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PHASE EQUILIBRIA OF THE TANTALUM-RICH REGION OF THE
TANTALUM-TUNGSTEN-HAFNIUM AND TANTALUM-
TUNGSTEN-ZIRCONIUM SYSTEMS

- Submitted to -

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ABSTRACT

The solid-state phase equilibria at 1600C(2912F), 1300C(2372F), and 1100C(2012F) are being determined for the tantalum-rich regions of the Ta-W-Hf and Ta-W-Zr alloy systems. Alloys for the investigation are prepared by arc melting charges consisting of a 60 w/o Ta-40 w/o W master alloy with appropriate additions of tantalum and hafnium or tantalum and zirconium. The as-cast alloys are homogenized by means of a high-temperature heat treatment in vacuo prior to the vacuum annealing treatments at the three isothermal levels. Metallographic examination of the annealed structures and X-ray diffraction studies of the alloys are the principal techniques employed in the investigation to determine the phase equilibria. The progress report summarizes the results of the metallographic examination of as-cast alloy structures and presents a discussion of the high-temperature vacuum annealing furnace and the annealing procedures used.

I. INTRODUCTION

This report is the second quarterly report describing the progress achieved on the research program, "Phase Equilibria of the Tantalum-Rich Region of the Tantalum-Tungsten-Hafnium and Tantalum-Tungsten-Zirconium Systems." The research program is sponsored by the United States Atomic Energy Commission, Contract No. AT(11-1)-1092-Task F. The research period covered by this report is from 1 December 1964 to 28 February 1965.

The objective of the research program is to establish the phase relationships in the tantalum-tungsten-hafnium and tantalum-tungsten-zirconium systems at three isothermal levels: namely, 1100C(2012F), 1300C(2372F), and 1600C(2912F). The accurate determination of the solid-solution boundary for the tantalum-rich region in each system will be the primary goal of the investigation. Other phase regions which may be located within the compositional area of interest will also be delineated and all phases in equilibrium with the tantalum-rich solid solution will be identified. Alloys which will be included in the investigation are in the composition range from 100 percent tantalum up to a maximum of 25 weight percent each of tungsten and hafnium or tungsten and zirconium.

Alloys are prepared by arc melting weighed charges of a master alloy (60 w/o Ta-40 w/o W) with additions of tantalum and hafnium or tantalum and zirconium in a non-consumable electrode arc furnace under an inert atmosphere. The as-cast alloys are homogenized in a tantalum-element, high temperature vacuum furnace prior to the isothermal anneals. Isothermal annealing treatments will be conducted in this same furnace for the 1300C(2372F) and 1600C(2912F) levels and in a nichrome-wire-wound resistance tube furnace for the 1100C(2012F) temperature level. Interpretations of the microstructures of the heat treated alloys will be made utilizing standard metallographic techniques and X-ray diffraction studies.

II. EXPERIMENTAL PROCEDURES

A. Alloy Preparation

All alloys for the first series of compositions in both the tantalum-tungsten-hafnium and tantalum-tungsten-zirconium systems were prepared by arc melting. Table I lists the alloy compositions from each system which have been made. Metallographic examination of the as-cast structures showed that incomplete melting resulted even after twelve melting cycles (the button ingot was inverted after each melting and solidification). Consequently, sixteen melting cycles were employed for the complete melting of each alloy. Weight loss data of the button ingot after the melting operation were used for compositional control purposes. No excessive weight losses were noted. Selected alloy specimens will be submitted for chemical analysis to provide compositional data with which to correlate the weight loss data.

B. Metallographic Examination

Each alloy button ingot was sectioned and a portion of the ingot was mounted in bakelite for metallographic examination. The mounted samples were ground on 80, 120, 400, and 600-grit abrasive rotating discs and then polished on rotating laps. Although a completely satisfactory metallographic procedure has not yet been developed, several alternative techniques currently are under investigation, including an electro-mechanical polishing operation to reduce or eliminate the flowed-metal conditions generally experienced with high-tantalum alloys.

The alloy structures were examined to determine if the alloys were completely melted. The amount and distribution of the phases present in the structures were also determined. It is apparent from the as-cast structure that the solid solubility of either zirconium or hafnium in the tantalum-rich ternary alloy is restricted to about five weight percent.

C. Homogenization Annealing

A tantalum-tube, resistance-element vacuum furnace which is capable of attaining temperatures in excess of 2000C(3632F) and an ultimate vacuum of 3×10^{-6} Torr at temperature is being used for the homogenization annealing treatments. Prior to the start of homogenization treatments incipient melting studies were conducted on two alloys

TABLE I

Alloy Compositions Prepared

Ta - W - Hf System			Ta - W - Zr System		
<u>% Ta</u>	<u>% W</u>	<u>% Hf</u>	<u>% Ta</u>	<u>% W</u>	<u>% Zr</u>
90	5	5	95	-	5
85	10	5	90	5	5
80	15	5	85	10	5
75	20	5	80	15	5
85	5	10	75	20	5
80	10	10	85	5	10
75	15	10	80	10	10
70	20	10	75	15	10
80	5	15	80	5	15
75	10	15	75	10	15
70	15	15	70	15	15
65	20	15	75	5	20
75	5	20	70	10	20
70	10	20	65	15	20
65	15	20	70	5	25
65	10	25	65	10	25
55	20	25	60	15	25
			95	5	-
			90	10	-
			85	15	-

from the tantalum-tungsten-zirconium system and three alloys from the tantalum-tungsten-hafnium system. As a result of these data, the following temperatures were chosen for the homogenization anneals:

<u>Ta-W-Zr Alloys</u>	<u>Temp. C</u>	<u>Ta-W-Hf Alloys</u>	<u>Temp. C</u>
Alloys containing < 15% Zr	1900	All Compositions	2200
Alloys containing > 15% Zr	1800		

Homogenization of the tantalum-tungsten-zirconium alloys has been completed (1 hour at temperature) and the homogenized buttons are being sectioned into several small pieces of a size suitable for the isothermal annealing studies. None of the ternary alloys has shown sufficient ductility to allow any significant cold reduction either prior to or subsequent to the homogenization anneal. Consequently, the alloys will be isothermally annealed without any attempt to introduce cold-work energy into the structure.

III. FUTURE WORK

Additional alloy compositions are being prepared in both ternary systems at 1.0 weight percent compositional increments in the region from 5 to 10 weight percent hafnium (or zirconium) and at 2.5 weight percent increments in the region from 2.5 to 22.5 weight percent tungsten. These alloys will be homogenized and prepared for isothermal annealing treatments according to the same procedures which were described earlier.

Alloy specimens will be prepared for the isothermal annealing treatments at 1100C (2012F) by wrapping the specimens individually in tantalum foil and then sealing the wrapped samples in a quartz capsule contained within another quartz capsule. The double capsule will provide the necessary strength for the containment of the samples for the long times at the elevated temperature. Both capsules will be filled with a partial pressure of purified argon to prevent collapse of the capsule walls during the annealing treatment.

The heat treatments will be done in a nichrome wire-wound Marshall tube furnace with temperature controlled by a Symplotrol controller. At the termination of the heat treatment period, the capsules will be dropped from the hot zone of the furnace and broken under water to quench the samples.

Isothermal anneals at the 1300C (2372F) and 1600C (2912F) temperature levels will be conducted in the high-temperature tantalum-resistance element tube furnace in high vacuum. Temperatures will be checked with an optical pyrometer.

As-cast and isothermally annealed alloy compositions will be sampled for the purpose of conducting X-ray diffraction studies. Identification of phases and lattice parameter determinations will be made.

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