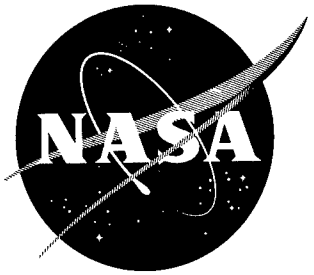
NASA TN D-1581



TECHNICAL NOTE

D-1581

EFFECT OF OXYGEN ON MECHANICAL PROPERTIES OF TUNGSTEN

By Joseph R. Stephens

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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National Aeronautics and Space Administration.
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SUMMARY

The effect of oxygen on the mechanical properties of commercial polycrystalline tungsten and zone-refined single-crystal tungsten was evaluated by means of tensile tests of oxygen-doped specimens in the ductile-to-brittle transition temperature range. The effect of oxygen as a surface oxide that might be encountered in normal working and forming operations was evaluated by means of a three-point loading bend test at room temperature.

Oxygen doping of the polycrystalline and single-crystal tensile specimens was accomplished by equilibrating the specimen and tungsten oxide powder in a sealed tungsten capsule at high temperatures. A desired oxygen concentration could be achieved by varying the time of doping in the temperature range of 3000° to 3600° F. Results showed that the commercial recrystallized rods containing 4 parts per million (ppm) oxygen had a ductile-to-brittle transition temperature (based on 50-percent reduction in area) of 450° F. Increasing the oxygen concentration to 10, 30, and 50 ppm increased the transition temperature to 660°, 840°, and 1020° F, respectively. The oxygen additions also produced a progressive lowering of the ultimate tensile strength. The mechanism for the effect of oxygen on the transition temperature and ultimate tensile strength is believed to be that of segregation of the oxygen atoms at the grain boundaries. The oxygen additions also caused a progressive lowering of the yield strength, which is believed to result from the interaction of oxygen with carbon atoms or other impurity atoms present in the tungsten lattice.

Oxygen additions to the single-crystal specimens had a slight effect on ductile-to-brittle transition temperature. There was very little change in the ultimate tensile strength or yield strength of the single-crystal specimens as a result of increasing the oxygen concentration. These results on high-purity single crystals support the belief that the detrimental effect of oxygen on ductility and tensile strength of polycrystalline tungsten is a result of grain boundary segregation of the oxygen.

A surface oxide that resulted from heating the specimens in oxygen at 1000° F did not affect the ductility of the tungsten rod. Oxidizing the rods at 1500° F increased the ductility of the bend specimens, which is believed to be a result of the removal of surface defects from the ground rod.

Typist: Pick up Rid's markings

INTRODUCTION

The effects of the interstitial elements carbon, oxygen, nitrogen, and hydrogen on the properties of tungsten, molybdenum, and chromium have prompted extensive investigations (refs. 1 to 9). The relatively poor room-temperature ductility and high ductile-to-brittle transition temperature of the group VI A metals (W, Mo, and Cr) are believed to be related to their low solubility for interstitial impurities, which normally occur in amounts near their solubility limits. Research on molybdenum (refs. 1 to 3) and chromium (refs. 4 to 7) has indicated that the presence of carbon, oxygen, or nitrogen increases the ductile-to-brittle transition temperature of the pure metals. In the case of molybdenum, it was reported (ref. 3) that less than 6 ppm oxygen severely embrittled cast specimens of the high-purity metal.

To date, there have been very few results reported on the effect of interstitial impurities on the mechanical properties of tungsten. The effects of carbon and oxygen on the tensile ductility of zone-refined tungsten crystals were studied by investigators at the Battelle Memorial Institute (ref. 8), but their results are limited because of the relatively few crystals available for study. They reported that oxygen additions and probably carbon additions up to 40 ppm lower but do not destroy the room-temperature ductility of single-crystal tungsten.

The purpose of this research was to investigate the effect of oxygen on the mechanical properties of tungsten and to study the interaction of this interstitial impurity with grain boundaries by comparing both high-purity polycrystalline and single-crystal materials. The effect of controlled amounts of oxygen added to tungsten by a doping procedure was evaluated by means of tensile tests in the ductile-to-brittle transition temperature range. The effect of oxygen as a surface oxide, which might be encountered in normal working and forming operations, was evaluated by means of a three-point loading bend test at room temperature.

EXPERIMENTAL PROCEDURE

Material

Tungsten rod (0.125-in. diam.) produced by powder metallurgy was obtained for this investigation from a commercial supplier. Chemical and spectrographic analyses indicated the following major impurities:

Element	C	O	Mo	Fe	K	Al	Si	Cr	Ni
Composition, ppm	8	4	20	10	20	10	10	10	10

Single crystals were grown from this rod by the electron-beam zone-melting technique (ref. 10). The rods were seeded to obtain the orientation as shown in figure 1 for all of the tensile specimens. The zone-melted length was approximately 7 inches; thus, three tensile specimens 2 inches in length were generally machined from each of the zone-melted rods.

Specimen Preparation

Buttonhead tensile specimens (fig. 2) were centerless ground from the commercial rods. For the single crystals the shoulders of the specimens were approximately 0.115 inch in diameter as a result of the material loss during the zone-melting process.

The polycrystalline tensile specimens were vacuum annealed at 3800° F for 2 hours in a dynamic vacuum of approximately 2×10^{-5} millimeter of mercury. The resulting grain size from this annealing treatment was approximately 0.05 millimeter in diameter. This relatively high recrystallization temperature was selected in order that all the specimens would be recrystallized at a higher temperature than any of the oxygen-doping temperatures in an attempt to produce a constant grain size for all the polycrystalline specimens. After doping, the specimens were electropolished from the 0.080-inch ground diameter to a diameter of 0.070 inch in order to remove the roughened surface produced during grinding and any surface oxide resulting from the doping process.

Bend-test specimens were also made directly from the 0.125-inch-diameter commercial tungsten rod by sectioning pieces to 1.250 inches in length.

Doping Procedure

Initial attempts at doping were conducted by heating a tungsten rod to temperatures ranging from 1700° to 2400° F in a vacuum of approximately 5×10^{-5} millimeter of mercury. After the rod had reached the desired temperature, oxygen was introduced into the vacuum chamber until the flow of oxygen brought the pressure to approximately 1×10^{-3} millimeter of mercury. The rod was held at temperature in the flowing oxygen for different time intervals to produce varying oxygen contents. This method proved unsuccessful, since the oxygen was found to be concentrated near the surface of the rod. Subsequent attempts to diffuse the oxygen into the tungsten rod by annealing it at a higher temperature resulted in a loss of the oxygen apparently by volatilization. A second method of oxygen addition to tungsten in controlled amounts was then developed that involved placing the rod in a tungsten capsule and packing tungsten oxide (WO_3) around the specimen. A tungsten plug was placed in the open end of the capsule, and the assembly was welded to give a vacuum-tight seal. Preliminary doping tests were carried out by heating encapsuled tungsten specimens at temperatures from 3000° to 4000° F for various time intervals. The specimens were analyzed for oxygen content after being removed from the tungsten capsules. From these tests, the time and temperature needed for a desired oxygen content was determined. In order to determine if the oxygen content were uniform across the cross section of the specimens, the tungsten slugs were electropolished to various diameters and then analyzed.

Chemical Analysis

The vacuum-extraction technique was employed to determine the oxygen content of the tungsten samples. In this method, a solid sample weighing approximately

2 grams is heated in a dry outgassed graphite crucible at 3632° F (2000° C). Under these conditions, oxygen diffuses to the surface and reacts with the graphite crucible to form carbon monoxide. The time interval necessary for holding the sample at 2000° C to assume complete extraction is determined by monitoring the system with an ionization gage and a strip chart recorder. The pressure is noted prior to dropping the sample into the crucible, and heating is continued until the original pressure is again reached. Standard vacuum-fusion techniques were used to analyze the evolved gases. Good reproducibility was achieved on both the undoped and doped polycrystalline and single-crystal specimens. The accuracy for this technique is generally stated to be approximately ±5 ppm at the low oxygen levels.

Surface Oxidation

The bend-test specimens were heated in an oxygen atmosphere in a tube furnace at 1000° and 1500° F for various time intervals. The 1000° F oxidizing treatment produced a thin blue oxide (WO_2) on the surface of the specimens with very little oxygen penetration. The 1500° F oxidizing treatment produced a thick adherent yellow oxide (WO_3) on the surface with the oxygen penetration dependent on the oxidizing time. Several series of specimens were tested in the oxidized condition for both oxidizing treatments. On a number of specimens the thick oxide formed at 1500° F was removed prior to the test by heating the specimens in a 10-percent sodium hydroxide solution at 180° F.

Mechanical Testing

Both the tensile tests and the bend tests were performed on an Instron tensile machine. The polycrystalline tensile specimens were heated in a vacuum capsule at a pressure of less than 10 microns and allowed to soak for approximately 10 minutes at temperature prior to testing (ref. 11). The single crystals tested above room temperature were treated in the same manner, while those tested below room temperature were cooled by using a mixture of dry ice and alcohol. The tensile specimens were tested at a crosshead speed of 0.005 inch per minute past the 0.2-percent yield and then at a crosshead speed of 0.05 inch per minute from yield to failure. Therefore, the reported yield strengths are at a strain rate of approximately 0.005 inch per inch per minute, while the percent elongation, percent reduction in area, ultimate tensile strength, and transition temperatures are based on a strain rate of approximately 0.05 inch per inch per minute. The bend-test specimens were all tested at room temperature using a three-point loading fixture with a span of 1 inch and loading pins having a diameter of 3/16 inch. The crosshead speed was held constant for the bend tests at a rate of 0.05 inch per minute. The final bend angle of the test specimen was used as the criterion to determine the effects of the oxidizing treatment.

RESULTS OF EXPERIMENTAL WORK

Oxygen Doping

Typical microstructures of the recrystallized commercial tungsten and of the

doped specimens shown in figure 3 reveal a uniform concentration across the diameter of the specimen; that is, when a second phase is present at the grain boundaries it does not appear to be concentrated near the surface of the rod. To ascertain that this was the case, the tungsten specimens were reduced to approximately one-fourth their original cross-sectional areas and then submitted for analysis (no. 2's in table I). Because of an adherent surface oxide, all specimens were electropolished at least 0.010 inch prior to analysis (no. 1's in table I). Table I lists the conditions of doping and the resulting oxygen contents. The results bear out the evidence in the photomicrographs, since the oxygen analyses of the bulk specimen and the specimen that has been reduced to approximately one-fourth of the original cross-sectional area are in good agreement.

Oxygen in Polycrystalline Tungsten

Effect on ductility. - The commercial tungsten rods containing 4 ppm oxygen were doped to give 10, 30, and 50 ppm oxygen. Table II lists the doping conditions and the oxygen contents. The tensile ductile-to-brittle transition temperatures were determined for each series of specimens, and the transition temperature was arbitrarily defined as the temperature at which 50-percent reduction in area would be observed. The data are plotted in figure 4 where it can be seen that the additions of small amounts of oxygen severely embrittle polycrystalline tungsten. Specimens having 10 ppm oxygen showed an increase in transition temperature to 660° F as compared with 450° F for the recrystallized commercial rod containing 4 ppm oxygen. Increasing the oxygen level to 30 and 50 ppm further increased the transition temperature to 840° and 1020° F, respectively.

Effect on strength. - The addition of increasing amounts of oxygen to tungsten progressively lowered both the ultimate tensile strength and the yield strength. Within the temperature range of these tests, the ultimate tensile strength shown in figure 5 was decreased approximately 30 percent for an oxygen content of 50 ppm. The yield strength over the same temperature range and for the same oxygen content was reduced approximately 50 percent as shown in figure 6.

Oxygen in Single-Crystal Tungsten

Effect on ductility. - The addition of oxygen to the single-crystal specimens did not produce a marked increase in the transition temperature as in the polycrystalline material. As is shown in figure 4, the addition of 20 ppm oxygen produced only an 80° F increase in the transition temperature over that of the zone-refined crystals having an oxygen content of approximately 2 ppm. Photomicrographs of the zone-melted crystals and the oxygen-doped crystals are shown in figure 7. It appears from the regular pattern of the oxide distribution that the oxygen is segregated at the subgrain boundaries in the doped crystal.

Effect on strength. - Limited comparisons indicated that the ultimate tensile strengths of the doped single-crystal tungsten were similar to those of the

zone-melted crystals. These results were unlike those obtained for the polycrystalline material where a large decrease in strength was observed.

Surface Oxidation

Oxidation at 1000° F. - Bend-test specimens of polycrystalline tungsten rods in the as-ground condition (room-temperature bend angle, approx. 5°) and electropolished condition (room-temperature bend angle, approx. 110°) were heated in oxygen for time intervals ranging from 1 to 10 hours. The thin blue oxide (WO_2) that formed on the surface did not affect the ductility of the tungsten rods; that is, the as-ground specimens fractured after only a 5° bend, and the electropolished specimens still underwent a 110° bend angle. These results agree with previous results of specimens tested in tension (ref. 11).

Oxidation at 1500° F. - Specimens were oxidized for time intervals of 10, 30, 50, and 100 minutes in order that increasing depths of oxygen penetration would result (or increasing thicknesses of oxide would form). The oxide was removed from one series of specimens by dissolution in a 10-percent sodium hydroxide solution at 180° F. Removal of similar amounts of metal from the surface by either oxidizing or electropolishing produced an improvement in ductility, as is shown in figure 8. A comparison of the bend angle as a function of deflection rate for the as-ground, oxidized, oxide removed, and electropolished surface treatment is shown in figure 9. Again, the oxidized and electropolished specimens displayed similar behavior.

Discussion of Results

Oxygen as an impurity element. - The hypothesis put forth in reference 12 can be applied to the present investigation to explain the low solubility of oxygen in tungsten and the presence of oxygen at the grain boundaries or subgrain boundaries for the single-crystal specimen. Briefly, this hypothesis predicts that the optimum condition for resonant bonding occurs when the number of electrons taking part in the bonding is equal to one-half the effective coordination number. Thus, in groups III A and IV A where the coordination number is 12, the optimum condition is not fulfilled, and metals in these groups can accept electrons. Hence, these metals depending on their valence and electronegativity have a relatively high solubility for electron donors. Although the metals of groups V A and VI A have a body-centered cubic structure with only eight nearest neighbors and six second nearest neighbors, it is assumed the effective coordination number is again approximately equal to 12. Therefore, the group V A metals can accept one electron, while the group VI A metals have reached the optimum bonding condition and the addition of an impurity element that would change the electron coordination number ratio would be energetically unfavorable. Thus, interstitials would be expected to be relatively insoluble in the group VI A metals, and if present they would tend to segregate at areas of disregistry such as grain boundaries or subgrain boundaries where the metal coordination number is less than that of the lattice.

From the preceding description, it appears that the proposed hypothesis for the solubility of electron donors in the group VI A metals can be applied to the addition of oxygen to tungsten. The segregation of the oxygen atoms to the grain boundaries follows, and the effects on the mechanical properties of tungsten as a result of oxygen additions can be considered from the point of view of impurity segregation at the grain boundaries. The presence of the oxide at the boundaries results in a brittle intergranular fracture (fig. 10(b)) as a result of crack propagation through the weak oxide phase. This brittle fracture is similar in character to the brittle intergranular fracture of the undoped specimens (fig. 10(a)), which suggests that oxygen segregated at grain boundaries in commercial tungsten leads to the brittle intergranular fracture. As the test temperature is increased, plastic deformation takes place as a result of dislocations being freed of their locking atmospheres (impurity atoms in the lattice) because of thermal vibrations at the higher test temperature; thus, the ductile-to-brittle transition temperature is observed. As the oxygen content is increased, the ease of crack propagation through the oxide phase is enhanced; thus, higher transition temperatures and lower ultimate tensile strengths are produced with increasing amounts of oxygen. A comparison of these results and those obtained for the single-crystal specimens were the embrittling effect and the effect on the ultimate tensile strength were much less is strong support for the idea of oxygen embrittlement of tungsten by a mechanism of grain boundary segregation of the oxygen.

The reduction of yield strength as the oxygen content is increased cannot be explained by the segregation of impurity atoms at the grain boundaries. As is shown in figure 11, the grain size of the specimens having the higher oxygen content increased compared with the original annealed grain size. The 4 and 10 ppm oxygen series had a grain size of 0.05 millimeter, while the grain sizes of the 30 and 50 ppm oxygen series increased to 0.06 and 0.12 millimeter, respectively. This change is not likely to account for the large change in yield strength associated with oxygen doping. A possible explanation for the reduction in yield strength is the interaction of oxygen with other impurity elements already present in the starting material. The most probable interaction would be with carbon that is present within the lattice. The removal of carbon or other impurity atoms from the lattice during the oxygen-doping treatment could be possible, and if this were the case it would be expected that the movement of dislocations would take place at a lower stress level. The driving force for diffusion of the carbon atoms from the lattice could be that, during the relatively long time of doping at the elevated temperatures, the carbon segregates at the grain boundaries as a result of the electronic considerations mentioned previously. Also, it has been demonstrated (refs. 8 and 13) that the removal of carbon from tungsten can be achieved by formation of carbon monoxide during either a melting or an annealing process in the presence of oxygen. To determine if this was the operating mechanism for the present investigation, a specimen containing 50 ppm oxygen was analyzed for carbon content. The starting material contained 8 ppm carbon, while the oxygen-doped sample contained 4 ppm carbon. This analysis was done by the method described in reference 14 and it is felt that the difference in the two values is significant; however, a more detailed study is required to confirm that the mechanism of carbon removal, as described previously, is taking place and hence is responsible for lowering of the yield strength. A drop in yield strength does not require complete removal of the

carbon or other impurities from the test specimen but only a shift from the lattice (where it can impede dislocation movement) to the grain boundary where its effect on the yield strength would be lessened. To determine if the carbon were being removed from the lattice, hardness readings were taken within the grains and across the grain boundaries. The results of these hardness readings are given in table III. For the polycrystalline material, the hardness of the grains decreased significantly with increasing oxygen content. These results support the hypothesis that lowering of the yield strength is due to the interaction of oxygen with an impurity previously dissolved in the tungsten lattice.

The absence of lowering of the yield strength of the single crystals or a reduction in hardness (table III) as the oxygen content is increased could result from a lower carbon concentration or from the absence of other impurities in the zone-refined tungsten.

Oxygen as surface oxide. - The increase in bend ductility of the tungsten specimens from the surface oxidation at 1500° F can very well be due to the same mechanism that is believed operative for electropolishing the surface, namely, the removal of surface defects that act as stress concentrators. At this temperature (1500° F), the diffusion rate of oxygen in tungsten is apparently so low that an oxide forms at the advancing interface of the diffusing oxygen. This oxide at the surface is effective in eliminating the peaks and valleys of the ground surface and hence removes the stress concentrators. Since the rod was ground as a finishing process after the final swaging, it is felt that any carbon or other impurities that would be concentrated near the surface during swaging would be removed during grinding and hence the removal of other impurities by oxygen, as described in reference 15, is unlikely to be the effective mechanism.

The absence of any effect from the oxidation treatment at 1000° F is probably a result of the very low oxidation rate at that temperature.

Thus, these oxidizing treatments indicate that heating tungsten in air at temperatures near 1500° F improves the ductility by effectively removing surface defects. Therefore, this treatment should be beneficial in forming or working operations. The ductility of tungsten is unaffected by exposure at 1000° F in air; therefore, a protective atmosphere is not needed for working or forming operations at this temperature.

CONCLUSIONS

From an investigation of the effect of oxygen on the mechanical properties of tungsten, the following conclusions are drawn:

1. Oxygen additions to polycrystalline tungsten result in a severe embrittlement. The ductile-to-brittle transition temperature increases with increasing oxygen content.

2. The ultimate tensile strength of polycrystalline tungsten is decreased as a result of oxygen additions.

3. The embrittlement associated with increasing oxygen content is believed to result from preferential segregation of oxygen to the grain boundaries. The high grain boundary solubility is probably associated with the low coordination number in the area of disregistry.

4. Oxygen added to polycrystalline tungsten having other impurities dissolved in the lattice can lower the yield strength by removal of these impurities (probably carbon) from the tungsten lattice. The removal of these impurities apparently has a greater effect on the yield strength than does the presence of oxygen either as an interstitial or as a second phase at the grain boundaries.

5. Oxygen embrittlement of single-crystal tungsten is much less severe than for the polycrystalline material. Oxygen in single-crystal tungsten does not significantly affect the ultimate tensile strength or yield strength.

6. Heating tungsten in air at 1000° F for forming or working operations does not cause embrittlement.

7. Heating tungsten in air at temperatures near 1500° F can enhance ductility if the tungsten is held at temperature for a sufficient period of time to allow oxidation to smooth the roughened surface layer.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, October 18, 1962

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TABLE I. - OXYGEN CONCENTRATION IN DOPED TUNGSTEN RODS

Specimen		Doping conditions		Chemical analysis		
Group	Number	Temperature, °F	Time, hr	Original area, sq in.	Analyzed area, sq in.	Oxygen content, ppm
III	1	2000	1	0.50×10^{-2}	0.385×10^{-2}	2
	2	2000	1	.50	.125	3
IV	1	2000	5	0.50×10^{-2}	0.385×10^{-2}	4
	2	2000	5	.50	.125	5
V	1	2000	10	0.50×10^{-2}	0.385×10^{-2}	5
	2	2000	10	.50	.125	7
VI	1	3000	0.5	0.50×10^{-2}	0.385×10^{-2}	2
	2	3000	.5	.50	.125	2
VII	1	3000	1	0.50×10^{-2}	0.385×10^{-2}	3
	2	3000	1	.50	.125	2
VIII	1	3000	5	0.50×10^{-2}	0.385×10^{-2}	10
	2	3000	5	.50	.125	9
XIII	1	3400	2	2.76×10^{-2}	2.400×10^{-2}	27
	2	3400	2	2.76	.785	31
IX	1	3500	1	1.23×10^{-2}	1.040×10^{-2}	26
	2	3500	1	1.23	.385	30
X	1	3500	5	1.23×10^{-2}	1.040×10^{-2}	35
	2	3500	5	1.23	.385	33
XI	1	3500	10	1.23×10^{-2}	1.040×10^{-2}	41
	2	3500	10	1.23	.385	38
II	1	4000	16	0.50×10^{-2}	0.385×10^{-2}	110
	2	4000	16	.50	.125	140

TABLE II. - DOPING CONDITIONS FOR

TUNGSTEN TENSILE SPECIMENS

Time, hr	Temperature, °F	Oxygen content, ppm
5	3000	10
3	3400	30
15	3500	50

TABLE III. - HARDNESS DETERMINATIONS IN

OXYGEN-DOPED TUNGSTEN

Oxygen content, ppm	Vickers hardness number[a]		
	Within grain	Across grain boundary	Single crystal
2	---	---	320
4	343	340	---
10	337	351	---
20	---	---	317
30	325	341	---
50	317	342	---

[a]An average of at least three indentations using a 1 kg load.

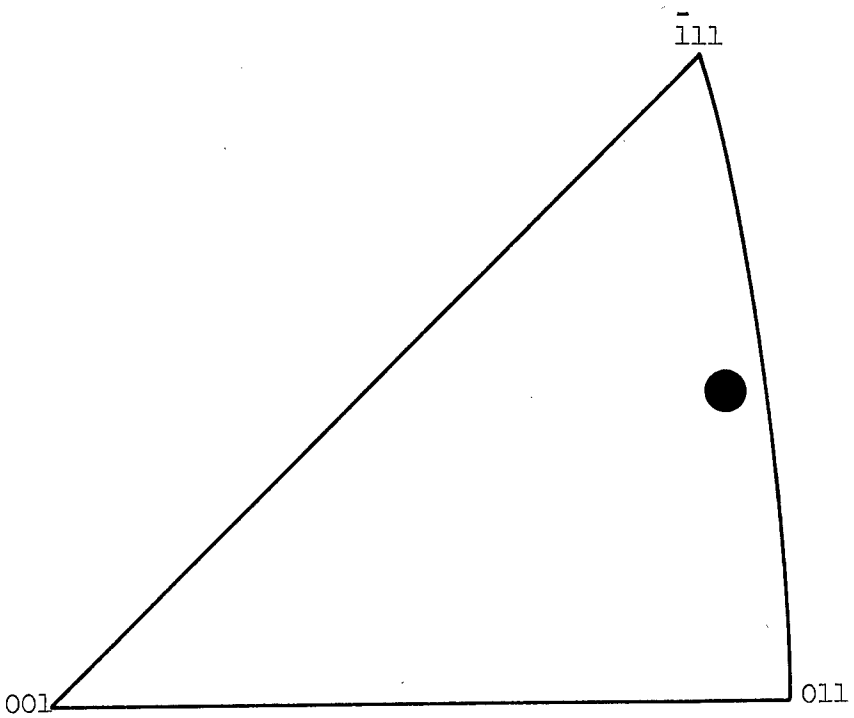


Figure 1. - Orientation of seeded single crystals.

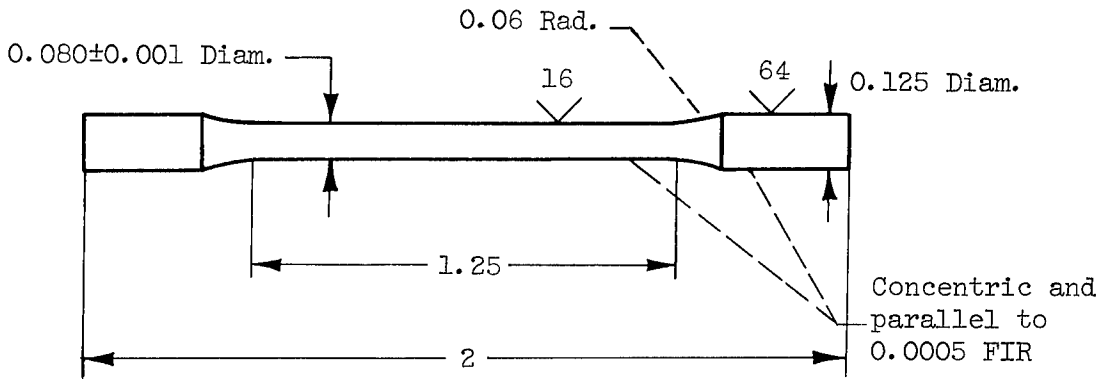
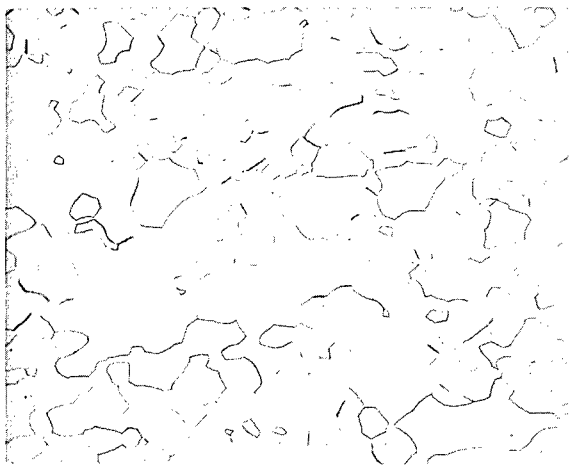
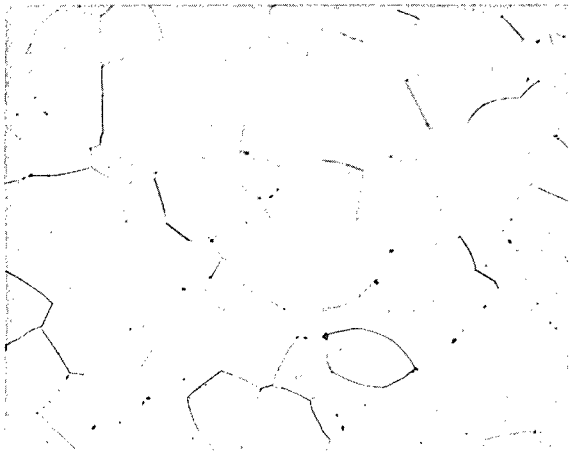


Figure 2. - Buttonhead tensile specimen. (All dimensions in inches.)

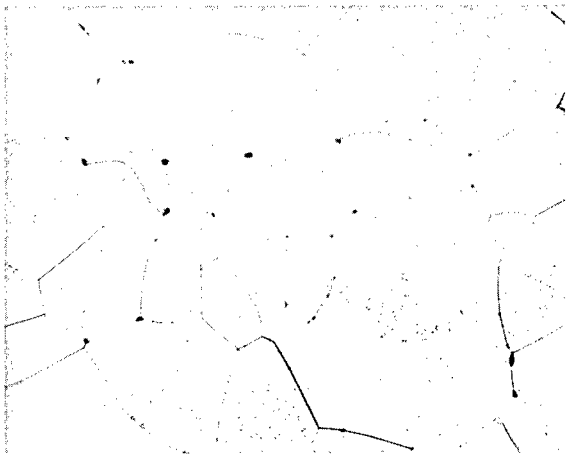


(a) Recrystallized commercial tungsten; undoped; 4 ppm oxygen.

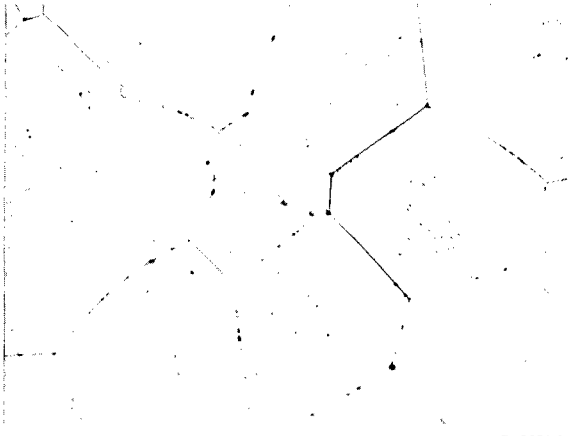
(b) 5 hours at 3000° F; 10 ppm oxygen.



(c) 1 hour at 3500° F; 28 ppm oxygen.



(d) 5 hours at 3500° F; 35 ppm oxygen.



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(e) 10 hours at 3500° F; 40 ppm oxygen.

Figure 3. - Typical microstructures of doped and undoped tungsten. ×250.

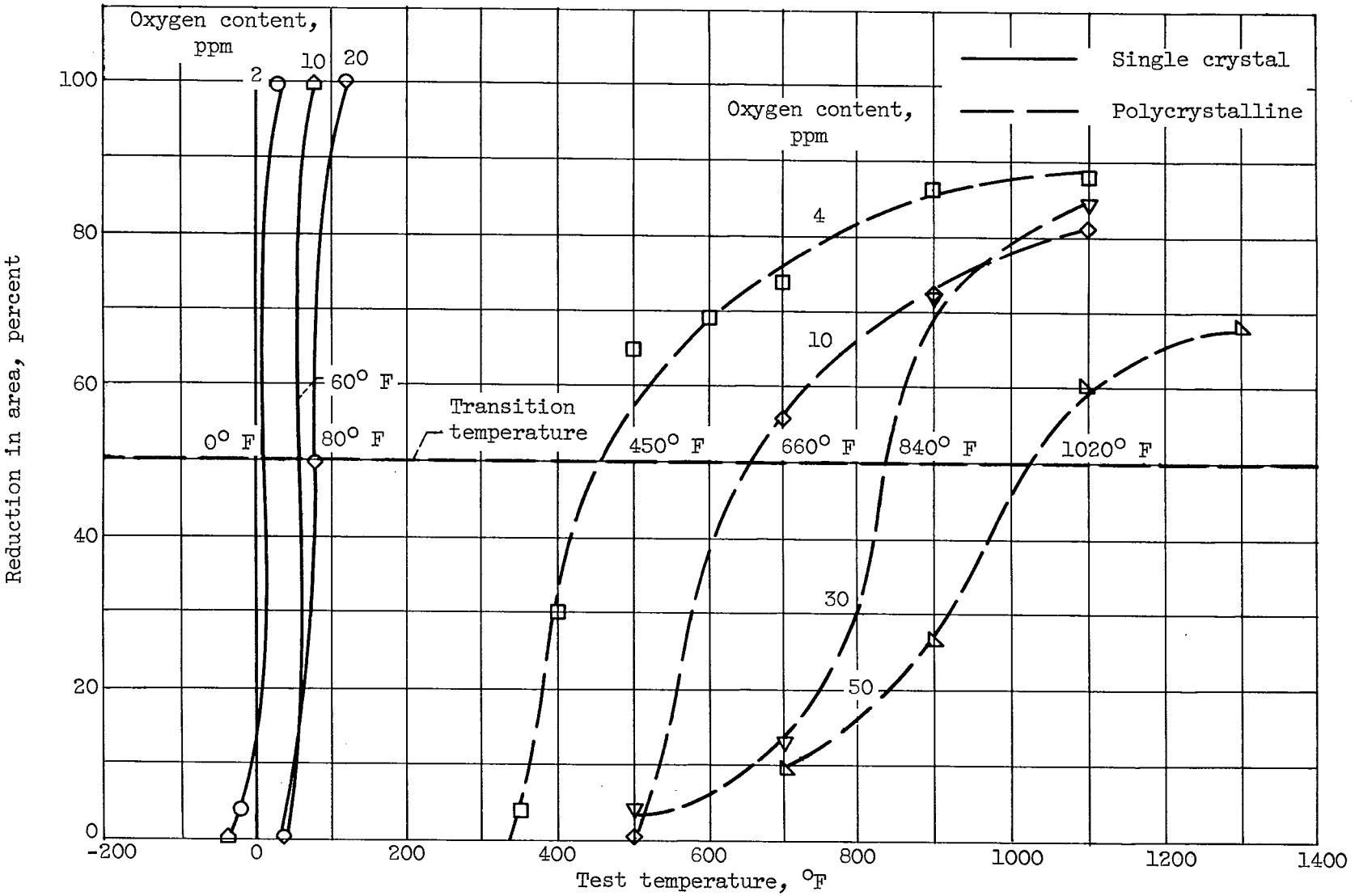


Figure 4. - Effect of oxygen on ductility of tungsten.

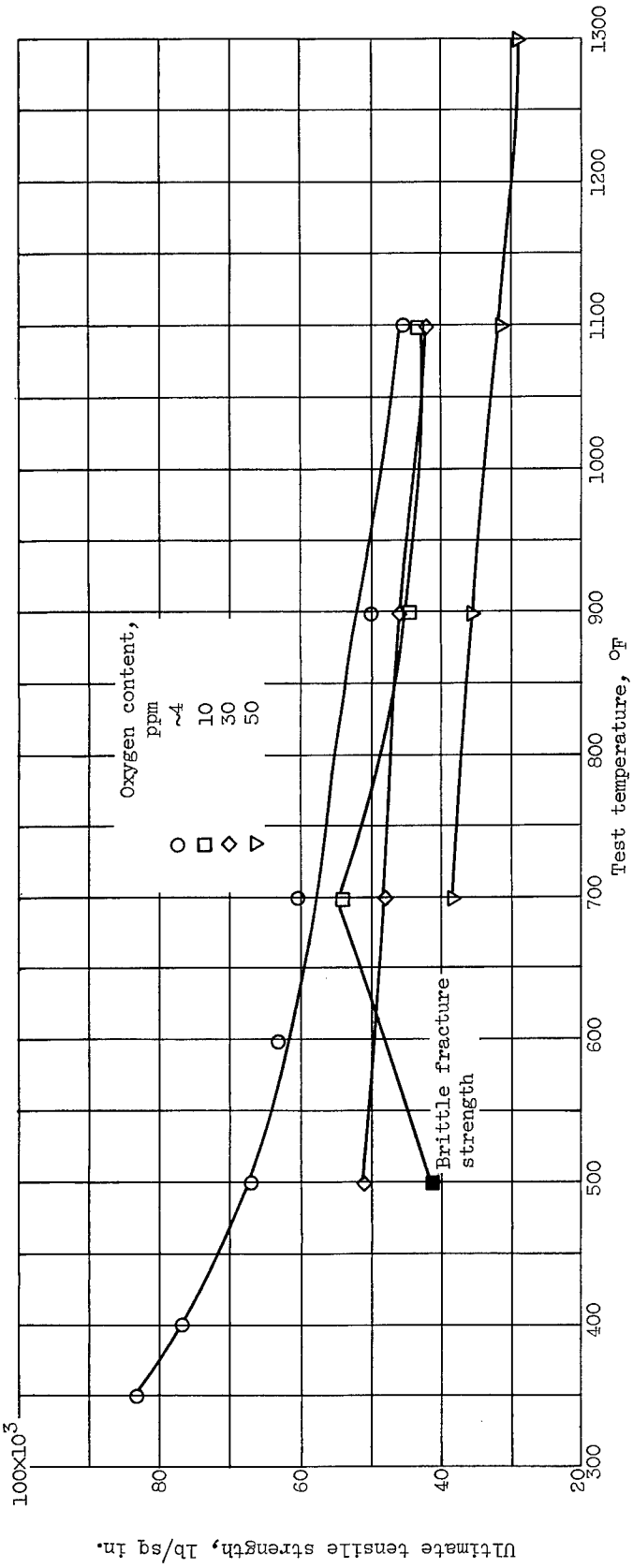


Figure 5. - Effect of oxygen on ultimate tensile strength of polycrystalline tungsten.

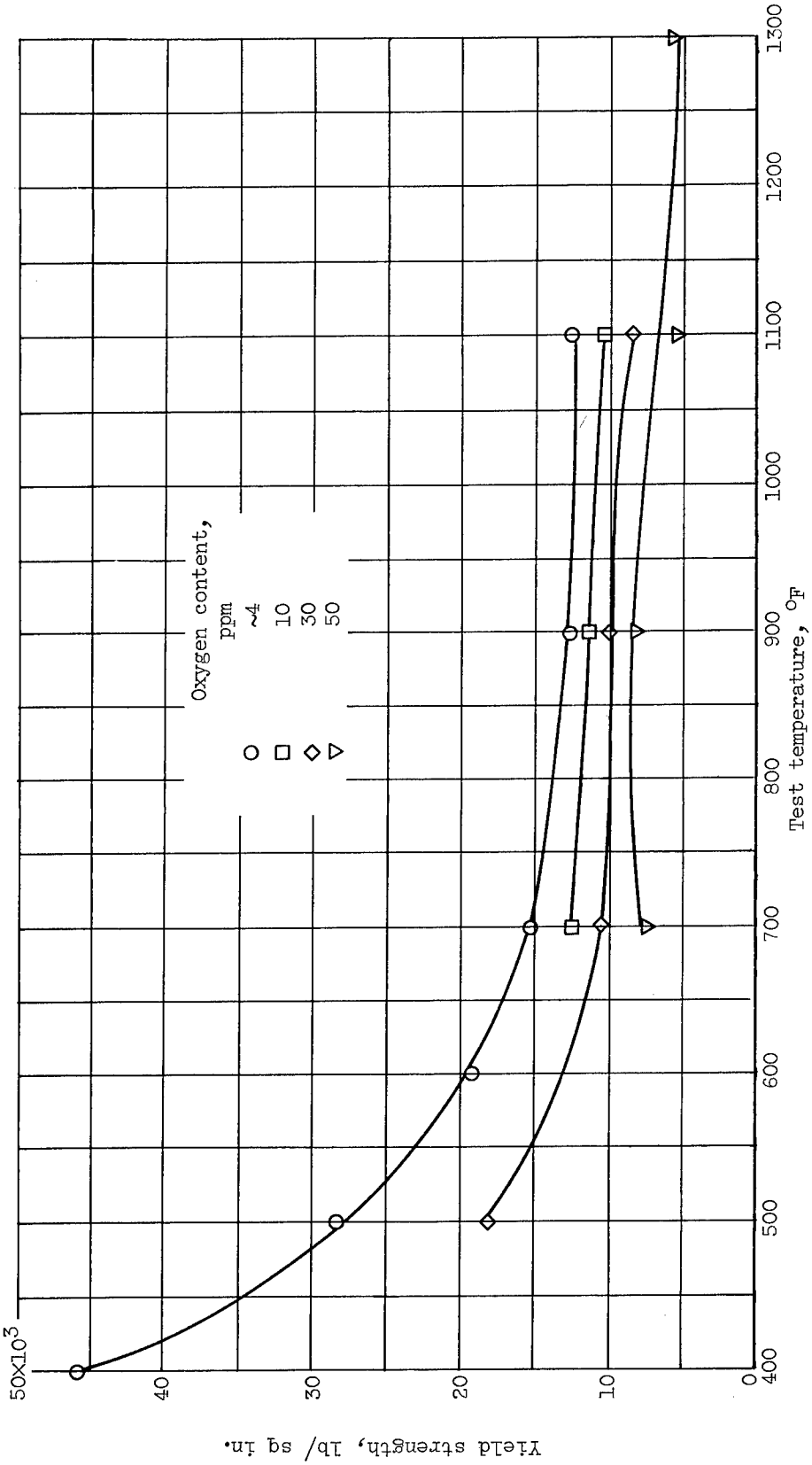
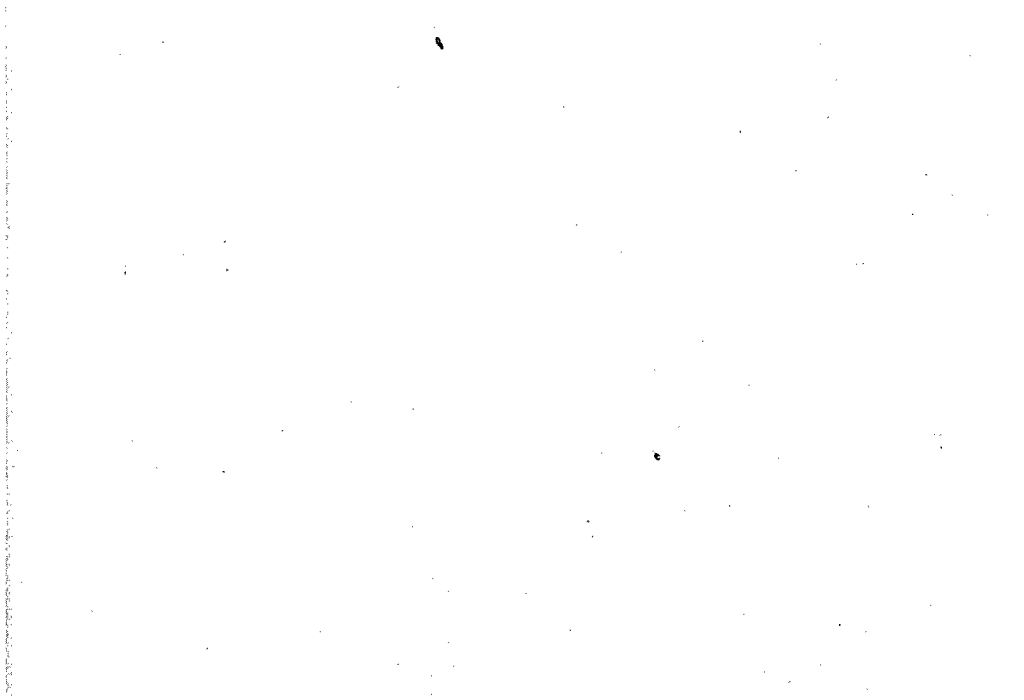
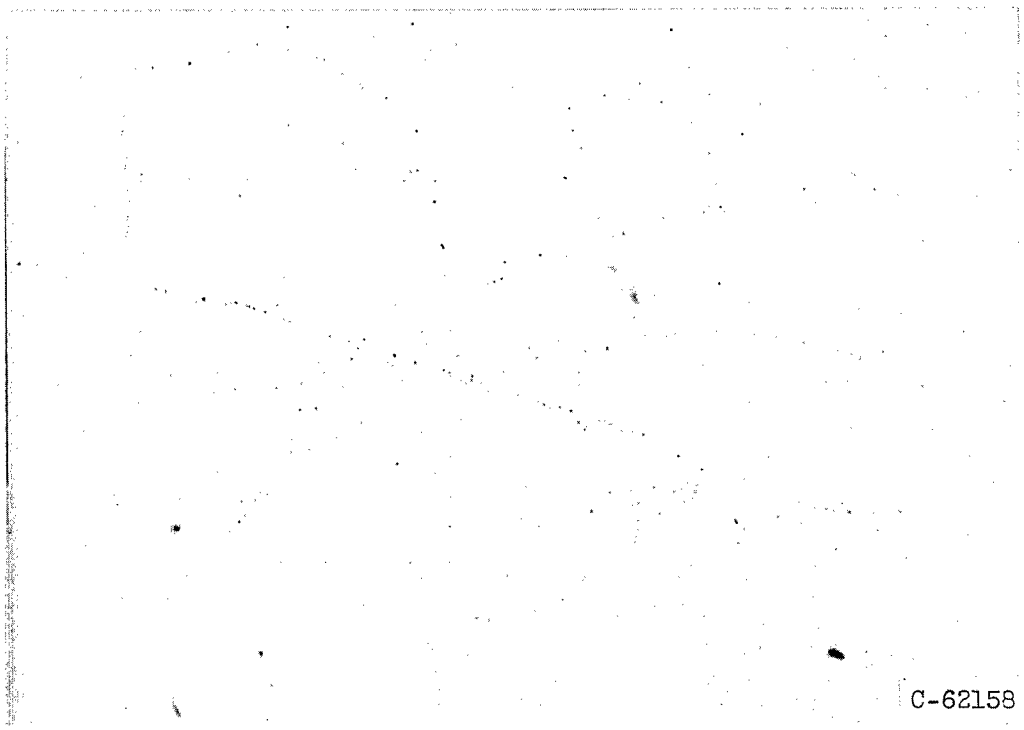


Figure 6. - Effect of oxygen on yield strength of polycrystalline tungsten.



(a) Zone-melted single crystal; 2 ppm oxygen.



(b) Doped, 20 ppm oxygen.

Figure 7. - Microstructures of undoped and doped single crystals. ×250.

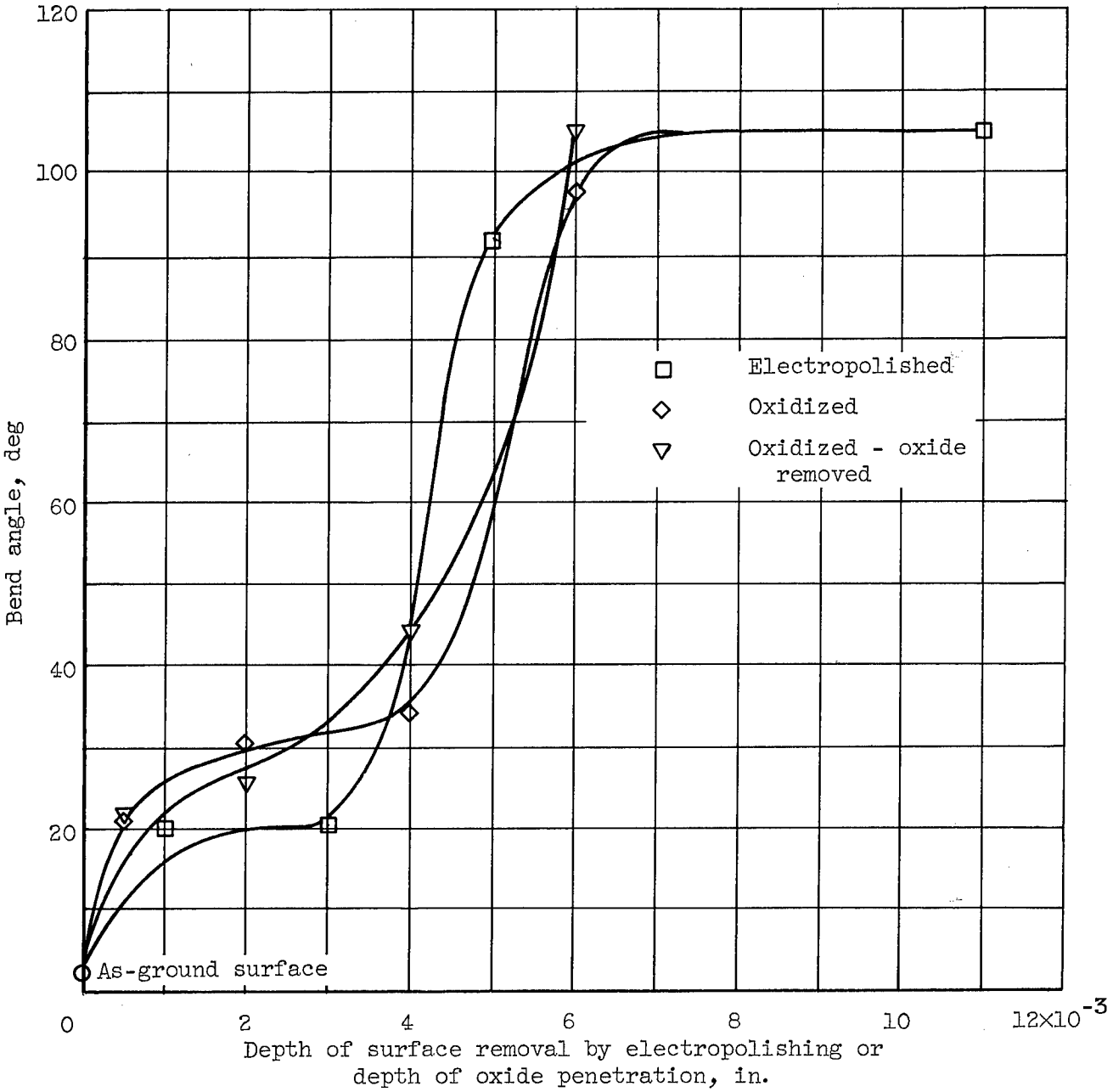


Figure 8. - Effect of oxidation at 1500 °F on ductility of tungsten.

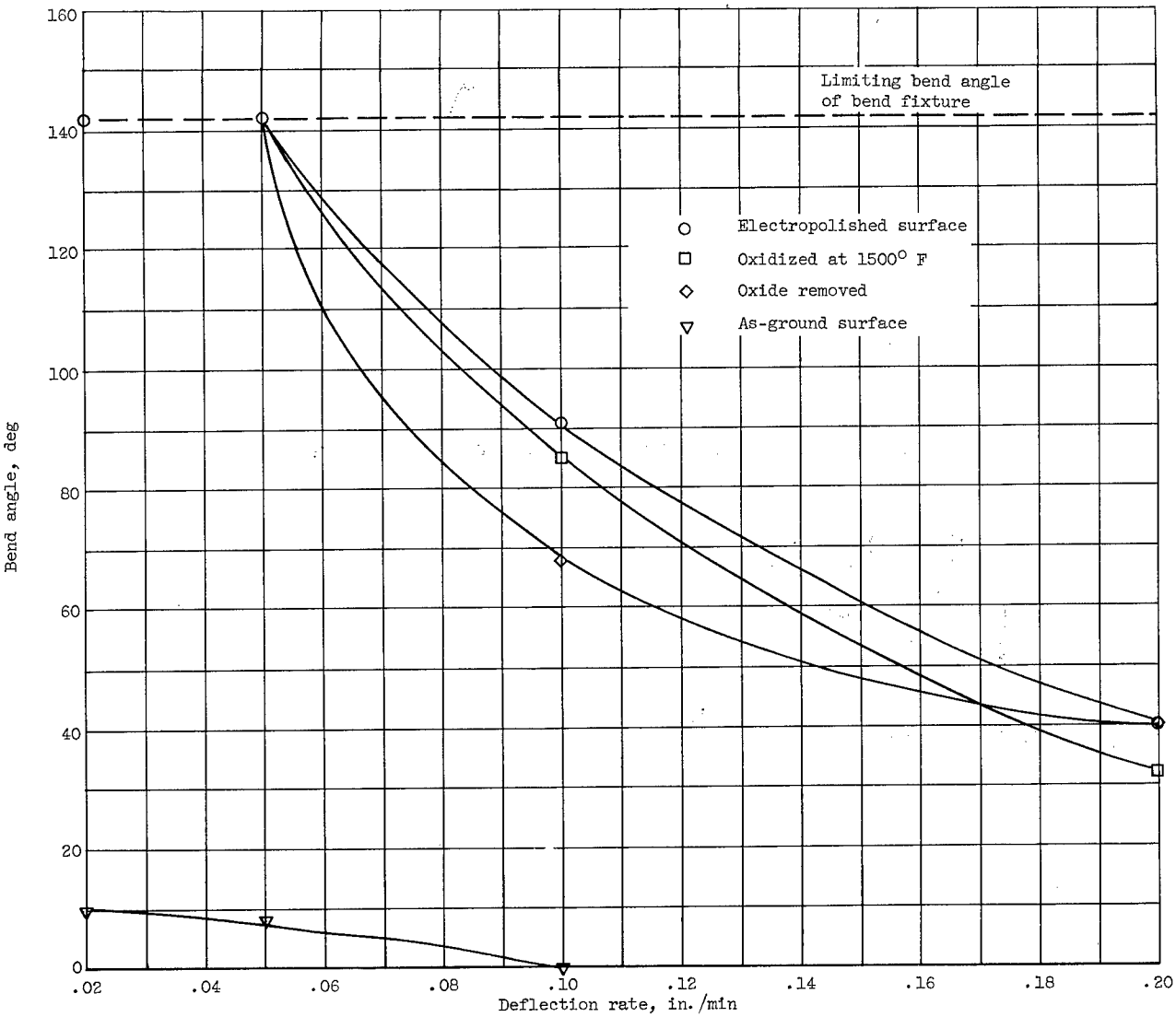
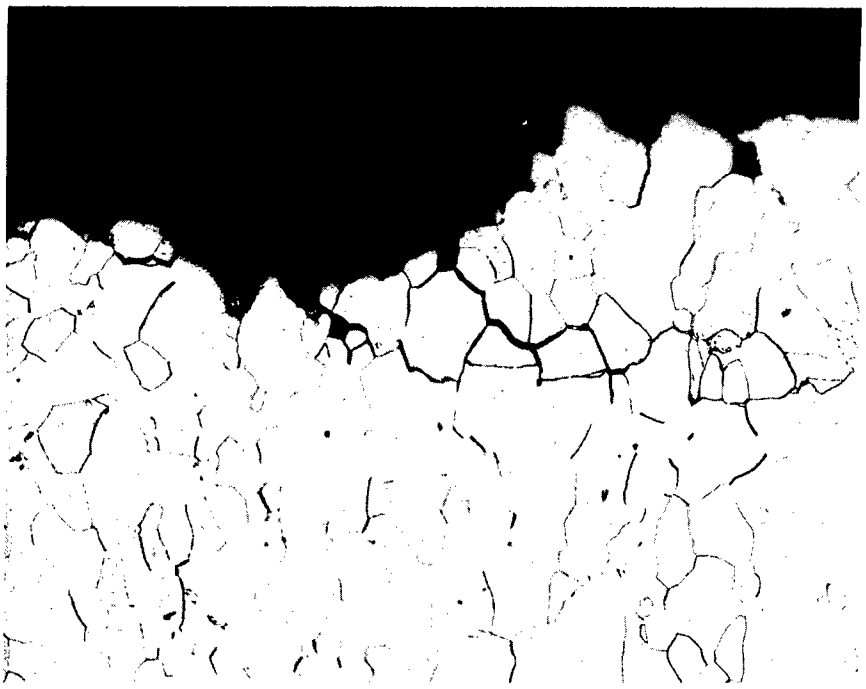


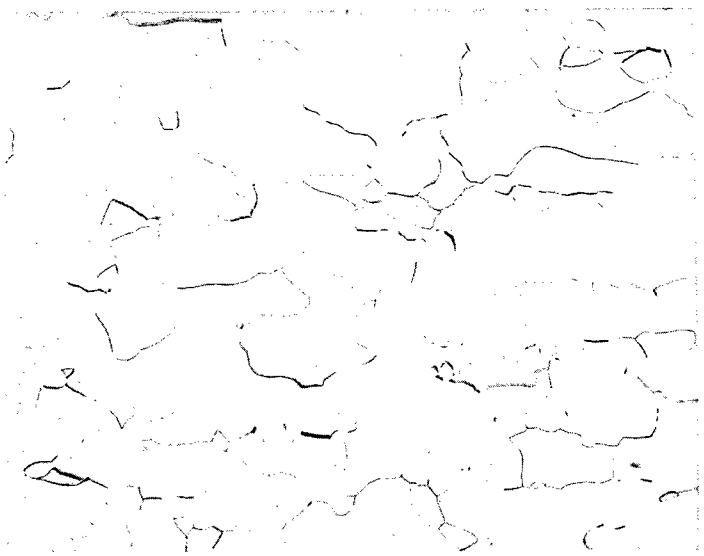
Figure 9. - Effect of surface oxide on ductility of tungsten.



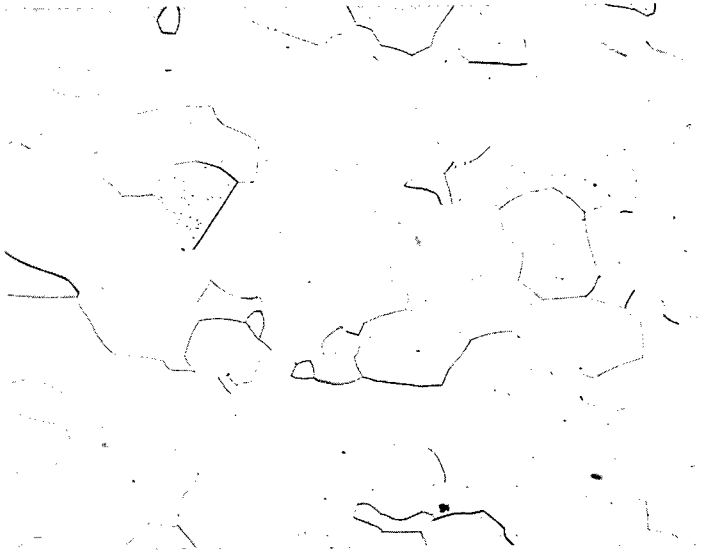
(a) Recrystallized commercial tungsten; undoped; 4 ppm oxygen.

(b) Doped, 30 ppm oxygen.

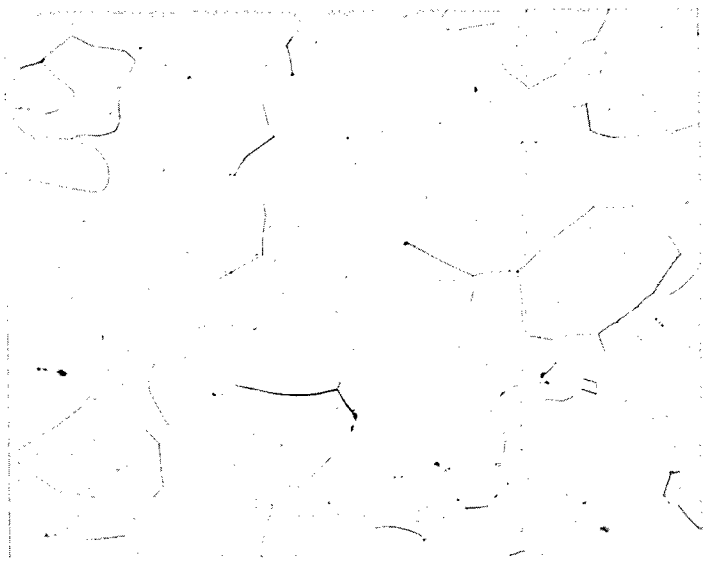
Figure 10. - Brittle intergranular fracture in doped and undoped tungsten. ×250.



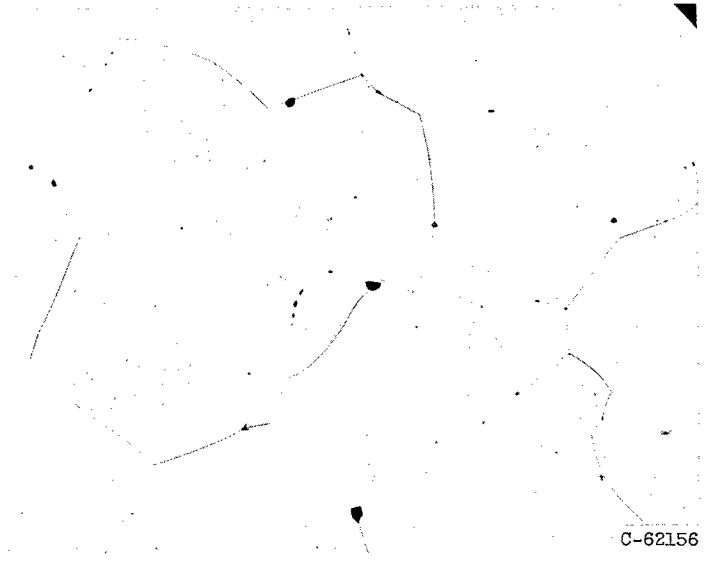
(a) Recrystallized commercial tungsten; undoped, 4 ppm oxygen.



(b) Doped, 10 ppm oxygen.



(c) Doped, 30 ppm oxygen.



(d) Doped, 50 ppm oxygen.

Figure 11. - Comparison of grain size of doped and undoped tungsten. ×250.