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# EFFECT OF REACTOR IRRADIATION ON DUCTILE-BRITTLE TRANSITION AND STRESS-STRAIN BEHAVIOR OF TUNGSTEN

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16. Abstract

The effect of reactor irradiation at 250° F (395 K) on the ductile-to-brittle transition behavior of polycrystalline tungsten in the recrystallized condition was investigated. Unirradiated material showed transition at 600° F (589 K), but when irradiated to $12\times10^{19}$ fast neutrons/cm$^2$ the ductile-to-brittle transition temperature increased about 50° F (28 K) with each increase in fluence of $1\times10^{19}$ neutrons/cm$^2$. At $15.3\times10^{19}$ fast neutrons/cm$^2$, the transition occurred at 1460° F (1067 K). Irradiation strengthened the metal and caused plastic strain to be concentrated within a small-volume element of the test specimen gage section.

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# EFFECT OF REACTOR IRRADIATION ON DUCTILE-BRITTLE TRANSITION AND STRESS-STRAIN BEHAVIOR OF TUNGSTEN

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## SUMMARY

The effect of reactor irradiation at 250° F (395 K) in the range of $3\times10^{19}$ to $18\times10^{19}$ neutrons per square centimeter, neutron energy greater than 1 MeV (160 fJ), on the transition from ductile to brittle behavior of recrystallized tungsten was investigated. Reduction-of-area measurements from tensile tests conducted in the range of 700° to 1800° F (644 to 1256 K) were used to determine a ductile-to-brittle transition temperature.

The ductile-to-brittle transition temperature for 40-percent reduction of area increased with increasing neutron fluence. Unirradiated material showed transition at 600° F (589 K), but when irradiated up to $12\times10^{19}$ neutrons per square centimeter, the ductile-to-brittle transition temperature increased at about 50° F (28 K) with each increase in fluence of $1\times10^{19}$ neutrons per square centimeter. At $15.3\times10^{19}$ neutrons per square centimeter, the transition occurred at about 1460° F (1067 K).

The transition from ductile to brittle behavior of unirradiated material took place over a temperature interval of 200° F (111 K). For fluences greater than $4\times10^{19}$ fast neutrons per square centimeter, the transition took place within 50° F (28 K).

The plastic strain accompanying ductile fracture for irradiated material was concentrated within a small-volume element of the test specimen gage section. The maximum reduction of area for irradiated material was not significantly different from that for unirradiated material. The total elongation of irradiated material, however, was decreased by at least a factor of 4.

The strength at the transition from brittle to ductile fracture was approximately constant - about 80 000 psi (55 100 N/cm$^2$) - for the fluence and test temperatures investigated.

## INTRODUCTION

Advanced space power system designs employ refractory metals and nuclear reactors as part of the power generation system. The refractory metals, particularly tungsten, exhibit a transition from ductile-to-brittle fracture behavior at temperatures well above room temperature. This transition in fracture behavior is a sensitive index for studying the effect of process and environmental exposure variables on the metal.

Investigations reported in the literature (refs. 1 to 2) are not in agreement as to the magnitude of the increase in the ductile-to-brittle transition temperature due to irradiation. A previous investigation by the present authors (ref. 3) revealed that reactor irradiation at about 200° F (366 K) to $1\times10^{20}$ fast neutrons per square centimeter severely embrittled polycrystalline tungsten. (All fast fluence values quoted herein are for neutron energy greater than 1 MeV (160 femtoJoule) unless otherwise noted.) The metal remained brittle at 700° F (644 K), the highest temperature employed in the program.

Since ductile fractures of irradiated material were not obtained at a test temperature of 700° F (644 K), an experimental program was undertaken to investigate the effect of test temperatures greater than 700° F (644 K). The reactor exposure of interest was $1\times10^{20}$ fast neutrons per square centimeter; however, the fluence actually achieved ranged between $2.9\times10^{19}$ and $17.9\times10^{19}$ fast neutrons per square centimeter.

Tensile tests conducted in the range of 700° to 1800° F (644 to 1256 K) were used for obtaining ductile and brittle fractures for both irradiated and unirradiated material. The unirradiated material was thermally cycled to duplicate the irradiated exposure conditions. Selected specimens for both exposure conditions were subjected to metallographic examination for comparison of fracture behavior.

## TEST MATERIAL

Test specimens were fabricated from 0.5-inch- (12.7-mm-) diameter sintered and swaged tungsten rod obtained from a commercial supplier. Spectrographic and chemical analyses of the as-received material are given in table I. (This same material was used in ref. 3.)

Test specimens (fig. 1) were first ground to the specified dimensions and then vacuum annealed for 1 hour at 3500° F (2200 K). The annealing produced an equiaxed grain with an average grain diameter of about 0.0031 inch (0.080 mm).

Following annealing, each test specimen was electropolished in accordance with the procedures and apparatus reported in reference 4. The electropolishing treatment reduced the gage diameter from 0.200 to 0.190 inch (5.1 to 4.8 mm).

TABLE I. - SPECTROGRAPHIC AND CHEMICAL ANALYSES OF TUNGSTEN TENSILE SPECIMENS

| Element | Composition, ppm |
|---|---|
| Aluminum | 10 |
| Boron | [a]<2 |
| Carbon | 22 |
| Calcium | [a]<10 |
| Chromium | [a]<5 |
| Copper | 2 |
| Iron | 5 |
| Potassium | [a]<10 |
| Manganese | [a]<1 |
| Molybdenum | 10 |
| Sodium | [a]<10 |
| Nickel | 5 |
| Oxygen | 11 |
| Phosphorous | [a]<20 |
| Lead | [a]<10 |
| Sulfur | [a]<10 |
| Silicon | [a]<3 |
| Tin | [a]<5 |
| Thorium | [a]<30 |

[a]Not detected, less than value given.

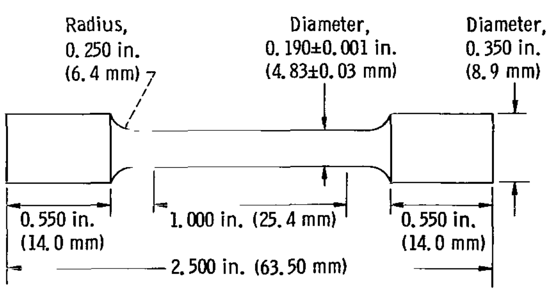



Figure 1. - Tungsten tensile specimen.

## EXPERIMENTAL PROCEDURE

The experimental program consisted of exposing test specimens in a eutectic sodium-potassium mixture (NaK-56) and then conducting tensile tests at various temperatures.

### Environmental Exposure

Specimen containers for environmental exposure were stainless-steel tubes (fig. 2) that were filled with NaK-56 and sealed by heliarc welding.

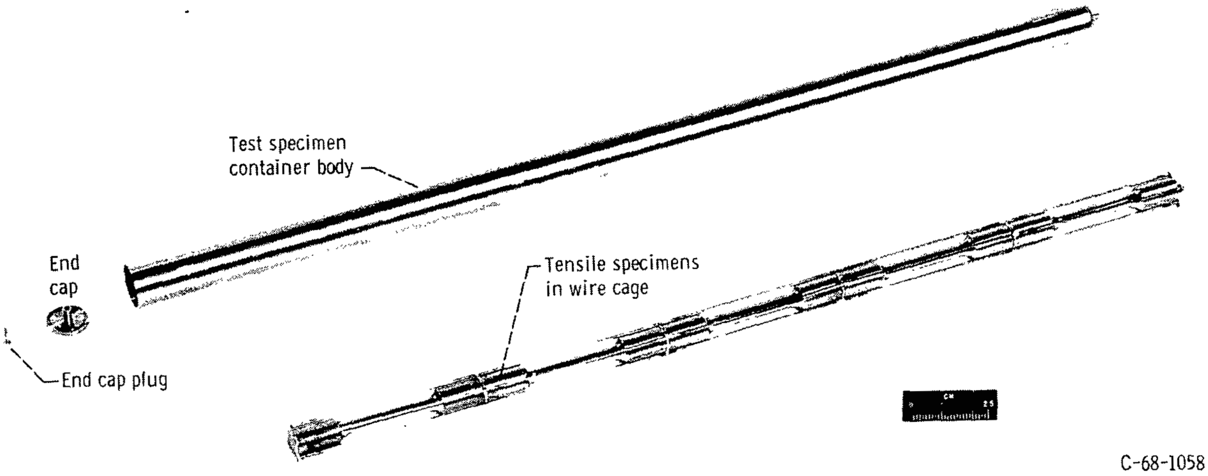



Figure 2. - Irradiation capsule with specimen container disassembled.

Irradiation exposure consisted of placing six NaK-56-filled tubes, each containing five test specimens, in an aluminum irradiation capsule (fig. 3). This capsule, except for slight internal modifications, is described in detail in reference 5. The irradiation capsule provides a 30-inch (76-cm) length wherein test specimens and flux monitor wires are positioned in tiers relative to the vertical orientation of the reactor (fig. 4). Each tier, relative to the horizontal orientation of the reactor, consists of three tensile specimens (fig. 4, positions A to C) and four sets of flux monitors (fig. 4, positions D to J). Each set of flux monitors consisted of two wires 0.030 inch (0.76 mm) in diameter by 0.5 inch (12.7 mm) long. One was composed of aluminum-0.5 percent cobalt and the other of 99.98 percent nickel.

The irradiation exposure was achieved during 873.2 hours of reactor operation dur-

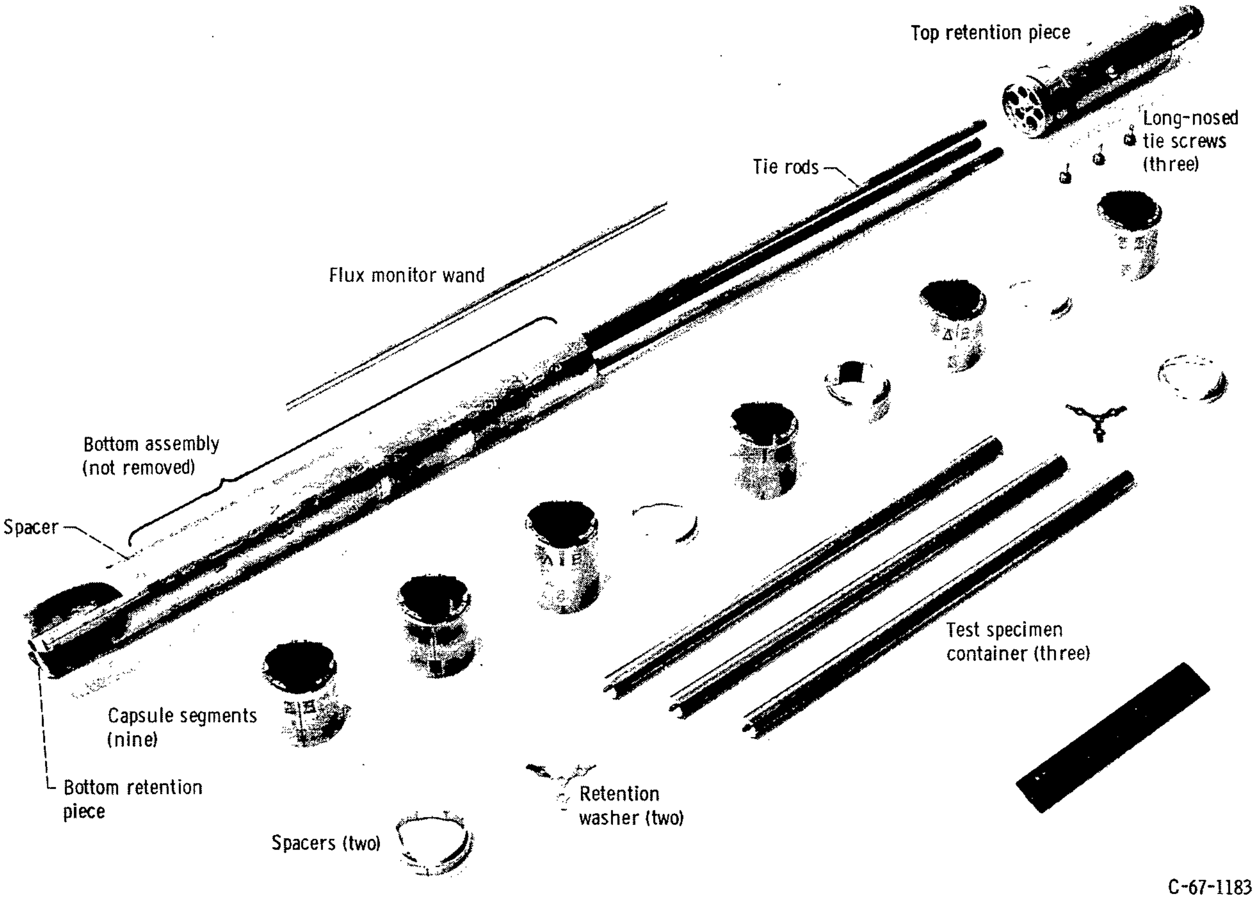



Figure 3. - Disassembled irradiation capsule.

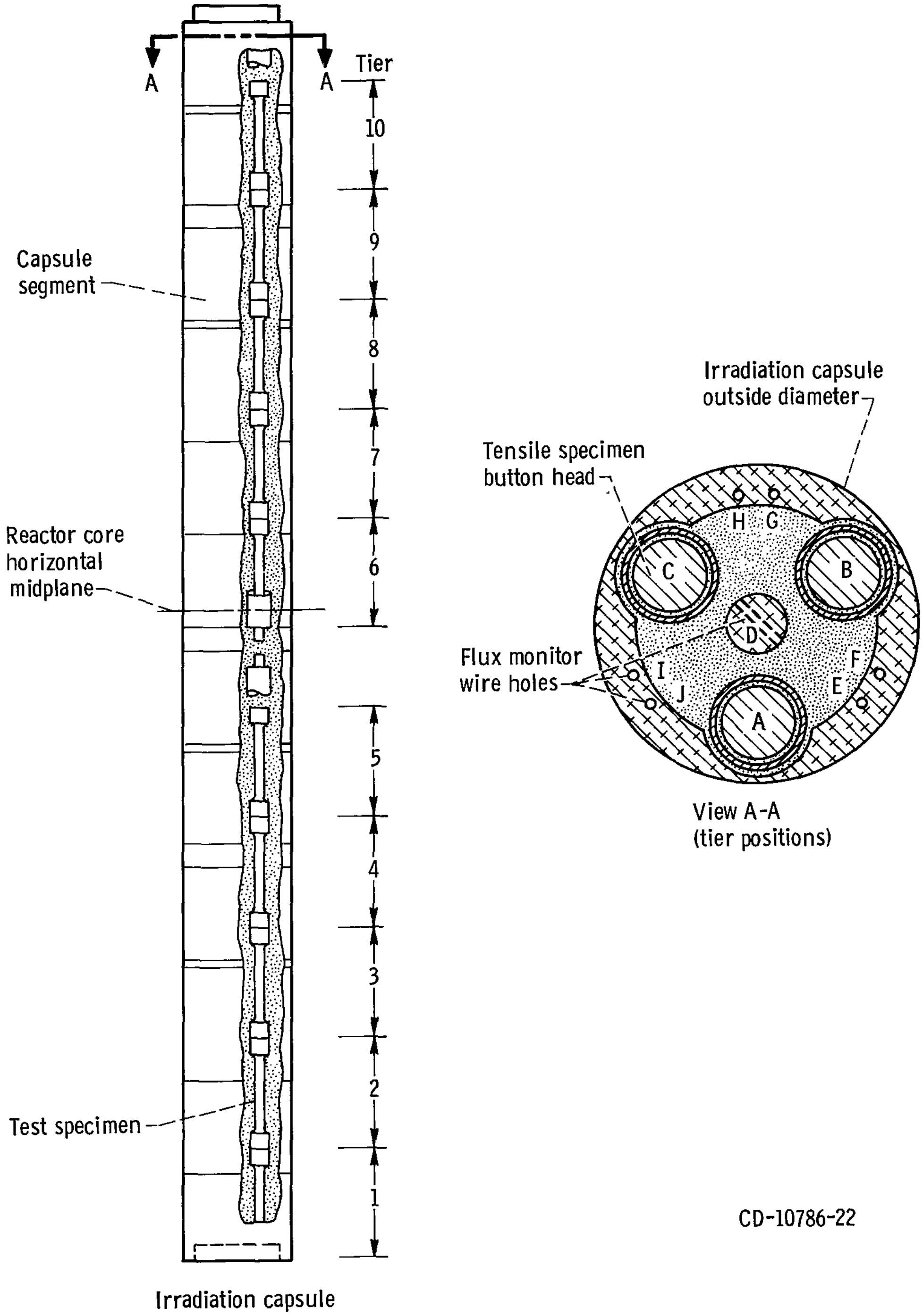



Figure 4. - Irradiation capsule loading scheme.

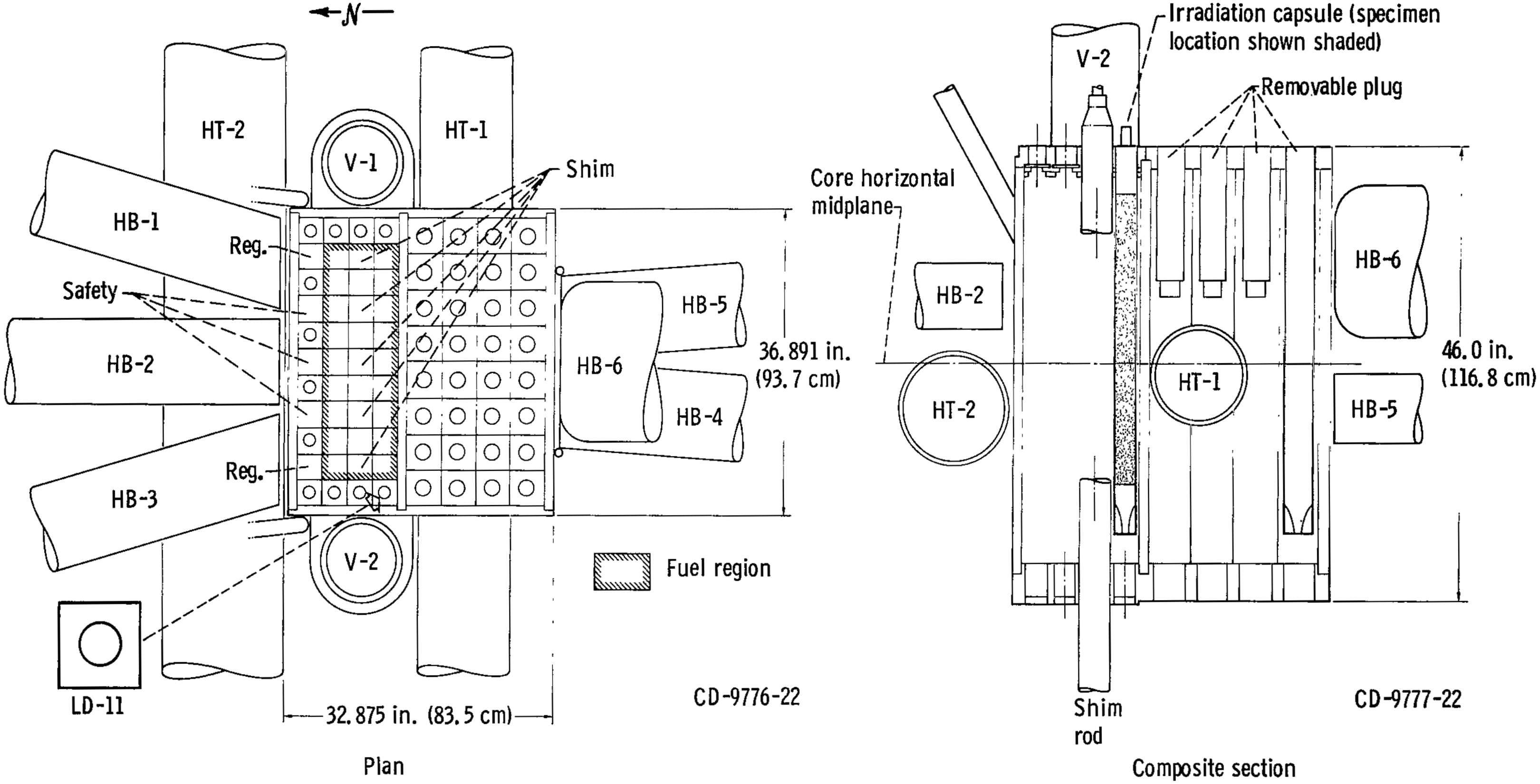



Figure 5. - NASA Plum Brook reactor core and irradiation test holes.

ing which time the irradiation capsule occupied position LD-11 of the Plum Brook Reactor (fig. 5). Test specimen containers were cooled by reactor primary water flowing through the capsule at about 20 feet per second (6.1 m/sec). The specimen temperatures were not monitored during irradiation, but, based on a heat-transfer analysis using a computer program (ref. 6), the maximum specimen temperature was about $250^{\circ}$ F (395 K).

The peripheral neutron fluences, determined by an activation analysis of the flux monitor wires, are given in table II and plotted in figure 6. Fluence values from monitor wires in position D (along the irradiation capsule centerline) were low, indicating a high degree of perturbation by the test specimens. For specimen exposure, the fast and thermal neutron fluences were taken from figure 6 at a position corresponding to the center of the test specimen gage length. These fluence values are given in table III and represent the average value obtained from the two monitor fluence curves between which the specimen lies (e.g., for tier position A, the specimen fast fluence is the average value from monitor fluence curves E and I, and the thermal fluence is the average from monitor fluence curves F and J).

Control specimens in NaK-56-filled tubes were thermally cycled in a furnace with time and temperature cycles duplicating those of the irradiation exposure.

TABLE II. - FAST AND THERMAL NEUTRON FLUENCE PROFILES ABOUT PERIMETER OF PLUM BROOK REACTOR LATTICE POSITION LD-11

| Distance from center of flux monitor wire to reactor core horizontal midplane | | Tier position[a] | | | | | |
|---|---|---|---|---|---|---|---|
| | | E | G | I | F | H | J |
| | | Neutron fluence,[b] neutrons/cm$^2$ | | | | | |
| in. | cm | Fast, $E > 1$ MeV (160 fJ) | | | Thermal | | |
| 13.8 | 35.1 | $0.16\times10^{20}$ | $0.18\times10^{20}$ | $0.12\times10^{20}$ | $1.10\times10^{20}$ | $0.93\times10^{20}$ | $0.90\times10^{20}$ |
| 11.4 | 29.0 | .43 | .33 | .21 | 1.75 | --------- | 1.40 |
| 8.5 | 21.6 | .84 | .64 | .44 | 2.80 | 2.65 | 2.35 |
| 6.2 | 15.7 | 1.20 | .90 | .63 | 4.00 | 3.80 | 3.25 |
| 3.5 | 8.9 | 1.60 | 1.27 | .79 | 5.50 | 5.20 | 4.40 |
| 1.4 | 3.6 | 1.90 | 1.60 | 1.00 | 6.10 | 6.50 | 5.10 |
| -1.1 | -2.8 | 2.15 | 1.75 | 1.10 | --------- | 7.80 | 6.60 |
| -3.5 | -8.9 | 2.05 | 1.85 | 1.10 | --------- | 7.30 | 6.20 |
| -6.4 | -16.3 | 2.05 | --------- | --------- | 7.00 | 6.60 | 5.70 |
| -8.8 | -22.4 | --------- | 1.45 | --------- | --------- | 5.90 | 4.60 |
| -11.4 | -29.0 | 1.10 | .96 | .62 | 4.65 | 4.25 | 3.55 |
| -13.5 | -34.3 | .57 | --------- | .34 | 3.80 | 2.85 | 2.55 |

[a]See fig. 4.
[b]See fig. 6.

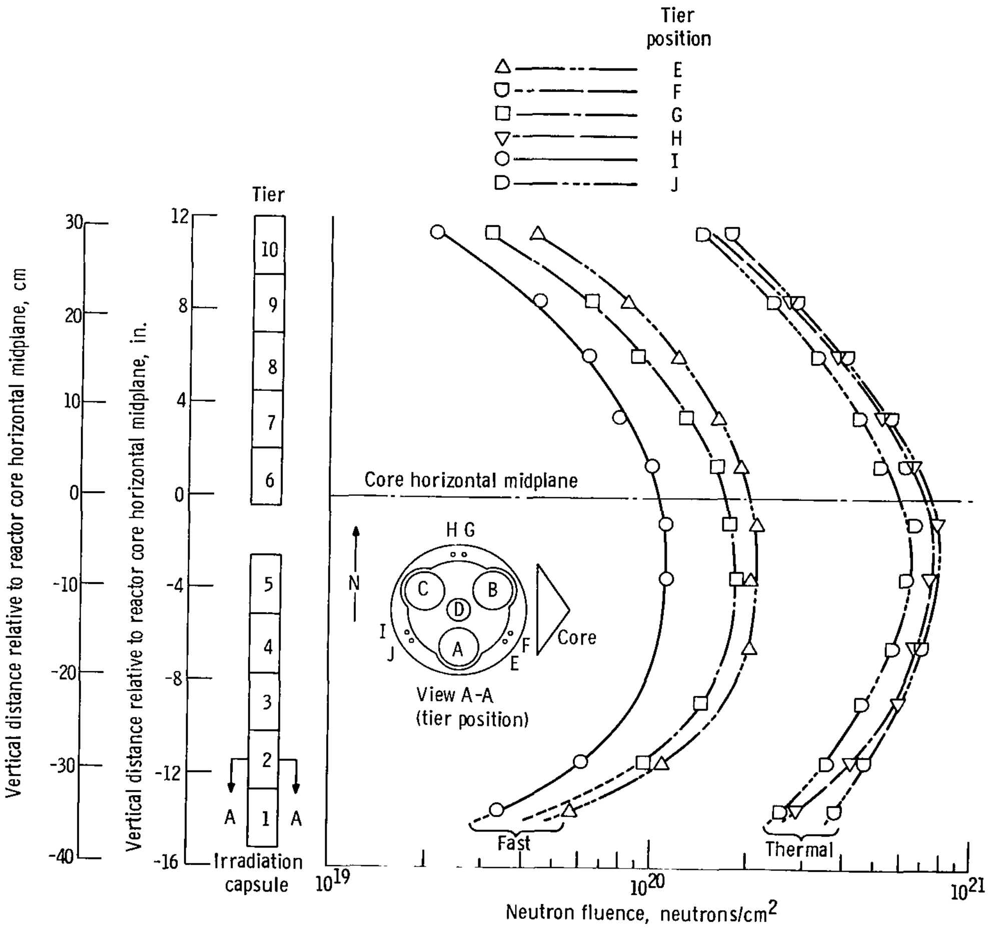



Figure 6. - Fast and thermal neutron fluence profiles about perimeter of irradiation capsule. Fast neutron energy, greater than 1 MeV (160 fJ).

TABLE III. - FAST AND THERMAL NEUTRON FLUENCES OF IRRADIATED TUNGSTEN TENSILE SPECIMENS

| Specimen | Position in irradiation capsule[a] | | Distance from reactor core horizontal midplane[b] | | Fluence, neutrons/$cm^2$ | | |
|---|---|---|---|---|---|---|---|
| | Tier | Position | in. | cm | Fast, $E > 1$ MeV (160 fJ) | Thermal | Thermal/fast |
| 70 | 1 | A | -14.1 | -35.8 | $3.7\times10^{19}$ | $2.9\times10^{20}$ | 7.83 |
| 71 | 2 | ↓ | -11.6 | -29.5 | 8.2 | 4.1 | 5.00 |
| 72 | 3 | | -9.0 | -22.9 | 12.7 | 5.3 | 4.17 |
| 73 | 4 | | -6.4 | -16.3 | 15.4 | 6.4 | 4.15 |
| 74 | 5 | | -3.9 | -9.9 | 16.3 | 7.2 | 4.41 |
| 85 | 6 | | .8 | 2.0 | 14.8 | 6.5 | 4.39 |
| 86 | 7 | | 3.3 | 8.4 | 12.3 | 5.0 | 4.06 |
| 87 | 8 | | 5.9 | 15.0 | 9.4 | 3.7 | 3.94 |
| 88 | 9 | | 8.4 | 21.3 | 6.4 | 2.6 | 4.06 |
| 89 | 10 | | 11.0 | 27.9 | 3.5 | 1.7 | 4.85 |
| 75 | 1 | B | -14.1 | -35.8 | $4.3\times10^{19}$ | $3.1\times10^{20}$ | 7.21 |
| 76 | 2 | ↓ | -11.6 | -29.5 | 9.8 | 4.3 | 4.39 |
| 77 | 3 | | -9.0 | -22.9 | 15.4 | 5.9 | 3.83 |
| 90 | 6 | | .8 | 2.0 | 17.9 | 7.0 | 3.91 |
| 91 | 7 | | 3.3 | 8.4 | 14.6 | 5.5 | 3.77 |
| 92 | 8 | | 5.9 | 15.0 | 11.1 | 3.9 | 3.51 |
| 93 | 9 | | 8.4 | 21.3 | 7.5 | 2.7 | 3.60 |
| 94 | 10 | | 11.0 | 27.9 | 4.0 | 1.7 | 4.25 |
| 81 | 2 | C | -11.6 | -29.5 | $7.4\times10^{19}$ | $3.8\times10^{20}$ | 5.14 |
| 82 | 3 | ↓ | -9.0 | -22.9 | 11.5 | 5.2 | 4.52 |
| 83 | 4 | | -6.4 | -16.3 | 13.8 | 6.2 | 4.49 |
| 84 | 5 | | -3.9 | -9.9 | 14.7 | 7.0 | 4.76 |
| 95 | 6 | | .8 | 2.0 | 13.2 | 6.2 | 4.70 |
| 96 | 7 | | 3.3 | 8.4 | 10.7 | 4.9 | 4.57 |
| 97 | 8 | | 5.9 | 15.0 | 8.0 | 3.6 | 4.50 |
| 98 | 9 | | 8.4 | 21.3 | 5.4 | 2.5 | 4.62 |
| 99 | 10 | | 11.0 | 27.9 | 2.9 | 1.6 | 5.51 |

[a]See fig. 4.
[b]Distance to center of test specimen gage length.

## Postexposure Testing

Postexposure testing of control specimens and irradiated specimens was performed during the same time period in the NASA Plum Brook Reactor Facility hot laboratory. All tests were performed by remote operations using the same test equipment for both unirradiated and irradiated material. The test apparatus and procedures are described in detail in reference 3 and will only be summarized herein.

Tensile tests were performed in flowing argon at temperatures in the range of 700° to 1800° F (644 to 1256 K) on a 10 000-pound (44 500-N) test machine adapted to remote operation. The time necessary to reach test temperature varied from 80 minutes at 700° F (644 K) to 400 minutes at 1800° F (1256 K), following which the specimen was soaked at temperature for 10 minutes prior to loading. Tensile tests were conducted at a constant crosshead speed of 0.2 inch per minute (5.1 mm/min), and load-time curves were obtained autographically. An extensometer was not employed during testing. Autographically recorded load-time curves were corrected for elastic strain of the pulling assembly and replotted as stress-strain curves.

Following fracture, photographs, both longitudinal and transverse views, were taken of the fractured specimens.

The reduction of area was determined from measurements before and after tensile testing. The specimen areas before tensile testing were calculated from diameter measurements obtained at room temperature following environmental exposure. These measurements were obtained by using an optical gage. The fracture area was determined from the transverse-view photograph, which included a reference scale for this purpose.

On completion of the tensile test, photographs of the microstructure (longitudinal) in the fracture zone of the test specimen were obtained. An appropriate section was cut from the test specimen with a small water-cooled abrasive cutoff wheel. The section was mounted in an epoxy, ground through four grits (220, 320, 400, and 600), and polished on a vibratory polisher. Etching was performed by swabbing for 4 seconds with Murakami's reagent.

# DISCUSSION OF TEST RESULTS

Test results may be considered from two separate but related viewpoints. These are the effect of irradiation on (1) the transition from ductile to brittle fracture and (2) the stress-strain behavior.

## Transition from Ductile to Brittle Fracture

The reduction of area following tensile testing at selected temperatures is frequently used to establish a characteristic ductile-to-brittle transition temperature (DBTT) for tungsten. This is accomplished by plotting reduction-of-area values against the test temperature and then selecting some value of the reduction of area to represent a DBTT. Figure 7 shows such a plot for the test data obtained herein with the temperature corresponding to a 40-percent reduction of area defined as the DBTT.

From figure 7 it can be seen that (1) the DBTT for a 40-percent reduction of area increases with increasing neutron fluence; (2) the reduction of area reaches a maximum value near 85 percent regardless of the exposure condition; and (3) the temperature range for transition from brittle to ductile behavior is reduced by irradiation.

Figure 8 shows the DBTT to increase at a rate of $50^{\circ}$ F (29 K) per $10^{19}$ neutrons per square centimeter up to $12\times10^{19}$ neutrons per square centimeter. This rate appears to increase for fluences greater than $12\times10^{19}$ neutrons per square centimeter.

Although the irradiated material showing ductile fracture had a high reduction-of-area value, this ductility was confined to a small-volume element of the test specimen

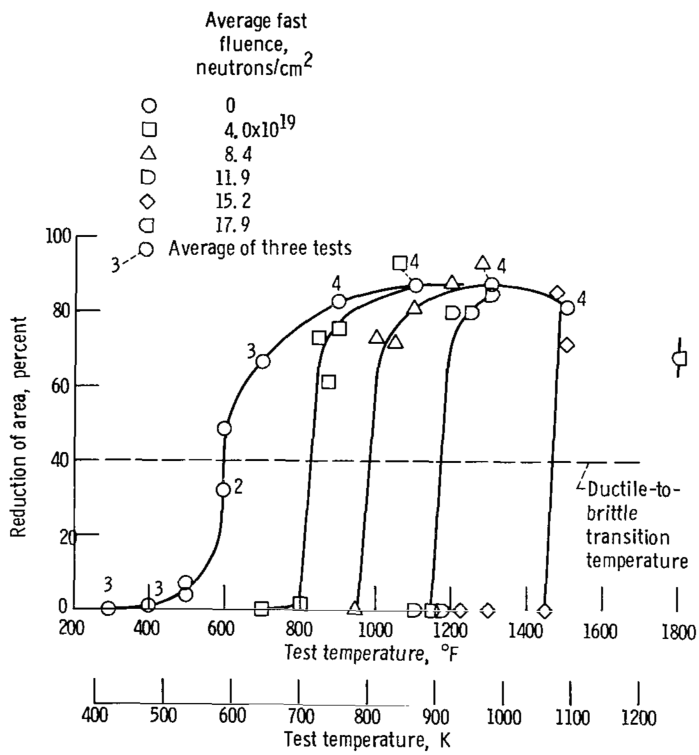



Figure 7. - Effect of test temperature on reduction of area of tungsten for constant neutron fluence. Neutron energy, greater than 1 MeV (160 fJ).

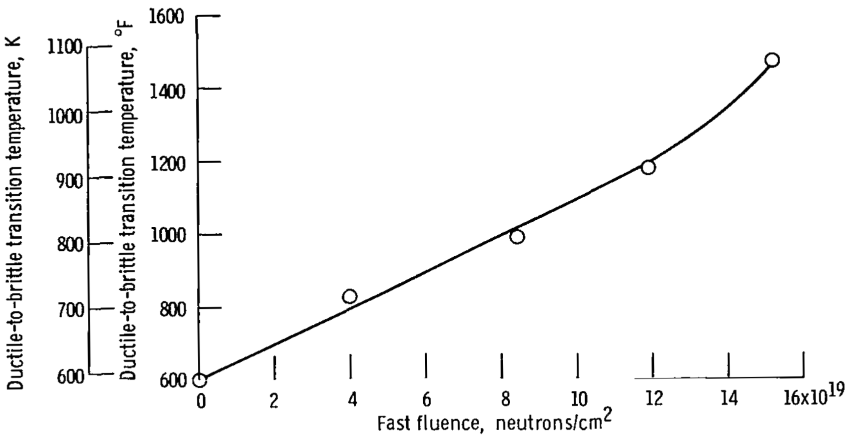



Figure 8. - Ductile-to-brittle transition temperature at 40-percent reduction of area as function of neutron fluence. Neutron energy, greater than 1 MeV (160 fJ).

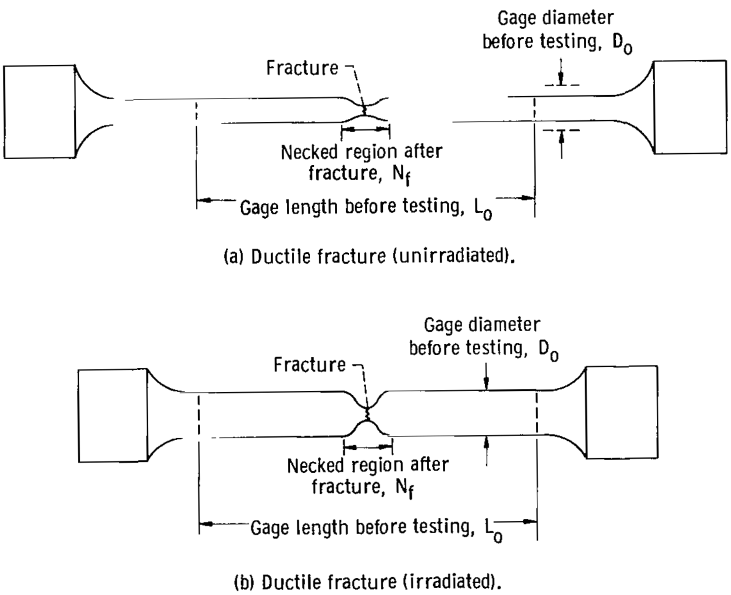
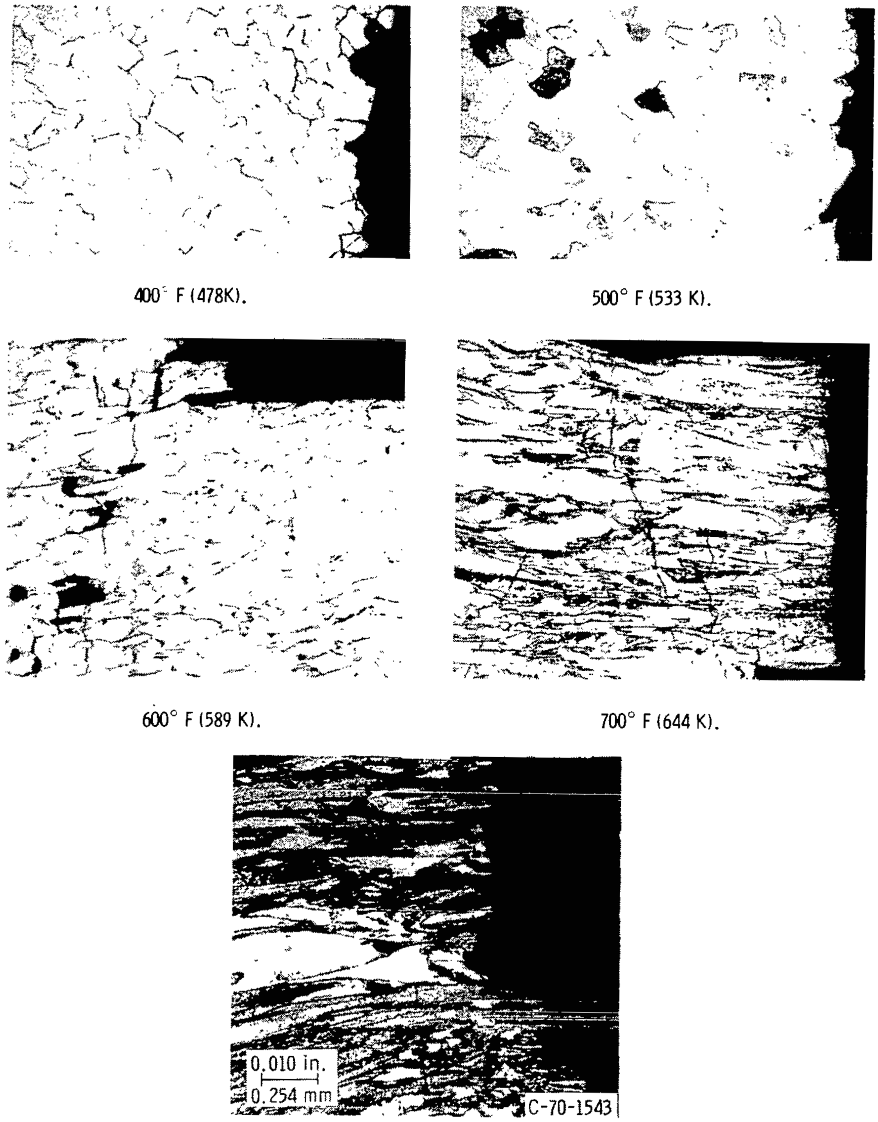



Figure 9. - Test specimen gage sections following tensile testing.

gage length. Figure 9 is a schematic representation of the ductile fracture for unirradiated and irradiated materials. It should be noted that, whereas the unirradiated material showed uniform strain and reduction of specimen diameter outside the necked region, the irradiated material showed little deformation except in the necked region. The difference in the elongation of the necked region for unirradiated and irradiated conditions was small.

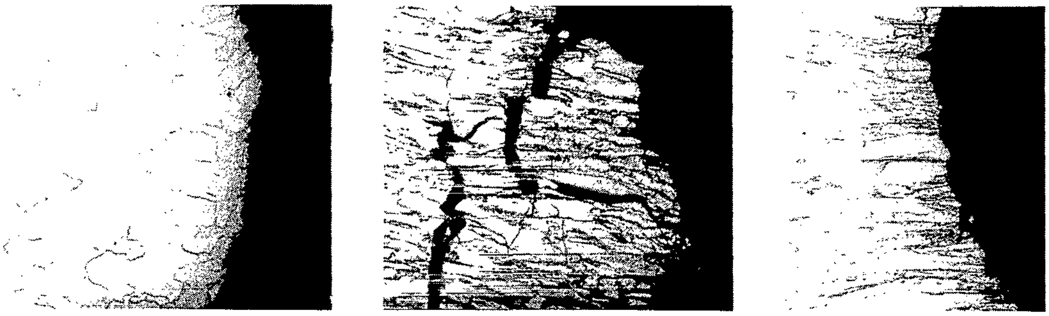
Microstructures of fractured specimens show no grain elongation accompanying brittle fractures, but for ductile fractures with at least a 40-percent reduction of area,




900° F (756 K).

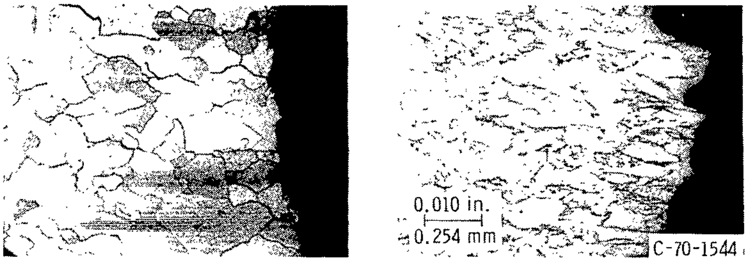
Figure 10. - Microstructure of unirradiated tensile fractures. Etchant, Murakami's reagent.

the grain elongation is appreciable. Figure 10 shows fracture microstructures for the unirradiated condition. Brittle fracture occurred at $400^{\circ}$ F (478 K), and ductile fracture with a 40-percent reduction of area occurred at $600^{\circ}$ F (589 K). The fracture microstructure at $500^{\circ}$ F (533 K) is typical of specimens that yielded and showed some plastic strain but did not achieve a 40-percent reduction of area. The microstructures of irradiated fractures (fig. 11) show that, for a fluence of $3.7\times10^{19}$ neutrons per square centimeter, the transition from brittle fracture to ductile fracture with appreciable grain elongation occurs within the interval $800^{\circ}$ to $850^{\circ}$ F (700 to 727 K). For a fluence of $15.4\times10^{19}$ neutrons per square centimeter, a comparable transition in fracture mi-



800° F (700 K). 850° F (727 K). 900° F (756 K).

(a) Fluence, $3.7 \times 10^{19}$ fast neutrons per square centimeter.




1450° F (1061 K). 1475° F (1075 K).

(b) Fluence, $15.4 \times 10^{19}$ fast neutrons per square centimeter.

Figure 11. - Microstructure of irradiated tensile fractures. Fast neutron energy, greater than 1 MeV (160 fJ); etchant, Murakami's reagent.

crostructure occurs between 1450° and 1475° F (1061 to 1075 K). Hence, the temperature range for transition from brittle fracture to ductile fracture with appreciable grain elongation is reduced by irradiation. For the unirradiated condition, the transition takes place over an interval of 200° F (111 K), whereas a comparable interval for irradiation conditions is 50° F (28 K).

Figure 12 is a summary plot showing the relation of test temperature and fluence for specimens tested in the present investigation, a previous investigation (ref. 3), and results reported in the literature (ref. 2) for comparable material. The DBTT curve defined by a 40-percent reduction of area (fig. 8) separates specimens failing by brittle fracture from those failing by ductile fracture for fluences greater than $4 \times 10^{19}$ neutrons per square centimeter. For fluences less than $4 \times 10^{19}$ neutrons per square centimeter, a few specimens yielded and showed some plastic strain but fractured without achieving a 40-percent reduction of area.

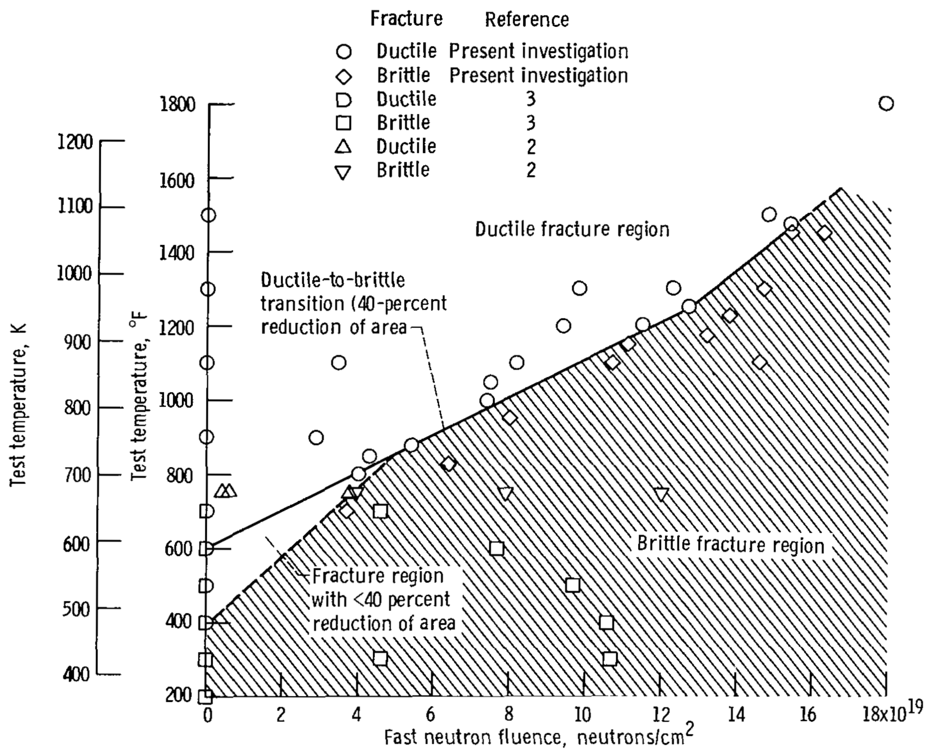



Figure 12. - Ductile-brittle fracture behavior of irradiated commercially pure tungsten. Neutron energy, greater than 1 MeV (160 fJ).

## Stress-Strain Behavior

The character of the stress-strain curve was also influenced by irradiation exposure and test temperature. Figure 13 shows typical stress-strain behavior for various fluences over the range investigated. The test temperature for the data illustrated was $1100^{\circ}$ F (867 K). The comparisons shown in figure 13 employ the strain at fracture as zero on the abscissa. This method of comparison clearly shows that the strength increases and the total elongation to fracture decreases with increasing fluence. The brittle fracture strength following $14.6\times10^{19}$ neutrons per square centimeter is 169 percent greater than the ultimate tensile strength of unirradiated material. From the comparisons shown in figure 13, it is clearly evident that, when irradiated to $3.5\times10^{19}$ neutrons per square centimeter, the initial strain-hardening region is compressed and the elongation occurs principally in the necking region. Irradiation to the higher fluence of $8.2\times10^{19}$ neutrons per square centimeter further compresses the initial strain-hardening region and initiates yield-point behavior without significantly altering the necking elongation.

Figure 14 shows that the stress-strain behavior with decreasing test temperature

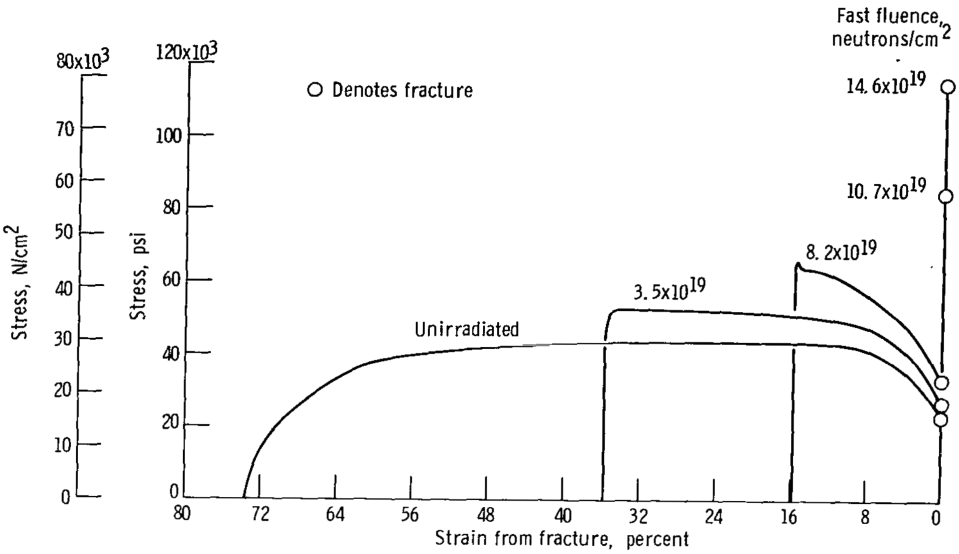



Figure 13. - Effect of neutron fluence on stress-strain behavior of tungsten for test temperature of 1100° F (867 K). Neutron energy, greater than 1 MeV (160 fJ).

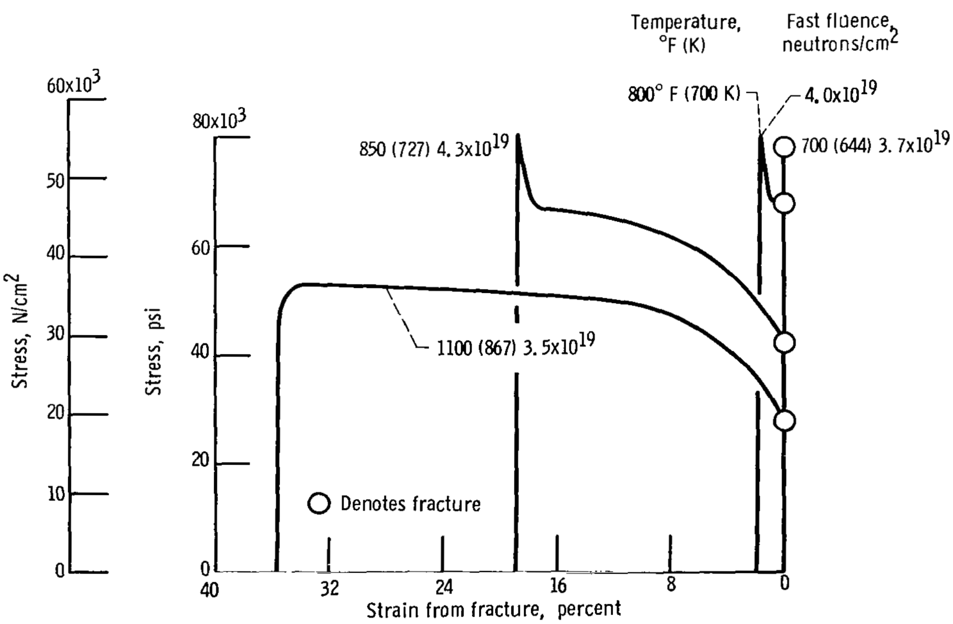



Figure 14. - Effect of test temperature on stress-strain behavior of tungsten irradiated to nominal fluence of $4.0\times10^{19}$ neutrons per square centimeter. Neutron energy, greater than 1 MeV (160 fJ).

for a nominal fluence of $4 \times 10^{19}$ neutrons per square centimeter is similar to that for increasing fluence. The yield-point phenomenon, however, is more pronounced at the lower test temperatures. Near the transition from ductile to brittle behavior, the necking elongation is sharply reduced. Moteff (ref. 2) reports comparable data for a test temperature of 752° F (673 K). In addition, he reports that brittle fracture occurs when the test specimen is shielded with 0.02-inch (0.5-mm) cadmium during irradiation to $3.9 \times 10^{19}$ neutrons per square centimeter. Such behavior suggests that some ductility is imparted by the thermal neutron transmutation of tungsten to rhenium.

The effect of test temperature on stress-strain behavior of irradiated tungsten can be more conveniently discussed in terms of engineering tensile properties. The engineering tensile properties of interest to this discussion are the yield strength, the ultimate strength, and the total elongation. Test data are compiled in tables IV and V.

Yield strength. - The yield strength as a function of test temperature is shown in figure 15. For the unirradiated condition, the yield strength is the 0.2-percent offset strength and shows a large temperature dependence below about 800° F (700 K). Above

TABLE IV. - ENGINEERING TENSILE PROPERTIES OF UNIRRADIATED TUNGSTEN TESTED IN TEMPERATURE RANGE 300° TO 1500° F (422 TO 1089 K)

| Specimen[a] | Test temperature[b] | | Brittle fracture strength | | Yield strength | | | | Lower yield point | | Ultimate tensile strength | | Reduction of area, percent | Total elongation,[c] percent |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | 0.2-Percent offset | | Upper yield point | | | | | | | |
| | °F | K | psi | N/cm² | psi | N/cm² | psi | N/cm² | psi | N/cm² | psi | N/cm² | | |
| CT-55 | 300 | 422 | $66.5 \times 10^3$ | $45.8 \times 10^3$ | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| CT-56 | 300 | 422 | 70.5 | 48.6 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| CT-60 | 300 | 422 | 69.5 | 47.9 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| CT-5 | 400 | 478 | 67.1 | 46.2 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 1 | 0 |
| CT-6 | 400 | 478 | 66.1 | 45.5 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 1 | 0 |
| CT-58 | 400 | 478 | -------- | -------- | $61.0 \times 10^3$ | $42.0 \times 10^3$ | -------- | -------- | -------- | -------- | $63.7 \times 10^3$ | $43.9 \times 10^3$ | 1 | 1 |
| CT-7 | 500 | 533 | -------- | -------- | 47.2 | 32.5 | -------- | -------- | -------- | -------- | 66.5 | 45.8 | -- | 9 |
| CT-8 | 500 | 533 | -------- | -------- | 51.5 | 35.5 | -------- | -------- | -------- | -------- | 65.5 | 45.1 | 3 | 4 |
| CT-9 | 500 | 533 | -------- | -------- | 48.4 | 33.3 | -------- | -------- | -------- | -------- | 65.0 | 44.8 | 6 | 5 |
| CT-10 | 600 | 589 | -------- | -------- | 35.2 | 24.3 | -------- | -------- | -------- | -------- | 60.4 | 41.6 | 49 | 54 |
| CT-11 | 600 | 589 | -------- | -------- | 39.8 | 27.4 | -------- | -------- | -------- | -------- | 61.6 | 42.4 | 33 | 50 |
| CT-12 | 600 | 589 | -------- | -------- | 35.2 | 24.3 | -------- | -------- | -------- | -------- | 58.9 | 40.6 | 33 | 52 |
| CT-13 | 700 | 644 | -------- | -------- | 22.5 | 15.5 | -------- | -------- | -------- | -------- | 54.8 | 37.8 | 68 | 63 |
| CT-15 | 700 | 644 | -------- | -------- | 23.0 | 15.8 | -------- | -------- | -------- | -------- | 55.1 | 38.0 | 68 | 74 |
| CT-59 | 700 | 644 | -------- | -------- | 22.4 | 15.4 | -------- | -------- | -------- | -------- | 56.0 | 38.6 | 68 | 64 |
| 100 | 900 | 756 | -------- | -------- | -------- | -------- | $21.0 \times 10^3$ | $14.5 \times 10^3$ | $20.5 \times 10^3$ | $14.1 \times 10^3$ | 49.3 | 34.0 | 83 | 71 |
| 101 | 900 | 756 | -------- | -------- | 14.8 | 10.2 | -------- | -------- | -------- | -------- | 51.3 | 35.3 | 82 | 68 |
| 102 | 1100 | 867 | -------- | -------- | 12.2 | 8.4 | -------- | -------- | -------- | -------- | 43.3 | 29.8 | 72 | 72 |
| 103 | 1100 | 867 | -------- | -------- | -------- | -------- | 15.7 | 10.8 | 15.1 | 10.4 | 42.0 | 28.9 | 88 | 59 |
| 104 | 1300 | 978 | -------- | -------- | 13.2 | 9.1 | -------- | -------- | -------- | -------- | 40.4 | 27.8 | 87 | 62 |
| 105 | 1300 | 978 | -------- | -------- | 12.0 | 8.3 | -------- | -------- | -------- | -------- | 40.5 | 27.9 | 87 | 71 |
| 106 | 1500 | 1089 | -------- | -------- | 10.3 | 7.1 | -------- | -------- | -------- | -------- | 38.5 | 26.5 | 76 | 66 |
| 107 | 1500 | 1089 | -------- | -------- | 9.2 | 6.3 | -------- | -------- | -------- | -------- | 38.0 | 26.2 | 81 | 67 |

[a]Specimens designated CT were tested in prior program (ref. 3).

[b]All specimens tested at 900° F (756 K) and above were termally cycled in NaK-56, then tested in argon. All specimens designated CT were tested in air with no prior environmental exposure.

[c]Elongation values calculated from stress-strain curves.

TABLE V. - ENGINEERING TENSILE PROPERTIES OF IRRADIATED[a] TUNGSTEN TESTED IN TEMPERATURE RANGE 700° TO 1800° F (644 TO 1256 K)

[Fast neutron energy, >1 MeV (160 fJ).]

| Specimen | Fluence, neutrons/cm$^2$ E > 1 MeV (160 fJ) | Test temperature[b] | | Brittle fracture strength | | Yield strength | | | | Lower yield point | | Ultimate tensile strength | | Reduction of area, percent | Total elongation,[c] percent |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | 0.2-Percent offset | | Upper yield point | | | | | | | |
| | | °F | K | psi | N/cm$^2$ | psi | N/cm$^2$ | psi | N/cm$^2$ | psi | N/cm$^2$ | psi | N/cm$^2$ | | |
| 99 | 2.9×10$^{19}$ | 900 | 756 | -------- | -------- | -------- | -------- | 65.1×10$^3$ | 44.9×10$^3$ | 56.5×10$^3$ | 38.9×10$^3$ | -------- | -------- | 75 | 36 |
| 89 | 3.5 | 1100 | 867 | -------- | -------- | 49.5×10$^3$ | 34.1×10$^3$ | -------- | -------- | -------- | -------- | 52.6×10$^3$ | 36.2×10$^3$ | 89 | 36 |
| 70 | 3.7 | 700 | 644 | 78.1×10$^3$ | 53.8×10$^3$ | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 94 | 4.0 | 800 | 700 | -------- | -------- | -------- | -------- | 80.5 | 55.5 | 67.9 | 46.8 | -------- | -------- | 1 | 2 |
| 75 | 4.3 | 850 | 728 | -------- | -------- | -------- | -------- | 80.8 | 55.7 | 67.4 | 46.4 | -------- | -------- | 73 | 19 |
| 98 | 5.4 | 875 | 742 | -------- | -------- | -------- | -------- | 78.5 | 54.1 | 67.1 | 46.2 | -------- | -------- | 62 | 19 |
| 88 | 6.4 | 825 | 714 | 90.1 | 62.1 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 81 | 7.4 | 1000 | 811 | -------- | -------- | -------- | -------- | [d]75.0 | [d]51.7 | 65.0 | 44.8 | -------- | -------- | 74 | 17 |
| 93 | 7.5 | 1050 | 839 | -------- | -------- | -------- | -------- | [d]67.9 | [d]46.8 | 65.1 | 44.9 | -------- | -------- | 72 | 16 |
| 97 | 8.0 | 950 | 783 | 85.1 | 58.6 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 71 | 8.2 | 1100 | 867 | -------- | -------- | -------- | -------- | [d]64.4 | [d]45.1 | 63.7 | 43.9 | -------- | -------- | 81 | 16 |
| 87 | 9.4 | 1200 | 922 | -------- | -------- | -------- | -------- | [d]66.4 | [d]45.7 | 60.1 | 41.4 | -------- | -------- | 88 | 16 |
| 76 | 9.8 | 1300 | 978 | -------- | -------- | -------- | -------- | 78.8 | 54.3 | 61.9 | 42.6 | -------- | -------- | 87 | 15 |
| 96 | 10.7 | 1100 | 867 | 84.6 | 58.3 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 92 | 11.1 | 1150 | 894 | 85.1 | 58.6 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 82 | 11.5 | 1200 | 922 | -------- | -------- | -------- | -------- | 89.6 | 61.7 | 68.1 | 46.9 | -------- | -------- | 81 | 14 |
| 86 | 12.3 | 1300 | 978 | -------- | -------- | -------- | -------- | 90.2 | 62.1 | 66.1 | 45.5 | -------- | -------- | 85 | 15 |
| 72 | 12.7 | 1250 | 950 | -------- | -------- | -------- | -------- | [d]72.7 | [d]50.1 | 70.6 | 48.6 | -------- | -------- | 80 | 14 |
| 95 | 13.2 | 1175 | 908 | 99.1 | 68.3 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 83 | 13.8 | 1225 | 936 | 90.7 | 62.5 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 91 | 14.6 | 1100 | 867 | 114.7 | 79.0 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 84 | 14.7 | 1300 | 978 | 88.6 | 61.0 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 85 | 14.8 | 1500 | 1089 | -------- | -------- | -------- | -------- | 84.0 | 57.9 | 60.4 | 41.6 | -------- | -------- | 72 | 14 |
| 77 | 15.4 | 1475 | 1075 | -------- | -------- | -------- | -------- | [d]77.5 | [d]53.4 | 59.6 | 41.1 | -------- | -------- | 85 | 13 |
| 73 | 15.4 | 1450 | 1061 | 79.4 | 54.7 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 74 | 16.3 | 1450 | 1061 | 79.9 | 55.1 | -------- | -------- | -------- | -------- | -------- | -------- | -------- | -------- | 0 | 0 |
| 90 | 17.9 | 1800 | 1256 | -------- | -------- | 43.5 | 30.0 | -------- | -------- | -------- | -------- | 72.2 | 49.7 | 68 | 23 |

[a]Irradiated in NaK-56 for 873 hr.
[b]All tests conducted in argon.
[c]Elongation values calculated from stress-strain curves.
[d]Strain, approx. 0.1 percent, accompanied yielding.

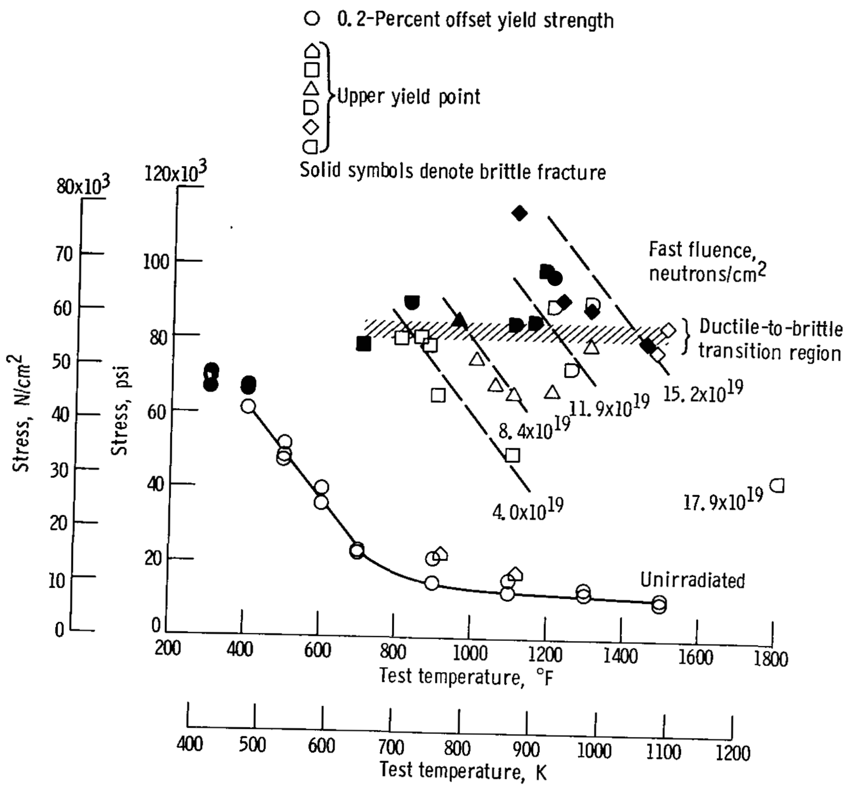



Figure 15. - Effect of test temperature on yield strength of unirradiated and irradiated tungsten. Neutron energy, greater than 1 MeV (160 fJ).

$800^\circ$ F (700 K), the temperature dependence of the yield strength is quite small. These results are in good agreement with data reported in the literature (e. g. , ref. 7).

The effect of irradiation on stress-strain behavior was to compress the strain-hardening region and initiate yield-point behavior. Hence, the irradiated test data shown in figure 15 are the upper yield point, and it is evident that irradiation increases the yield strength. The temperature dependence of the unirradiated yield strength between about $400^\circ$ and $800^\circ$ F (478 and 700 K) appears to be merely shifted to higher test temperature intervals for irradiated conditions.

The brittle fracture strengths, also plotted in figure 15, are generally higher than the unirradiated yield strengths and irradiated upper yield points. The increase in strength with decreasing test temperature appears to be a continuous function without regard to fracture mode. The strength at the transition from ductile to brittle fracture is approximately constant at about 80 000 psi (55 100 N/cm$^2$) regardless of test temperature and fluence. Irradiated material tested at temperatures in the range of $1200^\circ$ to $1300^\circ$ F (922 to 978 K), however, show this strength to be about 90 000 psi (62 000 N/cm$^2$).

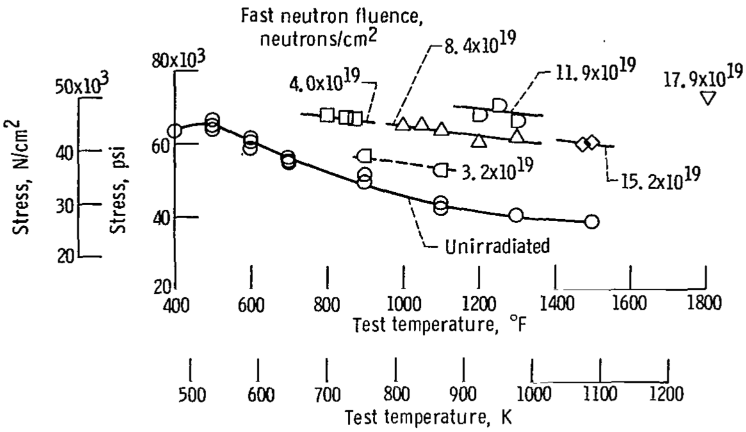



Figure 16. - Effect of test temperature on ultimate strength of irradiated tungsten. Neutron energy, greater than 1 MeV (160 fJ).

Ultimate strength. - The ultimate strength is defined for the purposes of this discussion as the point on the stress-strain curve showing plastic instability (i.e., the onset of necking). For the irradiated test data, this onset of necking corresponds to the lower yield point in all but two instances (see table V).

The ultimate strength as a function of test temperature is shown in figure 16. The unirradiated test data show that the ultimate strength is temperature dependent and decreases with increasing test temperature. The irradiated data show an increase in strength, but with increasing test temperature, the strength fell off at about 1100 psi (760 N/cm$^2$) per 100$^\circ$ F (56 K). The decrease in strength with increasing test temperature for irradiated material is in agreement with the trend of the unirradiated data at the higher test temperatures.

Total elongation. - The total elongation as a function of test temperature is shown in figure 17. The unirradiated data show that between 500$^\circ$ and 700$^\circ$ F (533 and 644 K)

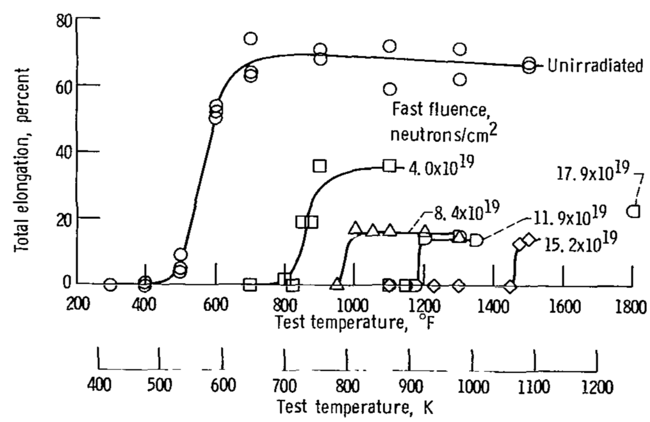



Figure 17. - Effect of test temperature on total elongation of unirradiated and irradiated tungsten. Neutron energy, greater than 1 MeV (160 fJ).

the elongation increases rapidly to values in the range of 65 to 70 percent. Above 700° F (644 K), the total elongation remains high and shows little temperature dependence. The irradiated test data, on the other hand, show the initiation of elongation to be shifted to higher test temperatures, and the temperature interval for achieving maximum elongation is markedly reduced. For fluences above about $8\times10^{19}$ fast neutrons per square centimeter, the total elongation tends to reach a maximum value of about 14 percent for the test temperatures investigated. This elongation occurs primarily in the necked region of the test specimen (fig. 9).

The behavior of the total elongation (fig. 17) is similar to that of the reduction of area (fig. 7), except for the maximum values of ductility. The reduction of area shows no difference in maximum values for unirradiated and irradiated conditions, while the maximum values for total elongation are reduced by at least a factor of 4 because of irradiation.

## Phenomenological Behavior

The data obtained during the present investigation and the comparable data reported by Moteff (ref. 2) provide a good phenomenological description of stress-strain and fracture behavior for tungsten irradiated at about 250° F (395 K). This behavior represents the synergistic effect for both thermal neutron and fast neutron interactions, with the lattice structure of the metal for test temperatures near the transition from ductile to brittle fracture. As a result of these neutron-metal interactions, the metal is strengthened, and plastic strain is concentrated within a small-volume element of the test specimen gage section. This strain concentration reduces the maximum value of the total elongation by at least a factor of 4 but does not alter the maximum value of the reduction of area.

The neutron-metal interactions with the lattice structure of the metal inhibit the onset of yielding, thereby raising the temperature required for plastic flow as well as increasing the stress required for yielding. The strength at the transition from ductile to brittle fracture is approximately constant - near 80 000 psi (55 100 $N/cm^2$) - for all irradiated test conditions, regardless of DBTT. The temperature dependence for yielding is not altered over the range of fluence and test temperature investigated.

The strength for plastic instability (i.e., ultimate strength) is also increased by irradiation.

## SUMMARY OF RESULTS

The effect of reactor irradiation at $250^{\circ}$ F (395 K) in the range of $3\times10^{19}$ to $18\times10^{19}$ fast neutrons per square centimeter on the ductile-to-brittle transition behavior of commercially pure polycrystalline tungsten in the recrystallized condition was investigated. Tensile tests conducted in the range of $700^{\circ}$ to $1800^{\circ}$ F (644 to 1256 K) were used to determine the transition from ductile to brittle fracture. Microstructures of the tensile fractures were also obtained for investigating changes in the fracture mode.

The principal test results obtained and the conclusions reached from this investigation are as follows:

1. The ductile-to-brittle transition temperature (DBTT) for a 40-percent reduction of area increases with increasing neutron fluence. The DBTT increases at a rate of $50^{\circ}$ F (28 K) per $10^{19}$ neutrons per square centimeter up to $12\times10^{19}$ neutrons per square centimeter.

2. The temperature range for transition from brittle fracture to ductile fracture with appreciable grain elongation was reduced by irradiation. For the unirradiated condition, the transition took place over an interval of $200^{\circ}$ F (111 K), whereas a comparable interval for irradiated conditions was $50^{\circ}$ F (28 K).

3. As a result of neutron irradiation, tungsten was strengthened and plastic strain was concentrated within a small-volume element of the test specimen gage section.

4. Irradiation caused an increase in the stress and temperature required for yielding but did not alter the temperature dependence of the yield stress.

5. The strength at the transition from brittle to ductile fracture was approximately constant - about 80 000 psi (55 100 N/cm$^2$) - for the fluence and test temperatures investigated.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 21, 1970,
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