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HIGH-TEMPERATURE CHEMICAL STABILITY OF REFRACTORY-BASE ALLOYS IN HIGH VACUUM

D. T. Bourgette

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D. T. Bourgette

ABSTRACT

Evaporation losses of a tantalum-base alloy (T-111) and five niobium-base alloys (FS-80, FS-85, B-66, C-129Y, and D-43) were determined for various periods of time at temperatures between 1200 and 1500°C. The experimental pressures ranged from 7×10^{-7} to 6×10^{-10} torr.

In general, there were no serious evaporation problems associated with these alloys, especially at temperatures of 1200 to 1300°C. The alloys exhibiting the greatest evaporation losses contained the higher vapor-pressure elements such as V, Zr, Y, and Hf. For example, B-66 (the most volatile alloy studied) lost 1.70 mg/cm^2 at 1500°C and 7×10^{-9} torr in 316 hr, while at 1200°C and 6×10^{-10} torr a weight loss of 0.077 mg/cm^2 was measured in 513 hr; vanadium and zirconium losses contributed 95% of the total weight loss. Evaporation occurred only at the surfaces; no subsurface voids were detected nor did evaporation occur at any preferential sites, such as grain boundaries or impurity inclusions at or near the surfaces.

The total weight change in the specimens appeared to be pressure and composition dependent. At 1200 to 1300°C and 10^{-7} torr, the niobium-base alloys FS-80 and D-43, containing only zirconium as the evaporating element, showed a weight gain, while at the same temperature and 10^{-9} torr a weight loss was observed. This behavior is attributed to interstitial contamination at 10^{-7} torr and purification at 10^{-9} torr. For those alloys containing more than one evaporating species, such as B-66 and C-129Y, a weight loss was observed at both the high and low test pressures at 1500°C. The evaporation losses in these cases overshadowed the changes in the interstitial contents.

Changes in the interstitial concentrations were dependent upon the pressure. At 1500°C and pressures of 10^{-9} torr or lower, carbon was retained while oxygen, nitrogen, and hydrogen were evolved. At a higher pressure of 10^{-7} torr and 1500°C, carbon was lost while the oxygen and nitrogen contents showed an increase. It is believed that the reaction of carbon and oxygen resulted in the loss of carbon at the higher pressures, while at 10^{-9} torr insufficient oxygen was present to appreciably reduce the carbon concentrations.

INTRODUCTION

The importance of refractory alloys in space applications has resulted in an experimental program designed to determine their compositional stability in vacuum. In space vacuums ranging from 10^{-7} to 10^{-16} torr, refractory alloys may become interstitially contaminated at the higher pressures and purified at the lower pressures when exposed for extended periods at elevated temperatures. In addition, the concentrations of other alloying elements may change because of evaporation.

The objective of many researchers in the space power program is to improve the high-temperature creep strength of various refractory alloys. The principal method considered is that of changing the interstitial content and the concentration of minor alloying elements.[1–3] Published data based on tests conducted over short periods, although helpful in understanding the strengthening mechanisms, do not reveal time-dependent reactions that might be deleterious. For example, application of such alloys in space may require thin-walled structural members that must operate over a design life of 10,000 hr or longer. Loss of interstitial elements in solution would disturb the equilibrium with interstitial precipitates. In an effort to reestablish equilibrium, the precipitates may dissociate, resulting in changes in alloy composition and a loss of dispersion strengthening.

To assess these potential problems, a study was undertaken to determine the evaporation behavior and changes in the concentration of interstitials in the ranges of 1100 to 1500°C and 10^{-7} to 10^{-10} torr. The commercial designation and nominal composition of the alloys selected for study are listed in Table 1.

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[1]R. W. Douglass, Research on the Technology of Tantalum Alloys with Maximum Strength in the 3000°F to 3500°F Range, Summary Report March 1–May 31, 1964, NRC Contract No. AF 33(657)-11255, Task No. 735101 (June 1964).

[2]D. Peckner, Mater. Design Eng. 54(7), 107–17 (1961).

[3]R. L. Ammon and R. T. Begley, Pilot Production and Evaluation of Tantalum Alloy Sheet, Summary Phase Report – Part II, WANL-PR-M-009 (July 1, 1964).

Table 1. Alloys Investigated in This Study

Commercial Designation	Nominal Composition (wt %)								Analyzed Interstitial Content (ppm)			
	Ta	Nb	W	Hf	Zr	V	Mo	Y	C	O	N	H
T-111	90		8	2					70	57	29	4
FS-80		99			1				100	180	78	10
FS-85	28	60	11		1				40	100	28	2
C-129Y[a]		79.9	10	10				0.1	75	150	60	5
B-66		89			1	5	5		110	97	250	4
D-43		89	10		1				980	63	89	6

[a]Alloy contained 0.4 wt % Zr as an impurity.

EXPERIMENTAL PROCEDURE

Specimen Preparation

From specimen stock approximately 0.004 to 0.005 in. thick, cylindrical 0.50-in.-diam × 0.75-in.-long evaporation specimens were made by spot welding in air. The specimens were degreased in acetone and chemically cleaned in a solution composed of H_2O, HNO_3, HF, and H_2SO_4. Each specimen was weighed and the geometric area was determined.

Evaporation Apparatus

Two similar pieces of experimental equipment were used in this investigation. Both systems allowed for collection of vapor deposits from individually induction-heated specimens held at temperature for various periods of time. The basic difference between the apparatus is in the pumping systems. The first apparatus, possessing a liquid nitrogen cold trap, oil diffusion pump, and mechanical pump combination, allowed for controlled variation in the pressure by regulating the power to the diffusion pump. This system was capable of pressures ranging from 10^{-6} to 10^{-9} torr at temperature.

The second system incorporated a 15 liters/sec ion pump that was turned on after a roughing pressure of 10^{-6} torr was attained using con-

ventional oil pumps. This system was capable of pressures of 10^{-9} to 10^{-10} torr at temperature, but the pressures were not controllable. The experimental pressure was the lowest pressure attainable at the desired temperature. The specimens in both systems were simultaneously heated using a series connected induction coil.

RESULTS AND DISCUSSION

Interstitial Stability

Results of the pre- and posttest chemical analyses showed that, for all the alloys studied, the change in the interstitial concentration varied with test pressure. At high vacuums of 10^{-9} torr, the carbon concentrations remained virtually unchanged. However, the oxygen and nitrogen concentrations at 10^{-9} torr decreased substantially. At greater pressures of 1×10^{-7} to 6×10^{-7} torr, the oxygen and nitrogen concentrations in the alloys increased, while the carbon content decreased, and in most instances these changes were severe. It is postulated that at the higher pressures the increased oxygen and nitrogen concentrations in the alloy resulted from the presence of these elements in the vacuum environments.

When oxygen was available in the environment, a reaction between the oxygen and the carbon in the alloy occurred. For example, the carbon-to-oxygen ratio for D-43 alloy decreased with an increase in pressure at 1500°C, as illustrated in Fig. 1. For alloy FS-80, the carbon-to-oxygen ratio at 2×10^{-9} torr increased with temperature, while at 5×10^{-7} torr the carbon-to-oxygen ratio decreased with an increase in temperature. Further, after several hundred hours, the carbon-to-oxygen ratios at low temperatures (1100 to 1200°C) are nearly the same for pressures between 5×10^{-7} and 2×10^{-9} torr. This behavior is illustrated in Fig. 2.

These results indicate that at temperatures between 1200 and 1500°C and in ultrahigh vacuums these alloys will lose nearly all of the oxygen (and nitrogen) while retaining nearly all the carbon. On the other hand,

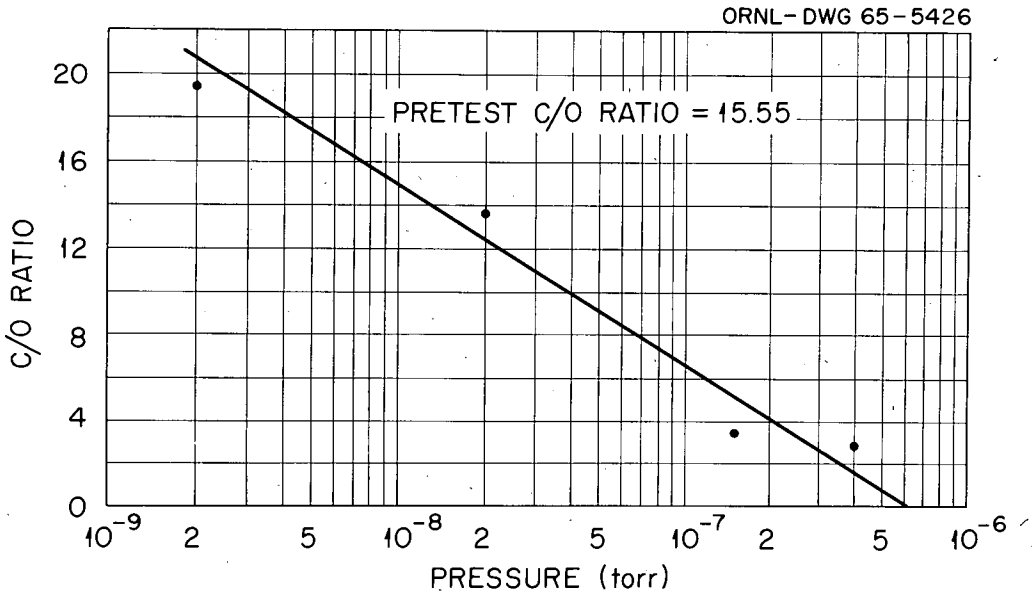

Fig. 1. Variation of the Carbon-to-Oxygen Ratio in D-43 Alloy as a Function of Pressure at 1500°C.

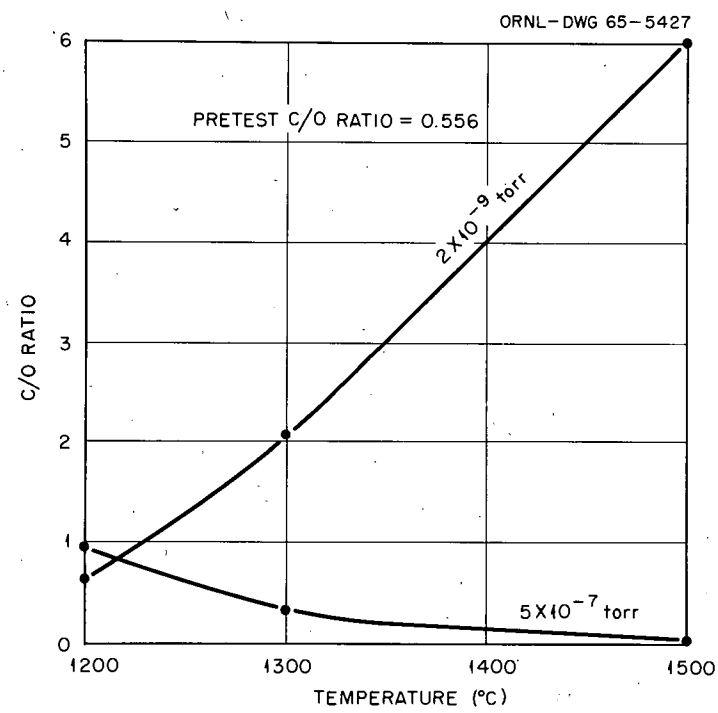

Fig. 2. Variation of the Carbon-to-Oxygen Ratio in FS-80 Alloy as a Function of Temperature.

when these types of refractory alloys are subjected to environments containing excessive amounts of oxygen (e.g., at higher pressures), then the carbon will decrease.

The individual analytical test results illustrating the behavior of the interstitial elements are tabulated in Table 2. A comparison of Table 2 with the initial concentration of interstitials as listed in Table 1 will show that in general the behavior discussed above is relevant to all of the alloys investigated in this study.

Table 2. Interstitial Concentration Changes Resulting from Tests Lasting from 500 to 750 hr

Alloy	Temperature (°C)	Pressure (torr)	Changes in Interstitial Concentration, ppm			
			Carbon	Oxygen	Nitrogen	Hydrogen
D-43	1500	2×10^{-9}	−50	−16	−56	−3
		2×10^{-8}	−50	−13	−66	−3
		1.5×10^{-7}	−400	+101	+351	−3
		4×10^{-7} [a]	−260	+197	+421	−5
	1300	1×10^{-9}	−20	−12	−63	−4
		5×10^{-7}	−380	+347	+244	−4
FS-80	1500	3×10^{-9}	−70	−175	−53	−6
		6×10^{-7}	−97	0	+92	−6
	1300	5×10^{-9}	−10	−137	−32	−7
		5×10^{-7}	−48	−40	+6	−4
	1200	1×10^{-9}	0	−20	−14	−5
		5×10^{-7} [a]	+190	+120	+42	−5
T-111	1500	2×10^{-9}	−10	−56	−8	−3
		5×10^{-7}	−30	+40	+171	−3
B-66	1500	7×10^{-9}	−80	−45	−150	−2
		5×10^{-7}	−90	−37	−10	−1
FS-85	1500	2×10^{-9}	0	−96	−19	−1
		5×10^{-7}	−21	+70	+222	−1
C-129Y	1500	2×10^{-9}	0	−74	−31	−4
		4×10^{-7} [a]	+385	+90	+1140	−4

[a]Cold trap became depleted of liquid nitrogen during the test.

Metallography

When certain refractory-base alloys undergo a severe change in concentration of interstitial carbon, oxygen, or nitrogen, a corresponding change in the size and quantity of precipitates containing these elements will also occur. For example, Table 2 shows that T-111 alloy lost oxygen and nitrogen at 1500°C and 2×10^{-9} torr, while the carbon content was not grossly affected. Examination of the corresponding microstructure (see Fig. 3a) shows a comparatively large grain-boundary precipitate accompanied by clear specimen surfaces. At 5×10^{-7} torr, however, carbon was lost, while the oxygen and nitrogen concentrations increased. Figure 3b shows that the resulting microstructure contained a very fine grain-boundary precipitate and that the specimen surfaces were now covered by a thin film. At 2×10^{-9} torr, the grain-boundary phase is believed to be carbides, while at 5×10^{-7} torr it is believed that both carbides and nitrides are present in the grain boundaries. The nitrides are present because of the increase in nitrogen concentration (Table 2). In addition, the film on the surfaces of the specimen tested at 5×10^{-7} torr is also believed to be a nitride because its etching characteristics are similar to those of certain grain-boundary precipitates.

A similar solid-solution alloy, T-222 (Ta–9.6 wt % W–2.5 wt % Hf), containing 0.010 to 0.013 wt % C exhibits two complex carbides.[4] At 1090°C a tantalum-rich, hexagonal $(Ta,W,Hf)_2C$ precipitates and is stable to 1316°C. At 1316°C a hafnium-rich, body-centered cubic $(Hf,Ta,W)_{1-x}$ precipitates and is stable to 1540°C. From the present results it also appears that after several hundred hours at 1500°C T-111 containing just 0.004 to 0.006 wt % C may possibly precipitate a carbide phase such as that illustrated in Fig. 3.

Microstructures of niobium-base alloy D-43 (Figs. 4 and 5) show pressure-dependent variations in the interstitial concentrations at 1300 and 1500°C. At both temperatures, the interstitial elements in the D-43 alloy exhibited a behavior similar to the interstitials in T-111 alloy.

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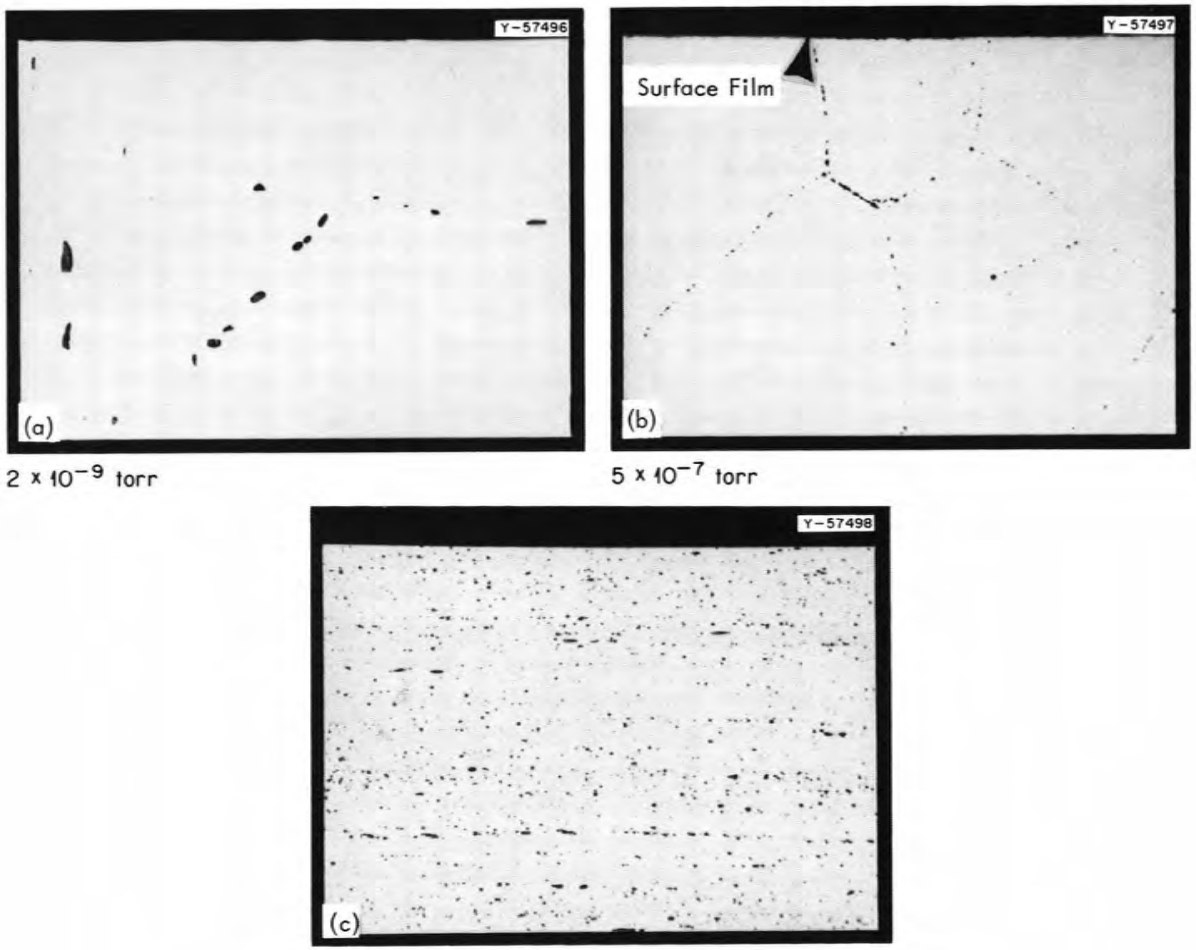
[4]R. L. Ammon and R. T. Begley, Pilot Production and Evaluation of Tantalum Alloy Sheet, Summary Phase Report – Part II, WANL-PR-M-009 (July 1, 1964).

PHOTO 69599



2 x 10^{-9} torr

5 x 10^{-7} torr

AS ROLLED TO 0.005 in. FROM 0.062 in.

Fig. 3. Microstructures of Alloy T-111 in the As-Rolled Condition and after 500 hr at 1500 °C as a Function of Pressure. Etchant: 60 parts H_2O, 20 parts H_2SO_4, 10 parts HF, and 10 parts HCl. 1000×.

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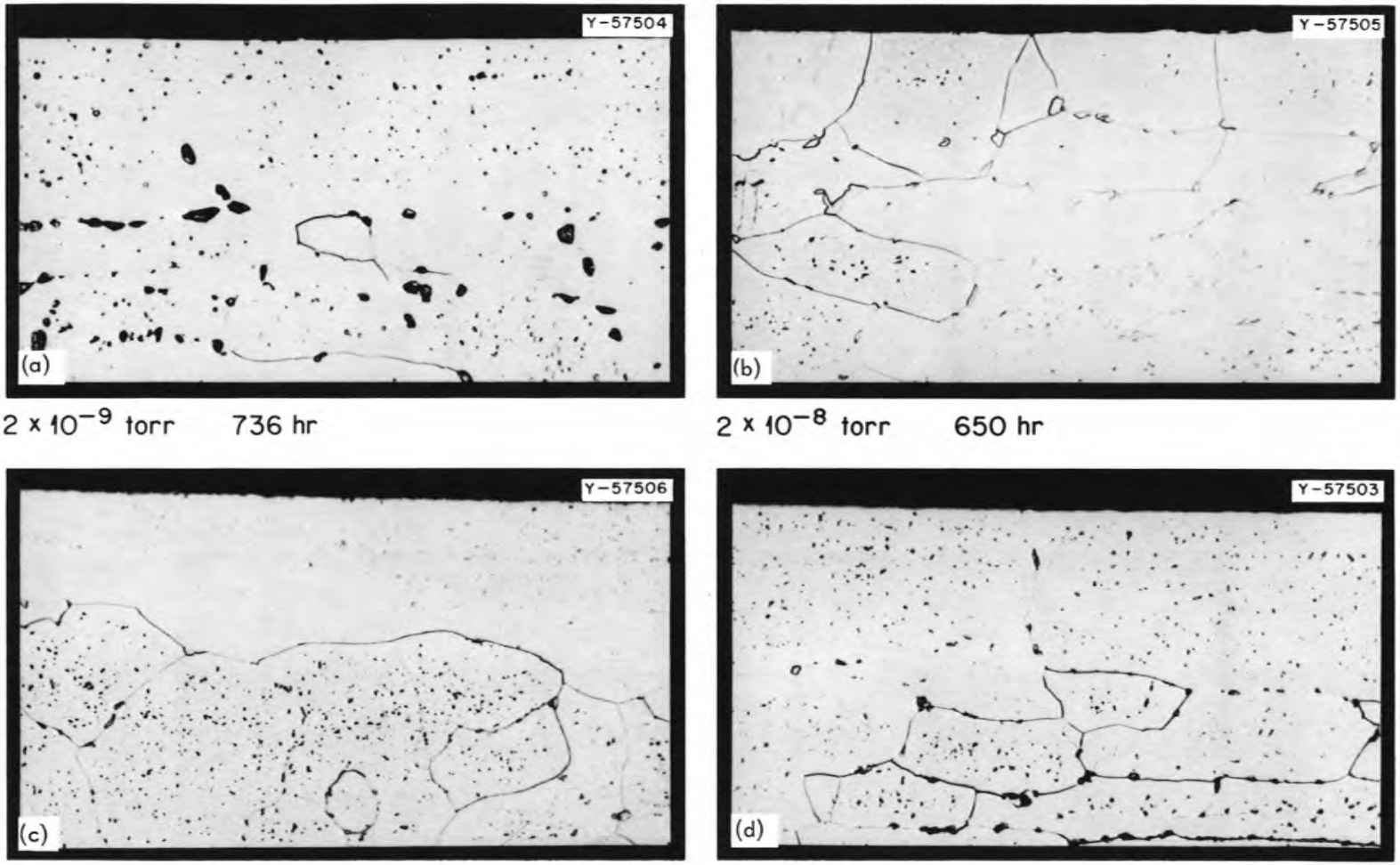

2×10^{-9} torr 736 hr

2×10^{-8} torr 650 hr

1.5×10^{-7} torr 650 hr

4×10^{-7} torr 736 hr

Fig. 4. Microstructures of D-43 Specimens Tested at 1500°C as a Function of Pressure. Etchant: 60 parts H_2O, 20 parts H_2SO_4, 10 parts HF, and 10 parts HCl. 1000×.

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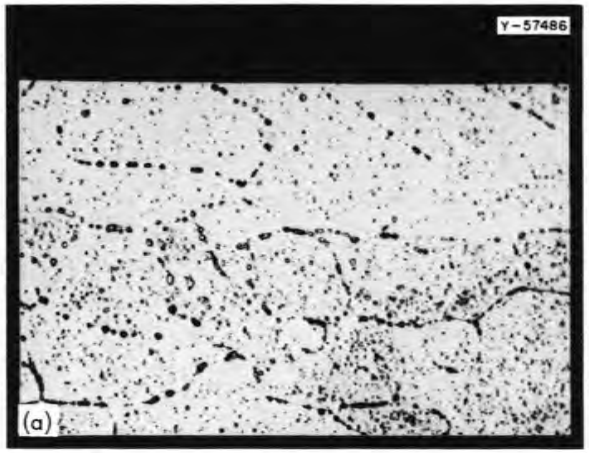
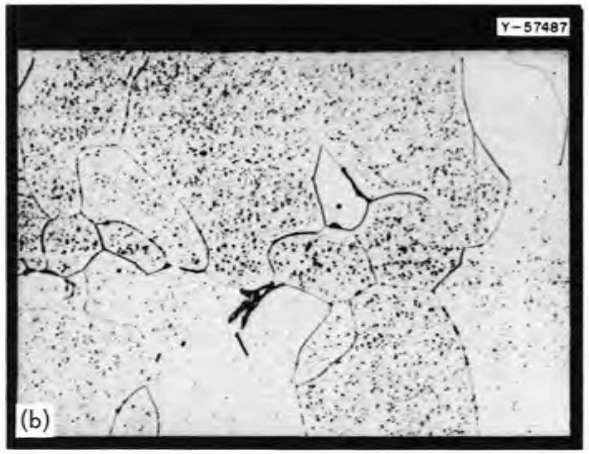
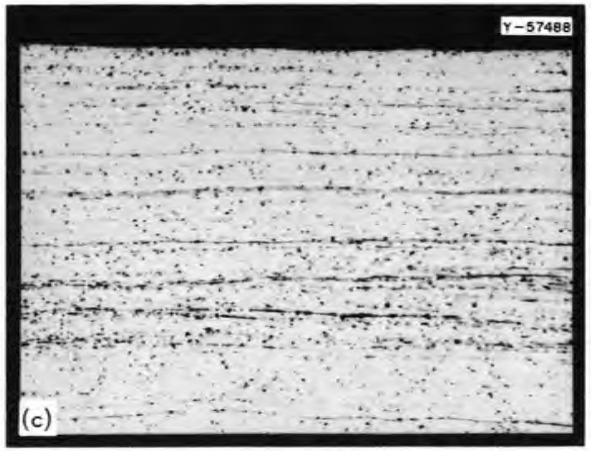


1×10^{-9} torr 376 hr



5×10^{-7} 376 hr



AS ROLLED TO 0.005 in. FROM 0.063 in.

Fig. 5. Microstructures of D-43 Specimens in the As-Rolled Condition and after 376 hr at 1300 °C as a Function of Pressure. Etchant: 60 parts H_2O, 20 parts H_2SO_4, 10 parts HF, and 10 parts HCl. 1000×.

At 1500 °C and pressures ranging from 4×10^{-7} to 1×10^{-9} torr, the microstructures (Fig. 4) and changes in the interstitial concentration (Table 2) indicated a close relationship between grain-boundary precipitates and carbon content. As the test pressure was increased the carbon concentration decreased (after several hundred hours), and this decrease was accompanied by a corresponding decrease in the size and number of grain-boundary precipitates. The sensitivity of D-43 alloy to changes in carbon content is more fully illustrated in Fig. 4c and d. Near the end of a 736-hr test, a failure to fill the liquid nitrogen cold trap during a 36-hr period resulted in a higher carbon content (Fig. 4d) when compared to a normal 650-hr test (Fig. 4c). This 140-ppm difference in carbon concentration resulted in a greater amount of grain-boundary precipitates (Fig. 4d). Microstructural dependency on test pressure also occurred at 1300 °C, as illustrated in Fig. 5. In a 376-hr test at 5×10^{-7} torr, the carbon concentration decreased from 980 to 600 ppm, thus resulting in a disappearance of the grain-boundary phase. At 1500 °C this disappearance of the grain-boundary phase occurred at a carbon level of 580 ppm, which was attained at 1.5×10^{-7} torr in 650 hr. These results indicate that this grain-boundary phase may be unstable at lower temperatures, provided it is the same phase that is present at 1500 °C.

No attempts were made to identify this grain-boundary phase; however, it has been reported[5–7] that Nb_2C has a hexagonal structure and precipitates at grain boundaries in niobium-base alloys containing relatively high carbon concentrations. Examination of this phase under polarized light revealed an anisotropic behavior. Although not conclusive, this is an indication that the grain-boundary phase illustrated in Figs. 4 and 5 might be Nb_2C. Further, Gerken[7] has shown that Nb_2C is a high-temperature phase and that heat treatment at 1200 to 1300 °C or a loss of carbon will decrease the amount of this phase present in the microstructure, a behavior consistent with the results of this study.

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[5]A. C. Barber and P. H. Morton, A Study of the Niobium-Zirconium-Carbon and Niobium-Zirconium-Oxygen Systems, Imperial Metal Industries K/RR/33/64 (May 7, 1964).

[6]D. O. Hobson, Aging Phenomena in Columbium-Base Alloys, ORNL-3245 (March 1962).

[7]J. M. Gerken, A Study of Welds in Columbium Alloy D-43, Thompson Ramo Wooldridge, Inc., TM-3865-67 (March 1964).

Gerken has also shown that at 1200 °C Nb_2C is converted to a complex (Nb,Zr)C, which appears as the fine "dot" phase in Figs. 4 and 5. Verification of these results has been completed at ORNL. An electron micrograph illustrating this converted carbide is presented in Fig. 6.

Metallographic examination to 2000× did not reveal any differences in etching characteristics, color, or precipitate morphology in specimens heat treated at 1500 and 1300 °C and 10^{-7} and 10^{-9} torr. The oxygen and nitrogen contents were low at 1×10^{-9} and high at 5×10^{-7} (see Tables 1 and 2), and because the metallographic features of the different samples were similar, speculation as to the presence of oxides or nitrides cannot be made for these test conditions.

The microstructures of the remaining alloys, FS-80, FS-85, C-129Y, and B-66, were similar for the same conditions of time, temperature, and pressure. At 1500 °C, grain growth was severe: the average grain size was approximately ASTM 1. No precipitates were detected at magnifications to 1000×, except in the case of C-129Y at 1500 °C and 4×10^{-7} torr. Failure to fill the liquid nitrogen cold traps resulted in massive precipitates. The increase in carbon and nitrogen (see Table 2) suggests the precipitation of carbides and nitrides.

The FS-80 alloy was also tested at the lower temperatures of 1200 and 1300 °C. Grain growth was not evident, and the average grain size was ASTM 6. Precipitation was evident at 1200 and 1300 °C for test pressures of 10^{-7} and 10^{-9} torr. Hobson[6] has reported that when 100 ppm O is present in the alloy, ZrO_2 will precipitate in FS-80 at 900 to 1100 °C.

Evaporation

At the outset of this investigation, a primary concern was evaporation of the alloys at their service temperatures. It was felt that substantial evaporation losses would result in loss of mechanical strength and adverse changes in emittance. Therefore, tests were conducted at temperatures slightly above the intended application temperatures and for times sufficient to reveal the seriousness of the problem. It was learned that at the application temperatures no serious evaporation problems would be encountered in ultrahigh vacuum environments. The results of the evaporation tests are summarized in Table 3.

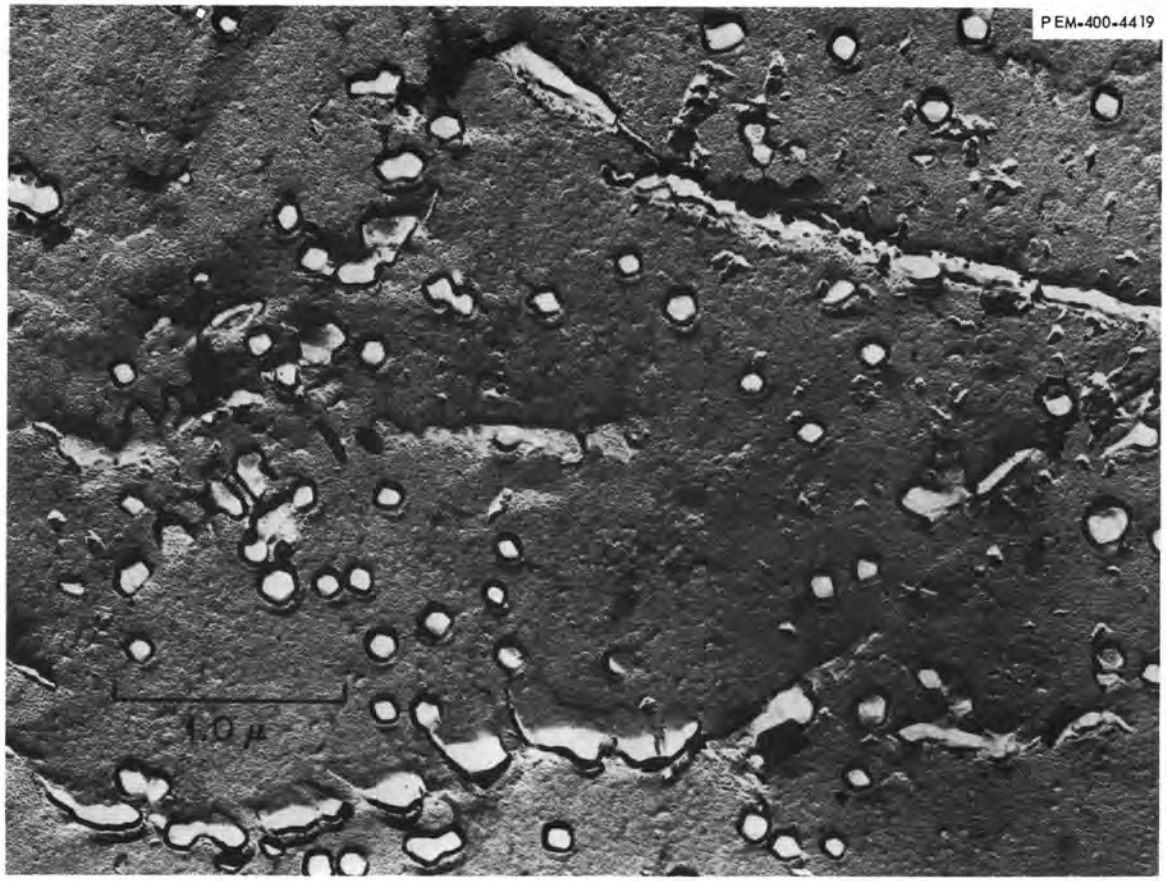

Fig. 6. Replicated Carbides (Nb,Zr)C *in situ*. Sample over-etched. Nb–10% W–1% Zr–0.1% C alloy annealed 2 hr at 3300°F. Aged 500 hr at 2200°F.

Table 3. Results of Evaporation Tests of Refractory-Base Alloys in High Vacuums

Alloy	Temperature (°C)	Pressure (torr)	Duration (hr)	Specimen Weight Change (mg/cm^2)	Composition of Deposit (wt %)
FS-80	1500	3×10^{-9}	512	−0.098	100 Zr
		6×10^{-7}	512	−0.130	100 Zr
	1300	5×10^{-9}	493	−0.012	a
		5×10^{-7}	493	+0.043	a
	1200	1×10^{-9}	1000	−0.024	a
		5×10^{-7}	1000	+0.051	a
	1100	1×10^{-9}	300	−0.003	a
B-66	1500	7×10^{-9}	316	−1.700	83.8 V–15.5 Zr–0.46 Mo–0.25 Nb
		5×10^{-7}	316	−1.600	82.9 V–12.2 Zr–0.46 Mo–0.17 Nb
	1200	6×10^{-10}	513	−0.077	93 V–7 Zr
		5×10^{-7}	494	−0.024	93 V–7 Zr
T-111	1500	2×10^{-9}	500	−0.079	92.6 Hf–5.8 Zr
		5×10^{-7}	500	−0.23	100 Hf
C-129Y	1500	2×10^{-9}	521	−0.16	30.5 Hf–8.1 Zr–21.2 Y[b]
		5×10^{-7}	521	−0.56	61.5 Hf–3.7 Zr–1.5 Y [b]
FS-85	1500	2×10^{-9}	667	−0.066	100 Zr
		5×10^{-7}	667	−0.100	100 Zr
D-43	1500	1×10^{-9}	736	−0.029	100 Zr
		2×10^{-8}	650	−0.029	100 Zr[c]
		1.5×10^{-7}	650	−0.030	100 Zr[c]
		4×10^{-7}	736	+0.019	100 Zr
	1300	1×10^{-9}	376	−0.016	100 Zr[c]
		5×10^{-7}	376	+0.018	a

[a]Not measurable.

[b]Sample weight uncertain.

[c]Sample weight very low; only one analysis completed.

In general, a good mass balance was obtained between the weight of the vapor deposit and the weight lost by the specimen. Errors of 8 to 12% were attributed to the formation of gaseous products, which were probably pumped out of the system. In those cases where an adequate mass balance could not be obtained, the deposit was so light that the accuracy and precision of the chemical analytical technique led to uncontrolled errors. The deposits, therefore, could not be identified.

At 1500°C, all of the investigated alloys (see Table 3) lost weight at both 10^{-7} and 10^{-9} torr. However, the weight loss (except in the case of D-43 and B-66 alloys) was greater at 10^{-7} torr than at 10^{-9} torr. It is postulated that the higher concentration of oxygen and nitrogen in the environment at the higher pressures resulted in the formation of volatile metal oxides and nitrides, which were pumped out of the system. In addition, decarburization, which was discussed previously, added to the weight loss. For those alloys investigated at lower temperatures (see Table 3), it was observed that all specimens lost weight at high vacuums of 10^{-9} to 10^{-10} torr. At poor vacuums of 5×10^{-7} torr, however, all samples except B-66 alloy gained weight even though a slight vapor deposit was observed on the collection tube. The data show that at temperatures of 1100 to 1300°C and pressures of 4×10^{-7} to 5×10^{-7} torr, the weight lost by evaporation of volatile compounds is overshadowed by the weight gain due to the increase in the interstitial concentration at these higher pressures.

The B-66 alloy contains two high vapor pressure elements, vanadium and zirconium, which evaporated at 1500 and 1200°C and 10^{-7} to 10^{-10} torr. The evaporation results (Table 3) showed that system pressure at constant temperature did not influence the evaporation of either element. At the different test pressures, the relative amounts of vanadium and zirconium in the vapor deposit were the same. The influence of temperature, however, is quite obvious in the evaporation results tabulated in Table 3.

The microstructures of the alloys studied did not contain any surface or subsurface voids, broadened grain boundaries, or any other phenomena that might indicate localized evaporation. In the case of

the FS-80 alloy, however, certain grain orientations evaporated at greater rates. This behavior is illustrated in Fig. 7 by the offset in the surface at the grain boundary.

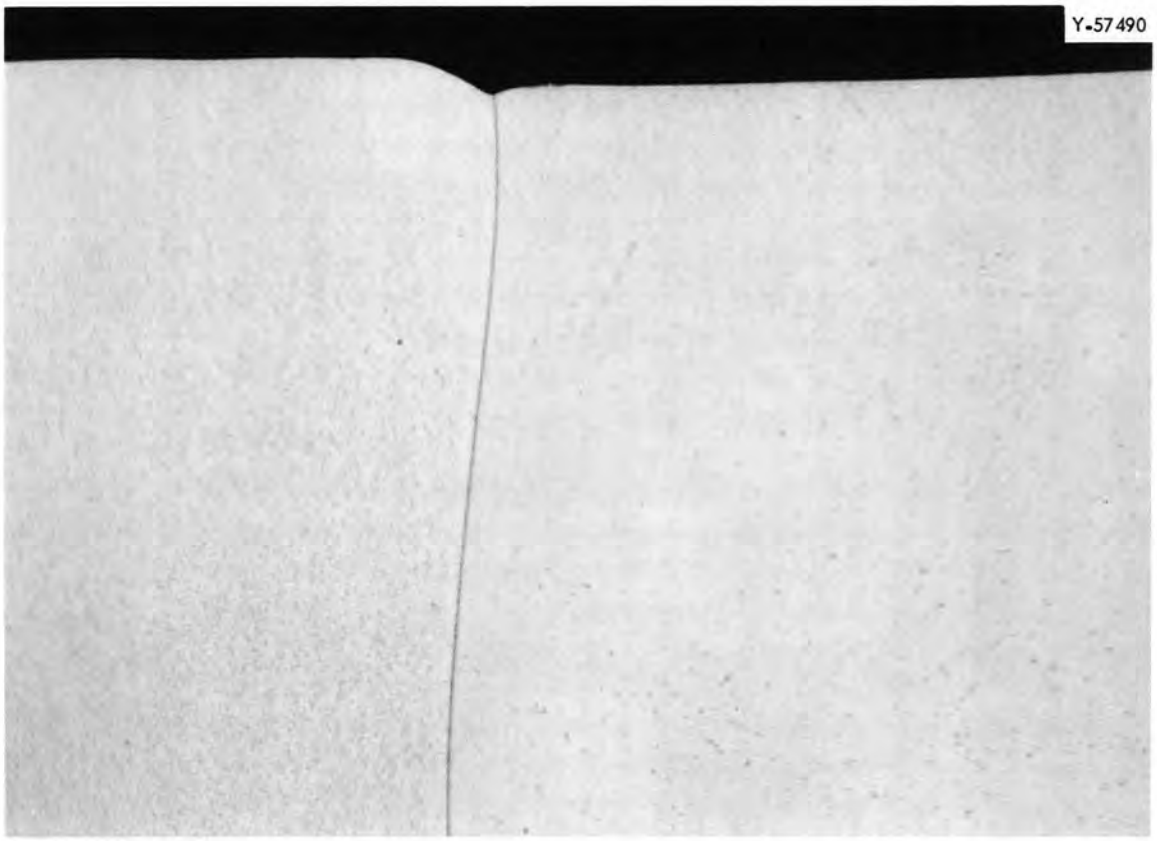

Fig. 7. Surface Irregularity Illustrating Preferential Evaporation from Certain Crystal Orientations in FS-80 Alloy at 1500 °C and 3×10^{-9} torr. Etchant: 60 parts H_2O, 20 parts H_2SO_4, 10 parts HF, and 10 parts HCl. 1000×.

CONCLUSIONS

The niobium- and tantalum-base alloys investigated in this study exhibited interstitial instabilities at 1300 to 1500 °C under high vacuums. At vacuums of 7×10^{-9} to 6×10^{-10} torr, the instability is a loss of oxygen, nitrogen, and hydrogen. At 1×10^{-7} and 6×10^{-7} torr, the concentration of oxygen and nitrogen increased, but the carbon content decreased over an extended period of time. At 10^{-9} torr or lower, the

carbon content remained virtually unchanged. It was concluded that a carbon-oxygen reaction occurred, resulting in carbon losses as CO over long testing periods. The gaseous elements were lost simply by diffusing to the specimen surfaces and desorbing into the vapor state.

Evaporation of the alloying constituents from the alloy surfaces was found to be low. It was concluded that at temperatures of 1000 to 1300 °C no serious evaporation problems would be encountered in high-vacuum environments. However, it was further concluded that exposures of several thousands of hours at 1300 to 1500 °C would result in severe losses of volatile species, such as hafnium and vanadium.

ACKNOWLEDGMENTS

The author is indebted to the many service departments in carrying out various phases of these experiments. The chemical analyses performed by W. R. Laing of the Analytical Chemical Chemistry Division and the metallography of W. H. Farmer and T. J. Henson proved to be necessary services in the interpretation of these experiments. The assistance of J. Newsome and R. L. Taylor in carrying out the experiments is appreciated.

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