NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Technical Report No. 32-818

Slip in Tungsten at High Temperatures

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ABSTRACT

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Single crystals of tungsten grown from powder-metallurgy swaged rod by high temperature annealing were deformed in tension at temperatures from 2500 to 5000°F. Orientation of specimen tensile axis, strained matrix, and deformation bands was determined optically by reflections from {110} etch pits. Slip traces were analyzed and slip direction determined. Results indicate that {110} <111>, {112} <111>, and {123} <111> type slip occur in tungsten over the temperature range investigated. Slip is orientation dependent occurring on that combination of slip plane and direction which has the highest critical resolved shear stress. "Overshooting" appears to be a general occurrence between 2500 and 5000°F. Deformation bands show rotation in a direction opposite to the rotation of the tensile axis. Author

I. INTRODUCTION

Deformation in body-centered cubic (bcc) metals has been reviewed by Maddin and Chen (Ref. 1), Keh and Weissman (Ref. 2), and most recently by Nabarro, Basinski, and Holt (Ref. 3). From these reviews it is apparent that some disagreement exists concerning the crystallography of plastic deformation in bcc metals. There is evidence of noncrystallographic slip, the crystal slipping on or near a noncrystallographic plane in the <111> zone for which the resolved shear stress is greatest. There is also evidence to indicate that crystallographic slip occurs in the planes of the same zone. On the other hand, there is agreement that the close-packed direction <111> is the slip direction in bcc metals. Further, in pure bcc metals (Ref. 4) and alloys (Ref. 5) the operative crystallographic slip systems are strongly dependent on temperature.

Both crystallographic and noncrystallographic slip have been reported (Ref. 6, 7, 8) in tungsten, a group VI-A bcc metal. The number of slip families which operate in the case of crystallographic slip, however, has been in question. The earliest study by Goucher (Ref. 6) of single-crystal tungsten tested at temperatures of 1800 to 5000°F led to the conclusion that only {112} <111> type slip operated. Raymond and Neumann (Ref. 7) recently reached the same conclusion studying deformation by rolling of plasma-flame single crystals at 1800°F; however, only [100] and [110] orientations were studied. Work at high temperatures by Leber and Pugh (Ref. 8) on tungsten and Chen and Maddin (Ref. 9) on molybdenum suggests that conjugate slip on nonparallel {110} planes may account for the slip on {112} and {123} planes found by others working with bcc metals.

The present paper presents evidence of three families or modes of slip in tungsten in the temperature range from 2500 to 5000°F: {110} <111>, {112} <111> and {123} <111>

II. EXPERIMENTAL PROCEDURE

A. Material

Standard microtensile specimens, 0.160 in. D by 0.640 in. gage length, were ground from commercial powder-metallurgy swaged tungsten rod, type MK. Specimens were heated for 10 min at 5150°F or 6 hr at 5400°F in vacuum of 1×10^{-5} torr or less to produce single crystals. At the lower temperature, an average of two out of seven specimens heated in a group developed single crystals throughout the specimen length as shown in Fig. 1, the rest remaining wholly polycrystalline. At the higher temperature, three or four out of seven specimens became single crystal. Other than to note that Laue X-ray photographs generally showed sharp, well-defined spots, no attempt was made to assess crystal perfection or measure dislocation density. Two tungsten single crystals grown from the melt by the plasma-arc process were also used for this study.

Table 1 shows the level of impurities in the two tungsten materials as determined commercially. The type MK tungsten after heat treatment was of exceptionally good purity. The plasma-flame tungsten "as received" had a notably high carbon content and more iron, aluminum and silicon than type MK tungsten.

Table 1. Impurity levels in type MK and plasma-flame single-crystal tungsten

Impurity element	Parts per million by weight		
	Type MK rod "as received"	Type MK single crystal specimen[b]	Plasma flame single crystal "as received"
O	10.4–13.2[a]	3.2	4.9–8.7
H	<0.1–0.4	0.1	—[c]
C	12–15	6.0	66–98
N	5–8	6.0	3–7
Fe	10	<10	20–40
Ni	<10	<10	<1
Si	10	<10	10–30
Al	<10	<10	20–40
Mo	<10	<10	—[c]

[a]Two values in this table always represent two determinations.

[b]Obtained by recrystallizing 10 min at 5150°F.

[c]Not determined.

B. Testing

Tests were carried out in a tensile testing apparatus (Ref. 10) at temperatures from 2500 to 5000°F under vacuum of 1×10^{-5} torr or less. The specimen, held in a hot-grip assembly, was soaked 10 min at test temperature before loading at a constant crosshead speed equivalent to a strain rate of 0.02/min.

Specimens strained to failure were used to determine slip directions. Other specimens strained from $\simeq$1 to $\simeq$20% in one or several increments were studied in order to follow the onset of conjugate slip.

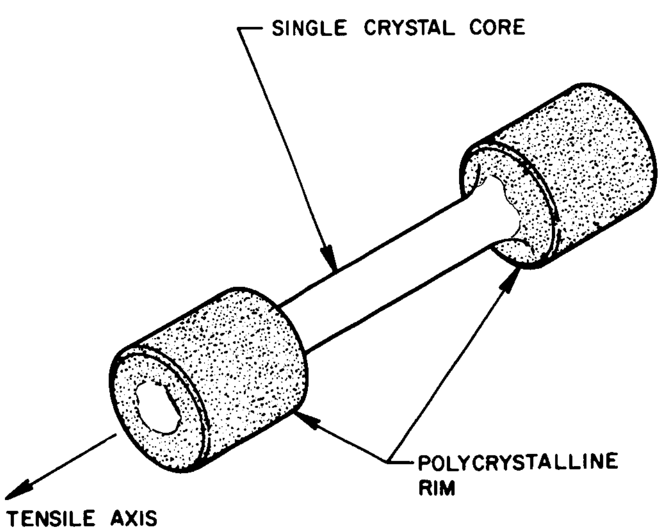


Fig. 1. Tensile specimen showing single-crystal core with polycrystalline outer rim

C. Determination of Orientation

Orientation of the tensile axis of the tungsten single crystals was determined optically by light reflection from {110} planes in a manner similar to Barrett's (Ref. 11). Surfaces were prepared by hand sanding with 600-mesh wet paper, electropolishing at 10 v, electroetching at 3 v in 2% NaOH solution, and macroetching in a 10% NaOH plus 10% $K_3Fe(CN)_6$ solution. This treatment exposed {110} planes as etch pits on the surface. The surface normal to the tensile axis was then viewed at 50X with a metallurgical microscope fitted with a universal petrographic stage. By a combination of rotation and tilt of the specimen, reflections were optimized for at least two and many times four {110} planes, depending upon specimen orientation. Readings were plotted on a stereographic net to obtain the orientation with an overall accuracy estimated to be ± 2 deg. Orientations determined from Laue back-reflection X-ray photographs gave the same order of accuracy as the optical method but the X-ray method, being much slower, was used only as a check.

D. Determination of Slip Direction and Slip Plane

Since the axis of a single crystal moves in the slip direction during tensile deformation (Ref. 12), slip directions were obtained from the orientation of the axis of the tensile specimen before and after deformation.

In determining slip planes, surface traces were measured on specimens which had been deformed $\simeq$1 to $\simeq$20% by a technique based on the "trace in two surfaces" method (Ref. 13).

The specimen was viewed normal to its axis at 50 or 100X, and the angle between the visible trace of the slip plane and the rod axis was measured as the specimen was rotated and translated along its axis. The angle between the trace and the rod axis together with the angle of rotation was plotted on a stereographic net to obtain the orientation of the slip plane. The pole of the slip plane was then plotted on a standard stereographic projection of the crystal.

III. RESULTS AND DISCUSSION

A. Slip Directions

Figure 2 shows a unit triangle divided into regions of preferred slip calculated from critical resolved shear stresses, assuming that all three families or modes of slip observed for bcc metals occur and that equal critical stresses are required for slip (Ref. 14, 15). Experimentally determined slip directions of eight tungsten single crystals strained to failure at temperatures from 2500 to 5000°F are shown in Fig. 3, with the approximate boundaries of Fig. 2 indicated by dashed lines. Open symbols represent the original orientation of the tensile axis of

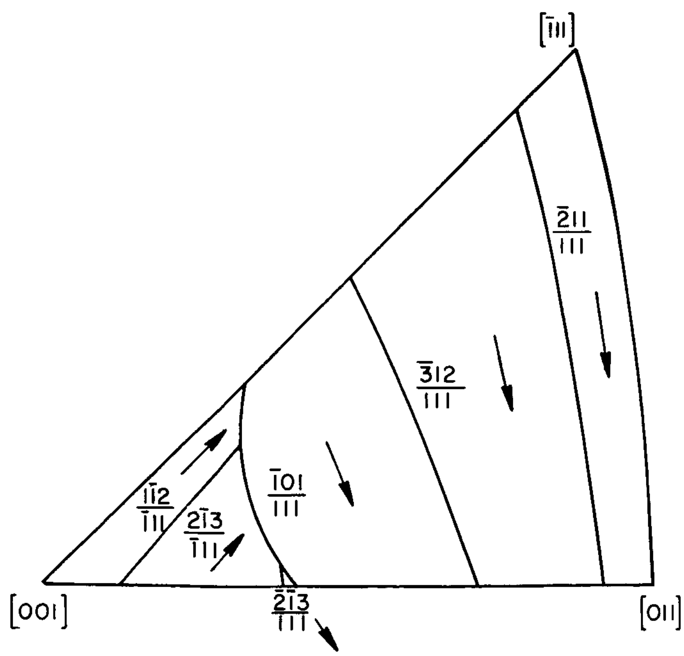


Fig. 2. Unit triangle divided into regions which have the greatest critical resolved shear stress for the combination of plane and direction indicated: three families of planes with the same critical stress

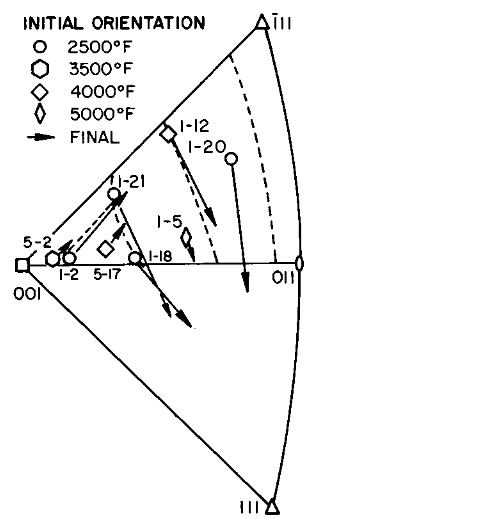
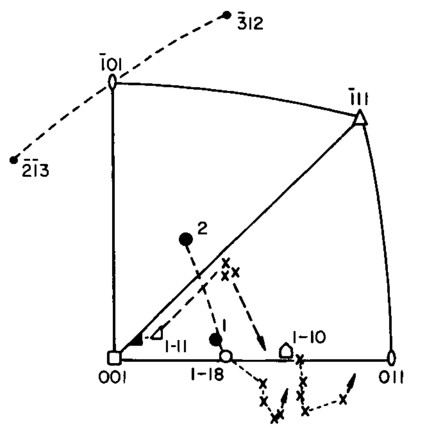
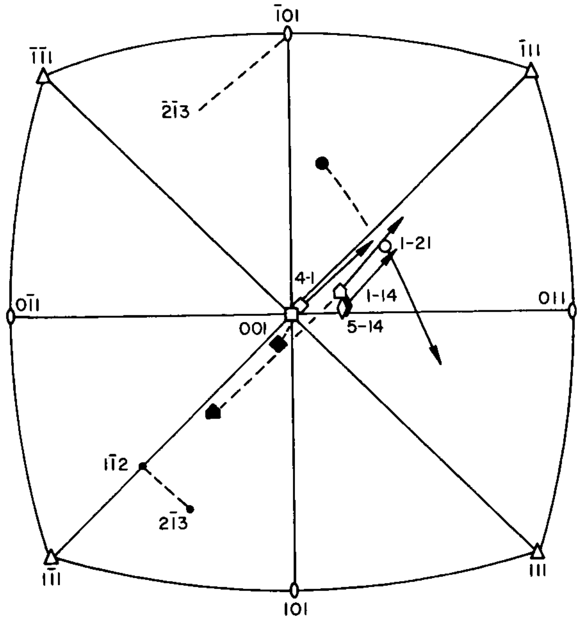


Fig. 3. Orientation in unit triangle of specimen tensile axis and slip direction of eight tungsten single crystals tested at temperatures from 2500 to 5000°F. Dashed boundaries from Fig. 2.



INITIAL ORIENTATION OF TENSILE AXIS
O 2500°F ⌂ 3000°F ◿ 4500°F
X MATRIX DURING ROTATION
→ FINAL ORIENTATION OF MATRIX
● ◢ ORIENTATION OF DEFORMATION BANDS

Fig. 4. Orientation of the matrix during rotation in the slip direction of three tungsten single crystals tested at 2500, 3000, and 4500°F (illustrative of "overshooting"). Also orientation of deformation bands



INITIAL ORIENTATION OF TENSILE AXIS
O 2500°F ⌂ 3000°F ◇ 4000°F ◊ 5000°F
→ FINAL ORIENTATION, MATRIX
●◆◆◆ DEFORMATION BANDS OR MOSAIC BLOCKS

Fig. 5. Orientation of tensile axis, deformation bands and mosaic blocks, and slip direction of four tungsten single crystals strained at 2500, 3000, 4000, and 5000°F

the specimen and the heads of the arrows represent the orientation of the tensile axis in a randomly selected section of the strained portion of the gage.

From the position of the heads of the arrows in Fig. 3, it is apparent that slip has occurred across critically resolved shear stress boundaries. This phenomenon, known as "overshooting," may be followed in greater detail in Fig. 4, where crosses show the orientation of the strained matrix as determined optically in several gage sections of the specimen beginning with a section near the point of failure. Crystal 1-18 slipped on the $(\bar{2}\bar{1}3)$ [111] system or possibly $(\bar{1}01)$ [111] system $\simeq 14°$ across the [001]–[011] join where normally conjugate slip on $(2\bar{1}3)$ $[\bar{1}11]$ might be expected to occur. Without overshooting the slip direction would be along the [001]–[011] join. Crystal 1-10 deforming on the $(\bar{1}01)$ [111] system exhibited 10 deg of overshooting before slip on the conjugate (101) $[\bar{1}11]$ system contributed to the slip direction. Overshooting from one region of family slip, $(1\bar{1}2)$ $[\bar{1}11]$, into another family region, $(\bar{1}01)$ [111], is illustrated by specimen 1-11. Overshooting appears to be a common occurrence in tungsten in the temperature range from 2500 to 5000°F. Further examples of slip directions and overshooting are shown in Fig. 5.

Specimens such as 1-11, which failed as the result of multiple slip, exhibited necking in two dimensions. Specimens which failed by conjugate slip on the other hand showed necking in only one dimension.

Experimental evidence of slip occurring on crystallographic planes is presented in Fig. 6, 7 and 8. In Fig. 6 and 7 two unit triangles, the right hand [001]–[011]–$[\bar{1}11]$ and the left hand [001]–$[0\bar{1}1]$–$[\bar{1}\bar{1}1]$ of a stereographic plot, are used to show original orientations in order to avoid crowding. Orientations of the specimen tensile axes before straining are represented by filled symbols and orientations of the poles of corresponding slip planes after deformation by open symbols.

Figures 6 and 7 taken together give evidence of slip occurring on {110}, {112}, and {123} family planes in the temperature range 2500 to 5000°F. In Fig. 6 the slip plane poles of specimens 3-25 and 5-21, which are within $\simeq 5°$ of $[\bar{1}01]$, indicate single slip on the $(\bar{1}01)$ plane. Similarly, the poles of specimens 7-7 and 4-7 indicate single slip on $(\bar{2}13)$ and $(2\bar{1}3)$ planes respectively; and the poles of specimens 6-11 and 7-22 indicate conjugate slip on {123}.

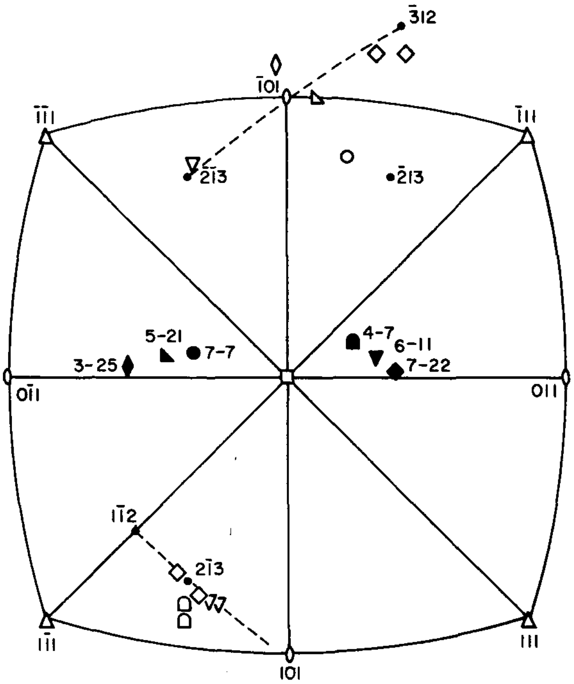


Fig. 6. Orientation of tensile axis and poles of slip planes of six tungsten single crystals strained at 2500, 4000, 4500, and 5000°F: two families of slip, {110} <111> and {123} <111>

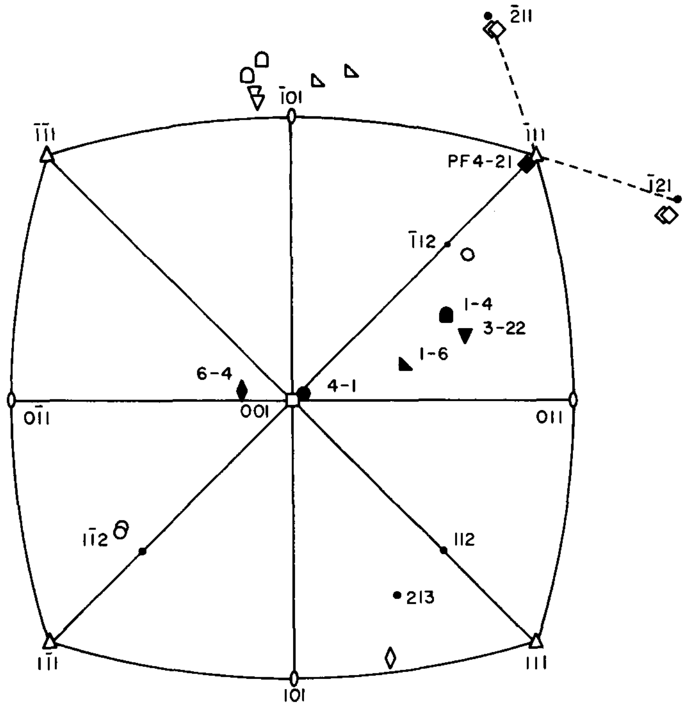


Fig. 7. Orientation of tensile axis and poles of slip planes of six tungsten single crystals strained at 3500, 3700 and 4000°F: three families of slip, {110} <111>, {112} <111> and {123} <111>

In Fig. 7 specimens 1-6, 1-4 and 3-22 exhibit single slip on the ($\bar{1}01$) plane, 4-1 and PF 4-21 (plasma-flame single crystal) exhibit conjugate slip on {112} planes, and 6-4 exhibits single slip on the (213) plane. The poles of specimens 1-6, 1-4, and 6-4 show deviations up to 10 deg from the crystallographic directions because the specimens were strained greater amounts than the other specimens. Strain beyond $\simeq$10% was associated with poorly defined slip traces.

The photomicrographs of Fig. 8 show examples of surface slip traces which were measured to obtain the data presented in Figs. 6 and 7. The photomicrograph of specimen PF 4-21 (Fig. 8a) strained at 3500°F shows conjugate slip traces, one straight and one wavy, both of the {112} family of planes. (The wavy trace appeared straight when the crystal was rotated until the view parallel to the slip plane was also normal to the slip direction.) Straight traces of the ($\bar{1}01$) plane are observed in Fig. 8b in the photomicrograph of specimen 3-22 strained at 3500°F. Specimen 4-7 (Fig. 8c) shows straight traces of the ($2\bar{1}3$) plane after straining at 4500°F, and specimen 7-22 (Fig. 8d) has conjugate {123} traces, one straight and one wavy, after straining at 2500°F. The accepted explanation for the difference in appearance of traces depending on viewpoint is that the straight appearance is the result of movement of edge dislocations while the wavy or branched appearance is due to screw dislocations.

B. Slip Families

Using the slip planes and slip directions as determined for specimens of various orientation, three slip families were found to operate, {110} <111>, {112} <111>, and {123} <111>. For example, specimen 4-7 which slipped on the ($2\bar{1}3$) plane (Fig. 6) considered together with specimen 5-17, which was oriented in the same boundary region and which slipped in the [$\bar{1}11$] direction (Fig. 3), indicates a ($2\bar{1}3$) [$\bar{1}11$] slip system of the

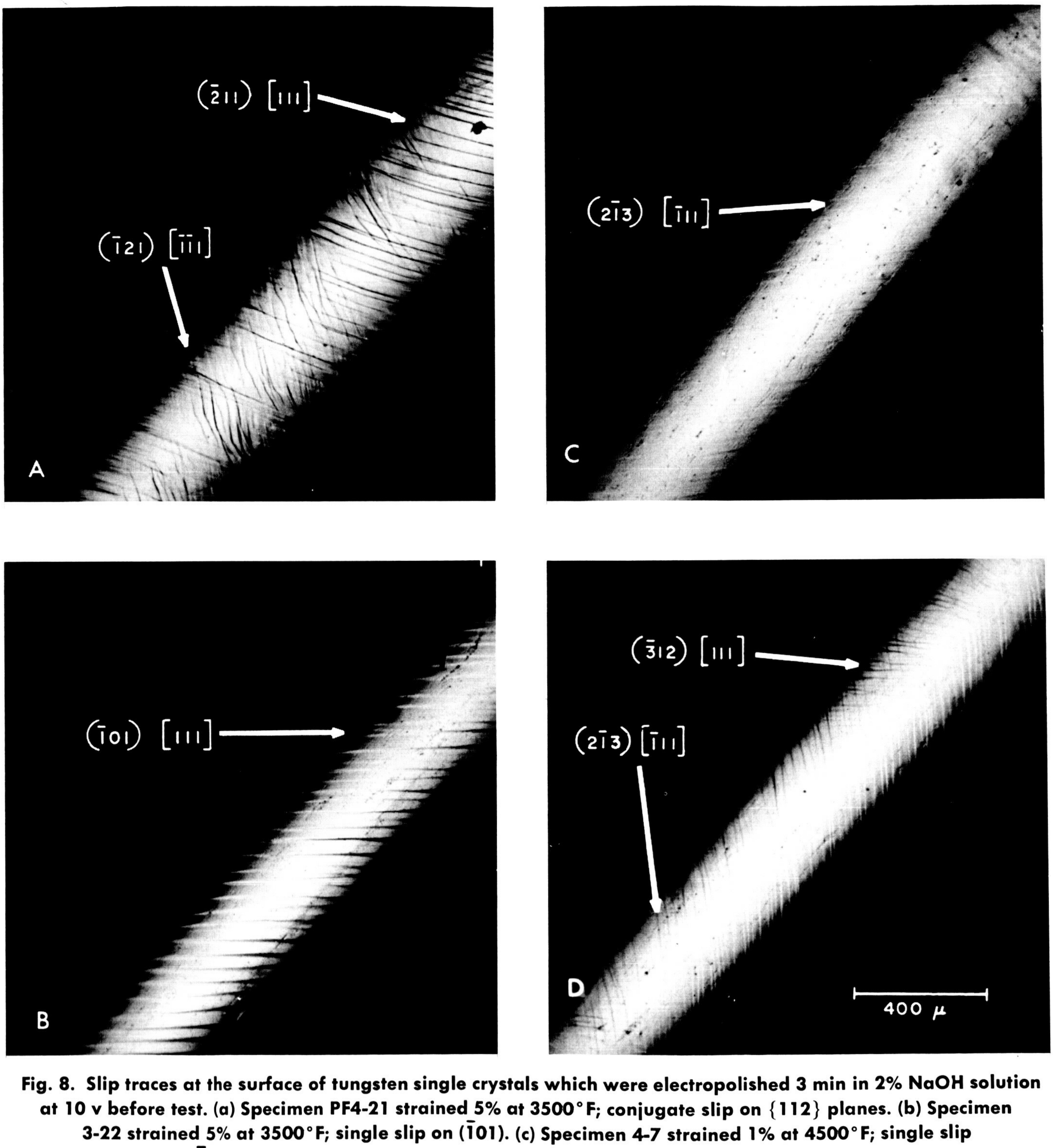


Fig. 8. Slip traces at the surface of tungsten single crystals which were electropolished 3 min in 2% NaOH solution at 10 v before test. (a) Specimen PF4-21 strained 5% at 3500°F; conjugate slip on {112} planes. (b) Specimen 3-22 strained 5% at 3500°F; single slip on $(\bar{1}01)$. (c) Specimen 4-7 strained 1% at 4500°F; single slip on $(2\bar{1}3)$. (d) Specimen 7-22 strained 1.7% at 2500°F; conjugate slip on {123} planes

$\{123\} \langle 111 \rangle$ family. The $(\bar{1}01)$ $[111]$ system was identified, for example, from specimen 1-6 which showed the active slip plane to be $(\bar{1}01)$ (Fig. 7) and specimens 1-21, 1-18, and 1-5 which showed the slip direction in the boundary region in which all these specimens fell to be $[111]$ (Fig. 3). An example of the $\{112\} \langle 111 \rangle$ family was found in crystal 4-1 (Fig. 5 and 7).

Incontrovertible evidence of a slip system is given by specimen 4-1, for which both the slip plane and slip direction were determined. After 1.5% strain at 4000°F, it showed primary slip on the $(1\bar{1}2)$ plane and after an additional 4% strain at the same temperature it gave evidence of secondary slip on the $(\bar{1}12)$ plane. The specimen was then strained to a total of 38% at 4000°F and sectioned, and the primary slip direction was determined to be $[\bar{1}11]$.

Although slip directions and poles of slip planes were determined using separate crystals with the exception noted, specimen 4-1, the experimental evidence taken altogether is in good agreement with families of slip shown in Fig. 2: $\{110\} \langle 111 \rangle$, $\{112\} \langle 111 \rangle$, and $\{123\} \langle 111 \rangle$. The evidence, which pertains to the temperature range from 2500 to 5000°F, strongly suggests that the critical stress is the same for slip by any of the three modes and shows that slip is dependent upon orientation in the manner expected from critical resolved shear stress calculations. The present experimental evidence is in contradiction with the findings of Leber and Pugh (Ref. 8) which indicated $\{110\} \langle 111 \rangle$ slip only and Raymond and Neumann (Ref. 7) which indicated $\{112\} \langle 111 \rangle$ only. However the conclusion of Ref. 8 might have been altered by knowledge of the slip direction, which was not determined.

Data of the present study and of Schadler (Ref. 16) give evidence of the temperature dependence of slip in tungsten. At low temperatures $\{110\} \langle 111 \rangle$ slip occurs, at room temperature $\{112\} \langle 111 \rangle$ occurs in addition to $\{110\} \langle 111 \rangle$, and at temperatures between 2500 and 5000°F $\{123\} \langle 111 \rangle$ in addition to the other two modes operates. The third mode, $\{123\} \langle 111 \rangle$, becomes operative somewhere between room temperature and 2500°F. This experimental evidence is contrary to the prediction, using the correlation of Andrade and Chow (Ref. 4), that only $\{123\} \langle 111 \rangle$ slip should occur in bcc metals above approximately one-half the homologous melting point.

C. *Deformation Bands and Mosaic Blocks*

The photomicrographs of Fig. 9 show a randomly selected cross section of the gage portion of a strained tungsten single crystal which exhibited deformation bands. Dark areas represent deformation bands; light areas, the strained crystal matrix. In Fig. 5 orientations of the strained matrix (arrowheads) and of the deformation bands (filled symbols), which were determined separately by the optical method, are shown together with the orientation of the unstrained specimen (open symbols). Filled circles labeled 1 and 2 represent the orientation of the deformation bands in two different sections of specimen 1-18. The photographs of Fig. 9 correspond with specimens in Fig. 5.

It appears in general that the orientation of the deformation bands lies on the great circle joining the orientation of the unstrained specimen and the slip direction, the bands lying however in a position opposite the slip direction. No explanation is offered at present for this phenomenon. However, Keh (Ref. 17) observed by X-ray using a 250 μ D collimator a similar increase in misorientation between matrix and deformation bands. His results suggest that the deformation bands move to the $[\bar{1}\bar{1}1]$ opposite the $[111]$ slip direction instead of along the great circle as indicated by the present data.

One specimen, specimen 4-1 which was strained in increments of 1.5% + 4% + 32.5% for a total of 38%, exhibited areas which appear to be recrystallized zones within the deformation band or at the band-matrix interface. These areas called "crystallites" which are represented in Fig. 9b by narrow dark areas varied in orientation, having either the orientation of the matrix or of the bands. The crystallites had diffuse $\{112\}$ reflections as well as specular $\{110\}$ reflections.

Specimen 5-14 tested at 5000°F (Figs. 5 and 9d) had an unusual appearance. It exhibited low angle boundaries resulting from extensive polygonization (light areas, Fig. 9d) and mosaic blocks which retained essentially the initial orientation (dark areas). The mosaic blocks may be areas of unslipped matrix between regions of coarse conjugate slip.

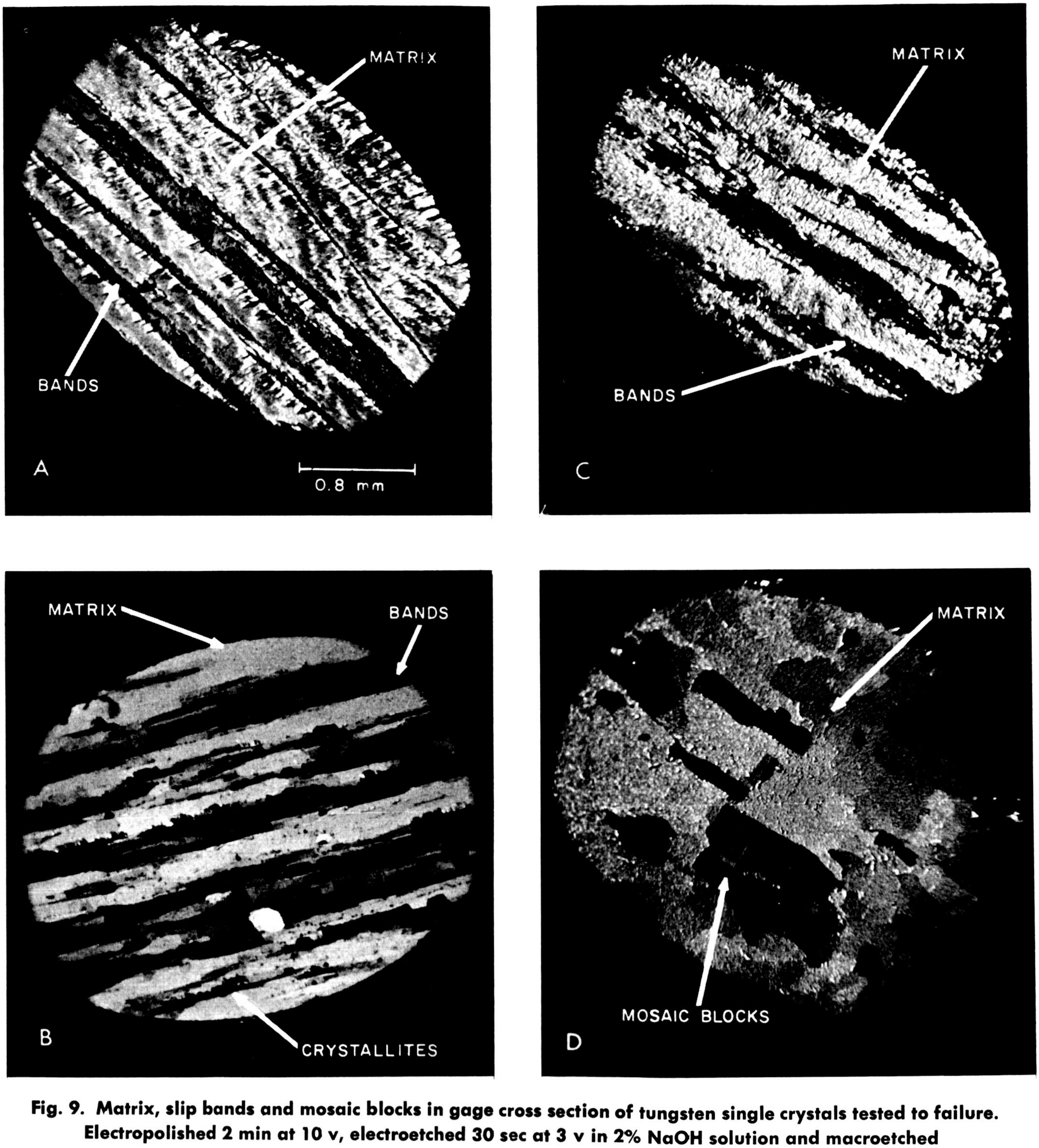


Fig. 9. Matrix, slip bands and mosaic blocks in gage cross section of tungsten single crystals tested to failure. Electropolished 2 min at 10 v, electroetched 30 sec at 3 v in 2% NaOH solution and macroetched 10 min in a 10% NaOH plus 10% $K_3Fe(CN)_6$ solution. (a) Specimen 1-21 tested at 2500°F. (b) Specimen 4-1 tested at 4000°F. (c) Specimen 1-14 tested at 3000°F. (d) Specimen 5-14 tested at 5000°F

IV. CONCLUSIONS

The following may be concluded from the foregoing experimental work:

1. In the temperature range 2500 to 5000°F, tungsten single crystals deform by crystallographic slip on 3 families of the type $\{110\} \langle 111 \rangle$, $\{112\} \langle 111 \rangle$ and $\{123\} \langle 111 \rangle$ at a nominal strain rate of 0.02/min.

2. Slip in tungsten is orientation dependent, occurring on that combination of slip plane and direction which has the highest critical resolved shear stress.

3. Slip in tungsten is temperature dependent. The $\{123\} \langle 111 \rangle$ type slip family becomes operative at some temperature between room temperature and 2500°F, however, in addition to rather than to the exclusion of $\{110\} \langle 111 \rangle$ and $\{112\} \langle 111 \rangle$ type slip.

4. "Overshooting" generally occurs and appears to be independent of orientation.

5. Deformation bands show orientations which indicate movement in a direction opposite to the slip direction along a great circle joining the original orientation and the slip direction.

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