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Quarterly Progress Report No. 5

Contract No. AT(11-1)-1092-Task B

December 1965

STUDIES OF THE EFFECT OF INTERSTITIAL ADDITION
ON THE PHASE EQUILIBRIA OF THE NIOBIUM-RICH REGION
OF THE NIOBIUM-TUNGSTEN-HAFNIUM
AND NIOBIUM-TUNGSTEN-ZIRCONIUM ALLOY SYSTEMS

RELEASED FOR ANNOUNCEMENT
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I. INTRODUCTION

This report is the fifth quarterly report describing the research activities at the Metallurgy Division of the Denver Research Institute of the program sponsored by the United States Atomic Energy Commission under Contract No. AT(11-1)-1092-Task B. The period covered by this report is from 1 September 1965 to 31 November 1965. The objective of this sponsored research is to study the effect of interstitial addition on the phase equilibria of the niobium-rich region of the niobium-tungsten-hafnium and niobium-tungsten-zirconium alloy systems. Specifically the interstitial elements under investigation are carbon and oxygen. This is a continuation of the program, "Determination of the Niobium-Rich Region of the Ternary Phase Diagrams, Niobium-Tungsten-Hafnium and Niobium-Tungsten-Zirconium" which was completed in August 1964.

The principal technique to be used in alloy-structure evaluation will be metallographic analysis. X-ray diffraction analysis will assist in identification of intermediate phases. Microhardness measurement will be made also. Two isothermal sections at 1600 and 1300 C (2912 and 2372 F) in the niobium-rich region of each alloy system will be studied at the interstitial levels of approximately 0.25, 0.1 and 0.05 w/o. The newly determined niobium-rich solid solution boundaries will be compared with the respective boundaries of the ternary alloy systems without addition of carbon and oxygen.

II. RESULTS AND DISCUSSION

A. Niobium-Tungsten-Zirconium Alloys with Carbon Additions

A number of the niobium-tungsten-zirconium alloys containing various amounts of carbon was subjected to X-ray diffraction analysis. The phases identified in the annealed alloys by X-ray powder technique are tabulated in Table I with the lattice parameters determined for the niobium-rich solid-solution phase and microhardness values.

In the alloys containing 25 w/o Zr, regardless of the tungsten content, (0, 1 or 25 w/o) the presence of α -Zr phase is tentatively identified because of the best fitting of a set of d-values with the reported values for the particular phase. These lines, however, may be accounted for by the Nb₂C phase also. It is felt that further study is necessary to make positive identification of the phase in question. On the other hand the above observation seems to be valid. Marcotte, Larsen and Williams¹ investigated the effect of oxygen on the phase equilibria of Nb-20/25 a/o Zr alloys in the temperature range of 1000 to 1200 C (1832 to 2192 F). With the addition of oxygen as low as 0.21 w/o (1.18 a/o), these alloys contained α -Zr. It appears that the effect of small carbon additions on the phase relationships of these alloys is parallel to that of oxygen.

B. Niobium-Tungsten-Hafnium Alloys with Oxygen Additions

The niobium-rich solid solution in the niobium-tungsten-hafnium alloy system became restricted by the addition of small amounts of oxygen. The preliminary results show that at 0.05 w/o oxygen (nominal) level the phase boundary of the terminal solid solution is located at less than 0.5 w/o W at 25 w/o Hf and at less than 1 w/o Hf at 25 w/o W at 1600 C (2912 F). At 1300 C (2372 F) the solid solution is further limited. Several alloys of this system have been submitted for powder X-ray analysis. The results are not yet available. Additional alloys are being prepared in order to better locate the solid-solution phase boundary.

C. Niobium-Tungsten-Zirconium Alloys with Oxygen Additions

Only cursory data are available for this alloy system. However, it is observed that the microstructures of some alloys annealed at 1600 C

¹ V. C. Marcotte, W. L. Larsen and D. E. Williams, "Phase Relationships of the Oxygen-Containing 20 and 25 Percent Zirconium-Niobium Alloys," J. Less-Common Metals, 7 (1964) 373-382.

(2912 F) suggest the occurrence of a reaction peculiar to this system. A few alloys are being prepared for X-ray diffraction analysis. Preparation of additional alloys for this system is in progress also.

TABLE I
X-Ray Data For Nb-W-Zr Alloys With Various Carbon Additions

Alloys, w/o	Heat Treatment 1. 1600C Anneal 2. 1300C Anneal	Powder X-Ray Data		Microhardness Data, DPH (10 kg Load)
		2nd. Phase Identified	Lattice Parameter a_0 for Nb _{ss} Phase, Å	
Nb-1W-25Zr-0.05C	1	ZrC+ α -Zr	3.344 $\pm$ 0.001	281
	2	--	--	253
Nb-1W-25Zr-0.01C	1	ZrC+ α -Zr	3.331 $\pm$ 0.002	289
	2	ZrC+ α -Zr	3.332 $\pm$ 0.002	260
Nb-5W-5Zr-0.25C	1	ZrC	3.305 $\pm$ 0.009	203
	2	ZrC	3.303 $\pm$ 0.001	173
Nb-5W-5Zr-0.01C	1	--	--	183
	2	ZrC+?	3.303 $\pm$ 0.003	164
Nb-10W-10Zr-0.25C	1	ZrC+?	3.291 $\pm$ 0.005	274
	2	--	--	281
Nb-10W-10Zr-0.01C	1	ZrC+?	3.304 $\pm$ 0.001	225
	2	--	--	228
Nb-15W-15Zr-0.25C	1	ZrC+?	3.295 $\pm$ 0.005	322
	2	--	--	304
Nb-25W-1Zr-0.01C	1	ZrC?	3.280 $\pm$ 0.006	251
	2	--	--	251
Nb-25W-25Zr-0.25C	1	ZrC+ α -Zr+W ₂ Zr	3.285 $\pm$ 0.021	383
	2	--	--	380
Nb-25W-25Zr-0.01C	1	α -Zr+W ₂ Zr	3.291 $\pm$ 0.001	394
	2	--	--	380
Nb-25W-0.25C	1	ZrC	3.278 $\pm$ 0.001	287
	2	ZrC	3.279 $\pm$ 0.005	276
Nb-1Zr-0.01C	1	--	--	113
	2	trace ZrC	3.302 $\pm$ 0.001	111
Nb-5Zr-0.1C	1	ZrC	3.308 $\pm$ 0.002	160
	2	--	--	137
Nb-25Zr-0.1C	1	ZrC+ α -Zr	3.334 $\pm$ 0.010	274
	2	--	--	249
Nb-25Zr-0.05C	1	--	--	283
	2	ZrC+ α -Zr	3.350 $\pm$ 0.004	243
Nb-25Zr-0.01C	1	--	--	276
	2	α -Zr+?	3.336 $\pm$ 0.018	240

III. FUTURE WORK

1. Studies of the effect of small carbon additions on the phase relationships in the niobium-rich region of the niobium-tungsten-hafnium and niobium-tungsten-zirconium systems are nearly completed. It is hoped that a summary report for this phase of the contract will be finished within the next quarterly report period.
2. Additional oxygen bearing alloys both for the niobium-tungsten-hafnium and niobium-tungsten-zirconium alloy systems are being prepared.
3. Isothermal annealing of these alloys will continue.

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