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QUARTERLY PROGRESS REPORT
A STUDY OF TUNGSTEN-TECHNETIUM ALLOYS
APRIL 1, 1965 - JULY 1, 1965

By

THE STAFF OF METALLURGY DEVELOPMENT SECTION
REACTOR AND MATERIALS TECHNOLOGY DEPARTMENT

Sponsored by the National Aeronautics and Space Administration
Project Management at Washington, D. C.
J. W. MALTZ, Office of Advanced Research and Technology

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INTRODUCTION

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

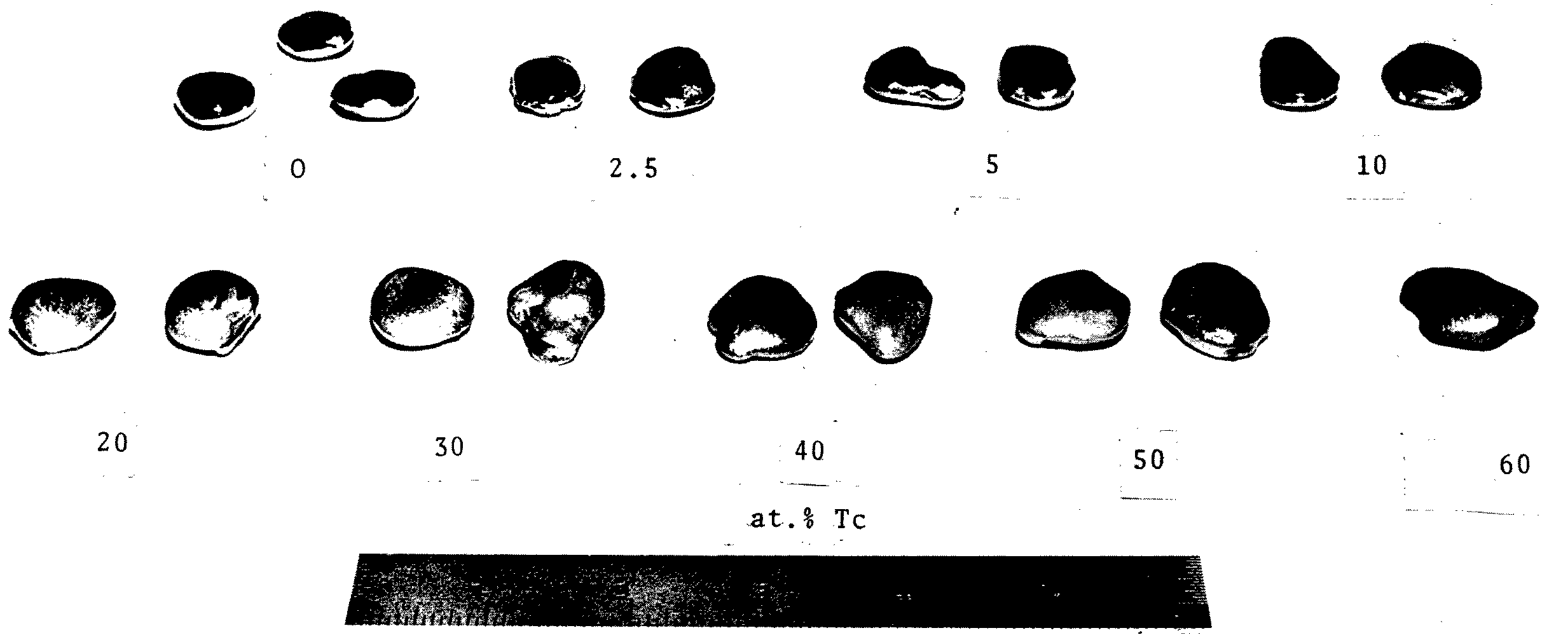
The objectives of the current work are to recover technetium from fission product wastes at Hanford Atomic Products Operation and reduce to purified metal; prepare W-Tc alloys containing up to 50 at.% Tc; fabricate the alloy ingots to sheet stock, assessing the effect of technetium on workability; and 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, melting of technetium and W-Tc alloys, and some initial observations of the alloy material.

CURRENT PROGRESS

During the past quarter the microstructure of a series of W-Tc alloys in the arc melted condition was observed, microhardness determined, and identification of phases present by X-ray diffraction completed.

One of the flatter surfaces of each button (Figure 1) was polished for these studies after mounting in bakelite. The grinding procedure was done entirely with a Buehler automet polisher. Water was used as the liquid vehicle for it was found to assist



W-Tc Alloys

FIGURE 1
Arc Melted Alloy Buttons

in the cleanup of the radioactive powder better than any other common liquid. Adhesive backed abrasive papers were used in the grades of 120, 320, 400, and 600 grit silicon carbide. Both the hardness and work hardening properties of the alloys made grinding difficult and several discs of paper in each grade were necessary for proper preparation.

Rough polishing followed the attainment of a good 600 grit finish. This was done in a vibratory polisher (Syntron) with a 12 in. bowl, using Metcloth, Linde A abrasive, and water. For pure technetium a solution of 2 wt% CrO_3 in water was found to work better as the liquid vehicle. The next step in polishing was also on the vibratory polisher using Microcloth, Linde A abrasive, and a mixture (4:1) of 2 wt% $Na_2Cr_2O_7$ and 2 wt% CrO_3, water solution. The material retained a layer of worked metal at this stage that required two additional steps to remove. The first of these used an acid polish or polish-attack on an 8 inch hand wheel. Linde A abrasive on Microcloth was used with an acid mixture of one part stock solution to nine parts water. The stock solution was: 500 cm^3 water, 100 g CrO_3, 70 cm^3 orthophosphoric acid, and 5 cm^3 H_2SO_4 acid. A chemical polish consisting of vigorous swabbing with a mixture of 45 parts HNO_3 acid, 45 parts water, and 7 to 10 parts concentrated HF was used to remove the final traces of scratches. Etchants used varied with alloy composition, but generally were as follows:

- Technetium - 10wt% $H_2C_2O_4$ - electrolytic 6 V for 2 to 5 sec.
- W to W-40 at.% Tc - Murakami's swab 2 sec.
- W-50 to 60 at.% Tc - 1 wt% NaOH electrolytic 3 V for 10 sec.

The structure of the as-cast alloys is shown in Figures 2 through 7. The alloys through 10 at.% Tc are equiaxed single phase. As noted in Figure 3, the material containing 20 to 40 at.% Tc is dendritic and severely cored due to the rapid cooling rate during freezing following arc melting. These compositions

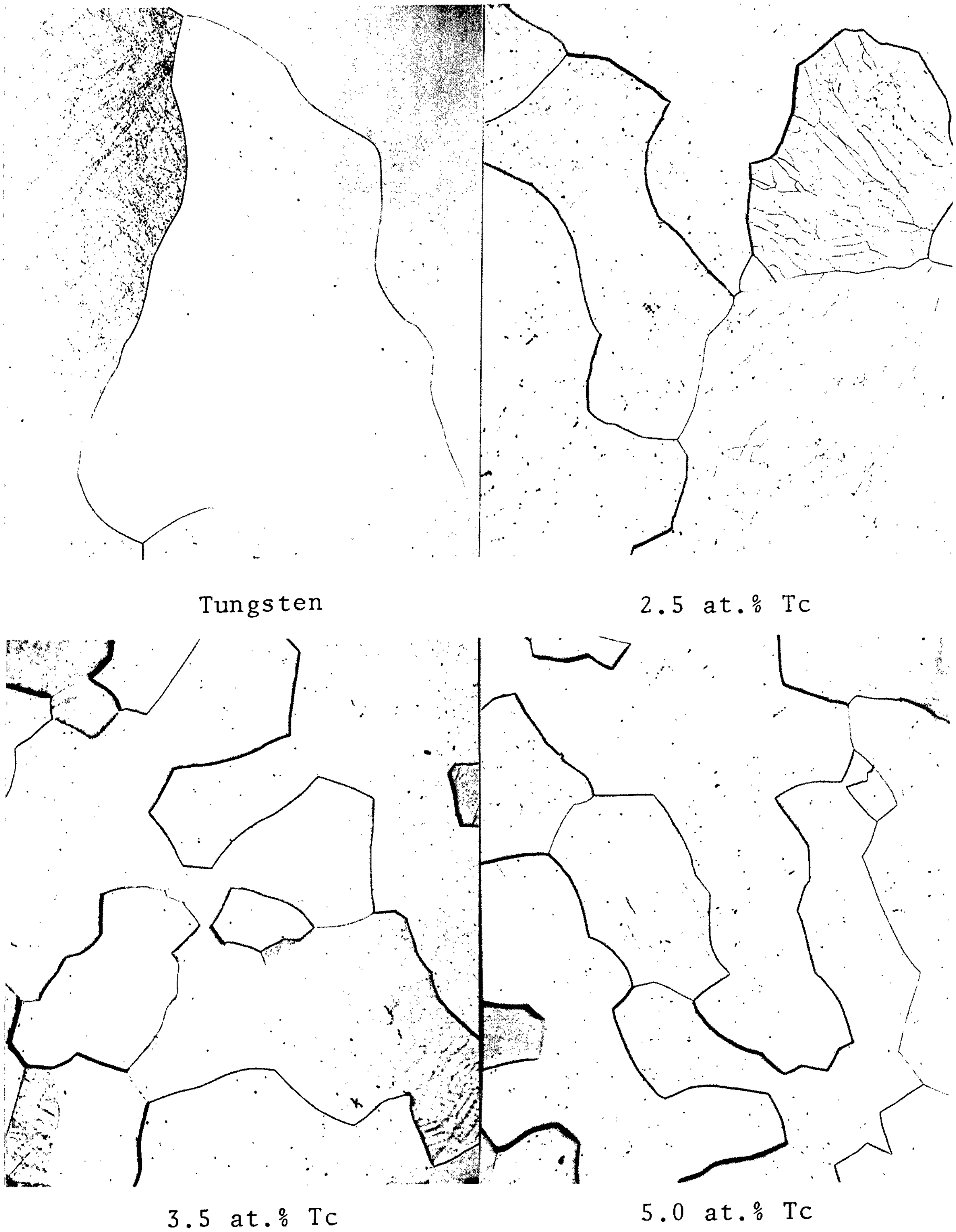
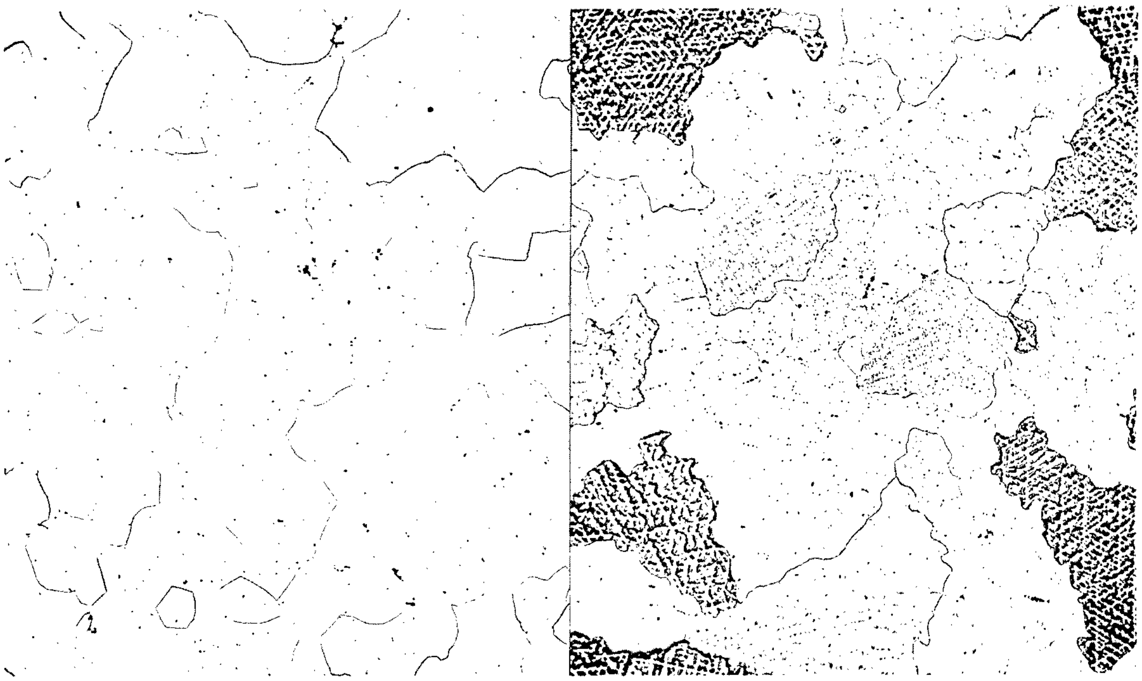
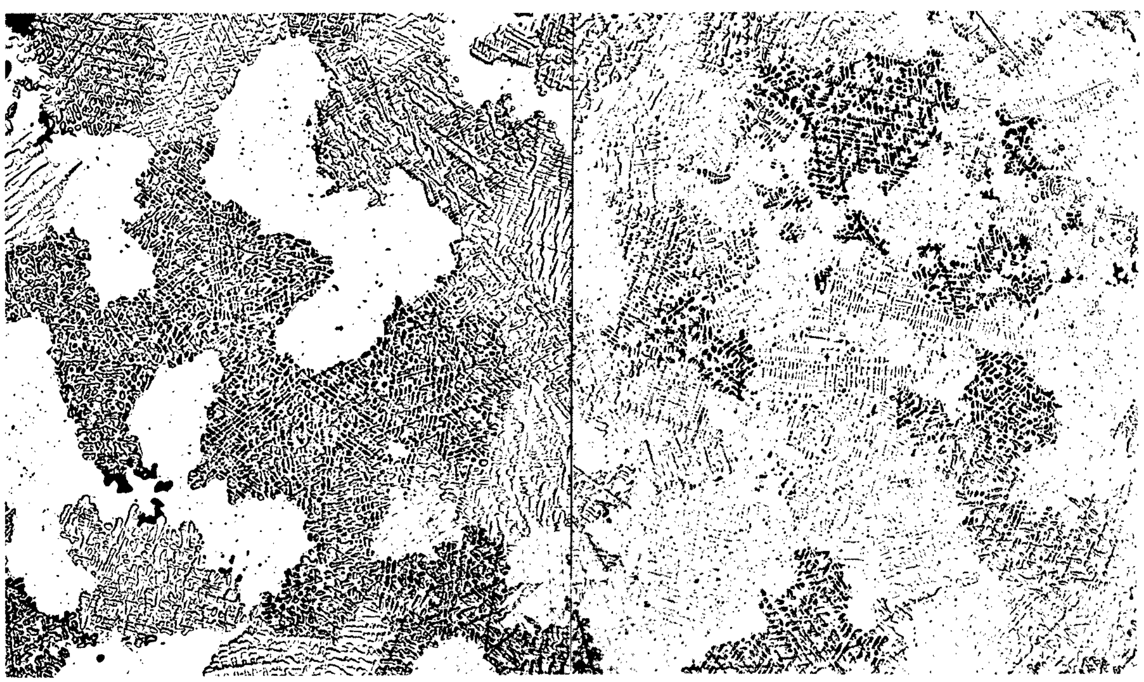


FIGURE 2

Microstructure of W-Tc Alloys.
Single Phase Solid Solution.
50X



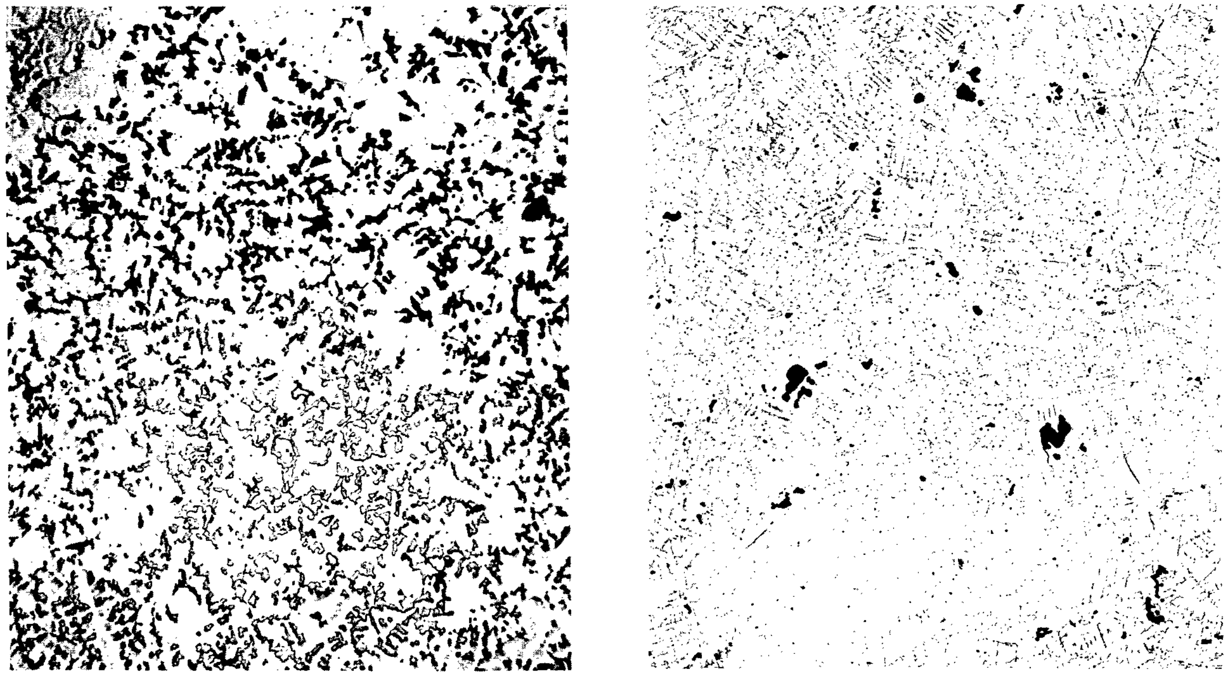
10 at.% Tc 20 at.% Tc



30 at.% Tc 40 at.% Tc

FIGURE 3

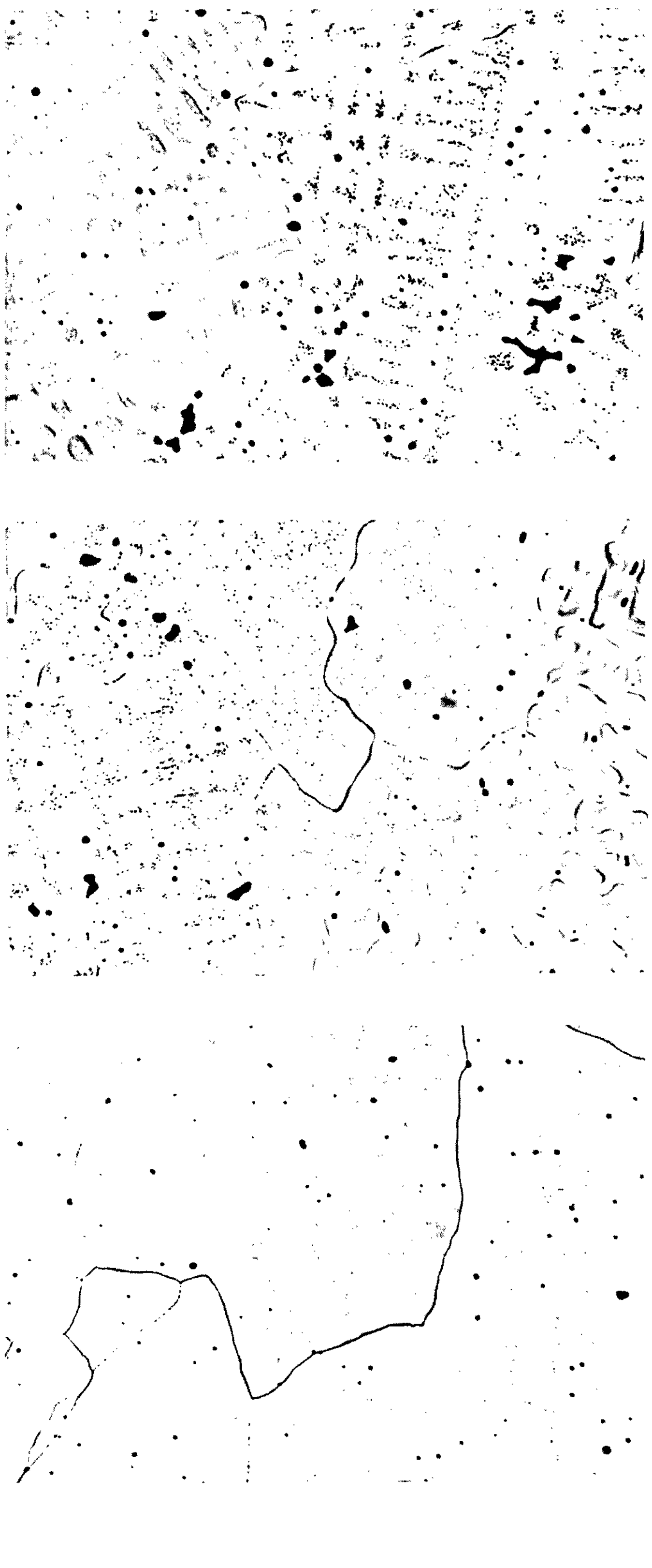
Microstructure of W-Tc Alloys.
Cored Single Phase Solid Solution.
50X



50 at.% Tc   60 at.% Tc

FIGURE 4

Microstructure of W-Tc Alloys.
Alpha Plus Sigma Structure.
50X



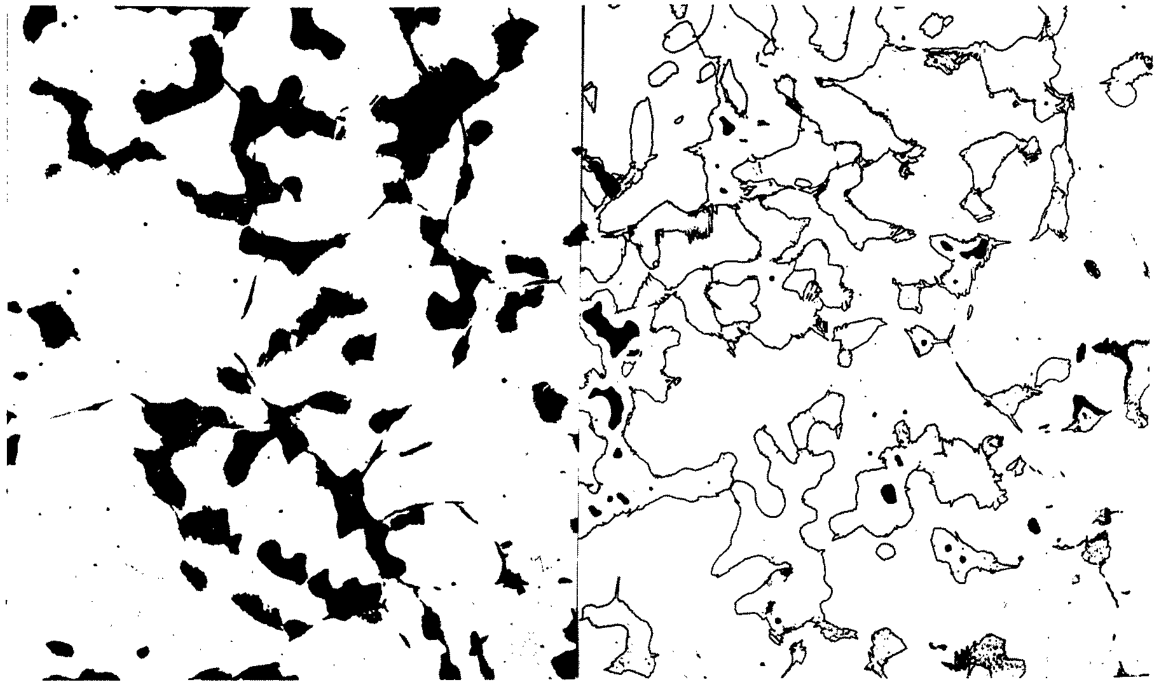
40 at.% Tc

30 at.% Tc

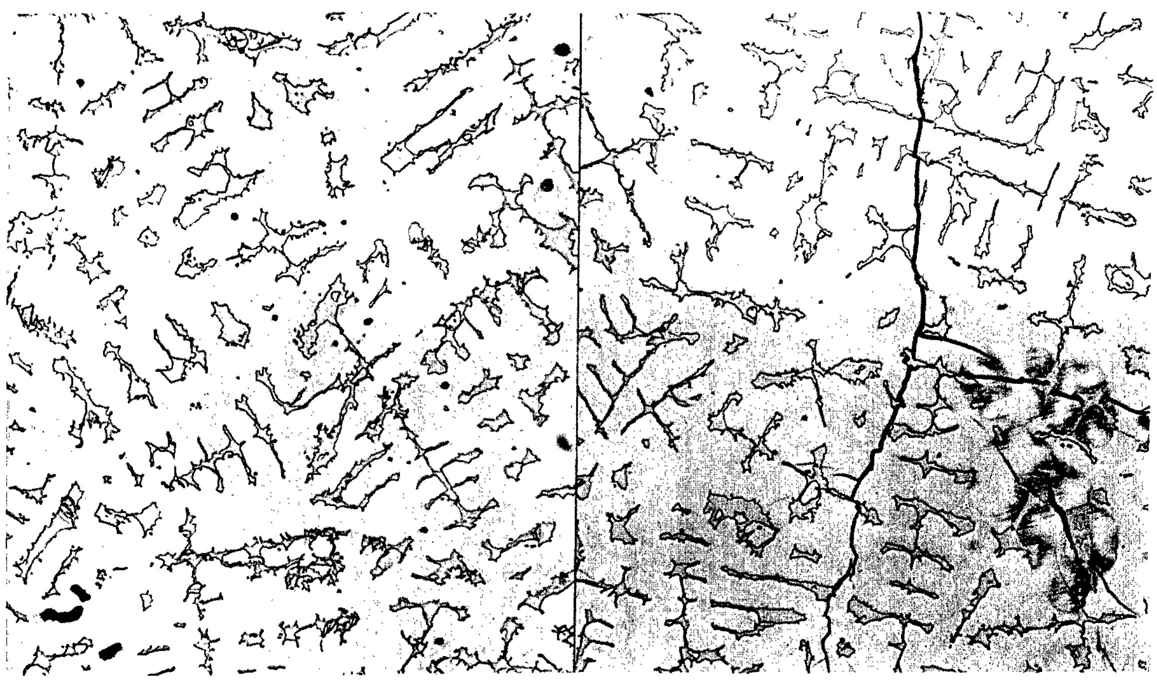
20 at.% Tc

FIGURE 5

Microstructure of Cored Single Phase Alloys
Showing Interdendritic Porosity
250X



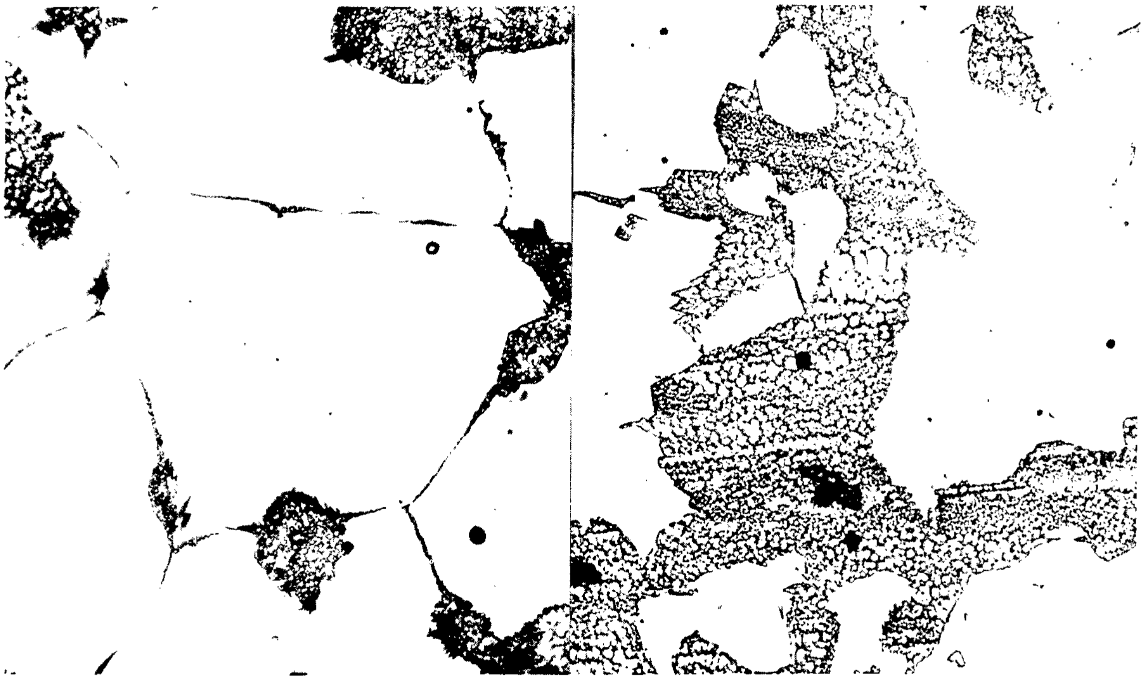
50 at.% Tc



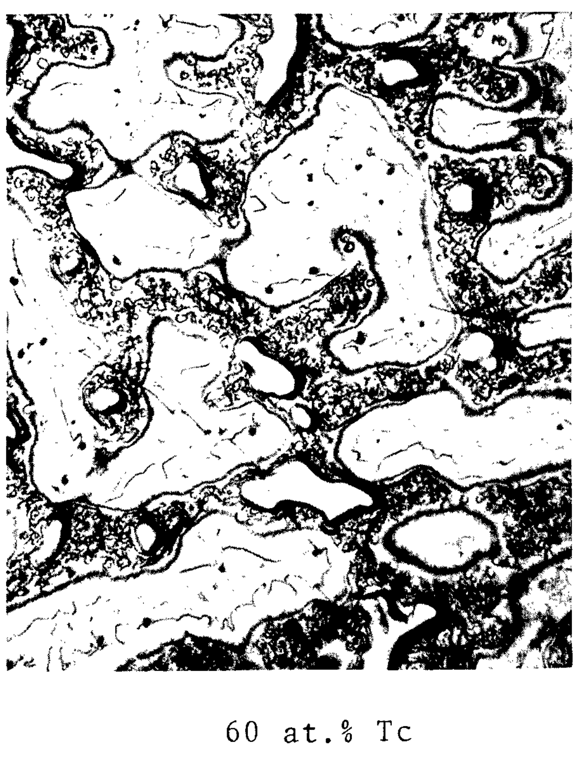
60 at.% Tc

FIGURE 6

Microstructure of Two Phase Alloys.
Note Brittle Failure in Sigma Phase of 60 at.% Alloy.



50 at.% Tc



60 at.% Tc

FIGURE 7

Microstructure of Two Phase Alloys.
Note Crack in Matrix Phase of 60 at.% Alloy.
750X

were still single phase solid solution and as shown in Figure 5, are subject to interdendritic porosity, primarily due to shrinkage. The 50and 60 at.% alloys appeared two phase in structure, the 50 at.% having a matrix of alpha solid solution. The last region to freeze in this alloy contains some porosity, as shown in Figure 6 and is intergranular. Some structure is evident in this region, shown in Figure 7 at higher magnification. The 60 at.% alloy is also two phase with the matrix being hard and brittle and having a structure similar to the minor phase of the 50 at.% alloy.

Microhardness data obtained are shown in Figure 8. An initial hardness decrease was observed with Tc additions as was noted with Rockwell hardness measurements. Increasing technetium content resulted in solid solution hardening to 20 at.% Tc. No twinning was observed around hardness indents to 10 at.% Tc but increased twinning deformation with technetium content from 20 to 40 at.% Tc is shown in Figure 9 that results in the hardness decrease in this range. The two phases present in the 50 at.% alloy had markedly different hardnesses and only the matrix deformed by twinning. The major phase of the 60 at.% alloy is extremely hard and brittle as shown by the cracking around the hardness indents illustrated in Figure 10. Note that the dendritic alpha phase present in this alloy is not cracked. The same evidence is shown in Figure 7 where a quenching crack is observed to stop at the boundaries of the alpha phase.

Identification of phases present and their lattice parameters was made on these polished surfaces. These results are given in Table I and Figure 11. These data indicate that under these nonequilibrium conditions, approximately 48 at.% Tc is taken in the bcc tungsten solid solution. The 50 at.% alloy had a slight indication of a second phase and the 60 at.% alloy was predominantly the tetragonal sigma phase. In Figure 11 the lattice parameters determined by Niemiec[1] for alloys quenched from 1800 °C, is also shown. The excellent correlation indicates that the buttons were effectively quenched from approximately 1800 °C during cooling on the water cooled hearth. The structure of the second phase of the 50 at.% alloy and of the matrix phase of the 60 at.% alloy have some of the appearance of a eutectic reaction between the alpha and sigma phases but no definite conclusions can be drawn from these nonequilibrium structures. Attempts will be made to further define these structures by electron microprobe analysis.

Several melts of pure tungsten and of a W-25 at.% Re alloy were made in the electron beam evaporator unit in preparation for remelting the W-Tc alloys.

LITERATURE REVIEW

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2. J. B. Darby and S. T. Zegler. "Comments on Superconducting Phases in the Mo-Tc System," International Journal of the Physics and Chemistry of Solids, vol. 23, pp. 1826-1828. December 1962.

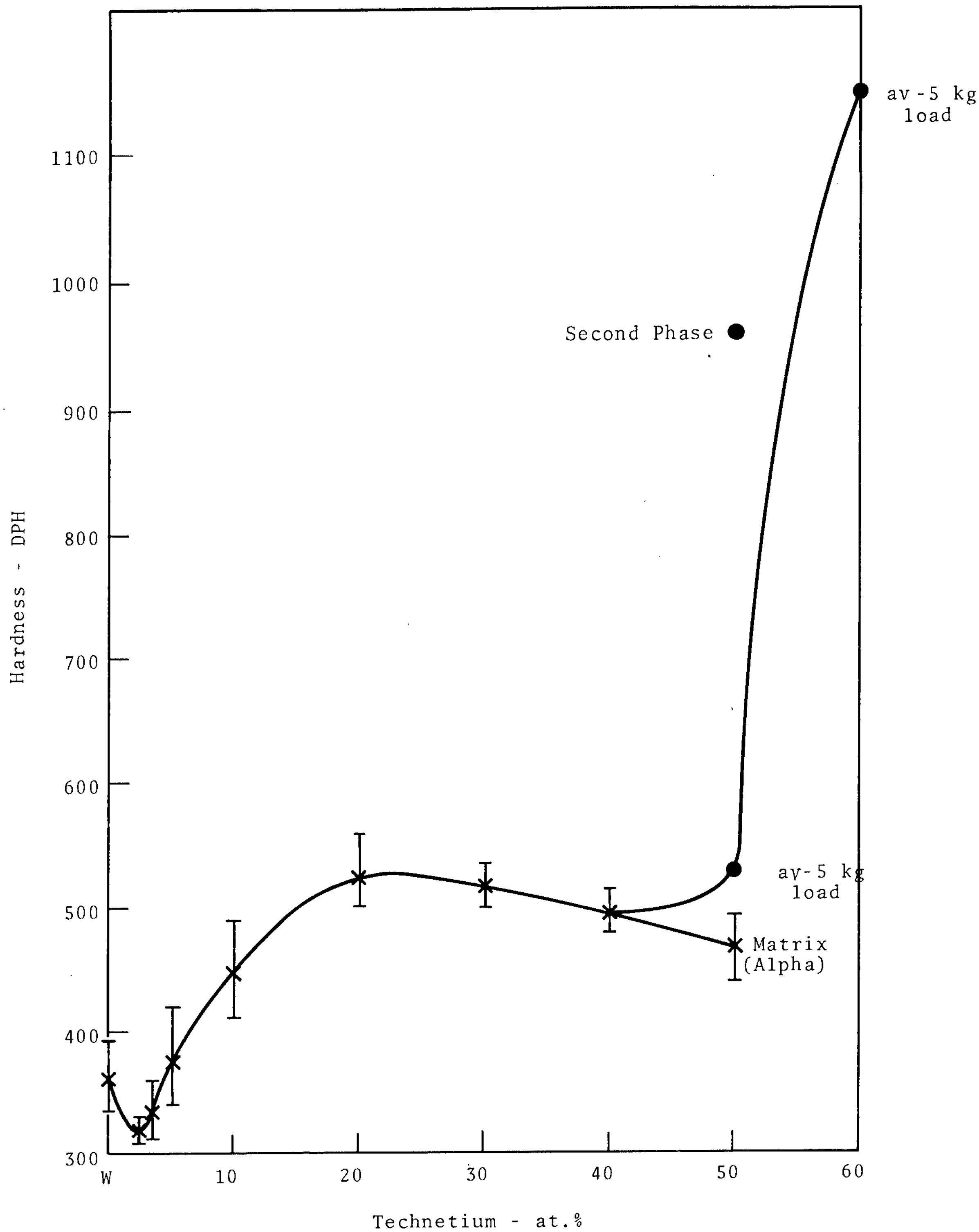
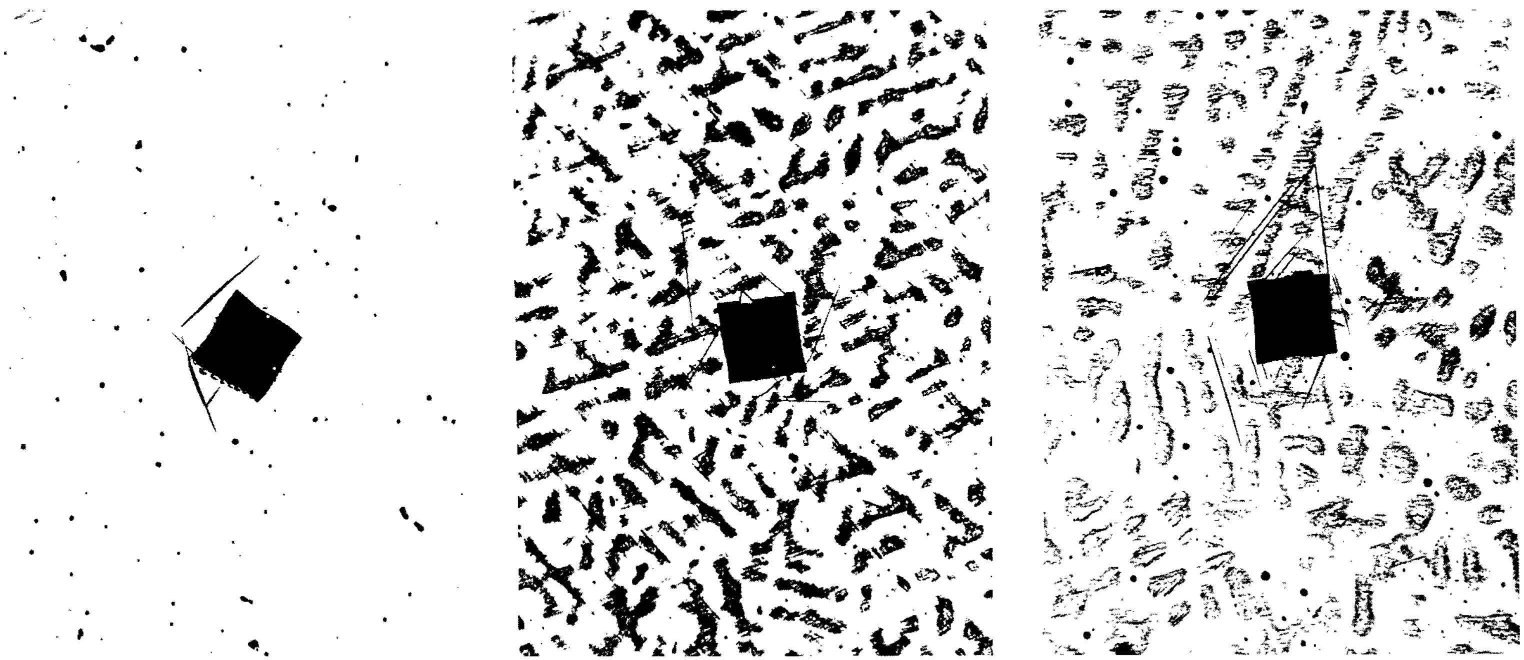


FIGURE 8

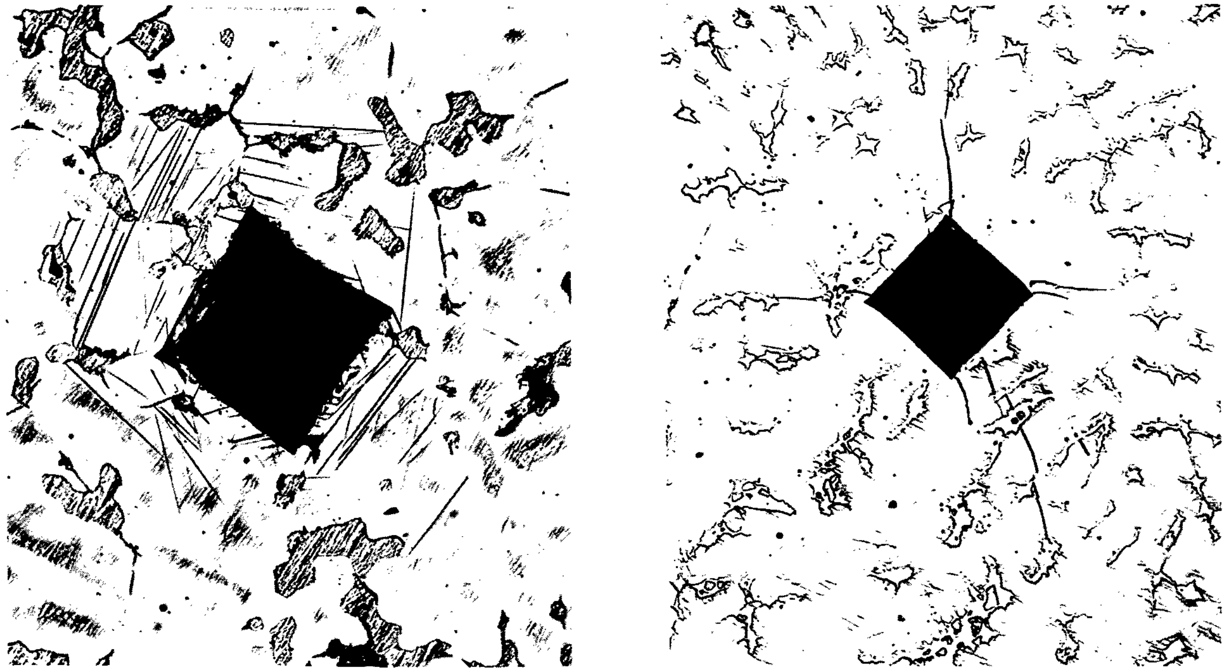
Microhardness of Tungsten - Technetium Alloys.



20 at.% Tc   30 at.% Tc   40 at.% Tc

FIGURE 9

Microhardness Indents in Solid Solution Alloys.
Note Increase in Twinning.
(1 kg load)
250X



50 at.% Tc   60 at.% Tc

FIGURE 10

Microhardness Indents in Two Phase Alloys.
Note Twinning in Solid Solution of 50 at.% Alloy
and Cracking in Sigma Phase of 60 at.% Alloy.
250X

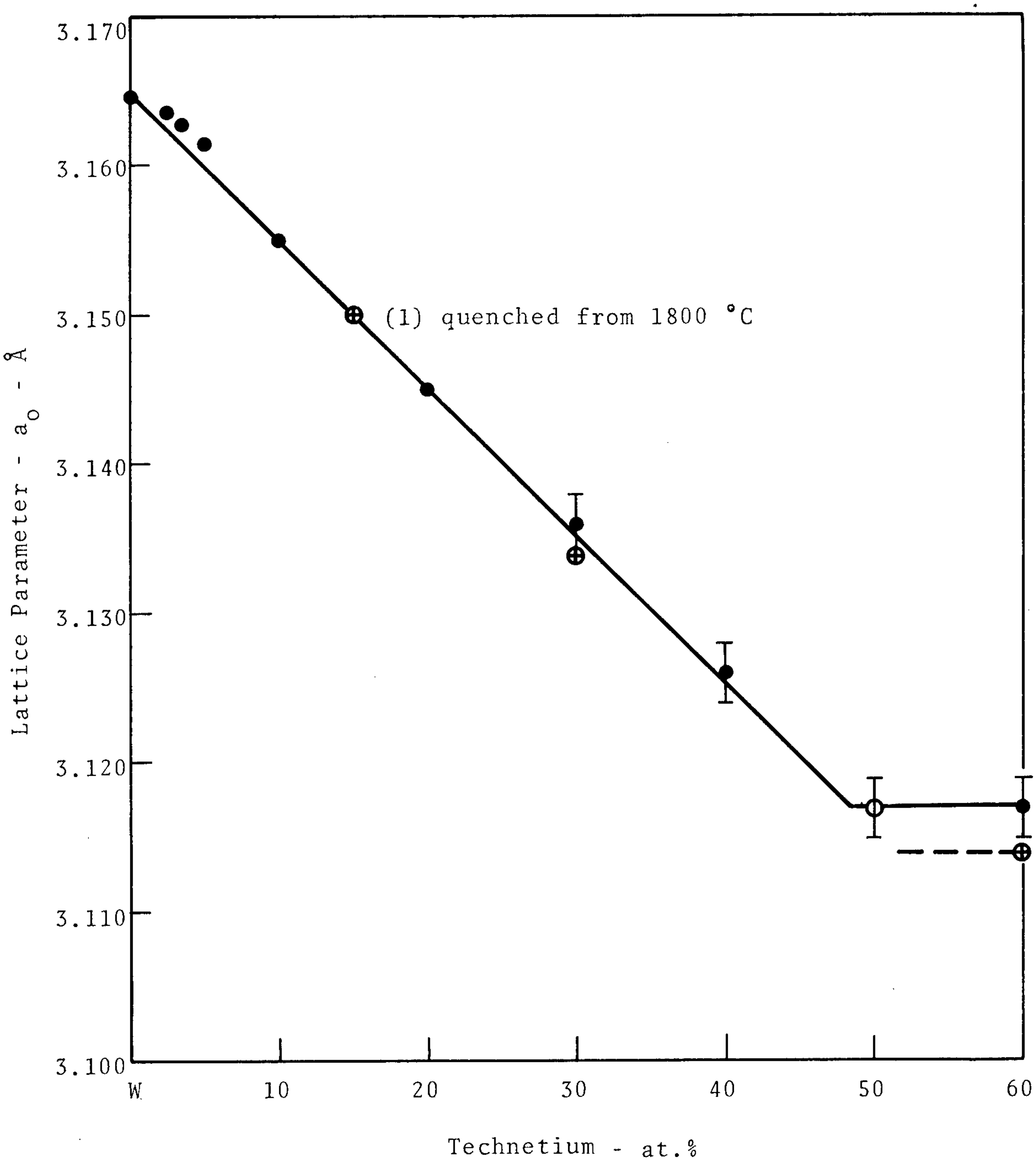


FIGURE 11

Variation of the Lattice Parameter of the Body Centered Cubic Solid Solution of Tungsten with Technetium Content

TABLE I

LATTICE PARAMETERS FOR PHASES IN THE W-Tc SYSTEM

Composition, at.% Tc	Phase	Lattice Parameters, Å
Tungsten	Alpha Solid Solution (body centered cubic)	a_o = 3.1649 ± 0.0001
2.5	Alpha Solid Solution (body centered cubic)	a_o = 3.1635 ± 0.0001
3.5	Alpha Solid Solution (body centered cubic)	a_o = 3.1628 ± 0.0001
5.0	Alpha Solid Solution (body centered cubic)	a_o = 3.1617 ± 0.0001
10.0	Alpha Solid Solution (body centered cubic)	a_o = 3.1553 ± 0.0002
20.0	Alpha Solid Solution (body centered cubic)	a_o = 3.147 ± 0.002
30.0	Alpha Solid Solution (body centered cubic)	a_o = 3.134 ± 0.002
40.0	Alpha Solid Solution (body centered cubic)	a_o = 3.126 ± 0.002
50.0	Alpha	a_o = 3.117 ± 0.002
	Sigma	- - -
60.0	Alpha	a_o = 3.117 ± 0.002
	Sigma (Tetragonal)	a_o = 9.520 ± 0.001
		c_o = 5.003 ± 0.001
		c/a = 0.525

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