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TECHNICAL NOTE

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AN EXPLORATORY INVESTIGATION OF SOME FACTORS INFLUENCING THE ROOM-TEMPERATURE DUCTILITY OF TUNGSTEN

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SUMMARY

Specimens having an initial diameter of 0.125 inch were cut from commercially pure sintered and swaged tungsten rods. The effect of various surface treatments on the ductility of tungsten was evaluated by the use of a bend test. The criterion used to determine the relative ductilities was the final bend angle of the specimens.

Results showed that the bend ductility of the tungsten specimens increased with increasing depths of the surface removed by electropolishing. When specimens electropolished to a depth sufficient to produce a marked increase in the ductility over that of the as-received surface condition were subsequently scratched with emery paper, the ductility achieved by electropolishing was greatly reduced. Removal of similar depths of the surface by grinding as were removed by electropolishing did not produce any appreciable increase in the ductility of the specimens. The ductility of specimens tested in the as-received surface condition and those having an electropolished surface exhibited a great sensitivity to deflection rate, the electropolished specimens being more ductile over the range of deflection rates tested. These exploratory results illustrated qualitatively the importance of surface condition on the room-temperature ductility of tungsten and indicated the importance of relatively fine scratches on the ductility of tungsten.

INTRODUCTION

Tungsten, because of its high melting point (6170° F) and good strength in the very high temperature range of 2500° to 3700° F (ref. 1), is being considered for use as a structural material in advanced aircraft and missiles. One of the main factors limiting the use of tungsten is its lack of ductility at room temperature. The mechanical properties of tungsten are known to be affected by the surface condition (ref. 2). Sedlatschek and Thomas (ref. 2) have reported that the transverse rupture strength and the ductility, as measured by deflection in a bend test of tungsten specimens 2.5 millimeters in diameter, were improved by electropolishing. Their transverse rupture strength measurements did not indicate any definite relation between the changes in mechanical properties and the thickness of the layers removed for diameter reductions from 20 microns (0.00079 in.) to 64 microns (0.0025 in.).

Reasons for the improvement of mechanical properties of tungsten by electropolishing the surface have not been well established. Possible beneficial effects of surface removal by electropolishing include the following:

(1) Electropolishing the surface decreases surface roughness, thus removing scratches that may act as stress-concentration sites and promote brittle fracture. The ductile to brittle transition temperature of another body-centered cubic metal, chromium, has been shown to be increased by scratching an electropolished surface (ref. 3).

(2) Electropolishing may remove contaminated surface layers in which cracks readily form and propagate.

(3) Electropolishing may remove surface layers having high concentrations of residual stresses in which cracks are readily generated.

The purpose of this preliminary investigation was to indicate some of the factors influencing the room-temperature ductility of tungsten in order to achieve a better understanding of the effects of surface condition.

For this study, specimens of 1/8-inch-diameter commercially pure, wrought, sintered tungsten were given various surface treatments and were then evaluated in room-temperature bend tests. The following effects were investigated:

(1) Influence of removal of increasing depths of the surface by electropolishing or by surface grinding

(2) Influence of different degrees of surface roughness achieved by abrading electropolished specimens with different grades of emery paper

(3) Influence of deflection rate on ductility of specimens with as-received (swaged and cleaned) and electropolished surfaces.

MATERIALS AND PROCEDURES

Materials

Commercially pure 0.125-inch-diameter tungsten rods were used for this investigation. The tungsten rods came from two separate powder-metallurgy lots, which will be designated lots B and G. Rods from lot B had been swaged and centerless ground to a 16-microinch average root-mean-square surface as measured by a surface analyser. Rods from lot G had been swaged and subsequently cleaned in a molten caustic dip and then rinsed in hot water. The resulting finish was 23-microinch average root-mean-square surface. All specimens were tested in the cold-worked condition. The fibrous structure shown in figure 1 is typical of the cold-worked tungsten specimens.

Specimens 2 inches long were cut from the 1/8-inch-diameter rods that were initially approximately 6 feet in length. It was noted that a great variation existed in the ductility of specimens cut from different rods from lot G when tested in the as-received surface condition.

Subsequent tests of the specimens from lot G after electropolishing revealed that the rods could be separated into two groups with regard to their bend ductility. Those that exhibited some ductility when tested in the as-received surface condition underwent a sevenfold increase in bend ductility after removal of 0.0025 inch from the surface by electropolishing. Those that exhibited only a very small amount of bend ductility when tested in the as-received surface condition showed no increase in ductility after removal of 0.010 inch by electropolishing.

Because of the variation in ductility among the rods, the number of specimens available for one series of tests was limited to those that could be cut from one rod. In view of the nature of the study, it was desirable to use specimens from those rods that exhibited some room-temperature bend ductility before any surface treatment.

Rods from lot B were available at the time the program was initiated and were used for the preliminary tests. The specimens from lot B were employed for two series of tests, the first being an investigation of the effect of removing various depths of the surface by electropolishing and the second being a study of the effect of removing similar depths of the surface by grinding. All other test specimens were sectioned from rods from lot G.

Specimens selected from one of the rods from lot B, one of the rods exhibiting some ductility from lot G, and one of the more brittle rods from lot G were analyzed for impurity content. The results of the spectrographic and chemical analysis are as follows:

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Element	C	Al	Cr	Cu	Fe	Mo	Ni	Si	O_2	N_2
Composition, ppm										
Lot B - ductile	123	<5	<5	<5	30	50	<5	<5	10	1
Lot G - ductile	42	12	<5	<5	15	12	<5	<5	5	1
Lot G - brittle	86	15	<5	<5	15	12	<5	<5	3	1

Procedure

Specimen preparation. - Before testing, the specimens were given one of the following treatments:

(1) Increasing depths of the surface removed by electropolishing: The apparatus developed for the electropolishing treatment permitted both rotation of the specimen and stirring of the electrolyte. This technique allowed dimensional tolerances to be held within ±0.0005 inch. Polishing conditions included the following:

(a) Electrolyte, 2 percent sodium hydroxide

(b) Current density, 3.0 amp/sq in.

(c) Rotation of specimen, 3 rpm

(d) Stirring of electrolyte

(e) Cathode, 0.125-inch-diameter Inconel rod, parallel and 2 in. away from test specimen

(f) Temperature of electrolyte, 25° C

(2) Specimens electropolished to a specified diameter and subsequently roughened with various grades of emery paper: The specimens in this series of tests were electropolished to a diameter of 0.115 inch and then scratched circumferentially with emery paper of three different grit sizes. The specimens were scratched until the entire electropolished surface appeared to be roughened.

(3) Increasing depths of the surface removed by grinding: The tungsten specimens were ground to the desired diameter using a 200-grit silicon carbide wheel for the finish grinding. The root-mean-square value of the ground surface as determined by the surface-roughness analyser was in the range of 25 to 30 microinches.

Testing. - All of the bend tests were performed at room temperature at deflection rates of either 0.02 or 0.05 inch per minute.

An Instron Tensile Testing Machine equipped with a 1000-pound tensile load cell was employed for testing the specimens. The bend fixture is shown in figure 2. The span L was 1.187 inches for one fixture and 0.813 inch for a second. The radius of the loading point and the reactants was 0.094 inch. The Instron Tensile Testing Machine is equipped with a strip-chart recorder that plots a load-deflection curve as the specimen is tested. Because of the design of the bend rig, it was necessary to apply a small load (i.e., less than 1 lb) to the specimen in order to assure accurate alinement of the specimen before testing. The tests were allowed to continue until the specimens either fractured or bent through an angle such that the specimen slipped through the bend rig.

Bend ductility and strength measurements. - The bend angles of the specimens were used as the basic criteria to determine the effect of the various surface treatments. The bend angles were measured after reconstructing the fractured pieces. In a few cases (some of the specimens having a bend angle of less than 25°), multiple fracture occurred, and accurate reconstruction of the three or more pieces was difficult. For these cases the bend angle was determined from the strip-chart recording of the test. The following equation was employed:

$$\text{Bend angle} = \theta = 180^{\circ} - 2\Phi$$

$$\tan \Phi = \frac{1}{2} \frac{L}{x}$$

where L is the span of bend fixture in inches and x is the travel of the crosshead in inches. Although x includes the elastic strain, a comparison of measured and calculated values of bend angles for specimens that had only a single fracture and bend angles less than 25° indicates that the calculated values were not in error by more than 3°.

Calculation of the maximum fiber stress in the specimen indicated that no definite correlation existed between depth of surface removed and calculated stress values, which is in agreement with the work of Sedlatschek and Thomas (ref. 2).

RESULTS AND DISCUSSION

Effect of Removal of Surface by Electropolishing

Tungsten specimens from lot G in the as-received (swaged and cleaned) surface condition were electropolished to diameters of

0.124, 0.122, and 0.120 inch (diameter reductions of 1, 3, and 5 mils) and tested by means of a bend test at room temperature using the bend fixture having a 0.813-inch span and at a deflection rate of 0.02 inch per minute. Figures 3 and 4 illustrate the effect on the bend ductility as a result of removing the surface by electropolishing. The 153° bend angle represents the maximum bend angle permitted by the bend fixture; specimens that bend to this angle without fracturing slip through the fixture without further bending. The results show that the bend ductility increases with increasing depth of surface removed by electropolishing in the range of 0.5 to 2.5 mils (reduction in diameter of 1.0 to 5.0 mils).

As previously indicated, improvement of the room-temperature ductility of tungsten rods by electropolishing could result from removal of a highly contaminated surface, removal of cracks and the roughened surface resulting from the swaging and grinding processes, or removal of a highly stressed surface layer. This set of experiments confirmed the results previously reported (ref. 2) that electropolishing substantially increases the room-temperature ductility of tungsten. The results show that diameter reductions (by electropolishing) of only 0.005 inch can produce as much as a sevenfold increase in bend angle. The results, however, provide little information as to why the electropolishing treatment is effective.

Examination with the electron microscope, however, indicated a significant decrease in surface roughness as the specimen was electropolished successive amounts. With a diameter reduction of only 1 mil, many irregular fissures were apparent (fig. 5). After removal of 3 mils, only a few fissures were evident, but many pits were noted. The surface of a specimen that had a diameter reduction of 5 mils by electropolishing showed a relatively smooth surface with only very shallow pits. Thus, the results of the electron-microscope examination strongly suggest that removal of surface roughness may be a main factor responsible for the improved ductility achieved by electropolishing.

The fact that electropolishing the more brittle rods from lot G did not result in such a marked increase in bend ductility indicates that some factor other than surface roughness also plays an important role in causing the room-temperature brittleness of tungsten. Differences in chemical composition, particularly the interstitial impurity content, are believed to influence the ductility of tungsten, but chemical analysis of specimens showing different degrees of bend ductility did not establish a definite relation between purity and room-temperature bend ductility.

In an attempt to establish more firmly that the ductility of tungsten is influenced by surface roughness, the following experiment was conducted: Specimens from lot G were electropolished to a diameter

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of 0.115 inch (reduction in diameter of 10 mils), in which condition they show excellent bend ductility (153° bend angle). The electropolished specimens were then scratched circumferentially with 3/0-, 1-, and 3-grit emery paper and subsequently tested in bending using the fixture with a 0.813-inch span and a deflection rate of 0.02 inch per minute. The root-mean-square values of the surface roughness resulting from the circumferential scratches were 7, 13, and 16 microinches, respectively, for specimens scratched with 3/0-, 1-, and 3-grit emery papers. The root-mean-square values of the as-received and electropolished surfaces were 24 and 4 microinches, respectively. The results of the tests are illustrated graphically in figure 6. Even the relatively shallow scratches resulting from scratching with 3/0 emery paper drastically reduced the ductility. This series of tests confirms the importance of surface roughness as being a major factor influencing the room-temperature ductility of tungsten.

Effect of Removal of Surface by Grinding

If removal of a surface layer containing a high concentration of impurities was responsible for the increase in ductility, removal of the surface by grinding would be expected to provide the same beneficial results as surface removal by electropolishing. In order to better determine the reasons for the resulting increase in ductility as a result of the electropolishing treatment, specimens were ground to diameters equal to those of the electropolished specimens.

A rod from lot B was selected for the source of material for this series of tests. One set of specimens was electropolished to diameters of 0.124, 0.120, 0.115, 0.110, and 0.105 inch (diameter reductions of 1, 5, 10, 15, and 20 mils, respectively), and the second set was ground to the same diameters. The specimens were tested using the bend fixture having a span of 1.187 inches and at a deflection rate of 0.05 inch per minute. Figure 7 shows a comparison of the results. The 125° bend angle is the limiting bend angle of this bend fixture. As with specimens from lot G, the electropolished specimens exhibited a rapid increase in bend ductility as the depth of the removed surface layer increased. Surface removal by grinding did not increase the bend ductility significantly. Since removal of the same amount of material by electropolishing and by grinding does not have similar effects on ductility, it is evident that a concentration of impurity atoms in the outer surface layer is not the sole mechanism for causing brittleness at room temperature. Although a concentration of impurity atoms in the outer surface layer may contribute to the lack of ductility of tungsten at room temperature, other factors (such as surface roughness) must also be involved.

Effect of Deflection Rate

It has previously been shown (ref. 4) that the ductility of tungsten, as well as other body-centered cubic metals, is decreased by increasing the strain rate. In order to determine whether electropolished specimens were as strain-rate sensitive as the as-received specimens, the following series of tests was run. Specimens in the as-received (swaged and cleaned) surface condition and those that had been electropolished to a diameter of 0.115 inch (diameter reduction of 10 mils) were tested at deflection rates ranging from 0.005 to 0.010 inch per minute in the bend fixture having a span of 0.813 inch. The results are shown in figure 8. It should be noted that tungsten exhibits a great sensitivity to strain rate regardless of the surface condition and that the electropolished specimens were more ductile than those in the as-received surface condition at any given deflection rate over the range of deflection rates tested.

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SUMMARY OF RESULTS

An investigation has been undertaken to study some of the causes of the room-temperature brittleness of tungsten. The results of this preliminary study are as follows:

1. Removing the surface of the tungsten specimens by electropolishing increases the room-temperature bend ductility. A definite correlation existed between depth of surface removed and increase in the bend angle, with as much as a sevenfold increase in bend angle resulting from diameter reductions of 0.005 inch by electropolishing.

2. Specimens electropolished to a sufficient diameter to produce good bend ductility were subsequently scratched circumferentially with emery paper, the result being to reduce the ductility greatly. This confirms the importance of surface roughness on the ductility of tungsten.

3. Removal of the surface by grinding rather than by electropolishing failed to produce any measurable increase in the room-temperature bend ductility of the tungsten specimens. The fact that removal of the surface enhances ductility only if the resulting surface is very smooth further indicates that the removal of surface roughness is primarily responsible for the increased ductility. Other factors such as a high concentration of impurities in the surface layer seem to be of less importance.

4. The room-temperature bend ductility of tungsten, in both the electropolished and the swaged and cleaned surface condition, is very

sensitive to deflection rate. The electropolished specimens were more ductile than the specimens having the as-received surface condition at any given deflection rate over the range of deflection rates tested.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, May 12, 1960

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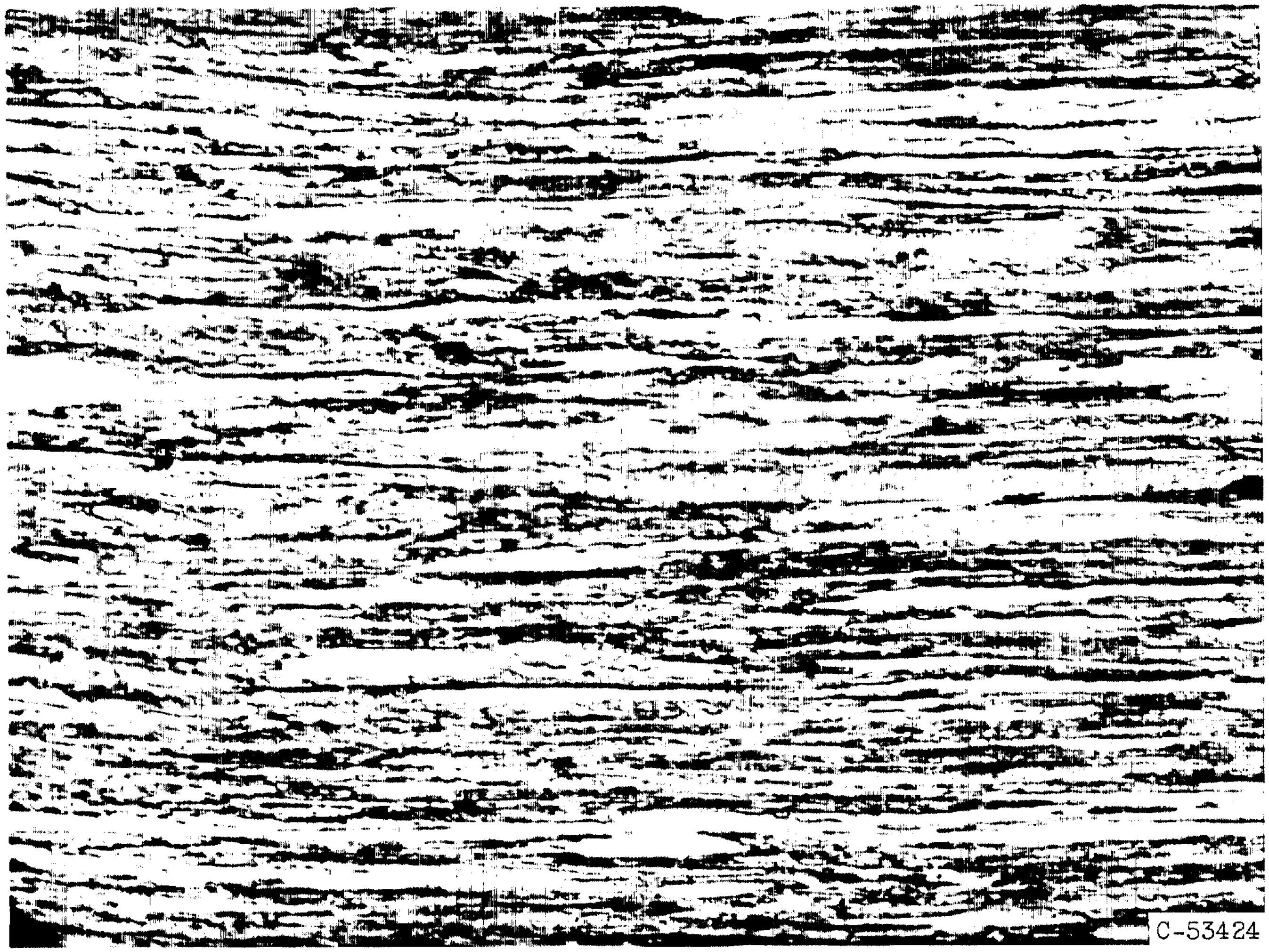

Figure 1. - As-received (swaged and cleaned) tungsten rod. X200.

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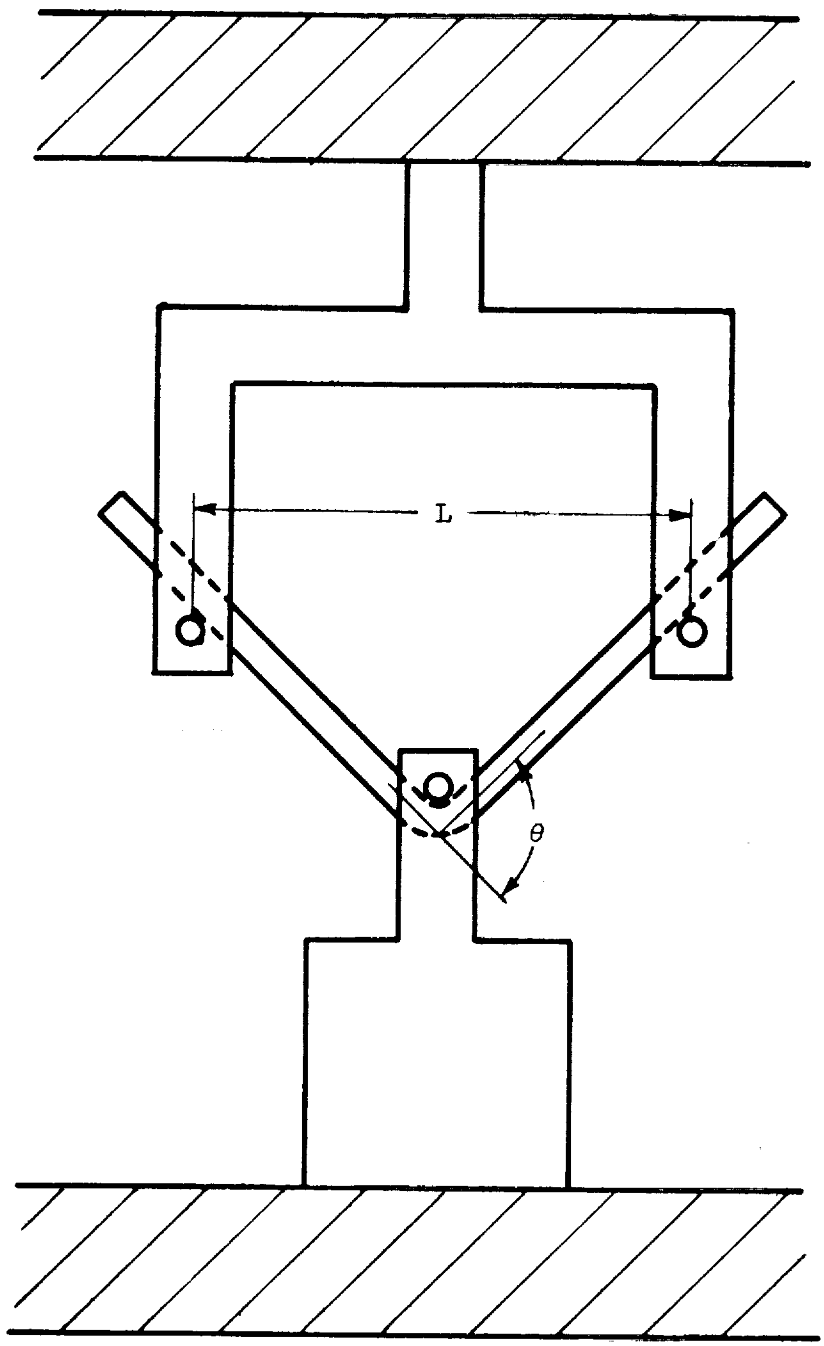


Figure 2. - Bend fixture.

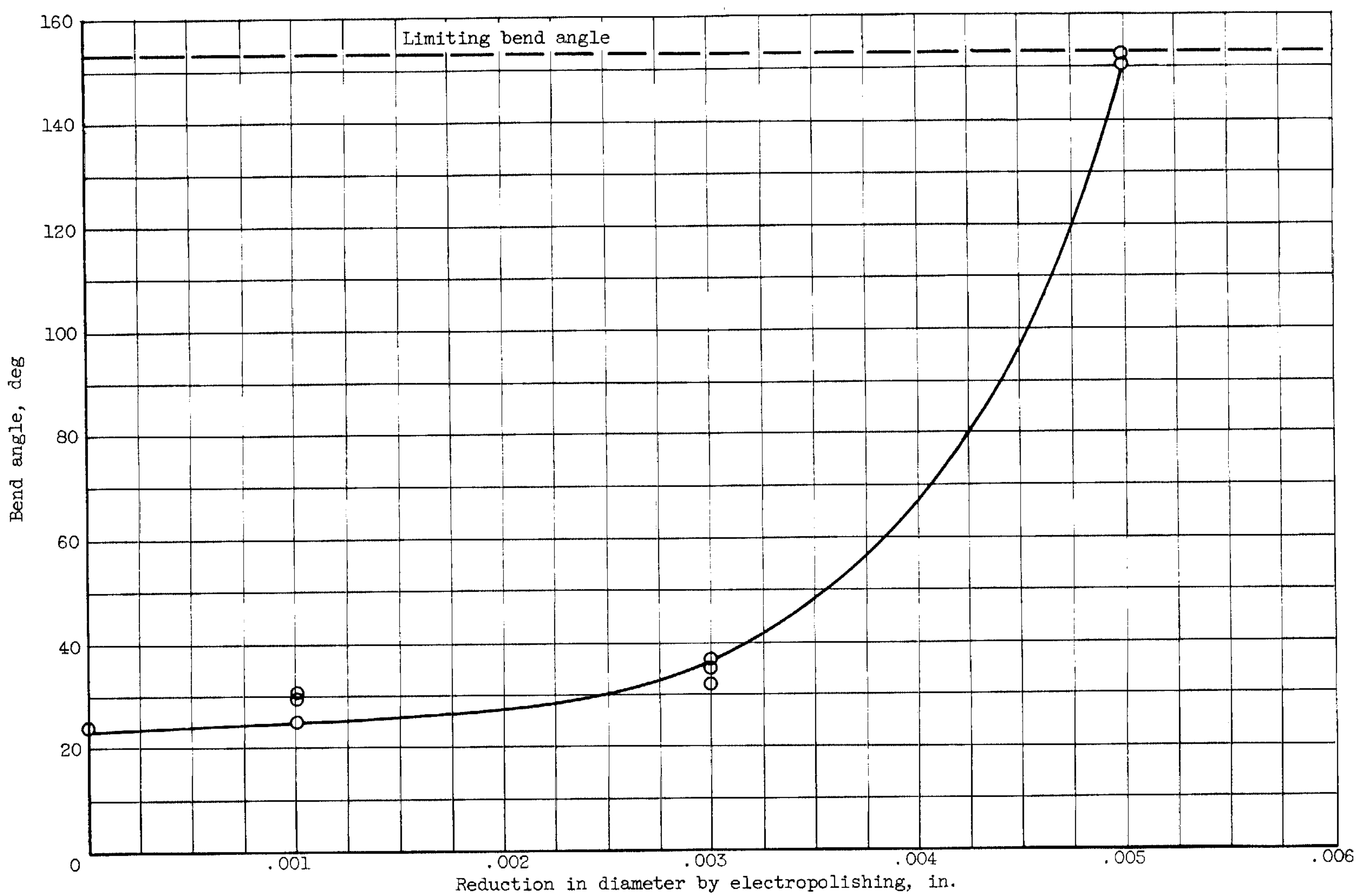
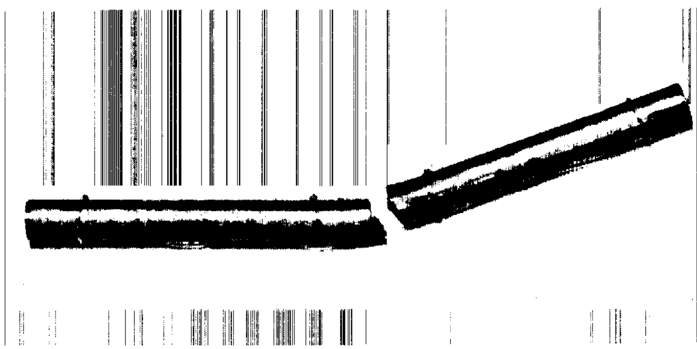
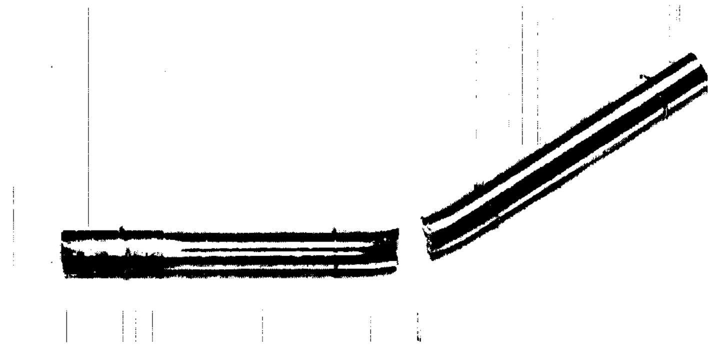


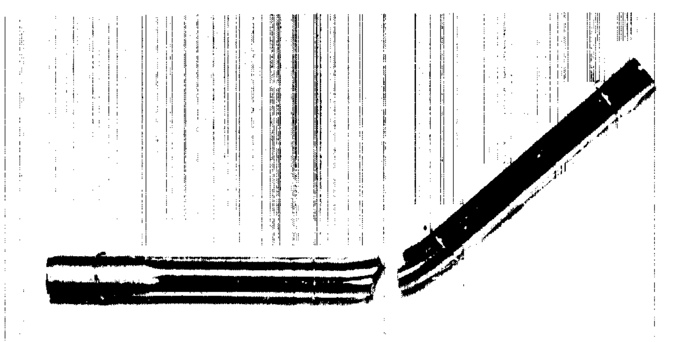
Figure 3. - Effect of removal of surface by electropolishing.



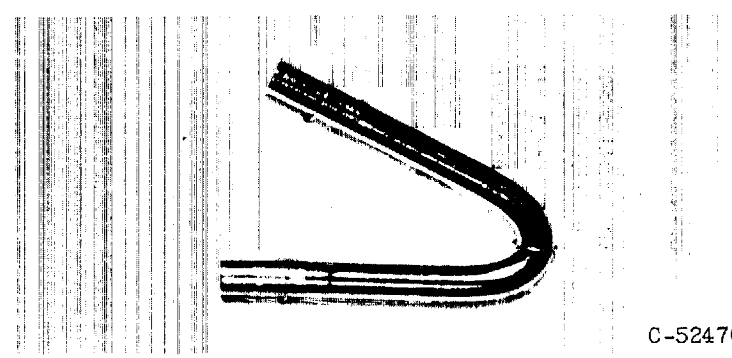
(a) As-received surface. Bend angle = 17°.



(b) Electropolished 0.001 inch. Bend angle = 30°.



(c) Electropolished 0.003 inch. Bend angle = 38°.



(d) Electropolished 0.005 inch. Bend angle = 153°.

Figure 4. - Effect of surface removal by electropolishing on room-temperature bend ductility of tungsten.

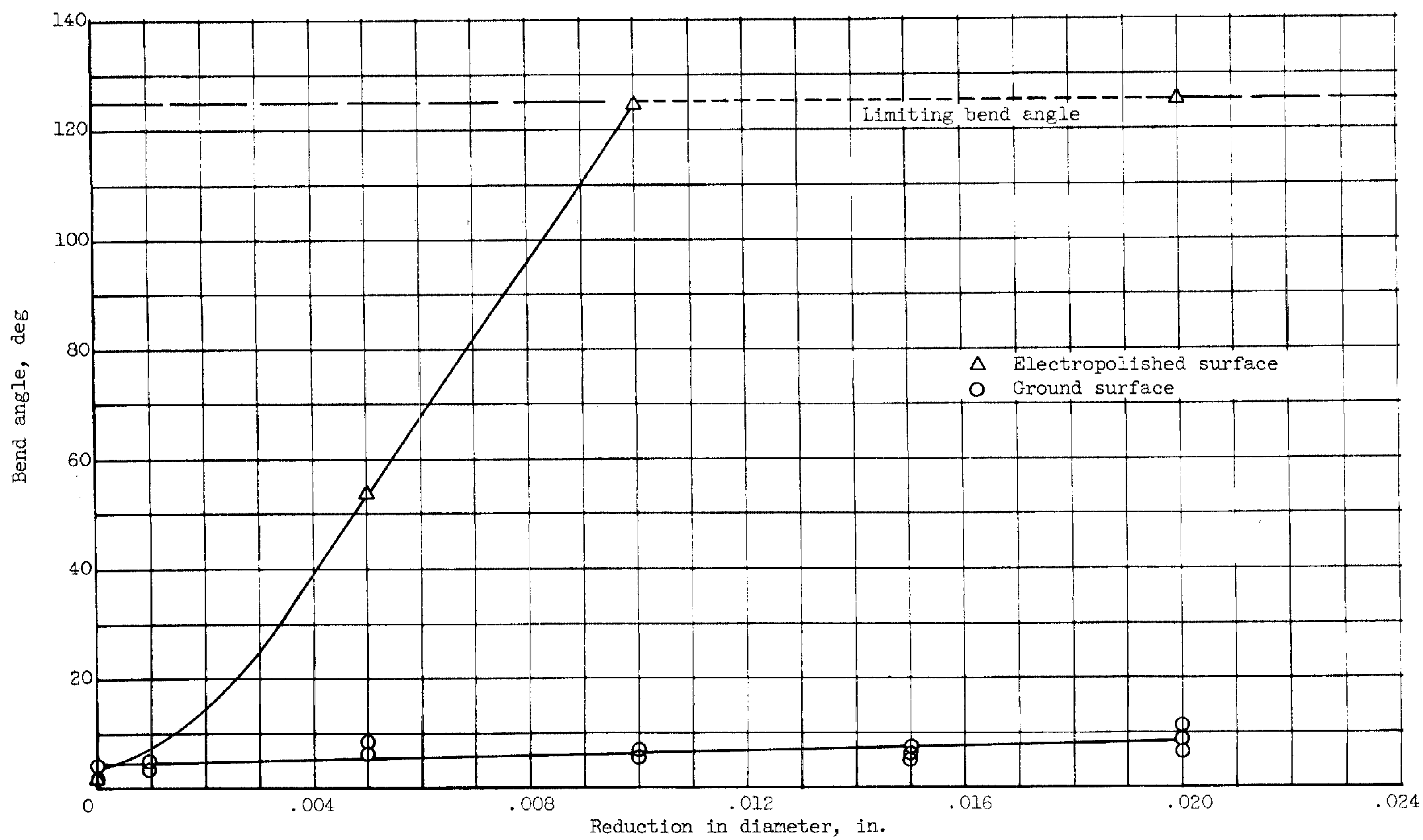


Figure 7. - Effect of surface roughness on ductility of tungsten.

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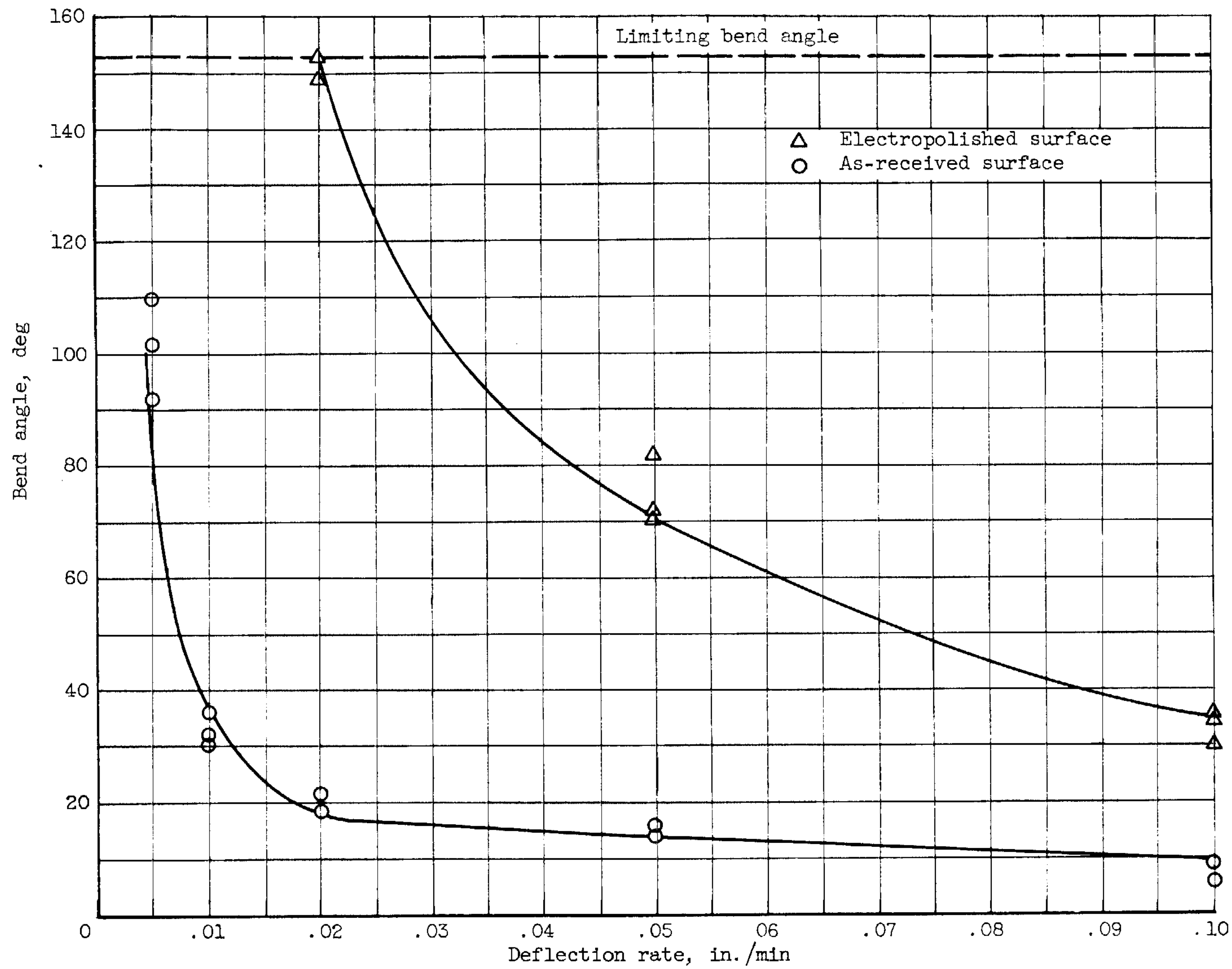

Figure 8. - Effect of deflection rate.