriented tungsten single crystals increased the proportional limit stress and decreased the flow stress and the rate of work hardening. In addition, while deformation in unalloyed $\langle 100 \rangle$ oriented crystals apparently involved both $\{110\}$ and $\{112\}$ slip, crystals with rhenium contents of 5 atomic percent or more deformed primarily by $\{112\}$ slip.

The mechanism by which high and low rhenium additions affect the mechanical properties of tungsten is still not well established. The present investigation was undertaken to determine by transmission electron microscopy the effects of low rhenium additions, 2 and 9 percent, and a high rhenium addition, 24 percent, on dislocation substructure in the early stages of deformation of polycrystalline electron-beam-melted tungsten. Unalloyed tungsten and a tungsten - 3 percent tantalum alloy were included for comparison.

EXPERIMENTAL PROCEDURES

Materials

Triple electron-beam-melted tungsten, tungsten - 2, 9, and 24 percent rhenium and tungsten - 3 percent tantalum were used for this investigation. Chemical analyses of the cast ingots are given in table I. A description of the starting metal powders and melting

TABLE I. - CHEMICAL ANALYSES OF

ELECTRON-BEAM-MELTED INGOTS

Element	Ingot composition (nominal wt. %)				
	Tungsten	Tungsten - 2 percent rhenium	Tungsten - 9 percent rhenium	Tungsten - 24 percent rhenium	Tungsten - 3 percent tantalum
	Ingot composition (analyzed wt. %)				
	--------	Tungsten - 1.9 percent rhenium	Tungsten - 9.1 percent rhenium	Tungsten - 24 percent rhenium	Tungsten - 3 percent tantalum
	Impurity content[a], ppm				
Oxygen	8	10	1	8	4
Nitrogen	7	3	3	3	5
Carbon	2	4	4	4	5
Aluminum	.1	--	.1	--	--
Beryllium	.1	--	.003	--	--
Calcium	.2	--	.1	--	--
Chromium	.2	--	2.2	--	--
Cobalt	.1	--	.01	--	--
Copper	.1	--	.01	--	--
Iron	.1	--	.5	--	--
Magnesium	.1	--	.001	--	--
Manganese	.1	--	.004	--	--
Molybdenum	1	--	95	--	--
Nickel	.1	--	.1	--	--
Niobium	.1	--	.3	--	--
Silicon	.1	--	.2	--	--
Titanium	.1	--	.02	--	--
Vanadium	.1	--	.03	--	--
Zirconium	.1	--	.01	--	--

[a]Oxygen analysis, vacuum fusion; nitrogen analysis, Kjeldahl; carbon analysis, combustion and conductometric; all other analyses, emission spectrography.

and fabrication procedures for unalloyed tungsten and the tungsten-rhenium alloys is reported in reference 5. The tungsten - 3 percent tantalum alloy was processed in a similar manner. Compression specimens measuring 0.300 inch (7.6 mm) in length by 0.130 inch (3.3 mm) in diameter were machined from swaged rods. All alloy specimens were annealed in a vacuum of 8×10^{-6} torr (10^{-2} N/m^2) for 1 hour at 3600° F (2255° K). The recrystallized grain size ranged from 0.06 to 0.08 millimeter in diameter for the alloy specimens. Unalloyed tungsten was annealed at 2400° F (1589° K) for 1 hour to produce a recrystallized grain diameter of approximately 0.12 millimeter. Specimens were electropolished in a 2 percent sodium hydroxide solution to a diameter of 0.125 inch (3.18 mm) to remove surface notches resulting from grinding and to improve reproducibility of the data. The ends of the compression specimens were ground flat, parallel to each other, and perpendicular to the longitudinal axes with 4/0 emery paper.

Compression Tests

The compressive stress-strain apparatus used for compression tests is shown schematically in figure 1. This apparatus is described in detail by Stearns and Gotsky (ref. 8). Room temperature compression tests were conducted at a crosshead speed of 0.01 inch per minute (0.25 mm/min). The apparatus could measure displacements of 5×10^{-7} inch (1.3×10^{-5} mm) and thus permit accurate reproducibility of predetermined strains for the compression specimens. Specimens were normally compressed to strains of 0.05, 0.1, 0.5, and 2.0 percent, unloaded, and prepared for observation in the electron microscope. In addition, unalloyed tungsten was strained to 5.0 percent and a tungsten - 9 percent rhenium specimen was strained to 1.0 percent. True stress - true strain curves were computer calculated from the load-compression plots by assuming uniform deformation and a constant volume for the compression specimens.

Thin Foil Preparation

Disks 0.015 inch (0.38 mm) thick were cut from deformed compression specimens by spark machining. The disks were cut at a 45° angle to the compression axes (plane of maximum shear stress) to maximize the probability of the foil plane coinciding with an operative slip plane. When these planes coincide and also satisfy diffraction conditions, the projected lengths of dislocations are maximized, and the substructure is more readily discernible. The disks were jet polished by means of a stream (jet) of electrolyte impinging upon the center of the disk, which produced a dimple in the disk. The disk was then inverted and the process repeated on the other side until the thickness between dimples was approximately 0.003 inch (0.076 mm). After dimpling, the disk was bath polished until a hole appeared at the thin spot between the two dimples. Electropolishing was stopped at the first appearance of light through the disk as seen through a ×20 telescope. The edge of the hole lends itself to observation in the electron microscope.

A solution of 2 percent sodium hydroxide was used as the electrolyte for both jet polishing and bath polishing. A direct current potential of 350 volts was used for the jet-polishing process and approximately 12 volts for the bath-polishing process. It was necessary to add 20 volume percent of 30 percent hydrogen peroxide to the 2 percent sodium hydroxide electrolyte, to electropolish the tungsten - 3 percent tantalum alloy.

An electron microscope operated at 100 kilovolts was used to observe the thin foils.

RESULTS

Stress-Strain Behavior

Specimens of unalloyed tungsten, tungsten - 2, 9, and 24 percent rhenium, and tungsten - 3 percent tantalum were compressed to total strains of 0.05, 0.1, 0.5, and 2.0 percent. Unalloyed tungsten was also compressed to 5.0 percent strain and tungsten - 9 percent rhenium to 1.0 percent strain. Figure 2 is a plot of true stress against true strain for unalloyed tungsten and each of the four alloys up to 2.0 percent strain.

The stress-strain curve for unalloyed tungsten in figure 2 is characterized by gradual yielding followed by rapid work hardening. The stress-strain curves for alloys of tungsten - 2 and 9 percent rhenium and tungsten - 3 percent tantalum exhibit a slight drop in stress at yielding, a large Luder's region, and the start of work hardening. The stress-strain curves for polycrystalline unalloyed tungsten and the dilute tungsten-rhenium alloys are similar to the curves for $\langle 100 \rangle$ oriented single crystals reported by Garfinkle (ref. 7) but show higher proportional limit stresses. In addition, slight work hardening occurred after the Luder's strain in this study, while Garfinkle observed practically no work hardening. These differences in behavior are attributed to the increased difficulty of slip caused by grain boundaries in the polycrystalline specimens.

Figure 2 shows that although tungsten - 3 percent tantalum has a very high proportional limit stress, the shape of the stress-strain curve is very similar to those for the two dilute tungsten-rhenium alloys. Discontinuous yielding in these alloys may be due to dislocation pinning by rhenium and tantalum atoms.

The stress-strain curve for the tungsten - 24 percent rhenium alloy is markedly different from those for the dilute alloys. This alloy deformed primarily by twinning after approximately 0.25 percent strain (fig. 3). Audible twinning occurred coincident with the horizontal displacements at A. Jaffee, Maykuth, and Douglass reported that twinning occurs in tungsten - 22 to 30 percent rhenium over the temperature range 70° to 750° F (294° to 672° K) (ref. 1).

Transmission Microscopy

The dislocation substructure for unalloyed tungsten after increasing amounts of strain is shown in figure 4. Short isolated dislocation loops were observed after a strain of 0.05 percent, as shown at A in figure 4(a). The partial loops at B are caused by intersection of the dislocation loops with the top and bottom surfaces of the foil. After a deformation of 0.1 percent, the dislocation density increased and the dislocations were characteristically straight over moderate distances (fig. 4(b)). Compressing to 0.5 percent strain produced a higher dislocation density than for the two lower strains and again straight dislocations were observed (fig. 4(c)). After 2 percent strain, the density increased still further, and dislocations were typically straight over moderate lengths (fig. 4(d)). However, the dislocations show many cusps as at A with some tangling of dislocations as at B. Figure 4(e) shows the substructure developed in unalloyed tungsten after 5.0 percent strain. Dislocations have become more tangled as at A. Some straight dislocations are observed as at B and frequent cusps as at C. A cell structure has not commenced to form even after this amount of deformation.

As noted previously, tungsten - 3 percent tantalum and tungsten - 2 and 9 percent rhenium exhibit yield drops and higher proportional limit stresses than unalloyed tungsten. Examination of these three alloys failed to reveal a significant number of dislocations after strains of 0.05, 0.1, and 0.5 percent. Figure 5 is an electron micrograph of tungsten - 3 percent tantalum after 0.5 percent strain and is typical of all three alloys after a similar strain.

After 2.0 percent strain, tungsten - 3 percent tantalum exhibited a uniform dislocation distribution (fig. 6). The structure is similar to that of unalloyed tungsten after the same amount of strain. Again the dislocations were fairly straight over moderate distances with frequent cusps, as at A. Dislocation tangling, as at B, appears to have occurred more frequently in this alloy than in unalloyed tungsten. The dislocation structure observed in unalloyed tungsten and tungsten - 3 percent tantalum is typical of body-centered cubic metals after small amounts of strain (ref. 9).

The dilute tungsten-rhenium alloys exhibited a marked difference in dislocation structure as compared with unalloyed tungsten and tungsten - 3 percent tantalum. After 2.0 percent strain, the tungsten - 2 percent rhenium alloy contained widely spaced cusps along the dislocation lines, as shown at A in figure 7. In contrast, the dislocation lines in unalloyed tungsten had a much smaller cusp spacing. In tungsten - 9 percent rhenium, widely spaced cusps were observed on dislocation lines after a strain of only 1.0 percent, as at A in figure 8(a). This alloy developed a cell structure after a strain of only 2.0 percent (fig. 8(b)). The cell walls A are heavily tangled, while the cell interiors are relatively free of dislocations except for occasional loops at B.

Increasing the rhenium content in tungsten to 24 percent resulted in localized deformation by slip as shown by the apparent dislocation pileup illustrated in figure 9(a) after 0.1 percent strain. Further deformation to 0.5 percent strain resulted in twinning (fig. 9(b)). Since dislocation pileups were observed prior to twinning, it is assumed that the band of dislocations at A preceded the twin at B. The substructure observations are in agreement with the stress-strain curve, which indicated twinning at strains greater than 0.25 percent. Figure 9(c) also shows deformation by both twinning and slip at 2.0 percent strain. Again, dislocations at A apparently precede the twin at B.

The dislocation densities were determined for unalloyed tungsten, tungsten - 3 percent tantalum, and tungsten - 2 and 9 percent rhenium by using the relation given in reference 10:

$$\rho = \frac{2n}{Lt}$$

where

ρ dislocation density, cm^{-2}

n number of intersections which dislocations make with six random lines

L total length of the six random lines over which dislocation intersections are counted, cm

t foil thickness (a value of 2000 Å used for the calculations)

The variation of dislocation density with strain is shown in figure 10. The data for unalloyed tungsten and tungsten - 9 percent rhenium apparently lie on straight lines passing through the origin. The dislocation density for tungsten - 9 percent rhenium after a strain of 2.0 percent was measured in the cell walls. The density for the alloy after 2.0 percent strain was approximately four times greater than for unalloyed tungsten after a similar amount of strain. The measured dislocation densities for tungsten - 3 percent tantalum and tungsten - 2 percent rhenium after 2.0 percent strain are similar to the dislocation density for unalloyed tungsten. The linear dependence of dislocation density on strain has been observed in other body-centered cubic metals; for example, in iron a linear relation existed to 7.0 percent strain (ref. 9). It was also reported that dislocation density increased with decreasing grain size. At 2.0 percent strain in iron a twofold increase in dislocation density resulted from a sevenfold decrease in grain size. In this study, at a similar strain, the fourfold increase in dislocation density corresponds to only a twofold decrease in grain size from unalloyed tungsten to the tungsten - 9 percent rhenium alloy. This indicates that a factor in addition to grain size must account for the observed increase in dislocation density.

DISCUSSION

Dilute Rhenium Additions

Transmission microscopy revealed that the primary effect of dilute rhenium additions to tungsten was to decrease the density of cusps along dislocation lines and to produce a cell structure after only 2.0 percent strain. Normally, in the early stages of deformation (0 to 10 percent) of body-centered cubic metals, dislocations are frequently kinked and nonuniformly distributed (ref. 11). As deformation proceeds, dislocations become more nonuniform and tangled, and finally a cell structure develops. The formation of a cell structure in body-centered cubic metals at room temperature normally occurs after much higher strains than 2.0 percent as observed in tungsten - 9 percent rhenium. For example, Keh (ref. 12) reported that a partial cell structure was observed in iron after 9.0 percent strain. Berghezan and Fourdeux (ref. 13) reported the formation of a cell structure in columbium after 5.5 percent strain at room temperature.

Alloying normally suppresses the formation of a cell structure and results in a more uniform dislocation distribution (ref. 11). In the present investigation an addition of 3.0 percent tantalum to tungsten resulted in a slight increase in dislocation density. However, the dislocation structure was very similar to that of unalloyed tungsten, with dislocations distributed nonuniformly and with no evidence of cell formation. Van Torne and Thomas (ref. 14) reported that alloying tantalum with molybdenum produced a tendency for dislocations to lie in straight lines in the alloy as compared with cell formation in unalloyed tantalum. They interpreted these results to indicate that molybdenum additions to tantalum increase the Peierls-Nabarro force. These results are exactly opposite to those observed herein for the tungsten - dilute rhenium alloys. Therefore, it is suggested that rhenium additions to tungsten have the opposite effect; that is, they decrease the Peierls-Nabarro force in tungsten. An exception to the normally observed alloying effect reported by Leslie, Michalak, and Aul (ref. 15) is that manganese additions to iron promote cell formation. In addition, the Charpy notch impact transition temperature was reduced as a result of dilute (0.02 to 1.9 at. %) manganese additions to iron (ref. 16). This behavior is similar to the effects of dilute rhenium additions to tungsten.

Transmission microscopy revealed that, in unalloyed tungsten after 2.0 percent strain, dislocations lines contained frequent cusps. Similar cusps in silicon iron (ref. 7) have been interpreted as jogs in screw dislocations. Gilman and Johnston (ref. 18) considered the creation of dislocations by a multiple cross-slip process. In this process, a portion of a screw dislocation moves to a parallel slip plane by cross slip. The segment of dislocation that connects the parallel screw dislocation segments thus becomes the jog. The jog height determines the movement of the dislocation and the resulting dislocation multiplication. Figure 11, taken from reference 18, summarizes the movement of screw dislocations as a function of jog height M to N. As shown in figure 11(a), dislocations with small jog heights move nonconservatively because of the edge character of the jog and thus produce point defects (vacancies or interstitials). An intermediate jog height (fig. 11(b)) results in production of dipoles, while at a jog height of over 300 Å, the dislocations can act as single-ended sources (fig. 11(c)).

The production of jogs can be accomplished by cross slip of the screw dislocations from the primary slip planes onto secondary slip planes. The frequency of jogs and the lack of evidence for secondary slip at the jogs would indicate that this mechanism could not account for all jogs observed in unalloyed tungsten. An alternative mechanism suggested by Low and Turkalo (ref. 17) is that screw dislocations lie on the maximum shear stress plane, cross slip to the minimum energy planes (i. e., close-packed planes), and thus produce jogs.

The high frequency of cusps or jogs observed in this study on screw dislocations in unalloyed tungsten is attributed to the high Peierls-Nabarro force normally associated with the body-centered-cubic lattice. Dislocations lie in the maximum shear-stress plane as a result of being acted upon by the applied shear stress. However, because of the high Peierls-Nabarro force, they frequently cross slip into the close-packed planes (i. e., minimum energy positions). These competing forces could produce the cusps or jogs observed in figure 4(d) for unalloyed tungsten. No dipoles or single-ended sources are observed in unalloyed tungsten. In tungsten - 2 percent rhenium, the cusps are widely spaced, and this is interpreted as indicating that the Peierls-Nabarro force has been lowered. Shear stress is the major force acting on the dislocation, and, therefore, cross slip into the close-packed planes is less frequent. In addition to the widely spaced cusps or jogs at A in figure 8(a) for tungsten - 9 percent rhenium, dipoles are observed at B and pinched-off loops at C. Dislocations acting as single-ended sources were seen in other areas of the foil. The increase in dislocation mobility due to a reduction in the Peierls-Nabarro force allows formation of these super jogs (jog height $\gg$ 1 or 2 Burgers vectors) which can act as single-ended sources. This may account for the increase in dislocation density in the tungsten - 9 percent rhenium alloy as compared with unalloyed tungsten, where such sources were not operative.

The cell structure in the tungsten - 9 percent rhenium alloy can be attributed to the reduced resistance of the lattice and an increase in dislocation density resulting from jogs acting as dislocation sources. These two effects would increase the frequency of dislocation intersections and result in tangling and cell formation as observed after 2.0 percent strain.

High Rhenium Additions

As expected, the tungsten - 24 percent rhenium alloy deformed primarily by twinning at room temperature. A significant observation in this study was the presence of localized bands of slip dislocations preceding a twin. A similar observation was made by Votava (ref. 20) on a chromium - 35 percent rhenium alloy. The compositions of both these alloys lie near the maximum solubility of rhenium in the solvent metal. This proximity to the solvus line may lead to a metastable structure such as clustering of rhenium atoms or preprecipitation of sigma. This metastability in turn could cause high localized stresses that can act as dislocation and twin sources.

CONCLUSIONS

A transmission microscopy study of tungsten and tungsten alloys yielded the following conclusions:

1. The moderately straight dislocations in unalloyed tungsten at low strains and frequently jogged dislocations at 2.0 percent strain are attributed to the high Peierls-Nabarro force normally associated with the body-centered-cubic lattice.

2. Alloying with 3.0 percent tantalum apparently has the normal alloying effect of suppressing cell formation. The substructure was characterized by a uniform distribution of moderately straight dislocations, very similar to the structure observed in unalloyed tungsten at a strain of 2.0 percent.

3. The presence of dipoles and jogs acting as single-ended sources in tungsten - dilute rhenium alloys and the observation of fewer jogs than in unalloyed tungsten strained a similar amount are attributed to a reduction in the Peierls-Nabarro force. The formation of a cell structure after only 2.0 percent strain in tungsten - 9 percent rhenium arises from multiplication of dislocations at jog sources that produces more intersections, tangling, and subsequent cell formation.

4. Twinning in tungsten - 24 percent rhenium is preceded by bands of dislocations. Clustering of rhenium atoms or preprecipitation of sigma may cause high localized stresses that can act as dislocation and twin sources.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 7, 1967,
129-03-02-05-22.

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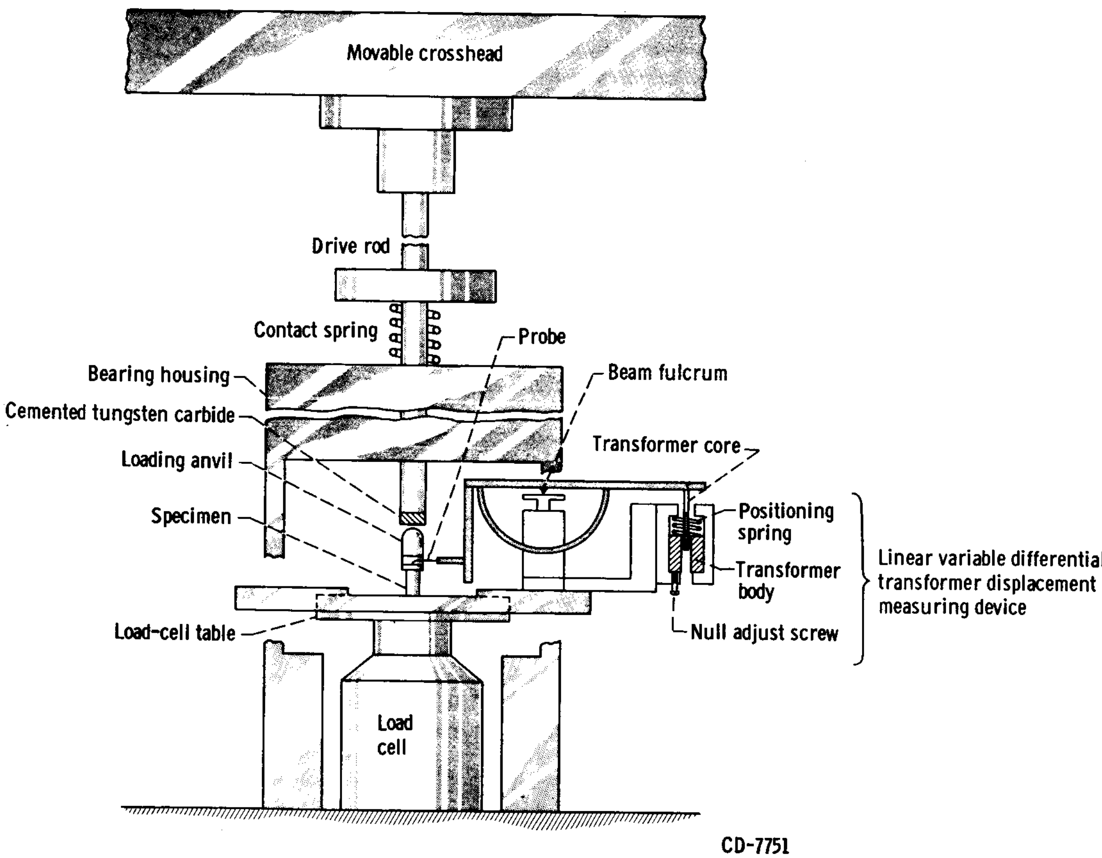


Figure 1. - Compressive stress-strain apparatus.

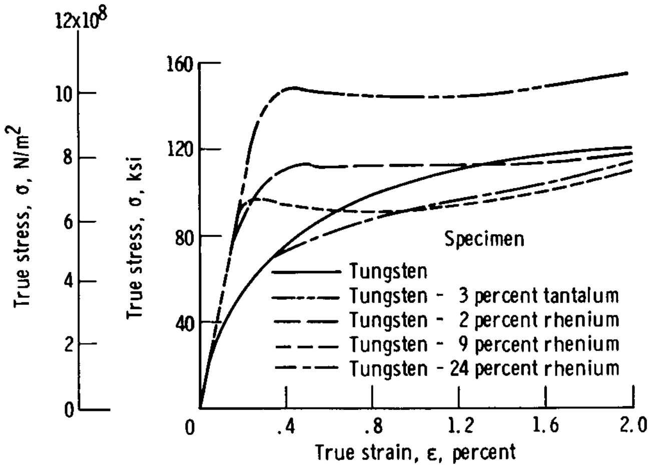


Figure 2. - Stress-strain curves for polycrystalline tungsten and tungsten alloys.

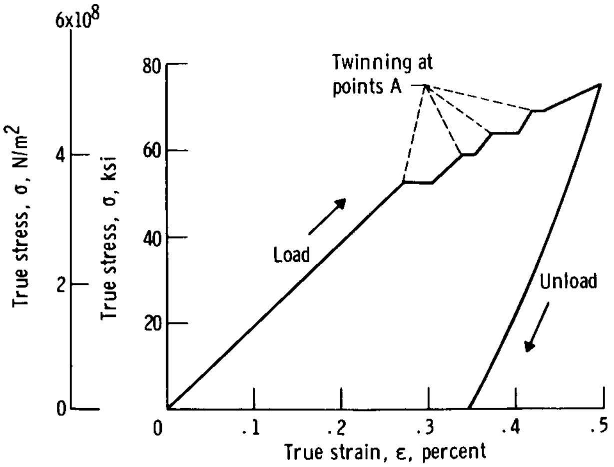
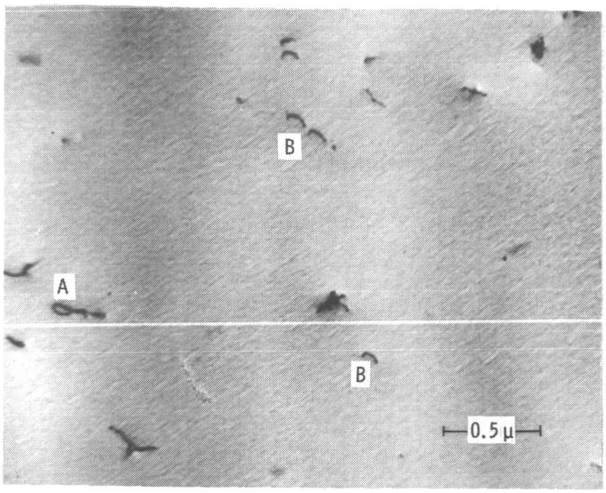
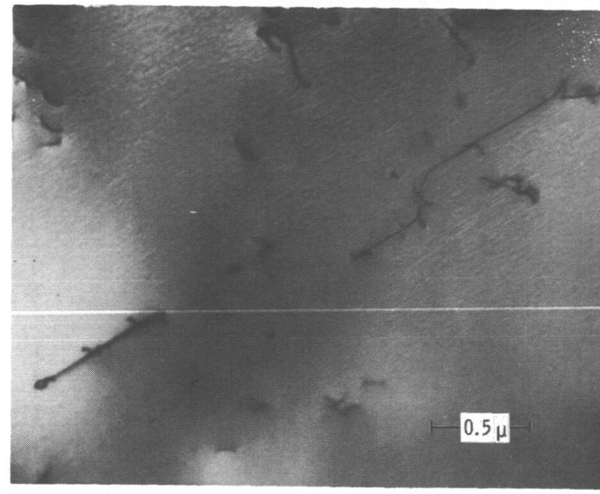


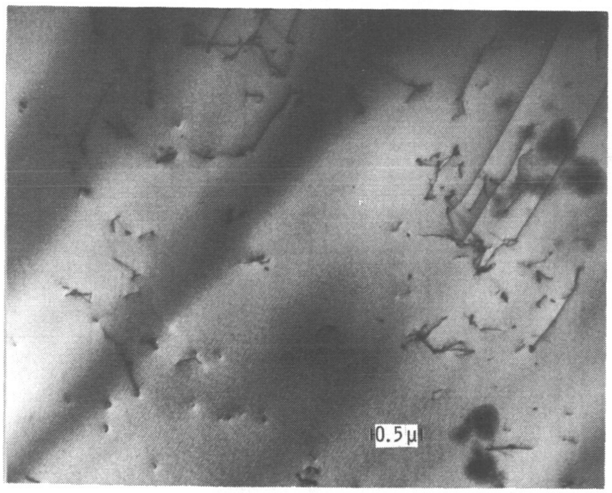
Figure 3. - Initial portion of true stress - true strain curve for tungsten - 24 percent rhenium.



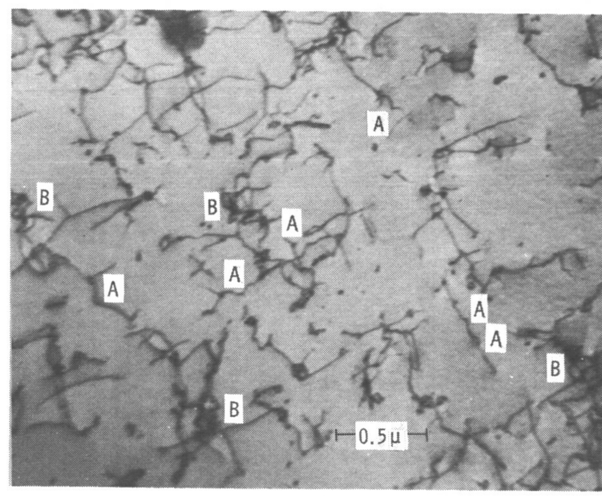
(a) Strain, 0.05 percent.



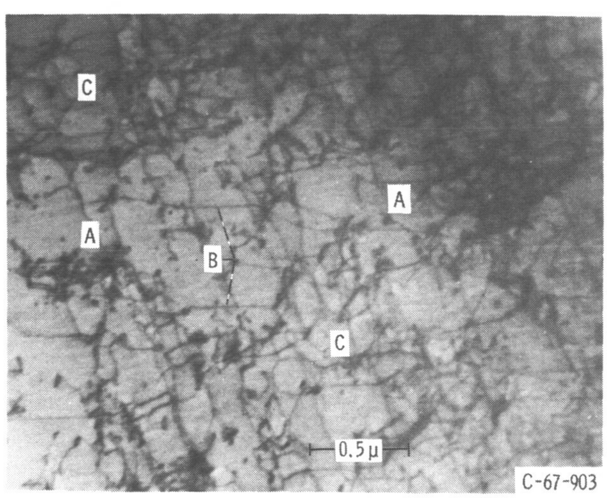
(b) Strain, 0.1 percent.



(c) Strain, 0.5 percent.



(d) Strain, 2.0 percent.



(e) Strain, 5.0 percent.

Figure 4. - Dislocation structure in unalloyed tungsten after testing in compression at room temperature.

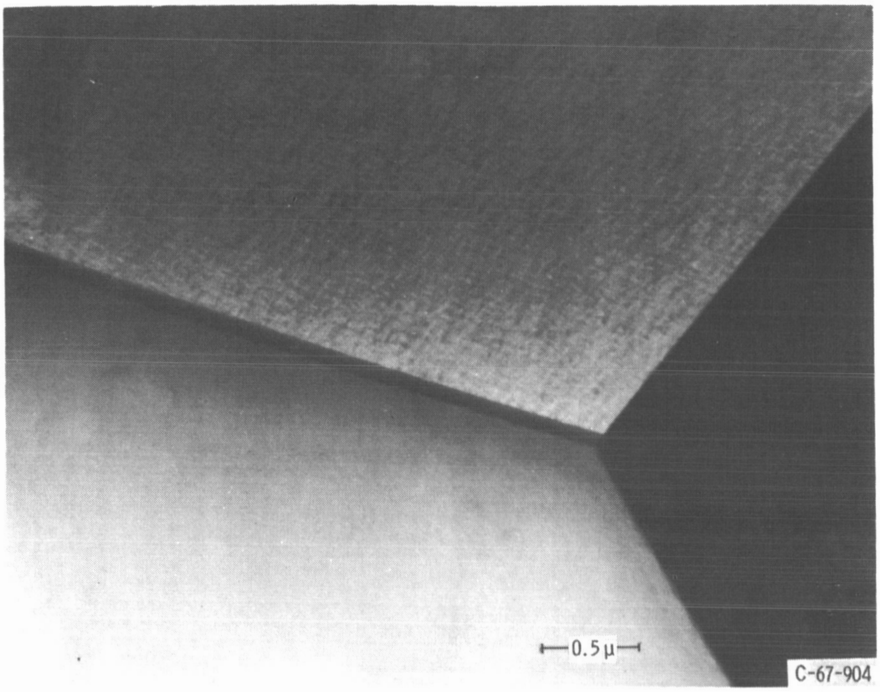


Figure 5. - Structure of tungsten - 3 percent tantalum after 0.5 percent compression strain.

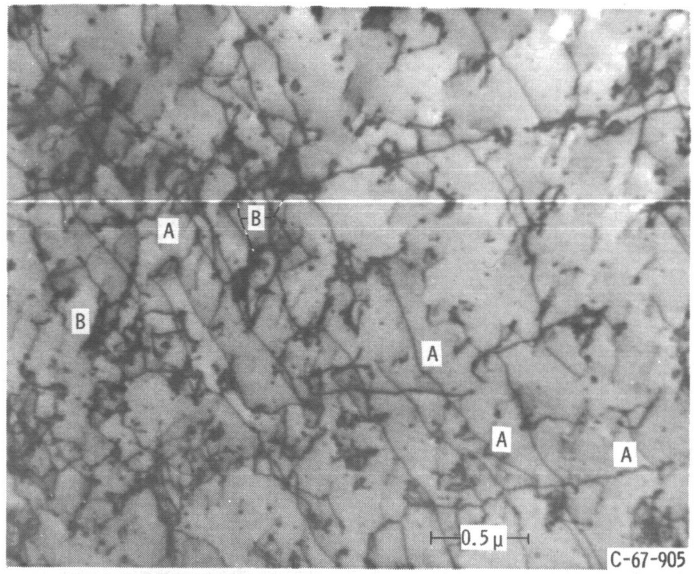


Figure 6. - Dislocation structure in tungsten - 3 percent tantalum after 2.0 percent compression strain at room temperature.

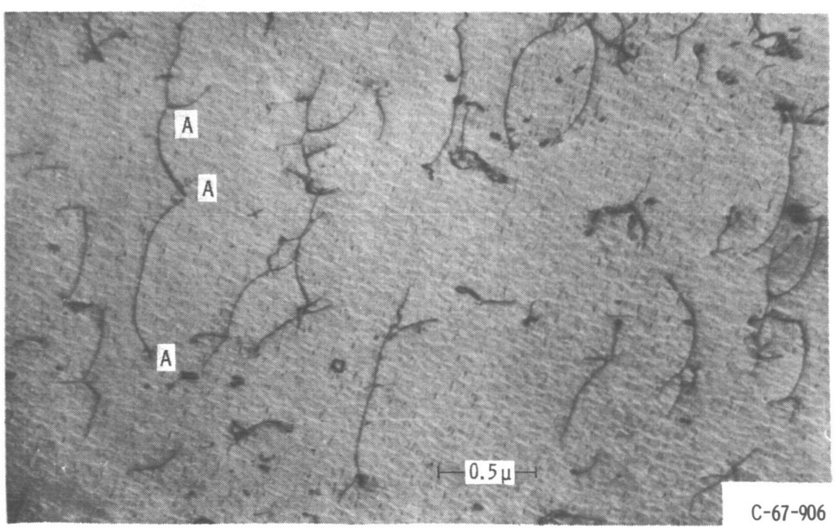
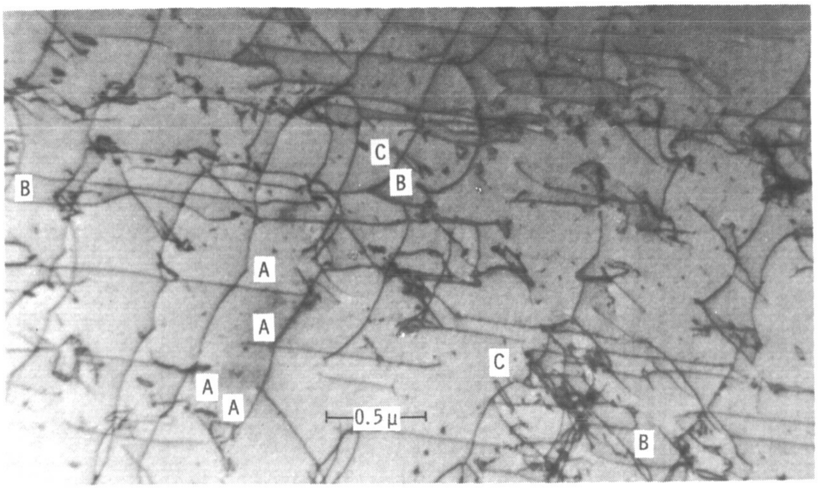
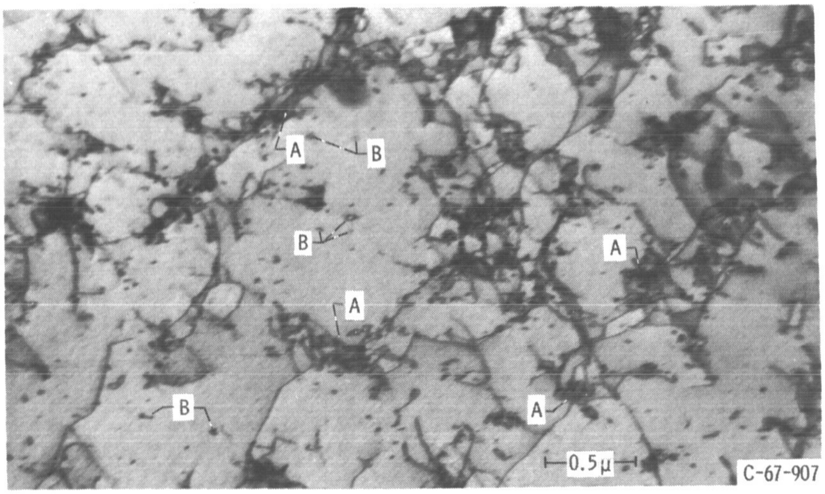


Figure 7. - Dislocation structure in tungsten - 2 percent rhenium after 2.0 percent compression strain at room temperature.

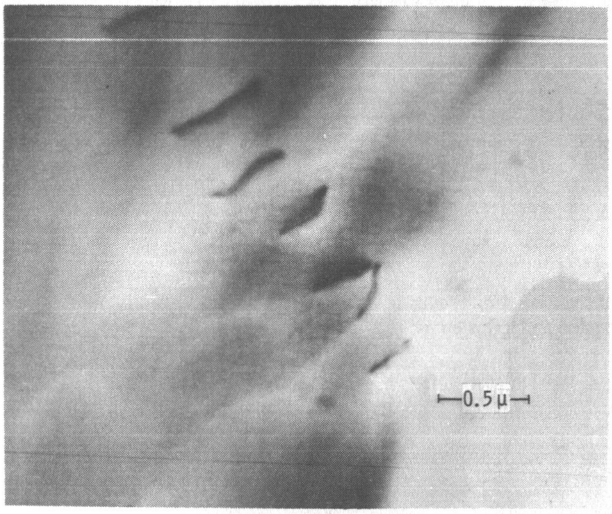


(a) Strain, 1.0 percent.

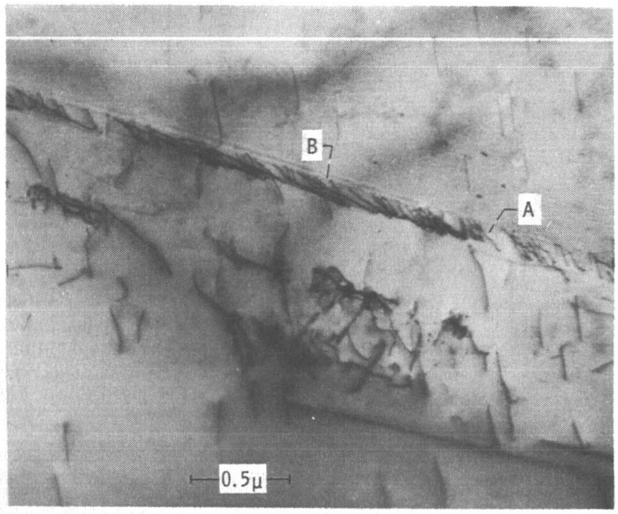


(b) Strain, 2.0 percent.

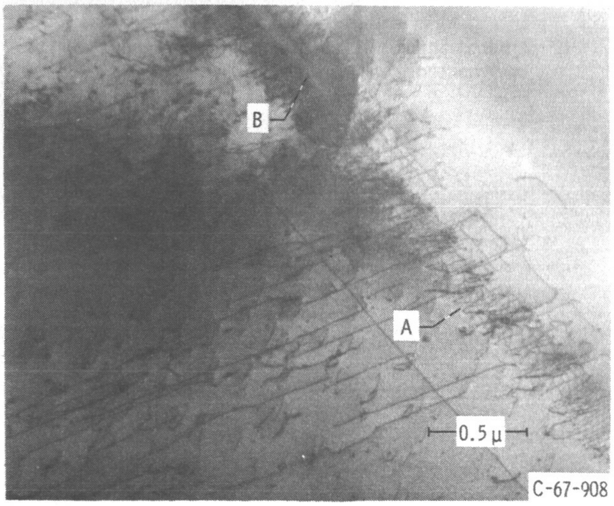
Figure 8. - Dislocation structure in tungsten - 9 percent rhenium after testing in compression at room temperature.



(a) Strain, 0.1 percent.



(b) Strain, 0.5 percent.



(c) Strain, 2.0 percent.

Figure 9. - Dislocation structure in tungsten - 24 percent rhenium after testing in compression at room temperature.

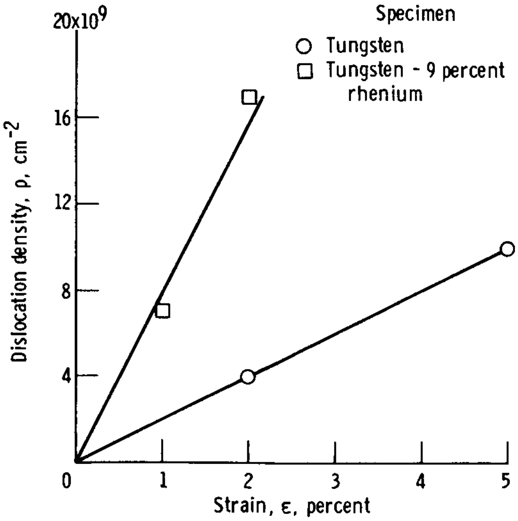
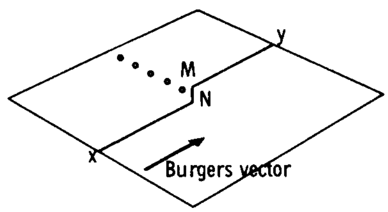
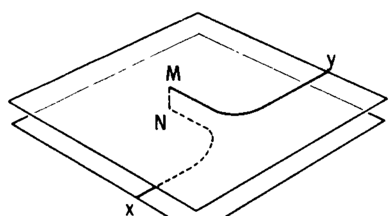


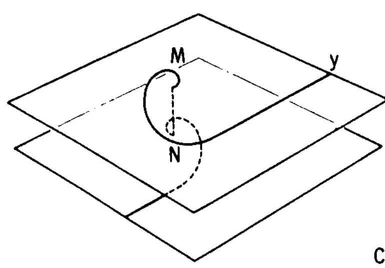
Figure 10. - Variation of dislocation density with strain for unalloyed tungsten and tungsten - 9 percent rhenium.



(a) Small jog; (M to N = 1 or 2 Burgers vectors) nonconservative movement, defects created.



(b) Intermediate jog; (M to N ≈ 200Å) dipole formed.



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(c) Large jog (superjog)(M to N>>1 or 2 Burgers vectors); M-y and N-x move independently and act as single-ended sources.

Figure 11. - Movement of screw dislocations with different height (M to N) jogs.

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