**CONCLUDING PROGRESS REPORT
A STUDY
OF TUNGSTEN-TECHNETIUM ALLOYS
OCTOBER 1, 1966-AUGUST 1, 1968**

R. G. NELSON
D. P. O'KEEFE

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By

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Chemistry and Metallurgy Division

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INTRODUCTION

Technetium is a sister element to rhenium and has many properties similar to those of rhenium. It is predicted that technetium and rhenium will have about the same effects on tungsten in regard to increase in workability, lowered ductile-to-brittle transition temperature, and improved ductility.

The objectives of the current work are to recover technetium from fission product wastes at Hanford and reduce it to purified metal; prepare W-Tc alloys containing up to 50 at. % Tc; fabricate the alloy ingots to sheet stock and assess the effect of technetium on workability; and perform metallurgical and mechanical property evaluation of the fabricated alloys.

Previous reports have described the separation and purification of 800 g of technetium metal powder, the melting of technetium and W-Tc alloys, some properties of the cast alloys, and forging of the cast alloys.

EXPERIMENTAL PROCEDURES

TUNGSTEN-TECHNETIUM ALLOYS

Fabrication

During this reporting period the forged alloys were rolled to 0.050 in. sheets and cut into bend test specimens. Ductile-to-brittle transition temperatures were then determined for most of the alloys. In addition, cold-work, recrystallization, and tensile data have been obtained on pure technetium.

Hot rolling of the W-Tc alloys and the pure technetium was performed on an 8 in., 4 high rolling mill shown in Figures 1, 2, and 3. A rolling temperature of 1500 °C was chosen for all of the material. Specimens were held in the furnace for 10 min between roll passes and returned to the furnace for 10 min after the last roll pass. The specimens were in a hydrogen atmosphere during heating and cooling, and a tungsten rod was used to push the specimens through the furnace and into the rolling mill. Temperature measurements were made using an optical pyrometer. Ten to fifteen percent reductions per roll pass were used down to the finished thickness of 0.050 in. The results of the hot working of the W-Tc alloys are summarized in Table I, and detailed rolling schedules for each of the 25 to 30 g forgings are given in the Appendix, Table A.I.

Successful sheet was produced from the following compositions: W-2.5, 3.5, 5, 10, 20 and 40 at.% Tc. Duplicate specimens of most of the compositions were rolled, and in the case of the W-10 and 20 at.% Tc one each failed during hot working. All of the W-30, 50, and 60 at.% Tc alloys failed during hot working. No apparent reason can be offered for the failure of both of the W-30 at.% Tc specimens while both of the W-40 at.% specimens were successfully rolled to finished sheet.

Microstructures* of the finished hot rolled sheet alloys are shown in Figures 4 to 7. Typical of all of these compositions is the extremely large recrystallized grain structure. A fine precipitate of the sigma phase is first noted in the W-40 at.% Tc alloy which is what one would expect from the equilibrium diagram shown in Figure 8.

Figures 9 to 11 are microstructures of the alloys that failed during hot working. The peculiar cubic cracking pattern was evident in the W-10, 20 and 30 at.% Tc specimens. The W-50 and 60 at.% Tc alloys show a considerable amount of the sigma phase to be present, and the W-60 at.% Tc alloy is extremely hard (1434 DPH).

* All alloy metallographic specimens were etched with Murakami's reagent with a 5 sec swab.

FIGURE 1. Schematic of Heating and Cooling Chambers for 8 in. Mill.

Neg 0642681-9

FIGURE 2. Eight Inch Rolling Mill Showing the Hydrogen Atmosphere,
Water-Cooled Exit Chamber.

Neg PNL-40583-9

FIGURE 3. Eight Inch Rolling Mill Showing the Hydrogen Atmosphere Preheat Furnace.

TABLE I. Summary of Hot Working of W-Tc Alloys

Alloy, at. %	Forging, 1700 °C	Rolling, 1500 °C	Number of Bend Specimens Produced
Pure W	ok	ok	5
W-25 Re	ok	ok	7 (3) ^(a)
W-2.5 Tc	ok	ok	5 (1)
W-3.5 Tc	ok	ok	3
W-5.0 Tc	ok	ok	4 (1)
W-5.0 Tc	ok	ok	4 (1)
W-10 Tc	One large edge crack	Broke upon third roll pass	0
W-10 Tc	ok	ok	3
W-20 Tc	Broke up	Did not roll	0
W-20 Tc	ok	ok	6 (2)
W-30 Tc	Severe edge cracking	Broke upon fourth roll pass	0
W-30 Tc	Severe edge cracking	Broke upon sixth roll pass	0
W-40 Tc	Edge cracking	ok	5 (2)
W-40 Tc	Edge cracking	ok	6 (3)
W-50 Tc	Severe cracking throughout	Did not roll	0
W-60 Tc	Broke up	Did not roll	

(a) Number of prepared specimens on hand that were not tested

Neg 468-5194

100X

Pure W

429 DPH

Neg 468-52535A

100X

W-25 wt% Re

461 DPH

FIGURE 4. Microstructure of Pure W and W-25 at. % Re Control Specimens Hot Rolled at 1500 °C to a 0.050 in. Thick Sheet (Note the large grain size of the W and the duplex structure of the W-25 at. % Re alloy.)

Neg 468-5192A

2.5 at.% Tc

100X

414 DPH

Neg 468-5193A

3.5 at.% Tc

100X

431 DPH

Neg 468-5231A

5.0 at.% Tc

100X

429 DPH

FIGURE 5. Microstructure of W-Tc Alloys Hot Rolled at 1500 °C to a 0.050 in. Thick Sheet
(Note the large grain size.)

FIGURE 6. Microstructures of W-Tc Alloys Hot Rolled at 1500 °C to a 0.050 in. Thick Sheet [Note the large grain size and substructure. Delta phase is not resolved at this magnification in the 40 at.% Tc Alloy (See Figure 7).]

Neg 468-5190B

250X

Forged 1700 °C

Neg 468-5374B

250X

Neg 468-5375C

750X

Rolled 1500 °C

40 at. % Tc

FIGURE 7. Microstructure of Forged W-Tc Alloy. (Forged and hot rolled at 1500 °C to a 0.050 in. thick sheet sigma phase is present in this alloy.)

FIGURE 8. Tungsten-Techneium Binary Equilibrium Diagram

Neg 468-5370B

100X Neg 468-5370A

100X

10 at.% Tc 408-439 DPH

Neg 468-5371A

100X Neg 468-5371B

100X

20 at.% Tc 348-502 DPH

FIGURE 9. Microstructures of W-Tc Alloys That Failed During Forging
(Note the peculiar cubic cracking pattern.)

Neg 468-5372A

100X

432 to 551 DPH

Neg 468-5378

1.5X

FIGURE 10. Microstructure of Forged 30 at.% Tc Alloy Failed by Laminar Split After the Fourth and Sixth Roll Passes.

Neg 468-5376 A

100X

Neg 468-5376B

750X

50 at.% Tc Sigma Dark, 813 DPH Alpha Light, 558 DPH

Neg 468-5191A

100X

Neg 468-5191C

500X

60 at.% Tc 1434 DPH (See microprobe and microhardness of W-40 and 60 at.% Tc alloys)

FIGURE 11. Microstructures of Forged W-Tc Alloys (These alloys failed during 1700 °C forging. Note the cored structure of the 50 at.% Tc alloy.)

Microhardness tests were made on all of the alloys and are shown in Table A. II. These data are plotted in Figure 12.

Ductile-Brittle Transition Temperatures

The effect of composition on the ductile-brittle bend transition behavior of the W-Tc alloy series was obtained for the alloy compositions that were successfully fabricated into sheet form. The bend tests were performed approximately as described previously in a report of this series.⁽¹⁾ Specimens 0.25 in. wide by approximately 7/8 in. long were cut from the sheet by electrical discharge machining. The long dimension was parallel to the direction of rolling. The tension side of each bend specimen was surface-ground through the grit series 280, 320, 400, and 600 to remove superficial cracks and other imperfections that were present in most of the sheet specimens. This resulted in a specimen thickness of 0.040 to 0.045 in.

The specimens were center-loaded as simple beams with 0.57 in. between supports and at a deflection rate of 0.020 in./min. Heating was accomplished with a resistance-wound split tubular furnace. The testing end-point was at the inception of cracking in the specimen, which was clearly indicated in each case by the drop in load on the Instron test machine weighing system, or when a deflection of about 0.140 in. had been reached without cracking. At a deflection of 0.140 in. the included angle of bend was about 100°. The bend specimens are shown in Figure 13 after testing. The three short specimens which are included were tested by bending into a V-block fixture which provided a short span. The bend angle in these specimens was converted to deflection over the 0.57 in. span for plotting with the other data.

The bend test results for the individual alloy compositions are shown in Figures 14 and 15. Pure tungsten and W-25 at. % Re specimens which were fabricated and tested as controls along with the technetium-containing alloys, are also shown. The extremely high bend transition temperatures for the pure tungsten and the completely brittle performance

FIGURE 12. Hardness of Wrought W-Tc Alloys

Neg PNL-0682622-1

FIGURE 13. Bend Test Specimens of Tungsten-Technetium Alloys

FIGURE 14. Bend Test Results for Electron-Beam-Melted, Hot-Rolled, 1500 °C Annealed W-Tc Alloy, 0.045 in. Thick Sheet.

FIGURE 15. Bend Test Results for Electron-Beam-Melted, Hot-Rolled, 1500 °C Annealed W, W-Tc, and W-Re Alloy, 0.045 in. Thick Sheet.

for W-25 at. % Re at 900 °C indicates the basically nonductile state that the fabrication procedure imparted to the tungsten-base alloys of this test series. Recrystallized tungsten sheet ordinarily has a bend transition temperature at about 350 °C. Obviously, the fabrication procedure, which was the same for all specimens, was very unfavorable for the production of ductile sheet from any tungsten-base alloy.

Even in this curiously brittle state, however, an effect of technetium additions is quite apparent in the bend test results. When a bend specimen deflection of 0.100 in. as a ductile-brittle transition point was used, the effect of composition on the transition was as shown in Figure 16. The minima in this curve occurs at compositions similar to those on the corresponding curve for rhenium additions in tungsten alloys annealed at 2000 °C. Transition temperature-composition curves developed by Klopp, et al.⁽²⁾ for fully annealed rhenium alloys are shown in Figure 16 for comparison.

Microprobe and Microhardness of W-40 and 60 at. % Tc Alloys

Microprobe analyses of two tungsten technetium alloys (40 at. % Tc and 60 at. % Tc) were conducted to gain more information about the W-Tc equilibrium diagram. Although an earlier analysis was performed on 50 and 60 at. % Tc alloys (described in a previous report of this series),⁽³⁾ the alloys were in the "as-cast" condition, and the results represented nonequilibrium conditions. It was felt that after a long-time anneal at relatively high temperatures, an equilibrium two-phase ($\alpha + \sigma$) structure might develop in the 60 at. % Tc alloy, and that an analysis of this structure would yield the phase boundaries. The 40 at. % Tc alloy was to be used for the purpose of calibrating the microprobe. The specimens were taken from alloy buttons that had been prepared by arc melting, were remelted in an electron beam furnace, and were subjected to the following thermal and mechanical treatment.

FIGURE 16. Bend Transition Temperatures of Annealed Tungsten-Technetium and Tungsten-Rhenium Alloys.

Heating:	5 hr to 1200 °C	Cooling:	3 hr to 800 °C
	16 hr at 1200 °C		5 hr to room temperature
	2 hr to 1700 °C		30 min to 1700 °C
	70 hr at 1700 °C		50% reduction in one forge pass
			Cool to room temperature.

Figure 17 illustrates the resulting microstructure of the 40 and 60 at.% alloys. The dark region in the 60 at.% alloy is near the surface of the specimen.

The microprobe analysis of 40 at.% Tc alloy, which was to be used for a calibration standard, indicated a variation in composition from grain to grain of as much as 2 or 3 at.% Tc. Thus, it was not possible to obtain an accurate calibration of the probe data, and the results for the 60 at.% Tc alloys may be systematically in error by 2 or 3 at.%.

In the 60 at.% Tc alloy, there were no indications of any significant differences in composition between the light "islands" and the darker, apparently two-phase regions between the "islands". There was, however, a variation of about 2 at.% Tc between the dark region near the surface and the center of the sample, the center being richer in technetium. This is probably due to the selective loss of technetium from the surface during heat treatment. The composition of the "islands" in the center of the specimen was determined to be approximately 63 at.% Tc. Unless diffusion is extremely sluggish in these alloys, one must assume that the "islands" are single-phase regions, either α or σ . On the basis of the earlier metallographic and microprobe results for the as-cast alloys, one would expect that the smaller two-phase regions are α and the "islands" are σ . This interpretation would place the boundary between the σ phase and $\sigma + \alpha$ region near 63 at.% Tc at 1700 °C, which, in view of Nemiec's⁽⁴⁾ data, would suggest that the range of stability of the σ phase increases with increasing temperature between 1200 °C and 1700 °C.

FIGURE 17. Microstructures of W-Tc Alloys Homogenized 70 hr at 1700 °C Then Forged at 1700 °C 50% Reduction, One Pass

Microhardness measurements were made to see if any differences in hardness were evident between the "islands" and the two-phase regions in the 60 at.% alloy. The hardness indentations, resulting from the use of a 100 g load are shown in Figure 18. As may be seen from the figure, it was possible to place some of the indentations in the two-phase regions and others in the "islands". The average readings were about 1050 DPH, and no significant differences in the readings were obtained. The use of a 1 kg load, however, yielded average microhardness readings of about 1430 DPH, and the use of a 1.5 kg load resulted in brittle cracks in the alloy. The indentations resulting from these tests are also shown in Figure 18. The high degree of brittleness suggests that the 60 at.% Tc alloy is predominantly σ phase. Microhardness measurements were also made on the 40 at.% Tc alloy. These measurements were made with both 1 kg and 1.5 kg loads. Typical indentations are shown in Figure 19. Twinning was observed for both the 1 kg and the 1.5 kg loads. The hardness values were the same regardless of twinning when the 1 kg load was used. The hardness values were about 60 DPH lower when twinning occurred and the 1.5 kg load was used. The average hardness for the 40 at.% Tc alloy was about 610 DPH.

Diffusion Couples

Three diffusion couple assemblies have been prepared so that more information about the W-Tc phase diagram could be gathered. Because the diffusion couple design in the absence of radioactive material was to be tested, a trial W-Nb diffusion couple was made first. The diffusion couple assembly, which makes use of the difference between the thermal expansion coefficients of molybdenum and tantalum to apply pressure to the W-Nb interfaces is shown in Figure 20. The tungsten and niobium disks were prepared metallographically by grinding and polishing both sides of each disk. The disks were cleaned in acetone and given a final rinse in ethyl alcohol before assembly. After assembly, the diffusion couple was heated to 1800 °C in a vacuum ($\approx 10^{-6}$ torr) for 4 hr. Although the threaded plug was easily removed after the anneal, it was necessary

FIGURE 18. Microhardness Indentations in W-60 at.% Tc Alloy
(Note cracking with the 1.5 kg load; 250X)

Neg 468-5190E

250X

610 DPH 1 kg Load

FIGURE 19. Microhardness Indentations in W-40 at.% Tc Alloys. (The twinning did not alter the hardness measurement with this load.)

FIGURE 20. Differential Expansion Jig for Producing W-Tc Diffusion Couples [A W-Nb diffusion couple was produced in a test run (See Figure 21) All dimensions in inches.]

to cut the bottom of the cylinder off in order to remove the W-Nb sandwich. Figure 21 shows the bond produced at one of the W-Nb interfaces.

FIGURE 21. A Test Diffusion Couple Using W-Nb in the Differential Expansion Jig Shown in Figure 20. (Porosity in the Nb is probably due to the Kirkendall effect. As-polished.)

Because the Kirkendall porosity observed by Hehemann and Leber⁽⁵⁾ in W-Nb diffusion couples is similar to that in Figure 21, it is believed that the porosity in Figure 21 is due to the Kirkendall effect rather than to surface preparation effects.

The jigs for the W-Tc diffusion couples are similar to that shown in Figure 20 except that threaded end caps are on both ends of the cylinder instead of one. The technetium wafers were made by punching disks, 3/16 in. in diameter, from electron-beam-melted technetium sheet, 0.029 in. thick, and metallographically polishing both sides. The tungsten disks were made from tungsten rod of the following specifications:

Fabrication History: Cold-pressed and sintered, hot-swaged, recrystallized, and warm-worked.

Chemical Specifications: 99.95% minimum W

Maximum Impurities (%): Al 0.001
Si 0.003
Fe 0.006
Cr 0.003
Ni 0.003
Cu 0.002
Sn 0.001
O₂ 0.010

Two of the diffusion couples have been assembled, placed in cans made from Cb-1 wt% Zr tubing, evacuated, and sealed. The purpose of the cans is to provide protection against contamination of the vacuum furnace. A third diffusion couple assembly of improved design has been fabricated but not assembled.

PURE TECHNETIUM FABRICATION AND RECRYSTALLIZATION STUDIES

The material for the work on pure technetium consisted of one, 6.7 g spherical shaped electron beam melted button and one, 31.1 g semiflat arc-melted button. The objective of this work was to produce enough sheet material for the determination of some mechanical properties and in so doing to provide as much information as possible on the mechanical working and annealing characteristics of technetium.

Electron-Beam-Melted Pure Technetium

The electron-beam-melted button was annealed at 1500 °C for 10 min and cold-pressed from 0.318 in. thickness to 0.126 in. thick sheet bar in 12 forging passes with 4 intermediate anneals at 1500 °C for 10 min in the 8 in. rolling mill furnace shown in Figures 1, 2, and 3. Table A.III gives the detailed cold-forging schedule followed. The cold-forged sheet bar was then cold-rolled from 0.126 in. to 0.070 in. in 11 passes with a 1500 °C 10 min anneal after the third pass at a 0.104 in. thickness. Rockwell

superficial hardness tests were taken on the sheet before and after the anneal and after each subsequent cold rolling pass. Converted BHN values ranged from 106 in the annealed condition to 376 in the 32.7% cold-worked condition. The detailed intermediate cold-rolling schedule followed is shown in Table A.IV.

It was realized that to determine the full cold-working capacity of technetium, a fine grained equiaxed starting structure was a requirement, and the 1500 °C annealing temperature used to date was too high. The small amount of material (less than 5 g, 0.070 in. thick) prohibited the cutting of samples for a recrystallization determination at this time. Consequently, the sheet was successively annealed at 400, 500, 600, 700, and 800 °C and Rockwell superficial hardness tests made after each ten minute anneal. The BHN conversions were 352, 327, 253, 152, and 108 BHN respectively, compared to 106 BHN obtained with a 1500 °C anneal. It was therefore concluded that after the 800 °C anneal the structure was optimized for additional cold work experimentation. During this annealing and all subsequent annealing studies a Pt + Pt-10% Rh thermocouple was used with a manual potentiometer corrected for room temperature.

The 0.070 in. thick annealed technetium sheet was cold rolled to 0.029 in. thick sheet in 19 passes. Rockwell superficial hardness tests were taken after each pass and the hardness increased from 108 BHN in the annealed condition to 476 BHN in the 58.5% cold-worked condition. The details of this final cold rolling of the electron-beam-melted technetium are shown in Table A.V. The left hand portion of Figure 22 shows how the hardness of electron-beam-melted technetium varies with increasing amounts of cold work.

The technetium sheet was now in a form where small samples (3/16 by 1/4 in.) could be sheared off for the determination of a more accurate recrystallization temperature. A ten minute anneal was given to separate samples for 300, 500, 600, 700, 800, and 900 °C.

FIGURE 22. Cold Rolling and Annealing of Technetium

Rockwell superficial hardness tests were made on each sample and their converted BHN values were 425, 354, 200, 115, 93, and 88 respectively. These data are tabulated in Table A. VI, and the right hand portion of Figure 22 shows how the hardness of a 58.5% cold-worked technetium sheet varies with increased annealing temperatures. Figure 23* is a series of microstructures of the cold-worked and annealed electron-beam-melted technetium. The dark areas in the worked structures are high concentrations of mechanical twins. Comparing Figures 22 and 23 it is apparent that the 800 °C annealing temperature produces a soft equiaxed recrystallized structure free of mechanical twinning.

Diamond Pyramid Hardness tests (1 kg load, 136°) were also made on the samples shown in Figure 23. These varied from 402 in the cold worked condition to 133 in the 900 °C annealed condition. See Table A. VI.

Arc-Melted Pure Technetium

The processing of the 31.1 g arc-melted button was nearly identical to that of the electron beam melted button. The breakdown was done by rolling at 1500 °C instead of cold rolling. This was performed in 7 roll passes going from 0.145 in. to 0.078 in. thick. See Table A. VII.

Intermediate cold rolling was performed from 0.078 in. to 0.068 in. thickness in 5 roll passes, with a total reduction of 12.8% (see Table A. VIII). Rolling was finally stopped because of some surface cracking.

Successive annealing of the cold-rolled sheet was used to optimize the structure for additional cold rolling experimentation. However, higher annealing temperatures were required to reach a full-soft condition (131 BHN). It took an 1100 °C anneal to reach the full-soft condition as compared to 800 °C for the electron-beam-melted material.

* All pure technetium metallographic specimens were first etched 15 sec in 45 HNO₃-45 H₂O-10 HF parts by volume to remove flow metal and scratches. This was followed by a 5 sec swab in Murakami's reagent.

FIGURE 23. Microstructures of Electron-Beam-Melted, Cold-Worked and Annealed Technetium. (The dark areas in the worked structures are high concentrations of mechanical twins.)

Final cold rolling of the arc-melted technetium was performed in 17 roll passes from a thickness of 0.070 in. to a thickness of 0.037 in. This gave a total of 45.6% cold work (see Table A.IX). Rockwell superficial hardness readings converted to BHN varied from 131 in the annealed condition to 442 in the 45.6% cold-worked condition. These values are plotted on the left hand side of Figure 22, and it is noted that the two materials have identical work-hardening characteristics. The cold-rolled sheet was trimmed by electrical discharge machining to approximately 2-3/4 by 3-1/4 in. The end trimmings were sheared into samples approximately 1/16 by 1/4 in. for the determination of a more accurate recrystallization temperature. Seven samples were annealed for 10 min at 7 different temperatures ranging from 300 to 1100 °C. Their resulting converted Breinell Hardness Numbers ranged from 442 in the 46.5% cold-worked condition to 106 in the 1100 °C annealed condition (see Table A.X). These values are plotted on the right hand side of Figure 22, and it is noted that the full-soft condition is attained at about 950 °C as compared to 800 °C for the electron-beam-melted technetium. This difference could be due to the greater amount of cold work or improved purity of the electron-beam-melted technetium. Comparing the microstructures of the cold-worked and annealed specimens of the arc melted material shown in Figure 24 with Figure 22, it is shown that a full-soft equiaxed structure free of mechanical twinning is attained with a 950 °C anneal. It is interesting to note that the intermediate anneal took place at 1100 °C with 12.8% cold work and the final anneal at 950 °C with 45.6% cold work. The electron-beam-melted technetium recrystallized at 800 °C with 32.7 and 58.6% cold work.

Tensile Properties of Technetium

Tensile properties were obtained for pure technetium that was arc melted and rolled to a 0.037 in. sheet in the as-rolled and annealed conditions. Processing details for the sheet are described in the section of this report covering fabrication and recrystallization studies. The

100X

250X

Anneal Temp °C (10 min)	46% Cold Work	600	700	800	840	875	950	1100
BHN	442	358	305	210	152	139	112	106

FIGURE 24. Microstructures of Arc-Melted, Cold-Worked and Annealed Technetium.
(The dark areas in the worked structures are high concentrations of mechanical twins.)

as-rolled sheet contained 46% reduction in thickness. This sheet was divided into two parts by EDM machining, and one portion was annealed at 950 °C for 10 min. This produced a fully recrystallized structure as shown in Figure 24.

Tensile specimens were machined to the dimension shown in Figure 25 by the EDM process. A Young's Modulus determination was made on the cold worked specimen using 1 in. Tuckerman strain gages. On the cold-worked specimen there was over 0.002 in. of elastic deflection available in the 1 in. gage length which permitted good accuracy in strain measurement. Gages were mounted on opposite side of the specimen and repeated measurements were made as the specimen position was reversed in the tensile grips. The strain values so obtained had a spread of about 4%. These were averaged in computing the modulus value of 40.9×10^6 psi.

FIGURE 25. Tensile Test Specimens Used for Pure Technetium

A modulus measurement could not be obtained on the annealed specimen because it was slightly bowed in the plane of the sheet. This could not be removed without imparting to the specimen considerable plastic strain, which was avoided in order to provide good yield and ultimate data for the annealed condition.

Tensile strength values were obtained using a strain rate of between 0.001 and 0.002 per minute through the yield point and a rate between 0.01 and 0.02 per minute beyond the yield.

Test results were as follows:

	<u>As-Rolled</u>	<u>Annealed</u>
Tensile Ultimate Strength, psi	219, 000	115, 800
0.2% Offset Yield Strength, psi	187, 000	46, 300
Percent Elongation in 1 in.	4	30
Young's Modulus, psi	40.9×10^6	

The stress-strain curves obtained on these single specimen tests are shown in Figure 26. The annealed specimen did not abruptly fracture completely, but gradually released the load as the fracture progressed. The fracturing process was accompanied by about a 0.010 in. extension. The annealed specimen failed outside the 1 in. gage marks, (Figure 27) but nearly all of the elongation occurred uniformly throughout the reduced section of the specimen, and the elongation value of 30% is fairly accurate. Dimensions at the fracture of those specimens were 0.175 in. by 0.032 in. for the annealed and 0.203 in. by 0.035 in. for the cold-worked specimen. Orthogonal strain computed from these are as follows:

Cold-Worked	Annealed
$\epsilon_x = +0.30$	$\epsilon_x = +0.16$
$\epsilon_y = -0.18$	$\epsilon_y = -0.14$
$\epsilon_z = -0.13$	$\epsilon_z = -0.06$

These very rough measurements indicate fairly isotropic strain behavior for the annealed specimen and a preference for strain in the plane of the sheet in the cold-worked material.

FIGURE 26. Tensile Curves for Arc-Melted Pure Technetium, 0.037 in. Sheet, Annealed and 46% Cold Worked.

Tc-46% Cold Worked

Tc-Annealed

Neg PNL-0682622-2

FIGURE 27. Tensile Test Specimens

Miscellaneous Measurements

Ultrasonic

After trimming and prior to cutting the tensile specimens from the cold-worked arc-melted technetium sheet, ultrasonic measurements were made of the longitudinal and shear wave velocities. These were found to be:

$$V_{Tc_L} = 252,000 \text{ in./sec} = 6400 \text{ m/sec}$$

$$V_{Tc_S} = 136,000 \text{ in./sec} = 3450 \text{ m/sec}$$

When a density of 11.493 g/cm^3 was used, some of the elastic properties were calculated to be as follows:

$$\text{Shear modulus, } 13.7 \times 10^{11} \text{ dyne/cm}^2 (19.8 \times 10^6 \text{ psi})$$

$$\text{Bulk modulus, } 28.9 \times 10^{11} \text{ dyne/cm}^2 (41.9 \times 10^6 \text{ psi})$$

$$\text{Poisson's ratio, } 0.285$$

$$\text{Young's modulus, } 35.2 \times 10^{11} \text{ dyne/cm}^2 (51 \times 10^6 \text{ psi})$$

$$\text{Lam's constant, } 19.8 \times 10^{11} \text{ dyne/cm}^2 (28.7 \times 10^6)$$

These results are not considered accurate. Better results would have been obtained if the technetium samples were much thicker, were flat, and parallel sided, and made to have smoother surfaces. The dynamic Young's modulus of $40.9 \times 10^6 \text{ psi}$ as determined in the tensile test described earlier is considered an accurate measurement.

Surface Dose Rate

Surface dose rate measurements were also made prior to tensile testing. These were made using a standard extrapolation chamber having a window 7 mg/cm^2 thick. The thickness of the sample was 0.037 in. , which is essentially infinite for the $0.292 \text{ MeV } E_{\text{max}}$ beta particle of ^{99}Tc . The measured surface dose rate was 1250 rad/hr , with an estimated total error of no more than $\pm 5\%$. Almost the entire surface dose contribution was from the beta particle emission.

Sea Water Corrosion

A small sample of technetium 0.037 by 3/8 by 3/8 in. weighing approximately 1 g has been immersed in 2 liters of agitated synthetic sea water for over 1000 hr and shows no detectable change in weight or in visual appearance.

DISCUSSIONS AND CONCLUSIONS

It was realized at the beginning of this program that the 1500 °C working temperature would result in high ductile-to-brittle transition temperatures. It was not within the scope of this program to search for optimum working schedules for each of the alloys investigated, but rather to determine if technetium had the same ability to impart ductility to tungsten as does rhenium. The fact that the technetium lowered the transition temperature from 675 °C for pure tungsten to 280 °C for the 3.5 at.% Tc alloy indicates a ductilizing effect of technetium on tungsten. Comparing the recrystallized transition temperatures versus rhenium alloy content as reported by Klopp⁽²⁾ with the results of this investigation show that technetium additions lower the ductile-to-brittle transition temperature of tungsten in a manner very similar to rhenium. The similarity is even more surprising when one considers that the ingot size, fabrication histories, testing methods, and bend criteria of the two investigations were considerably different. In the recrystallized W-Tc alloys it appears that compositions in the range of 3.5 and 20 at.% are the most effective in lowering the bend transition temperature.

Pure technetium is a soft ductile refractory metal with a melting point of 2150 °C.⁽⁶⁾ At room temperature in the annealed condition it has a hardness of 90 to 110 BHN (converted), a tensile strength of 115,800 psi, and a yield strength at 0.2% offset of 46,300 psi with an elongation of 30%. Technetium can be cold-rolled to at least a 58% reduction without serious edge cracking. The tensile strength of 45% cold-rolled technetium is 219,000 psi, and the 0.2% offset yield strength is 187,000 psi with an elongation of 4% and a Young's Modulus of 40.9×10^6 psi. Cold-worked metal profusely exhibits mechanical twinning.

Technetium-99 has a high surface dose rate of 1250 rad/hr of a relatively low energy beta emission that is easily shielded with two pair of rubber surgeon's gloves. Skin burns are a possibility if technetium is handled with bare hands.

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APPENDIX A
TABLES A.I THROUGH A.X

TABLE A.I. Hot-Rolling Schedules for W-Tc Alloys

Pass No.	Down Set, in.	Thickness, in.	Temperature, °C	Remarks
Specimen, Pure W				
Thickness, 0.140 in.				
1	0.020	0.115	1554	Edge Cracks
2	0.020	0.099	1521	
3	0.010	0.085	1527	
4	0.010	0.075	1504	
5	0.010	0.067	1504	
6	0.010	0.064	1500	
7	0.010	0.060	1493	
8	0.010	0.055	1504	
9	0.010	0.047	1500	
Back into furnace for 10 min, cooled				
Specimen, W-25 at. % Re				
Thickness, 0.157 in.				
1	0.020	0.157	1500	Didn't Go Through Rolls Just Contacted
2	0.020	0.157	1500	
3	0.020	0.150	1500	
4	0.020	0.145	1520	
5	0.020	0.133	1515	
6	0.020	0.118	1520	
7	0.020	0.104	1500	
8	0.015	0.090	1500	
9	0.015	0.074	1500	
10	0.010	0.069	1500	
11	0.010	0.057	1500	
12	0.008	0.049	1500	
Back into furnace for 10 min, cooled				
Specimen, W-2.5 at. % Tc				
Thickness, 0.134 in.				
1	0.020	0.125	1570	One Edge Crack No More
2	0.020	0.115		
3	0.010	0.097		
4	0.006	0.094	1500	
5	0.010	0.085	1500	
6	0.010	0.073	1500	
7	0.010	0.054	1500	
8	0	0.047	1500	

TABLE A. I. (contd)

Pass No.	Down Set, in.	Thickness, in.	Temperature, °C	Remarks
Specimen, W-3.5 at. % Tc				
Thickness, 0.138 in.				
1	0		1570	Not Touched
2	0		1570	
3	0.010	0.136		End Crack
4	0.010	0.125	1515	
5	0.010	0.117	1500	
6	0.010	0.107	1500	
7	0.010	0.097	1500	
8	0.010	0.088	1500	
9	0.010	0.079	1500	
10	0.010	0.069	1500	
11	0.010	0.060	1500	
12	0.006	0.053	1500	
13	0.006	0.046	1500	

Back into furnace for 10 min, cooled

Specimen, W-5 at. % Tc
Thickness, 0.150

1		0.150	1495	Not Rolled
2	0.010	0.150	1515	Just Touched
3	0.010	0.129	1500	Large Crack
4	0.010	0.120	1500	End Crack
5	0.010	0.113	1500	
6	0.010	0.101	1500	
7	0.010	0.093	1500	
8	0.010	0.086	1500	
9	0.010	0.078	1500	
10	0.010	0.067	1500	
11	0.010	0.058	1500	
12	0.010	0.047	1500	

Back into furnace for 10 min, cooled

TABLE A.I. (contd)

<u>Pass No.</u>	<u>Down Set, in.</u>	<u>Thickness, in.</u>	<u>Temperature, °C</u>	<u>Remarks</u>
Specimen, W-5 at. % Tc				
Thickness, 0.120 in.				
1	0	0.120	1550	Just Touched
2	0.010	0.108	1530	
3	0.010	0.099	1510	
4	0.010	0.090	1510	
5	0.010	0.080	1510	
6	0.010	0.071	1510	
7	0.010	0.062	1510	
8	0.007	0.054	1520	
9	0.006	0.052	1510	Straightened One Small Crack
10	0	0.048	1510	

Back into furnace for 10 min, cooled

Specimen, W-10 at. % Tc
Thickness, 0.160 in.

1	0-0.010	0.150	1510	Just Touched
2	0.010	0.140	1510	
3	0.010	0.128	1510	
4	0.010	0.121	1510	
5	0.010	0.110	1500	
6	0.010	0.099	1500	
7	0.010	0.090	1510	
8	0.010	0.080	1500	
9	0.010	0.073	1505	Cross Rolled Crack Opened
10	0.010	0.063	1505	
11	0.008	0.055		
12	0.008	0.047	1505	

Back into furnace for 10 min, cooled

Specimen, W-10 at. % Tc
Thickness, 0.213 in., severe crack

1	0.003	0.207	1500	Broke Up
2	0.010	0.198		
3	0.010	0.189	1500	

Back into furnace for 10 min, cooled

TABLE A. I. (contd)

Pass No.	Down Set, in.	Thickness, in.	Temperature, °C	Remarks
Specimen, W-20 at. % Tc				
Thickness, 0.160 in.				
1	0-0.010	0.150	1505	
2	0.010	0.141	1505	Slight End Crack
3	0.010			Stuck to Roll
4	0	0.128		Straightening Pass
5	0.005	0.126	1505	Several Edge Cracks
6	0.005	0.120		
7	0.010	0.110	1500	Piece Broken Out
8	0.010	0.102	1500	
9	0.010	0.091	1500	
10	0.010	0.082	1500	
11	0.010	0.075	1500	
12	0.010	0.063	1500	
13	0.010	0.052	1500	Stuck to Roll
14	0.004	0.049	1500	

Back into furnace for 10 min, cooled

Specimens, W-30 at. % Tc
Thickness, 0.182

1	0.010	0.174	1515	
2	0.010	0.164	1510	
3	0.010	0.155	1525	
4	0.010	0.150	1500	Split By Lamination Rolling Stopped

Back into furnace for 10 min, cooled

Specimen, W-30 at. % Tc
Thickness, 0.0175

1	0.010	0.167	1505	
2	0.010	0.164	1500	
3	0.010	0.154	1508	
4	0.010	0.147		
5	0.010	0.138	1500	
6	0.010	0.122	1500	Piece Hit Lip of Cooling Chamber Cracked by Lamination. Stopped Rolling

Back in furnace for 10 min, cooled

TABLE A. I. (contd)

Pass No.	Down Set, in.	Thickness, in.	Temperature, °C	Remarks
Specimen, W-40 at. % Tc				
Thickness, 0.180 surface cracks one side				
1	0	0.180	1515	Just Touched
2	0.010	0.170	1510	
3	0.010	0.164	1515	
4	0.010	0.156	1515	
5	0.010	0.146	1505	
6	0.010	0.137	1505	
7	0.010	0.129	1505	
8	0.010	0.120	1510	Surface Cracks Are Not Getting Larger
9	0.010	0.110	1505	
10	0.010	0.097	1510	
11	0.010	0.089	1510	
12	0.010	0.078	1515	
13	0.010	0.070	1510	
14	0.010	0.063	1515	
15	0.010	0.054	1515	Some Surface Flaking
16	0.006	0.047	1503	

Back into furnace for 10 min, cooled

Specimen, W-40 at. % Tc

Thickness, 0.166 in.

1	0-0.010	0.153	1505	No Cracks Edges Are Rough
2	0.010	0.146	1510	
3	0.010	0.139		
4	0.010	0.132	1507	
5	0.010	0.120	1508	
6	0.010	0.113	1510	
7	0.010	0.102	1508	
8	0.010	0.090	1510	
9	0.010	0.082	1510	
10	0.010	0.073	1510	
11	0.010	0.065	1512	
12	0.010	0.057	1510	
13	0.009	0.051	1515	

Back into furnace for 10 min, cooled

TABLE A. II. DPH Tests on Wrought W-Tc Alloys
(1 kg Load 136 °)

	W Pure	W-.25 wt. % Re	W-2.5 at. % Tc	W-3.5 at. % Tc	W-5 at. % Tc	W-10 at. % Tc	W-20 at. % Tc	W-30 at. % Tc	W-40 at. % Tc	W-50 at. % Tc	W-60 at. % Tc
	426	487	426	414	414	408	432	534	600	631	1488
	421	432	414	414	414	414	502	551	600	510	1407
	439	444	371	451	451	426	348	544	502	534	1407
		479	444	444	444	432	362	544	534	854 P	
						439				772 P	
Av	429	461 s	414 s	431 s	431 s	424 f	411 f	543 f	559 s	558 f	
										813 P	1434 f
					398	402	432	439	600		
					366	444	421	432	600		
					408	426	432	466	589		
					426	444	408	472	600		
									611		
									611		
									589		
					400 s	429 s	423 s	452 f	600 f		

s = sheet specimen

f = forged specimen

P = sigma phase

A.6

BNWL-865

TABLE A. III. Cold Breakdown Forging of Electron-Beam-Melted Pure Technetium

<u>Pass No.</u>	<u>Thickness, in.</u>	<u>Load, tons</u>	<u>Remarks</u>
0	0.318	-	
1	0.275	-	Load did not register
2	0.262	3	
3	0.255	5	
4	0.247	5	1500 °C, 10 min anneal after this pass
5	0.227	6	
6	0.213	8	
7	0.206	10	1500 °C, 10 min anneal after this pass
8	0.182	10	Appeared to be very ductile. Numerous edge passes were made to square the work. 1500 °C, 10 min anneal after this pass.
9	0.169		Draw forging on this and the remaining passes.
10	0.147		
11	--		About one third of sheet broke off. Pass was stopped. 1500 °C anneal, 10 min
12	0.126		Specimen moved to cold rolling mill.

TABLE A. IV. Intermediate Cold-Rolling of Electron-Beam-Melted Pure Technetium

<u>Pass No.</u>	<u>Down Set, in.</u>	<u>Thickness, in.</u>	<u>Reduction, %</u>	<u>Hardness</u>	
				<u>Rockwell^(a)</u>	<u>BHN^(b)</u>
1	0.004	0.120			
2	0.010	0.113			
3	0.010	0.104		52 (N)	301
	1500 °C, 10 min anneal			61.5 (T)	106
4		0.098	5.8	78 (T)	190
5		0.091	12.5	47 (N)	259
6		0.088	15.4	48.5 (N)	272
7		0.083	20.2	53 (N)	309
8		0.079	24.0	56.1 (N)	337
9		0.076	26.9	54.5 (N)	323
10		0.073	29.8	58 (N)	362
11		0.070	32.7	59 (N)	372

Cracked from edge across one end, cold-rolling stopped.

(a) Superficial scales 30 N and 30 T

(b) Converted BHN

TABLE A. V. Final Cold-Rolling of Electron-Beam-Melted Pure Technetium

Pass No.	Thickness, in.	Reduction, %	Hardness	
			Rockwell ^(a)	BHN ^(b)
0	0.070	0	62 (T)	108
1	0.068	2.85	44 (N)	241
2	0.066	5.70	46 (N)	253
3	0.065	7.10	50.7 (N)	291
4	0.062	11.40	52.5 (N)	305
5	0.059	15.70	54.7 (N)	324
6	0.054	18.50	57.0 (N)	352
7	0.053	24.30	58.2 (N)	364
8	0.052	25.70	59.0 (N)	372
9	0.050	28.50	62.5 (N)	413
10	0.047	32.8	62.8 (N)	416
11	0.045	35.7	63.2 (N)	421
12	0.042	40.0	63.5 (N)	424
13	0.040	42.8	65.5 (N)	447
14	0.037	47.1	66.2 (N)	457
15	0.036	48.5	65.5 (N)	447
16	0.035	50.0	65.5 (N)	447
17	0.033	52.8	66.2 (N)	457
18	0.030	57.1	68.5 (N)	488
19	0.029	58.5	67.5 (N)	476

(a) Superficial scales 30 N and 30 T

(b) Converted BHN

TABLE A. VI. Final Recrystallization of Electron-Beam-Melted Pure Technetium

Annealing Temperature, °C	Hardness		
	Rockwell ^(a)	BHN ^(b)	DPH ^(c)
Room 58.59% coldwork	67.5 (N)	476	402
307	63.6 (N)	425	414
503	57.2 (N)	354	323
602	39.3 (N)	200	229
701	64.0 (T)	115	164
803	55.4 (T)	92.5	124
900	53.7 (T)	88	133

(a) Superficial scales 30 N and 30 T

(b) Converted BHN

(c) 1 kg load 136°

TABLE A. VII. Hot Breakdown Rolling of Arc-Melted Pure Technetium

Pass No.	Down Set, in.	Thickness, in.	Temperature, °C	Remarks
0		0.145		
1	0.010	0.145	1507	Did not touch
2	0.010	0.129	1511	
3	0.010	0.116	1507	
4	0.010	0.106	1513	
5	0.010	0.097	1515	
6	0.010	0.086	1511	
7	0.010	0.078	1505	1500 °C, 10 min anneal after last pass.

TABLE A. VIII. Intermediate Cold-Rolling of Arc-Melted
Pure Technetium

Pass No.	Thickness, in.	Reduction, %	Hardness	
			Rockwell ^(a)	BHN ^(b)
0	0.078	0	67 (T)	125
1	0.076	2.56	78.5 (T)	193
2	0.073	6.4	48 (N)	269
3	0.072	7.7	51.5 (N)	294
4	0.070	10.3	51 (N)	290
5	0.068	12.8	55 (N)	327

Rolling was stopped because of surface cracking which was more severe near the ends.

TABLE A. IX. Final Cold-Rolling of Arc-Melted
Pure Technetium

Pass No.	Thickness, in.	Reduction, %	Hardness	
			Rockwell ^(a)	BHN ^(b)
0	0.070	0	68 (T)	131
1	0.068			
2	0.068			
3	0.064	5.9	46.4 (N)	256
4	0.062	8.8	52.0 (N)	301
5	0.061	10.3	54.0 (N)	318
6	0.059	13.2	54.5 (N)	323
7	0.058	14.7	56.0 (N)	337
8	0.056	17.6	57.7 (N)	362
9	0.053	22.1	58.5 (N)	367
10	0.051	25.0	58.7 (N)	374
11	0.050	26.4	60.0 (N)	382
12	0.047	30.8	61.0 (N)	392
13	0.045	33.8	64.0 (N)	430
14	0.043	36.7	63.6 (N)	425
15	0.041	39.4	63.5 (N)	424
16	0.039	42.2	64.5 (N)	436
17	0.037	45.6	65.0 (N)	442

(a) Superficial scales 30 N and 30 T

(b) Converted BHN

TABLE A.X. Final Recrystallization of Arc-Melted
Pure Technetium

Annealing Temperature, °C	Hardness		
	Rockwell ^(a)	BHN ^(b)	DPH ^(c)
Room 45.6 % coldworked	65 (N)	442	394
601	57.6 (N)	358	319
700	52.5 (N)	305	279
801	40.6 (N)	210	218
839	73.0 (T)	152	209
878	69.9 (T)	139	177
952	63.1 (T)	112	151
1099	61.5 (T)	106	164

(a) Superficial scales 30 N and 30 T

(b) Converted BHN

(c) 1 kg load 136°

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