Technical Report No. 32-632

Effect of Grain Size and Impurities on Tensile Strength and Ductility of Tungsten from 2500 to 5000°F in Vacuum

J. L. Taylor

D. H. Boone

O. W. Simmons

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J. L. Taylor

D. H. Boone

O. W. Simmons

Clarence E. Levoe, Chief
Materials Section

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PREFACE

This Report presents the results of one phase of research carried out at the Jet Propulsion Laboratory, California Institute of Technology, under Contract NAS 7-100, sponsored by the National Aeronautics and Space Administration. D. H. Boone is presently employed by the Advanced Materials Research and Development Laboratory, Pratt and Whitney Aircraft, North Haven, Connecticut; and O. W. Simmons is with the Metallurgical Department, California State Polytechnic College, San Luis Obispo, California.

ABSTRACT

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The effect of grain size and impurity level on the tensile strength and ductility of powder-metallurgy tungsten was determined by comparing recrystallized coarse- and fine-grained undoped tungsten with recrystallized fine-grained doped tungsten. Tests were conducted in vacuum at a strain rate of 0.02/min from 2500 to 5000°F. Both fine-grained materials were about the same strength and both were stronger than the coarse-grained material. Ductility for the doped (higher impurity level) fine-grained tungsten was lower than for the undoped fine-grained tungsten in which stress-induced grain growth occurred more readily. Stress-induced grain growth correlates in a general way with ductility measured by either reduction in area or elongation. High and low values of stress-induced grain growth are associated with high and low values of ductility, respectively. In the intermediate temperature region, between approximately 50 and 65% of the absolute melting point, where a ductility minimum occurs, grain boundary sliding and its effects, i.e., void nucleation, growth, and intercrystalline fracture, appear to predominate to reduce ductility in both fine-grained materials.

Author

I. INTRODUCTION

Recent studies indicate that grain size affects the high-temperature tensile strength and ductility of tungsten. Investigation (Ref. 1) of high-temperature tensile and creep properties of several lots of tungsten sheet showed that the fine-grained sheet was stronger but less ductile than the coarse-grained sheet. Similar dependence of tensile strength and ductility on grain size was suggested by the results of a study (Ref. 2) of recrystallized doped powder-metallurgy tungsten, recrystallized arc-melted and extruded tungsten, and annealed plasma-flame single-crystal tungsten, all in rod form.

Most of the data on high-temperature deformation appear in the creep literature. Although this Report is concerned with a tensile study, some of the grain-size effects found in creep may be pertinent. Sherby's correlation (Ref. 3) of creep data shows that for many metals the creep rate increases linearly with the square of the grain diameter, although metals such as molybdenum (Ref. 4) and aluminum (Ref. 5) show reduced creep rates for coarse-grained materials. Some of the conflicting data may have resulted from testing cold-worked or partially recrystallized structures which are unstable under test at higher temperatures.

Stress-induced grain growth appears to correlate with an observed increase in ductility for several metals (Ref. 6 and 7). Previous data for tungsten (Ref. 2) appear to hold the same relationship.

The investigation described herein was directed toward obtaining experimental evidence of the effect of grain size on the tensile strength and ductility of recrystallized powder-metallurgy tungsten in rod form. Data on stress-induced grain growth and ductility were also obtained. The effect of impurities on grain growth during testing was investigated by comparing doped with undoped powder-metallurgy tungsten.

II. MATERIALS

Specimens for this investigation were ground from 0.5-in. dia rods produced commercially from a single lot of undoped tungsten powder. The powder was hydrostatically pressed and sintered in bars, 1.6 in. in dia × 12 in. in length, to 93-95% of theoretical density. These were rolled and swaged to produce the 0.5-in. rods. Starting and finishing swaging temperatures were ≃2900 and ≃2400°F, respectively, with 3-min anneals between passes. Specimens of the doped powder-metallurgy tungsten (Ref. 2), used as a comparison material for this study, were made from 3/8-in. swaged rod.

The impurity levels for the doped and undoped materials as determined by an independent laboratory are given in Table 1. In the doped material, aluminum values are higher, and iron, silicon, and nitrogen values are slightly higher than in the undoped material. Carbon values are higher in the undoped tungsten.

A few analyses (not reported) made on specimens after they had been recrystallized in vacuum indicate that in both the doped and undoped tungsten oxygen and iron values were slightly lowered.

Table 1. Impurity levels in powder-metallurgy tungsten

Composition parts/million by weight

Material	O	N	C	Fe	Al	Si	Mo
Powder-metallurgy[a] (undoped)	1.0-6.4	2-10	2-14	10-20	< 10	< 10	30-50
Powder-metallurgy[b] (doped)	1.3-5.0	5-16	2-4	10-40	20-40	< 10-20	30-50

[a] Sixteen samples from 8 rods.
[b] Nine samples from 3 rods.

III. APPARATUS AND PROCEDURE

The apparatus and test procedure used for this investigation are described in Ref. 8. The apparatus provides radiation heating, in vacuum, for a standard specimen (Ref. 9) (0.640-in. gage lg by 0.160-in. dia) held in a hot-grip assembly. The furnace is heated by induction. Since first reported the apparatus has been improved (Ref. 8) with a ball-nut lead screw and a vacuum system which has a higher pumping capacity and lower ultimate pressure, $\simeq 5 \times 10^{-6}$ torr, at test temperatures. An engineering stress-strain curve is obtained for each test from an x-y plotter which records simultaneously the outputs from a load cell and an extensometer. A shoulder-strain correction, necessary because the extensometer measures grip separation, is determined by measurement, before and after testing, of the distance between marks on the specimen shoulder and gage. When voids occur as the result of testing, uniform void development up to the point of necking is assumed, and the corresponding change in volume is applied to obtain a corrected true-stress-true-strain curve. Void volume was determined by the lineal-intercept method during grain-size measurements.

A. Grain-Size Measurements

Grain size was measured by the lineal-intercept method, counting grains in four photomicrographs for

each specimen. The photomicrographs were of longitudinal and transverse sections in the unstrained head and in the strained gage section of the tested specimen, in an area just outside the zone disturbed by fracture. Grain growth, which may occur to some extent because of the temperature of the test, was excluded by using only photomicrographs of the tested specimen. Increase in grain size is defined as the difference in grain size between the undeformed specimen head and the uniformly deformed gage section divided by the grain size in the head, expressed as a percent.

B. Recrystallization

Specimens were recrystallized in vacuum at temperatures higher than the highest test temperature in order to stabilize the structure with respect to temperature alone. Exceptions were made for doped tungsten recrystallized at 5150°F and tested at 5400°F and undoped tungsten recrystallized at 4500°F and tested at 4500°F. Different temperatures were used to obtain the two nominal grain sizes in the undoped powder-metallurgy tungsten.

One group of undoped tungsten specimens was heated for 18 hr at 1000°F in vacuum at 10^{-5} torr as an initial recovery treatment. The specimens were subsequently recrystallized at 3900°F in vacuum at $\simeq 5 \times 10^{-6}$ torr for 20 min. The recovery treatment insured stable fine-grained structures. It was found that material not so treated occasionally showed abnormal secondary grain growth. Average grain size in the unstrained heads of the tested specimens was 550 grains/mm^2 (0.038 mm average dia). A longitudinal section, representative of the "as recrystallized" condition, from the unstrained head of a specimen is shown in Fig. 1.

Another group of undoped tungsten specimens was recrystallized in vacuum at $\simeq 5 \times 10^{-6}$ torr for 15 min at 4500°F or 10 min at 5150°F. All these specimens had coarse-grained to abnormal-grained structures as the result of random secondary recrystallization, with grain size varying from < 1 to 20 grains/mm^2 (> 1.0 mm to 0.226 mm average dia). Although it was difficult to associate a particular grain size with one or the other recrystallization temperatures, specimens recrystallized at 4500°F showed some tendency to be finer grained. A macrostructure typical of these specimens is shown in Fig. 2.

The doped powder-metallurgy tungsten (Ref. 2) used for comparison in this Report was recrystallized at 5150°F for 10 min. Grain size in the unstrained heads of the tested specimens averaged 280 grains/mm^2 (0.060 mm average dia). This material was recrystallized and tested in the apparatus before improvements were made, in the vacuum range from 2×10^{-4} to 9×10^{-4} torr, a fact not considered to have a significant bearing upon the results presented.

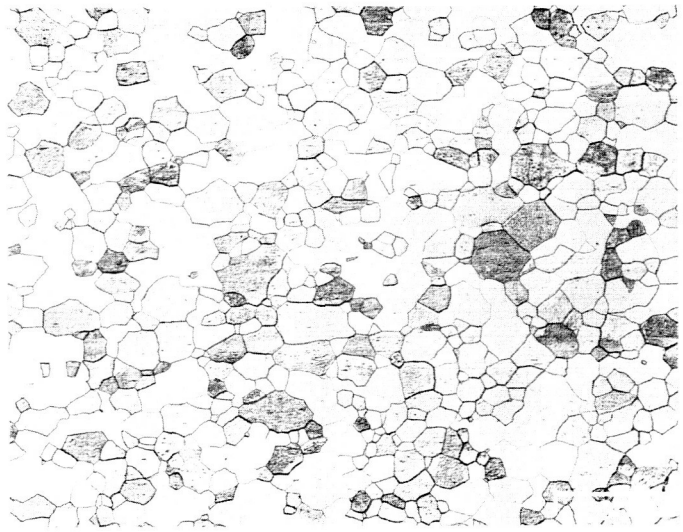

Fig. 1. Longitudinal section of head, undoped powder-metallurgy tungsten specimen recrystallized at 3900°F. Etched 1 min in 30-cm^3 lactic acid, 10-cm^3 HNO_3, and 10-cm^3 HF

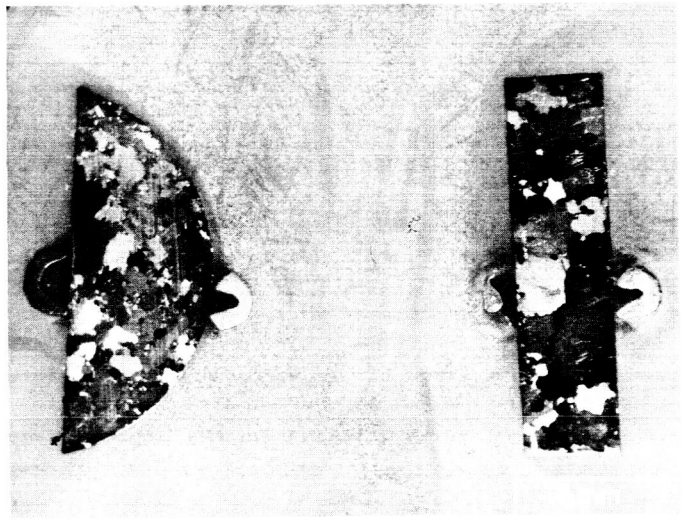

Fig. 2. Longitudinal section and cross section of undoped powder-metallurgy tungsten recrystallized at 4500°F. Etched 1 min in 30-cm^3 lactic acid, 10-cm^3 HNO_3, and 10-cm^3 HF

IV. RESULTS

Strain-hardening and tensile data for the undoped powder-metallurgy tungsten are presented in Fig. 3-6, along with comparison data for doped powder-metallurgy tungsten from Ref. 2. All these data were obtained at a strain rate of 0.02/min.

A. Strain-Hardening Exponent

The true-stress-true-strain flow curve may be described by the expression

$$\sigma = K\epsilon^n$$

where σ is the true stress, K is the strength coefficient, ϵ is the true strain, and n is the strain-hardening exponent.

The value of n is determined graphically from the slope of the line which best fits the log true-stress versus log true-strain data up to the point of maximum stress. Figure 3 shows how the strain-hardening exponent varies with temperature. These values, in view of the assumptions made for corrections mentioned earlier, should be considered as indicative of trends, and are offered here as a means of comparing the various tungsten materials. Of interest are the shapes of the three curves which show minima and maxima within the intermediate temperature region from $\simeq 0.5$ to $\simeq 0.65\ T_m$ (T_m is the melting point, °K) where a ductility minimum occurs for tungsten. The two fine-grained tungsten materials show quite similar strain-hardening curves while the coarse-grained material shows its minimum at a lower temperature ($\simeq$2800°F) and its maximum at a higher temperature ($\simeq$3700°F) than the other two. The coarse-grained material also exhibits less overall change for the n value.

B. Ultimate Tensile Strength

Figure 4 presents ultimate tensile strength as a function of temperature and shows that the fine-grained tungsten is stronger than the coarse-grained tungsten. Except at 2500°F, the curves for fine-grained undoped (550 grains/mm^2) and fine-grained doped (280 grains/mm^2) powder-metallurgy tungsten lie close together, as might be expected since grain size differs by less than a factor of two. Testing of the fine-grained undoped material was limited to 3800°F because the recrystallization temperature was 3900°F. The curve for the coarse-grained undoped powder-metallurgy tungsten (< 1 to 20 grains/mm^2) lies decidedly below the others. It is drawn through duplicate data points at 4500 and 5000°F and through the average of at least four data points at the other temperatures. This curve represents essentially the base-line tensile strength to be expected from coarse-grained (recrystallized) commercially pure tungsten. Data (Ref. 2) for coarse-grained arc-melted and extruded tungsten and plasma-flame single-crystal tungsten straddle the coarse-grained undoped powder-metallurgy tungsten curve, with most of the arc-melted and extruded data lying slightly above.

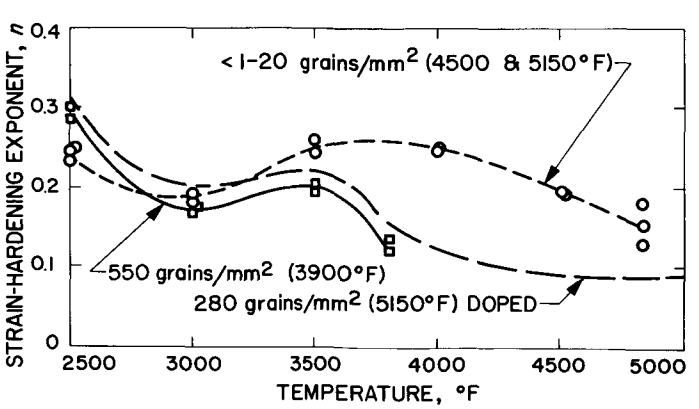

Fig. 3. Strain-hardening exponent as a function of temperature for recrystallized undoped powder-metallurgy tungsten in two grain sizes. Recrystallization temperature in parentheses. Doped tungsten data from Ref. 2

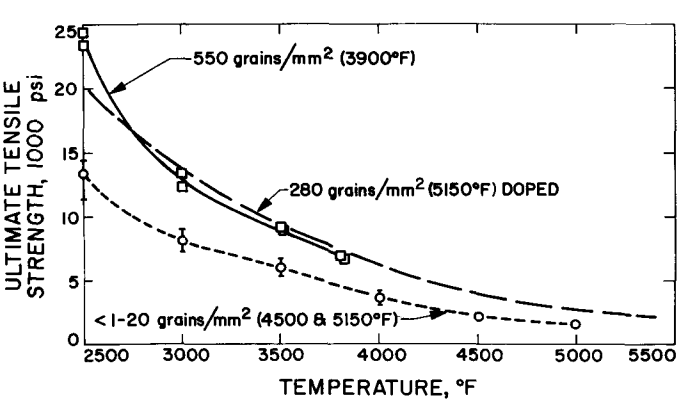

Fig. 4. Ultimate tensile strength as a function of temperature for recrystallized undoped powder-metallurgy tungsten in two grain sizes. Recrystallization temperature in parentheses. Doped tungsten data from Ref. 2

C. Reduction in Area

Figure 5 illustrates reduction in area as a function of temperature. Essentially a 100% reduction in area was obtained for the coarse-grained undoped tungsten at all test temperatures. Similarly, nearly 100% reduction-in-area curves were obtained for other coarse-grained tungsten materials (Ref. 2) tested at a strain rate of 0.02/min and for the fine-grained doped tungsten (Ref. 2) when tested at 2.0/min. A knife-edge, single-crystal type fracture was observed at all temperatures studied including 2500°F.

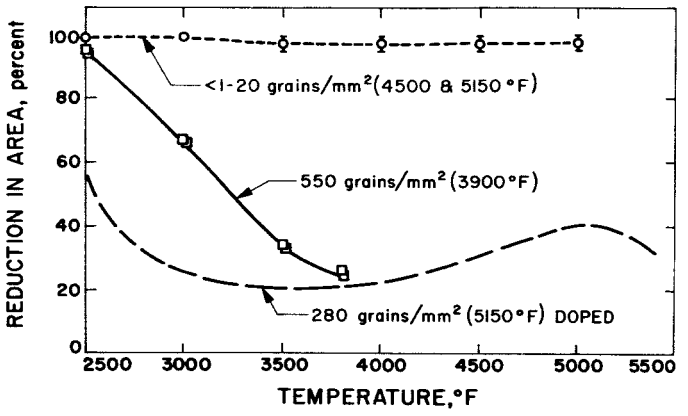

Fig. 5. Reduction in area as a function of temperature for recrystallized undoped powder-metallurgy tungsten in two grain sizes. Recrystallization temperature in parentheses. Doped tungsten data from Ref. 2

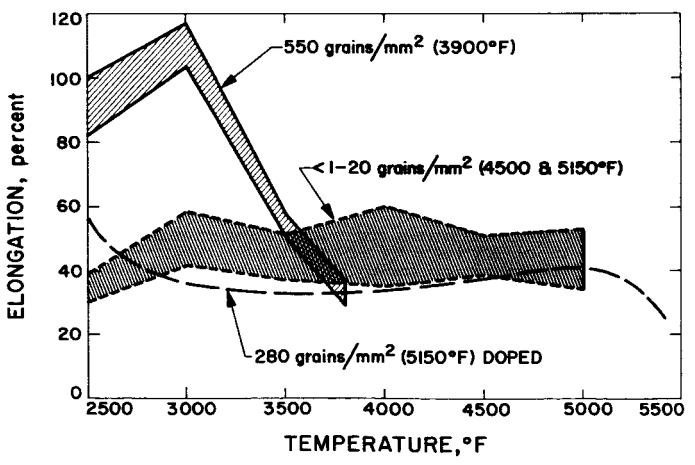

Fig. 6. Elongation as a function of temperature for recrystallized undoped powder-metallurgy tungsten in two grain sizes. Recrystallization temperature in parentheses. Doped tungsten data from Ref. 2

The shape of the curve for the fine-grained undoped material in Fig. 5 suggests a ductility minimum at about 3800°F. The fine-grained undoped material shows considerable uniform elongation before necking to give a "cup and cone" fracture and high reduction-in-area values at 2500 and 3000°F. At 3500 and 3800°F the fractures are intercrystalline, with little or no necking, similar to those for the doped powder-metallurgy tungsten in the same temperature range.

D. Elongation

The elongation data given as a function of temperature in Fig. 6 show nearly as great a ductility difference for the undoped materials having different grain sizes as the reduction-in-area curves do, but in the opposite direction. Elongation is higher for the fine-grained undoped tungsten up to approximately 3000°F where it falls rapidly to values slightly lower than those for the coarse-grained tungsten. In the coarse-grained material, early necking results in comparatively little change in elongation across the entire temperature range.

For the fine-grained undoped material, a band of values represents data scatter from rod to rod within the single lot. Rod-to-rod data scatter was not appreciable for either ultimate tensile strength or reduction in area versus temperature curves. The data scatter within the band for the coarse-grained undoped material can be attributed to: (1) the specimens being made from three different rods, (2) variation in coarse-grain size, (3) difference in recrystallization temperature, (4) possibly, heating and cooling rates.

E. Grain-Size Increase

Figure 7 shows that the undoped fine-grained tungsten exhibits a greater grain-size increase than the doped fine-grained material, which has a higher impurity level. The curve for doped tungsten is somewhat different from that shown in Ref. 2 because of the addition of data points (not shown); however, the trend remains the same. Grain-size increase was not measured for the coarse-grained undoped tungsten because of the difficulty in obtaining meaningful measurements.

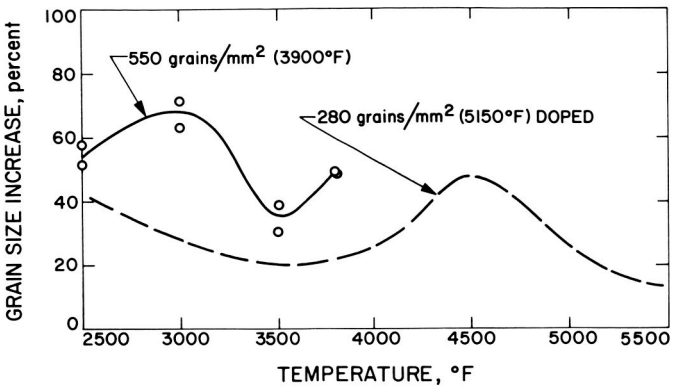

Fig. 7. Grain-size increase as a function of temperature for recrystallized undoped powder-metallurgy tungsten. Recrystallization temperature in parentheses

F. Photomicrographs

The microstructures corresponding to decreasing reduction-in-area ductility and increasing temperature for the undoped fine-grained tungsten are shown in Fig. 8-11. Figure 8 presents the microstructure of a specimen tested at 2500°F, which, as compared to the microstructure of an unstrained head in Fig. 1, shows grain elongation, grain growth, and substructure. The substructure shows a tendency to align itself on a maximum shear-stress plane, namely 45 deg to the tensile axis which is oriented right and left parallel to the plane of the figure.

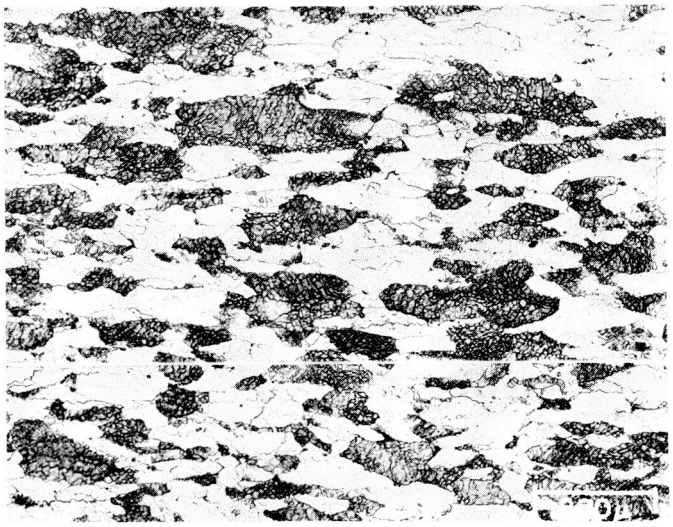

Fig. 8. Longitudinal section of gage, undoped powder-metallurgy tungsten specimen recrystallized at 3900°F and tested at 2500°F. Etched 1 min in 30-cm³ lactic acid, 10-cm³ HNO_3, and 10-cm³ HF

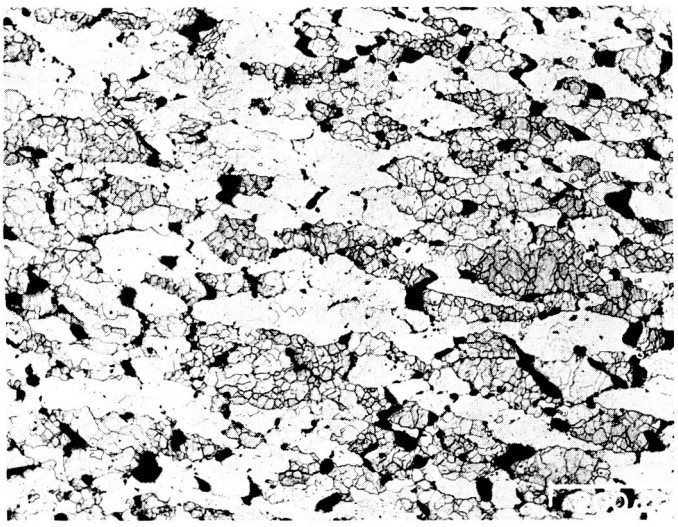

Fig. 9. Longitudinal section of gage, undoped powder-metallurgy tungsten specimen recrystallized at 3900°F and tested at 3000°F. Etched 1 min in 30-cm³ lactic acid, 10-cm³ HNO_3, and 10-cm³ HF

The microstructure of a specimen tested at 3000°F, shown in Fig. 9, is similar to that shown in Fig. 8 with two exceptions: (1) it has a slightly coarser more randomly oriented substructure and (2) there are somewhat rounded voids, a few within grains.

In Fig. 10, the microstructure of a specimen tested at 3500°F, where elongation and reduction in area are considerably lower, exhibits little grain elongation. Voids are larger and more numerous and lie chiefly on boundaries normal to the tensile axis. Void shapes are somewhat rounded by electropolishing. The substructure is quite randomly oriented and is much coarser.

The microstructure of a specimen tested at 3800°F, (Fig. 11), resembles that of the unstrained head, (Fig. 1), except that large voids resembling cracks have been introduced at grain boundaries situated at right angles to the tensile axis and the substructure is evident. Although not readily apparent in the photomicrograph, strain-induced grain growth has increased over that occurring at 3500°F. Evidence of grain-boundary sliding is apparent in Fig. 10 and 11, and the authors have also observed it qualitatively by means of a scratchmark technique.

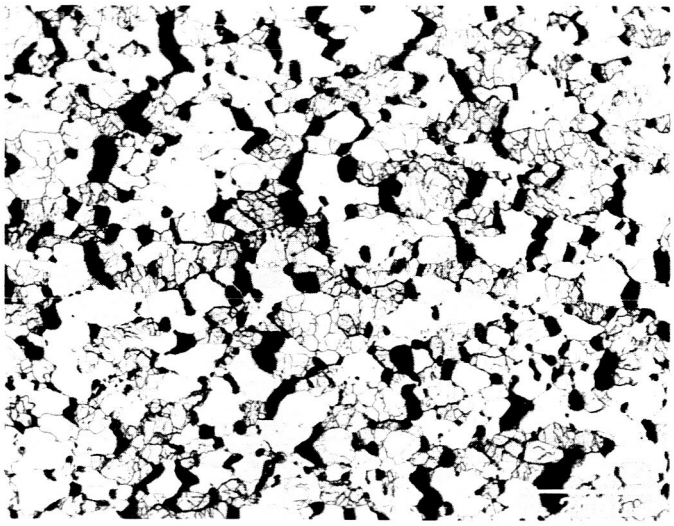

Fig. 10. Longitudinal section of gage, undoped powder-metallurgy tungsten specimen recrystallized at 3900°F and tested at 3500°F. Etched 1 min in 30-cm³ lactic acid, 10-cm³ HNO_3, and 10-cm³ HF

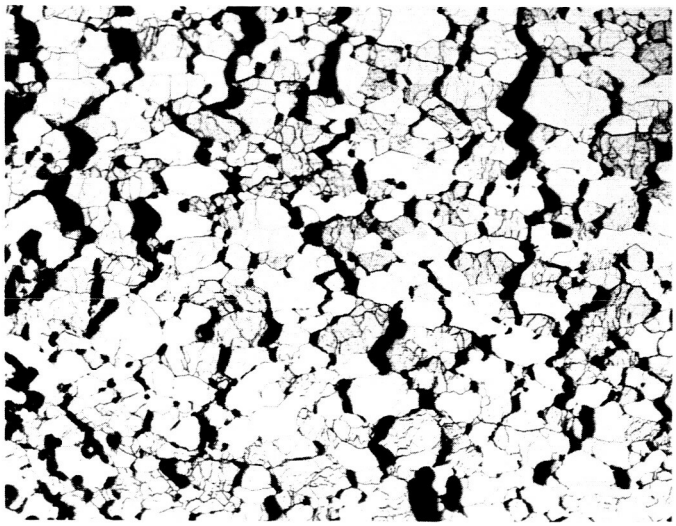

Fig. 11. Longitudinal section of gage, undoped powder-metallurgy tungsten specimen recrystallized at 3900°F and tested at 3800°F. Etched 1 min in 30-cm³ lactic acid, 10-cm³ HNO_3, and 10-cm³ HF

V. DISCUSSION

Considering the difference in strength between the fine- and coarse-grained undoped tungsten of the same material lot, shown in Fig. 4, it may be questioned whether this is an effect of grain size or of some other factors. Such factors may be: (1) grain-boundary energy resulting from the different recrystallization temperatures used; (2) an impurity distribution resulting from cooling from different recrystallization temperatures, and (3) the possibility of single-crystal behavior of the coarse-grained specimen because of there being so few grains in the cross-section.

Taking the first possible factor, other than grain size, for the strength difference, it is known that the energy of the grain boundaries can affect certain deformation processes. In this case, the random distribution of low- and high-energy boundaries in the material recrystallized at 4500 and 5150°F is not expected to be significantly different from that in the material recrystallized at 3900°F because these temperatures are well above the minimum recrystallization temperature of $\simeq$2400°F.

Again comparing the undoped tungsten in two grain sizes: distribution of impurities, especially carbon, at grain-boundary sites probably occurred to a greater extent in the coarse-grained material because it was furnace cooled from a higher recrystallization temperature than the fine-grained material. If true, this distribution would tend to lessen rather than contribute to the strength difference between the materials because impurities as stable second-phase particles favorably sized are known to pin grain boundaries, thereby strengthening the material.

The stress-strain curves of the coarse-grained material ($<$ 1 to 20 grains/mm² or $<$ 1 to 16 grains in the cross section) indicate polycrystalline behavior as distinct

from single-crystal behavior illustrated by Dieter (Ref. 10). Thus, a valid comparison with the fine-grained material can be made.

Grain size is the only factor believed to contribute significantly to the difference in strength observed. Further evidence of the strengthening effect of fine grain size is found in the recent work of Klopp et al (Ref. 11). They showed for swaged and annealed arc-melted tungsten ranging in grain size from < 1 to $\simeq 120$ grains/mm^2 that the finer-grained material was stronger from 2500 to 4000°F in both creep and tensile tests.

The decided difference in ductility, as shown by the reduction-in-area curves in Fig. 5, between the doped and undoped fine-grained powder-metallurgy tungsten probably can be attributed primarily to stress-induced grain growth, which occurs more readily in the undoped material (Fig. 7). The difference in initial grain size (550 grains/mm^2 in the undoped material versus 280 grains/mm^2 in the doped) probably plays a minor part in contributing to the difference in ductility in this case. Between the fine-grained and the coarse-grained undoped material there is also a marked difference in ductility, and in this case the difference is attributed to grain size, 550 grains/mm^2 versus < 1 to 20 grains/mm^2. The grain-size effect may be exaggerated because in the coarse-grained material, at fracture, a coarse grain has already grown to occupy essentially the entire cross section. It may be noted that there is a similarity in the shapes of the grain-size increase curve (Fig. 7) and elongation curve (Fig. 6) for the fine-grained undoped and fine-grained doped tungsten, except at 3800°F for the undoped tungsten. Increasing ductility, measured by reduction-in-area and/or elongation, above the intermediate temperature region has been associated with stress-induced grain growth in tungsten (Ref. 2) and noted in Monel (Ref. 6), copper (Ref. 7), and aluminum alloys (Ref. 12). This increasing ductility is also accompanied by decreasing void nucleation and/or growth. The present data strongly suggest that stress-induced grain growth and ductility are also associated *below* the intermediate temperature region.

For doped tungsten the decrease in both reduction-in-area and elongation above $\simeq$5000°F (Fig. 5 and 6) appears to be associated with less increase in grain size during the test. Stress relaxation, high recovery rates, and low stress levels probably account for the observed diminution of stress-induced grain growth.

Thus, it appears that reduction in area and elongation of recrystallized fine-grained powder-metallurgy tungsten correlate in general with stress-induced grain growth from 2500°F (and possibly lower) to some temperature above 5000°F. In tungsten-3% rhenium (Ref. 13) there was little or no stress-induced grain growth, and ductility decreased continuously in and above the temperature range usual for a ductility minimum.

Stress-induced grain growth, which reaches a maximum in the undoped fine-grained tungsten at 3000°F, is apparently little affected by the voids which occur during the testing (Fig. 9). The presence of rounded voids within grains would appear to indicate that voids nucleated and grew early, before being isolated and limited in size by stress-induced grain growth. The voids, rounded by increasing deformation, surface tension effects, and, possibly, vacancy condensation, have probably exaggerated the elongation values while lowering the reduction in area to 65%. At 3500°F in the region of the ductility minimum, where more and larger voids (Fig. 10) are evident as compared to lower temperatures, there is still a 35% increase in grain size.

It appears that in the ductility minimum region, where grain-boundary sliding as well as stress-induced grain growth occur, the effects of grain-boundary sliding predominate to lower the ductility. Grain-boundary sliding and its associated void formation are enhanced by higher impurity levels and fine recrystallized grain size (Ref. 14) and occur in a fairly narrow intermediate temperature region.

The results of the present investigation are consistent with current thinking about the role of grain boundaries in creep or tension, as summarized by Conrad (Ref. 15). In his summary, the experimental evidence is rather conclusive that grain-boundary sliding is necessary for void formation. It has been shown for aluminum (Ref. 16) that grain-boundary sliding is maximized in a critical stress range, and it has been suggested that for many metals this stress range coincides with the intermediate temperature range.

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