NASA TECHNICAL MEMORANDUM

NASA TM X-1252

NASA TM X-1252

EFFECT OF ORIENTATION ON DUCTILE TO BRITTLE TRANSITION OF TUNGSTEN SINGLE CRYSTALS

by Ralph G. Garlick

Lewis Research Center

Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JULY 1966

NASA TM X-1252

EFFECT OF ORIENTATION ON DUCTILE TO BRITTLE TRANSITION OF TUNGSTEN SINGLE CRYSTALS

By Ralph G. Garlick

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Clearinghouse for Federal Scientific and Technical Information
Springfield, Virginia 22151 – Price $1.00

EFFECT OF ORIENTATION ON DUCTILE TO BRITTLE TRANSITION OF TUNGSTEN SINGLE CRYSTALS

by Ralph G. Garlick

Lewis Research Center

31348

SUMMARY

An investigation was conducted of the ductile-brittle transition behavior of zone-refined tungsten single crystals as a function of orientation. Tensile-axis orientations ⟨100⟩, ⟨110⟩, and ⟨111⟩ were studied.

The transition behavior was determined to be highly orientation dependent. Crystals of the ⟨110⟩ orientation exhibited a sharp ductile-brittle transition based on reduction-in-area measurements and showed the lowest transition temperature, 205° K. Crystals of the ⟨100⟩ tensile axis exhibited a much more gradual transition with temperature from ductile to brittle behavior, with a transition temperature of about 380° K, based on a 50-percent reduction in area. The transition temperature of the ⟨111⟩ crystals was intermediate between those of the other orientations. Specimens of all orientations exhibited measurable plastic deformation at temperatures far below their respective ductile-brittle transition temperatures.

Orientation also significantly affected the tensile flow characteristics of the crystals. The ⟨110⟩ oriented specimens exhibited a high yield strength and a yield-point drop, and necking almost immediately followed yielding. In contrast, ⟨100⟩ and ⟨111⟩ crystals exhibited no yield-point drop and, for crystals tested above the transition temperature, showed considerable uniform elongation before necking. These orientations also exhibited conventional work-hardening behavior.

INTRODUCTION

Because of its high melting point and general refractory properties, tungsten is being investigated as a possible material for advanced aircraft and missiles. A major deterrent to its use is its lack of ductility at room temperature. It is believed that the low ductility may be associated with interstitial impurities. Support for this possibility has been found for the other group VI A metals molybdenum (ref. 1) and chromium (ref. 2). Evidence of similar effects of impurity elements in tungsten was found by Stephens (ref. 3), where the ductile to brittle transition temperature of single-crystal tungsten was increased 44 K degrees by addition of as little as 20 parts per million (by weight) of oxygen.

Some properties of high-purity tungsten have been determined at various laboratories through studies of zone-refined single crystals. Although investigators have reported on slip (refs. 4 to 6) and twinning (refs. 7 and 8) of these single crystals, a study of the ductile to brittle transition behavior has not been reported. The present investigation was initiated to determine the transition behavior of single-crystal tungsten as affected by crystal orientation at a constant low level of impurities.

MATERIALS AND PROCEDURE

Single-crystal tungsten rods 1/8 inch in diameter were produced from undoped commercial sintered and swaged rod by electron-beam-floating zone-melting, as previously described by Witzke (ref. 9). Rods $6\frac{1}{2}$ inches long were given one zone-melting pass at a zone-traverse rate of 6 inches per hour. Chemical analyses before and after zone melting are given in table I. This analysis is typical for all crystals used. Chemical analyses along the length of a zone-melted rod revealed no zoning of impurities, and it is believed that purification was obtained chiefly through vaporization. Another indication of impurity reduction during zoning was an electrical resistivity ratio (resistivity at room temperature/resistivity at 4.2° K) of about 11 000 for zone-refined metal compared with a ratio of less than 1000 for unmelted rod.

Crystals of $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ rod-axis orientations were produced by seeding. Orientation was controlled to within 5° of that desired. Orientations were determined by Laue back-reflection X-ray techniques.

TABLE I. - ANALYSES OF IMPURITIES IN TUNGSTEN

Element	Sintered and swaged tungsten rod	Zone-melted single-crystal tungsten
	Impurity content[a] by weight, ppm	
Carbon	8	6
Oxygen	4	2
Aluminum	2.5	.05
Iron	5	.04
Molybdenum	100	44
Nickel	1.2	.02

[a]Carbon was determined by a conductometric method; oxygen, by vacuum fusion; and metallic impurities, by emission spectroscopy.

Three buttonhead tensile specimens were produced from each zone-melted rod. The specimen configuration, as illustrated in figure 1, was produced by grinding the reduced section to a 0.075-inch diameter and then further reducing the diameter about 0.005 inch by electrolytic polishing in a 2-percent sodium hydroxide solution. Laue patterns indicated that grinding stresses were removed by this polishing. Tensile tests of the single crystals were performed on a tensile testing machine

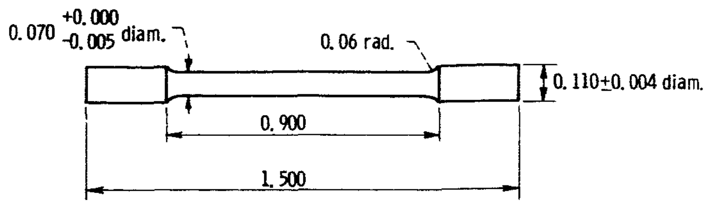

Figure 1. - Single-crystal tensile specimen. (All dimensions are in inches.)

at a cross head speed of 0.01 inch per minute. Tests below room temperature were conducted in an apparatus similar to that described by Wessel (ref. 10), which utilized a controlled spray of liquid nitrogen for cooling. For temperatures above room temperature, specimens were heated in air in a resistance-wound furnace. All temperatures were measured by thermocouples and are accurate to ±3 K degrees.

The fractured surfaces were viewed through a microscope at a magnification of 100. By the use of a filar eyepiece, the dimensions of the fractured surfaces were obtained (diameter for circular fractured sections, major and minor axes for elliptical fractured sections). From these dimensions, the percent reduction in area was calculated.

RESULTS AND DISCUSSION

The variation of ductility with test temperature for crystals of ⟨100⟩, ⟨110⟩, and ⟨111⟩ orientations is shown in figure 2. It is apparent that the variation of ductility with temperature is highly orientation dependent. Crystals of the ⟨110⟩ tensile axis exhibited a very sharp transition from ductile to brittle behavior, while those with a ⟨100⟩ axis showed a very gradual decrease in ductility with decreasing temperature. Because of the limited number of ⟨111⟩ tensile-axis specimens tested, the transition behavior for this orientation could not be determined with a high degree of certainty. The curve shown in

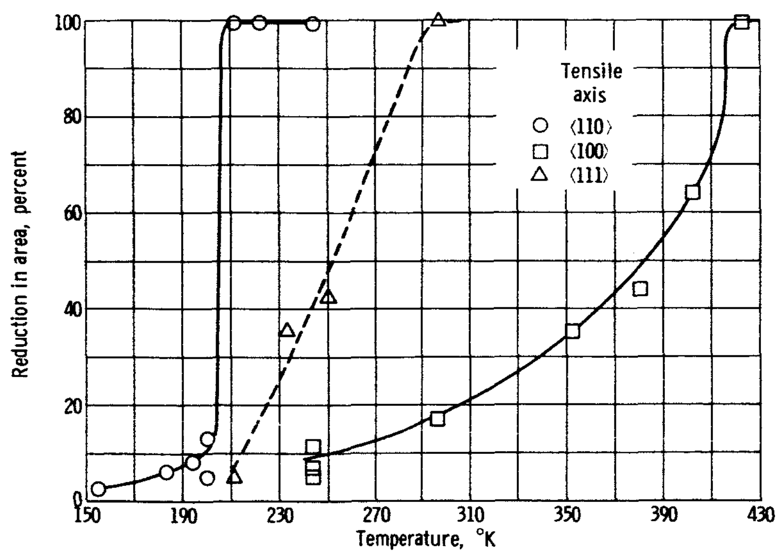

Figure 2. - Reduction in area as function of temperature for single-crystal tungsten.

figure 2 is believed to be the best fit for the data; however, the transition may be sharper than this. No evidence was found of slip traces for small deformation (less than 2 percent elongation). A similar difficulty in determining slip traces has been reported in references 11 and 12.

The ductile to brittle transition temperature, defined here as that temperature at which reduction in area is 50 percent, was 205° K for $\langle 110 \rangle$ and about 380° K for $\langle 100 \rangle$ oriented crystals. The transition temperature of $\langle 111 \rangle$ orientation crystals could not be accurately determined but was certainly intermediate between those of $\langle 110 \rangle$ and $\langle 100 \rangle$. These results are not in agreement with those of Beardmore and Hull (ref. 12), where necking (and therefore appreciable ductility) was observed at 20° K for all orientations around the edge of the unit stereographic triangle. It should be noted that their specimens were much smaller (0.4- by 0.02-in. cross section) than those used in the present investigation and that a perfect crystal surface was necessary for necking to occur.

Normally, the ductility of a polycrystalline body-centered-cubic metal decreases rapidly toward zero at temperatures slightly below the ductile-brittle transition temperature. This was not the case for high-purity single-crystal tungsten, since the crystals exhibited measureable ductility at temperatures appreciably below their ductile to brittle transition temperature. For example, $\langle 110 \rangle$ crystals retained significant ductility (about 2 percent reduction in area and 1.5 percent elongation) at 155° K, the lowest test temperature, 50° K below the ductile-brittle transition temperature. The $\langle 100 \rangle$ orientation also exhibited appreciable ductility far below its ductile-brittle transition temperature, showing a 5- to 10-percent reduction in area and about the same percent elongation at 244° K, well below the 380° K ductile to brittle transition temperature. This ductility below the transition temperature was also evident in stress-strain curves; that is, all samples deformed beyond the elastic limit. It is apparent that definition of the ductile to brittle transition temperature as some arbitrary percent reduction of area does not adequately describe the transition from ductile to brittle behavior for single-crystal tungsten, because truly brittle behavior is not observed for single-crystal tungsten even well below such an arbitrarily defined transition temperature.

For each orientation, fracture of specimens that exhibited a 99-percent reduction in area was of the knife-edge type, while fracture of specimens that exhibited little reduction in area occurred by cleavage on a $\{100\}$ plane. Specimens with intermediate reductions in area were necked, where fracture occurred by cleavage in the necked region.

In view of the different ductile to brittle transition behaviors noted for the three orientations, it is of interest to note the differences in stress-strain behavior. Stress-strain curves for the three orientations at room temperature are shown in figure 3. (As used herein, stress is the load indicated on tensile testing machine/original cross-sectional area, and strain is the movement of tensile testing machine crosshead/original

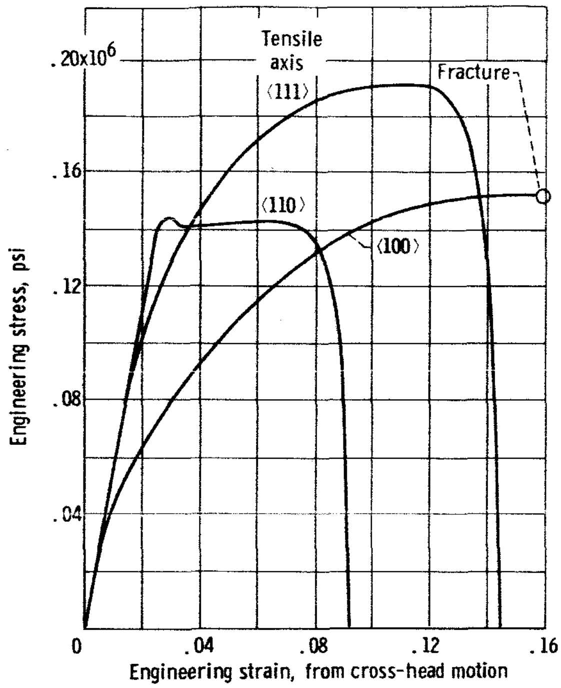

Figure 3. - Representative stress-strain curves at 298° K.

gage length of the specimen.) The general features apparent in figure 3 are characteristic of the crystals at all temperatures investigated. The ⟨110⟩ crystals exhibited a yield-point drop and a flat plastic region at all temperatures, while ⟨100⟩ and ⟨111⟩ crystals exhibited no yield-point phenomena and showed considerable strain-hardening. Similar observations have also been reported in references 11 and 12.

These observed differences in stress-strain behavior, particularly the presence or absence of a yield point, are indicative of differences in basic flow mechanisms among the three orientations. Although a description of these mechanisms is not possible from the results of this study, such different mechanisms might also account for the variations observed in ductile-brittle transition behavior of the three orientations.

SUMMARY OF RESULTS

From a study of the effect of orientation on the ductile to brittle transition temperature of single-crystal tungsten, the following results were obtained:

1. The ductile-brittle transition temperatures, based on 50 percent reduction in area, varied with orientation as follows: 205° K for the ⟨110⟩ orientation, 380° K for the ⟨100⟩ orientation, and intermediate between these values for the ⟨111⟩ orientation.

2. The transition from ductile to brittle behavior with temperature was extremely dependent on orientation. The ⟨110⟩ orientation showed a sharp transition, while the ⟨100⟩ orientation showed a very gradual transition.

3. The tensile flow characteristics of single-crystal tungsten were highly orientation dependent. The ⟨110⟩ oriented crystals showed a high yield stress and a yield-point drop, and necking occurred immediately after yielding. In contrast, crystals of the ⟨100⟩ and ⟨111⟩ orientations showed no yield drop and exhibited appreciable uniform elongation before necking.

4. All orientations showed significant ductility well below their ductile to brittle transition temperatures.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 13, 1966.

REFERENCES

1. Olds, L. E.; and Rengstorff, G. W. P.: Effects of Oxygen, Nitrogen, and Carbon on the Ductility of Cast Molybdenum. Trans. AIME, vol. 206, Feb. 1956, pp. 150-155.

2. Smith, W. H.; and Seybolt, A. U.: Effects of Impurities on the Ductility of Chromium. Ductile Chromium, ASM, 1957, pp. 169-179.

3. Stephens, Joseph R.: Effects of Interstitial Impurities on the Low-Temperature Tensile Properties of Tungsten. NASA TN D-2287, 1964.

4. Schadler, H. W.: Deformation Behavior of Zone-Melted Tungsten Single Crystals. Trans. AIME, vol. 218, Aug. 1960, pp. 649-655.

5. Leber, Sam; and Pugh, J. W.: High-Temperature Slip in Tungsten. Trans. AIME, vol. 218, Oct. 1960, pp. 791-793.

6. Garlick, R. G.; and Probst, H. B.: Investigation of Room-Temperature Slip in Zone-Melted Tungsten Single Crystals. Trans. AIME, vol. 230, Aug. 1964, pp. 1120-1125.

7. Schnitzel, R. H.; and Keith, G. H.: Deformation Twins in Single Crystals of Tungsten. J. Less Common Metals, vol. 4, 1962, pp. 98-101.

8. Wolff, Ursula E.: Twinning and Fracture in Tungsten Single Crystals at Room Temperature. Trans. AIME, vol. 224, Apr. 1962, pp. 327-333.

9. Witzke, W. R.: The Zone Melting of Tungsten by Electron Bombardment. Proc. Symposium on Electron-Beam Melting, Alloyed Res. Corp., 1959, pp. 73-81.

10. Wessel, E. T.: Refrigeration Techniques and Apparatus for Very Low Temperatures. Refrigerating Eng., vol. 57, no. 10, Oct. 1957, pp. 37-45.

11. Rose, R. M.; Ferriss, D. P.; and Wulff, J.: Yielding and Plastic Flow in Single Crystals of Tungsten. Trans. AIME, vol. 224, Oct. 1962, pp. 981-990.

12. Beardmore, P.; and Hull, D.: Deformation and Fracture of Tungsten Single Crystals. J. Less Common Metals, vol. 9, no. 3, Sept. 1965, pp. 168-180.

NASA-Langley, 1966 E-2649