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SPACE-POWER-SYSTEM MATERIAL COMPATIBILITY TESTS OF SELECTED REFRACTORY METAL ALLOYS WITH BOILING POTASSIUM

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ABSTRACT

The compatibility of potential niobium and tantalum tubing alloys with boiling potassium was investigated in the range of temperature representative of the upper operational temperatures of a turboelectric space power system. Two types of compatibility test were employed: (1) reflux capsule tests for materials screening, and (2) a forced convection two-phase loop test in which power-system flow rate and temperature gradient service conditions were simulated.

In the reflux capsule tests, eight alloys were studied: B-33 (Nb-4V, nominal alloy composition in weight percent); SCb-291 (Nb-10 Ta-10W): Nb-1Zr; D-43 (Nb-10W-1Zr-0.1C); FS-85 (Nb-28Ta-10W-1Zr); Ta-10W; T-111 (Ta-8W-2Hf); and T-222 (Ta-9.6W-2.5Hf-0.01C). Many of the alloys were tested in both the as-worked and recrystallized conditions. The initial oxygen content of the alloys was less than 150 ppm and that of the potassium less than 20 ppm. Tests were conducted over the temperature range of 1800° to 2400° F for times up to 10,000 hours in vacuums of 10^{-7} to 10^{-8} torr. In general, the gettered alloys (i.e., those containing the reactive elements Zr or Hf) were resistant to corrosive attack, while the ungettered alloys all exhibited significant corrosion. Since the presence of oxygen is known to increase alkali metal corrosion (probably via the formation of a soluble alkali-metal - container-metal - oxygen complex), the observed corrosion resistance of the gettered alloys can be attributed to a reaction of the getter element with available oxygen to form a stable oxide and thereby prevent oxygen from entering into the corrosion reaction. No stress corrosion effects were observed in tests on D-43 and B-33 alloy reflux capsules specially stressed to produce 15% and 45% creep strain, respectively, in sections of the capsule wall.

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In the forced convection two-phase loop test, the material of construction was Nb-1Zr alloy. The loop was run in a vacuum of 2×10^{-8} torr for 5000 hours under the following operating conditions: potassium flow rate, 0.1 gal/min; saturation temperature, 1850° F; superheat temperature, 2000° F; condensing temperature, 1425° F; and subcool temperature, 800° F. No measurable potassium corrosion was observed in any portion of the loop.

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INTRODUCTION

Interplanetary space travel and longtime orbital and lunar-based manned space stations will require large quantities of electricity for propulsion, communications, and life support equipment. Advanced nuclear turboelectric generator systems in the megawatt range will be needed to provide the electrical power for these operations. Such systems will require containment materials having adequate creep and corrosion resistance under the conditions of space operation at temperatures in the range of 1800° to 2400° F and for times in excess of 10 000 hours. At present, potassium is a favored choice for the system working fluid, and niobium and tantalum alloys are candidates for the containment material.

As part of the NASA technology program for advanced space power systems, studies have been initiated at the NASA Lewis Research Center, Cleveland, Ohio (LeRC) and at the General Electric Laboratory, Cincinnati, Ohio (GE) to determine the compatibility of potential tubing alloys with potassium. Two types of compatibility tests have been employed: (1) a reflux capsule test for material screening (LeRC and Ge) and (2) a forced convection two-phase loop test in which power system fluid flow and temperature conditions are simulated (GE).

Presented herein are initial findings of these tests. Reflux capsule test results are given for 8 niobium- and tantalum-base tubing alloys. The alloys were of both the gettered (i.e., containing hafnium or zirconium) and nongettered varieties: B-33 (Nb-4V, nominal alloy composition in weight percent); SCb-291 (Nb-10Ta-10W); Nb-1Zr; D-43 (Nb-10W-1Zr-0.1C); FS-85 (Nb-28Ta-10W-1Zr); Ta-10W; T-111 (Ta-8W-2Hf); and T-222 (Ta-9.6W-2.5Hf-0.01C). Many of the alloys were tested in both the as-worked and recrystallized conditions. Tests were conducted over the temperature range of 1800° to 2400° F for times up to 10 000 hours. Data from reflux capsule tests to determine the possible effect of stress on corrosion are also presented. In these tests, three niobium-base alloys, B-33, D-43, and FS-85 were tested at 2200° F up to 1000 hours at an initial stress of about 4000 psi. Lastly, results are presented of a test run of a forced convection boiling potassium loop. The loop, constructed of Nb-1Zr alloy, was operated at 1850° F saturation temperature for 5000 hours.

REFLUX CAPSULE TESTS

The corrosion resistance of the potential space-power-system tubing alloys to potassium was determined by means of reflux capsules, the capsule wall serving as the test specimen. In these tests, potassium is vaporized from a pool at the bottom of the capsule, rises, and is condensed near the top of the capsule. Condensate flows down the capsule wall to join the bulk liquid, completing the circuit.

1. PROCEDURE

The potassium used was purified by the hot-getter method [1] to an oxygen level of less than 20 ppm. The alloys, their composition, and their condition are given in table I. The so called nongettered alloys, B-33, SCb-291, and Ta-10W, were specifically analysed for the presence of getter-element contaminants, namely, Ta, Zr, and Hf. Heats 1, 2, and 3 of the B-33 alloy were found to contain small amounts of Zr.

Capsules of two sizes were used: a small capsule, 1/2-inch outside diameter by $1\frac{3}{4}$-inch by 0.040-inch wall (LeRC), and a large capsule, 1-inch outside diameter by 11-inch by 0.080-inch wall (GE). The choice of capsule size was somewhat arbitrary, reflecting mainly the test preferences and techniques employed by each laboratory. The small capsules were machined from rod stock, while the large capsules were fabricated by roll forming and tungsten-inert-gas welding of 0.080-inch sheet.

To preclude air contamination of the capsules and their contents, capsule loading and sealing were performed in special vacuum facilities, at pressures of about 10^{-5} torr. A volume of potassium equal to about 1/3 the internal volume of the capsule was introduced into each capsule. A cap was placed over the top of the capsule and electron beam welded to the capsule. This procedure is described in detail in the LeRC motion picture (C-241, available on request) entitled Vacuum Handling of Air Sensitive Space-Power-System Materials.

In the small capsule tests, in order that weld material might be exposed to the condensing vapor as well as the boiling potassium liquid pool, some capsules were positioned with the cap weld at the top and others inverted.

Testing was conducted in high-vacuum chambers at pressures in the range of 10^{-8} torr. Heat was supplied to each capsule by a tantalum heater. Electric power to the heaters was regulated by automatic control equipment to maintain a set temperature. Capsule temperatures, measured with platinum - platinum-13%-rhodium thermocouples, were continuously recorded throughout the test duration. Estimates of the condensation rate and condensate film velocity within the large capsules are 1 lb/h and

0.09 ft/sec, respectively.

At the conclusion of a test, the capsules were opened and the potassium removed. X-ray, chemical, and metallographic analyses were made on selected capsule sections.

2. RESULTS

Test results are summarized in Table II. Several general corrosion or corrosion-related results can be noted:

1. The alloys containing the gettering elements, zirconium or hafnium, were found to be markedly more resistant to corrosive attack than the nongettered alloys. A typical comparison of corrosion results between gettered and nongettered alloys can be seen in Figures 1 and 2 for niobium- and tantalum-base alloys, respectively.

2. Corrosion attack, when it occurred, was mainly intergranular. However, in the case of B-33, heats 1 and 2, and SCb-291 significant solution attack also was observed. In the case of the alloys tested at the more extreme conditions of temperature and time, a small amount of leaching or alloying elements from the condensate zone was manifest as a black film deposit in the region below the condensate zone (e.g., FS-85 capsule, Fig. 1).

3. Grain growth was more pronounced in the top sections of the capsules than in the bottom, even though the capsule temperature was lower at the top than at the bottom.

4. The gettered alloys generally showed less second-phase precipitation in the capsule top section than in the bottom and a precipitate deficient zone along the inner edge of the top sections.

STRESS CORROSION TESTS

1. PROCEDURE

For purpose of stress corrosion testing the standard reflux capsule was altered by machining down the wall thickness just above the midregion, as shown in Fig. 3. Internal pressure in the capsule resulting from the vapor pressure of the capsule imposed an initial hoop stress of about 4000 psi on the thin-wall section, causing it to creep (see Fig. 3). Three niobium-base alloys, B-33, D-43, and FS-85, were tested for stress corrosion at 2200° F for up to 1000 hours. In each stress corrosion test, as a reference, a standard capsule of the same material was tested at the same temperature and time as the stress corrosion capsule.

2. RESULTS

A summary of the test results is included in Table II. In general, no stress corrosion effects were found over the range of conditions tested.

B-33 (heat 1)

The nature and extent of corrosion attack in the stress corrosion capsule test at 2200° F for 1000 hours and 2300° F for 380 hours were the same as in the standard capsule. The 2300° F thin-walled capsule failed by creep rupture at a creep strain of 45%.

D-43

No effect of stress or corrosion was observed at 2200° F or at 2300° F (Fig. 3) for 1000-hour tests. The 2200° F and 2300° F thin-wall capsules underwent creep strains of 3.5% and 37%, respectively.

FS-85

No stress corrosion effects were seen in the capsule test at 2300° F for 1000 hours and a creep strain of 2.7%.

PUMPED LOOP TEST

The initial screening of potential space-power-system materials is readily accomplished in capsule tests, as previously described. The final materials selection, however, must be made on the basis of proof tests which simulate closely the temperature and flow conditions of an actual power system. Described below is the first of the forced convection two-phase corrosion tests in the NASA's advanced space-power-system technology program.

The prototype test system was fabricated from Nb-1Zr alloy (the only refractory metal alloy available as tubing at the start of the test program in 1963). The composition of the alloy was as follows: 0.87 Zr; 33 ppm C; 260 ppm O; 47 ppm N; 1 ppm H; balance, Nb.

The corrosion test system basically consisted of two liquid metal loops, a sodium heater loop and a potassium boiling and condensing loop, mounted in a vacuum chamber. A vacuum of 2×10^{-8} torr or better was maintained in the chamber during the test duration of 5000 hours.

In the heater loop, sodium, circulated by an electromagnetic pump, was heated by an electric resistance heater. From the heater, the sodium flowed into a tube-in-tube counterflow boiler where heat from the sodium was transferred to the potassium. The sodium loop operated at a maximum temperature of 2130° F, a minimum temperature of 1990° F, and a flow rate of 910 lb/h.

In the second loop, potassium, discharged from an electromagnetic pump, was preheated by an electric resistance heater before entering the boiler. In the boiler, the potassium converted from liquid to superheated vapor. The vapor passed through the turbine simulator (the pressure drop, heat extraction section) and on into the condenser. Condensed vapor then returned through the subcooler to the pump. The operating conditions were as follows: saturation temperature, 1850° F; superheat temperature, 2000° F; condensing temperature, 1425° F; flow rate, 31 lb/h, and liquid velocity in the boiler, 0.8 ft/sec

Figure 4 shows a view of the tube-in-tube boiler and photomicrograph of a typical section of the tube wall following the 5000-hour test. No corrosion or erosion attack was found in any portion of the potassium loop.

On the sodium side of the boiler a small amount of mass transfer deposit was found on the outside diameter of the inner tube. The deposit was in the relatively short section where the greatest temperature drop in the sodium is estimated to have occurred. The 1/2-mil deposit consisted primarily of zirconium. There were no other manifestations of corrosion in the sodium loop.

DISCUSSION

1. EFFECT OF OXYGEN ON CORROSION

The prominent role of oxygen in the dissolution mass-transfer reactions of alkali metals has been established by previous investigations. It has been found (1) that oxygen enhances the corrosion of various materials by Na and Li [2 to 5] and (2) that the presence of oxygen increases the measured solubility of elements such as Fe, Mo, Ta, and Nb in Li, Na, and K [6 to 8]. In the corrosion tests reported here, a similar "oxygen effect" is believed to account for the salient test result, namely, the gettered alloys were markedly more resistant to corrosion by potassium than were the nongettered alloys. The effectiveness of the gettered alloys in reducing corrosion appears to stem from the effectiveness of the getter element in scavenging and immobilizing oxygen through the formation of a stable oxide, i.e., ZrO_2 or HfO_2.

It has been suggested that the mechanism of oxygen-enhanced mass transfer involves a container-metal - alkali-metal complex oxide as a reaction intermediate [2,5]. Evidence for the existence of complex oxides has been obtained by Horsley in the Fe-Na-O system [2] and by Tyzack in the Nb-Na-O system [3]. Pertinent to the potassium corrosion tests reported herein are the results of a recent study at LeRC [9] in which tantalum, containing 1200 to 2400 ppm oxygen, was exposed to potassium at 1800° F for 100 hours In all cases the Ta suffered corrosion and complete oxygen depletion; the corrosion product isolated from the potassium was a white solid characterized as $3K_2O \cdot Ta_2O_5$. These results support the contention that, when oxygen is available, potassium corrosion attack occurs via the formation and subsequent dissolution of a Ta (or Nb) complex oxide.

In potassium corrosion tests reported herein, available oxygen potentially can be found either in the potassium, as K_2O or in the refractory alloy, as refractory metal oxide or as interstitial oxygen Potassium oxide is not considered a prime source of oxygen, since the amount of oxygen present as K_2O accounts for 3% or less of the total available oxygen. The same can be said for the refractory metal oxides, since oxides are not likely to form when the oxygen concentration of the alloy is below the solubility limit. Thus, interstitial (i.e., dissolved) oxygen is the most probable source of available oxygen.

The location and distribution of dissolved oxygen will depend largely on regional differences in solubility and diffusion rates which in turn depend on the temperature and the alloy composition and structure. When oxygen is more or less uniformly distributed throughout an alloy, uniform solution attack (such as exhibited by the B-33 alloys, heats 1 and 2) would be expected. Alternately, oxygen may concentrate in certain preferred regions such as grain boundaries [10], specific crystal planes [11], or lines of dislocations [12]. Corrosion attack, then, would be expected to follow these oxygen enriched paths. Although transgranular attack has been observed in tests of Ta in K [9] and Ta and Nb in Li [4], corrosion of the preferred path type when observed in the tests reported herein was exclusively intergranular.

The results for the two nongettered alloys tested indicated that the sensitivity to intergranular attack, considering time, temperature, and initial oxygen content, is Ta-10W > SCb-291. (The B-33 alloys used in this study cannot be considered as nongettered, since they contained Zr as a fortuitous impurity.) The order of penetration sensitivity of the Ta alloy, Ta-10W, and the Nb alloy, SCb-291, in potassium was the same as that found for Ta and Nb in Li [4]. No explanation can be offered for the difference in sensitivities.

2. GETTER-RELATED CORROSION THRESHOLD

As discussed previously, the corrosion resistance of the gettered Nb- and Ta-base alloys appears to result from the ability of the getter element to scavenge oxygen. If this is the case, a getter-related corrosion threshold should exist which may be defined in terms of the minimum amount of getter required to immobilize the oxygen. An estimate of the minimum amount of getter required to prevent corrosion must necessarily take into account interstitials such as N and C as well as O. The reason for this is the N and C, conceivably, may also react with the getter and thereby compete with O for available getter. (Reaction, as described here, may entail weak association or "clustering" as well as compound formation between getter and interstitials.) Evidence supporting the possibility of competitive reactions of oxygen and other interstitials with the getter element can be found in the results of two pertinent studies. First, internal friction measurements of a Nb-0.9 Zr alloy, containing 0.015% N and <0.07% O, have indicated that all of the O and part of the N are associated with the Zr [13]. Second, in a study of the Nb-Zr-C system, containing <0.015% O, one of the four phases found in the Nb corner of the system was tentatively identified as $Zr_2(OC)$ [14]. Thus, on the basis of the foregoing discussion, the amount of getter required to prevent corrosion should depend (1) on the extent to which the getter is partitioned between oxygen and other interstitials and (2) on the amount of interstitials present.

The premise of a getter-related corrosion threshold can be examined by considering the three heats of B-33 alloys Each heat, because of a fortuitous presence of Zr as an impurity, contained a small amount of Zr (viz., 0.02%, 0.05%, and 0.19% for heats 1, 2, and 3, respectively). Also, each heat showed a different corrosion response; the order of corrosion attack was heat 1 > heat 2, and no attack was found for heat 3. The previous data show that there is a qualitative relation between Zr content and corrosion. However, to convincingly verify the premise of a getter-related corrosion threshold, a quantitative relation is needed This relation may be achieved by comparing the corrosion results with the actual Zr content of the alloy Zr_A and with the amount of Zr theoretically required to prevent corrosion Zr_{th}. For each heat, Zr_{th} can be calculated from the interstitial composition given in Table I together with an estimate of the partition functions for the Zr-(O,N,C) reactions. (The interstitial H is neglected here because of the instability of pertinent hydrides at the test temperatures.) Since no quantitative information is available for the partition functions, two limiting cases will be considered. In case 1, it will be assumed that all the O, N, and C react to form ZrO_2, ZrN, and ZrC, respectively; in case 2, it will be assumed that only the oxygen reacts and forms ZrO_2. The true value of Zr_{th} is expected to be between the high value of Zr_{th}, case 1, and the low value of Zr_{th}, case 2.

A comparison of corrosion and Zr content for the three heats of B-33 alloy is given in table III. Shown are the test time, the corrosive attack, Zr_A, and the values calculated for Zr_{th}, cases 1 and 2. These results generally substantiate the premise of a getter-related corrosion threshold. When $Zr_A > Zr_{th}$ there is corrosion attack which tends to increase as the difference between Zr_A and Zr_{th} increases.

3. CORROSION BY ELEMENTAL DISSOLUTION

If the amount of getter available is greater than the amount of getter required to prevent corrosion, dissolution mass transfer cannot occur through the formation and dissolution of a container-metal complex oxide. Instead, the path of mass transfer attack will be restricted to direct dissolution of container metal in K. The capsule test results suggest that the dissolution potential of the container metal is very much lower than that of the container-metal complex oxide. Even at the upper test temperature of 2400° F, no gross dissolutions was observed.

The only indications of dissolution attack in the getter alloys were (1) the <1-mil-grain boundary penetrations in the 2400° F tests of he FS-85 and T-111 alloys and (2) the black film found on the inner surface at the middle third region of the capsules tested at the more extreme time and temperature conditions. In the former instance, the penetrations may possibly reflect grain boundary grooving arising from surface energy differences between grain and grain boundary at this temperature. In the latter instance, the black film presumably results from deposition of material leached out of the condensation region. The black film in the T-222 capsules was identified as $K_2Ta_4O_{11}$ plus traces of WO_3. In the 10 000-hour Nb-1Zr capsule, the black deposits were identified as predominantly Zr. Analyses were not made of the films found in the remaining capsules. However, in all of the capsules in which films were found, indirect indications of Zr (or Hf) leaching were noted (viz., less second-phase precipitation and acceleration of grain growth at the potassium-refractory alloy interface of the condensing region).

4. EFFECT OF STRUCTURE ON CORROSION

Alloy structure (i.e., worked or recrystallized) did not noticeably influence the gettered-alloy test results. Structure, however, did markedly affect the extent of attack on the ungettered Ta-10W alloy. The worked structure of the Ta-10W, heat 1, tested at 1800° and 2200° F showed no attack, while in the same capsules the recrystallized weld zone exhibited extensive intergranular penetration. The recrystallized Ta-10W, heat 2, tested at 1800° and 2200° F exhibited intergranular attack throughout the entire capsule.

When the mode of corrosion attack is intergranular, it is likely that a worked structure provides corrosion resistance from purely mechanical considerations. Disruption of the original grain boundaries during the process of cold working leads to a discontinuous network of grain boundaries and hence a discontinuous network of potential corrosion paths.

The protection offered by a worked structure is probably quite limited in duration at temperatures of 2000° F and above, since in this temperature range Nb- and Ta-base alloys recrystallize in a relatively short time. For the Ta-10W capsule, failure by grain boundary penetration at the weld occurred so rapidly that there was not time for recrystallization of the worked structure. However, in the case of the SCb-291 alloy, corrosion attack was more moderate, and recrystallization had time to take place. As expected, the initially recrystallized weld zone was deeply penetrated, and the subsequently recrystallized portions of the capsule showed intergranular penetration to a lesser extent.

CONCLUSIONS

The results of the capsule screening tests indicate that the gettered Nb- and Ta-base alloys have excellent corrosion resistance to boiling potassium in the range of temperature and time required in the operation of an advanced space power system. The corrosion resistance of the gettered alloys is attributed to the reaction of the getter element with available oxygen to form a stable oxide and thereby preclude the formation and subsequent dissolution of a potassium - refractory-metal - oxygen corrosion intermediate. It was demonstrated that as long as the amount of getter is greater than the amount needed to tie up all the oxygen present, no significant corrosion will occur.

The negative findings in tests for the possible effect of stress on corrosion are encouraging, inasmuch as thermally and mechanically induced stresses are inherently present in power systems. None of the three Nb alloys tested, B-33, D-43, or FS-85, showed any stress corrosion effects.

The results of the Nb-1Zr loop test support the general findings of the capsule tests concerning the corrosion resistance of gettered alloys. This is all the more significant inasmuch as the loop test conditions were more severe than those imposed on capsule tests, namely, a much larger temperature gradient and ~10 times greater liquid flow velocity. Further, it seems likely, given the low elemental dissolution potential inferred from the gettered alloy capsule results, that the effect of temperature gradient or flow velocity will not be a major problem at temperatures below 2400° F.

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TABLE I. - COMPOSITION AND CONDITION OF CAPSULE MATERIALS

Alloy	Heat	Condition	Composition						ppm			
			Weight percent						C	O	N	H
			W	Zr	Hf	V	Nb	Ta				
SCb-291	--	Worked	8.37	----	----	----	Balance	9.69	14	101	119	30
B-33	1	Worked	----	0.02	----	4.88	↓	-----	64	142	52	17
B-33	2	Recrystallized	----	0.05	----	5.14	↓	-----	63	113	57	18
B-33	3	Worked	----	0.19	----	4.24	↓	-----	111	159	77	15
Nb-1Zr	1	Worked	----	0.91	----	----	↓	-----	81	200	75	8
Nb-1Zr	2	Recrystallized	----	1.29	----	----	↓	-----	40	144	28	5
D-43	--	Worked	9.4	1.0	----	----	↓	-----	870	56	44	4
FS-85	--	Worked	8.53	0.80	----	----	↓	26.95	58	148	41	10
Ta-10W	1	Worked	8.09	----	----	----	-------	Balance	41	48	9	15
Ta-10W	2	Recrystallized	9.70	----	----	----	-------	↓	33	37	37	4
T-111	--	Worked	7.87	----	2.31	----	-------	↓	37	25	13	33
T-222	--	Worked	8.45	----	2.31	----	-------	↓	141	48	18	4

TABLE II. - SUMMARY OF CAPSULE TEST RESULTS

Alloy (heat)	Temperature, °F	Time, h	Number capsules tested	Corrosive attack, (a)	Remarks (b)
SCb-291	1800	1,000	2	Ig, 1.5, W	P
				Ig, 6, W(inv.)	P
	2200	1,000	2	Ig, 16, L; Sol, C	R, D(Nb and W, by X-ray diffraction)
B-33(1)	2200	1,000	2(c)	Sol, 3.5, C	R, G
	2300	380	2(c)	None	R, G
B-33(2)	1800	2,000	2	None	----
	2200	2,000	1	Sol, 1, C	G
B-33(3)	1800	2,000	2	None	R
	2200	2,000	2	None	F, R, G
Nb-1Zr(1)	1800	2,000	2	None	G
Nb-1Zr(2)	2000	10,000	1(d)	None	G, Z, D(Zr, by Electron Beam Probe)
D-43	2200	1,000	2(c)	None	P
	2200	2,000	1	None	F, P
	2300	1,000	2(c)	None	F, P, Z
	2300	2,000	1	None	F, P, Z
	2000	10,000	1(d)	None	-------
FS-85	2200	4,000	2	None	F, R, Z
	2300	1,000	2(c)	None	F, R, Z
	2300	2,000	2	None	F, R, Z
	2400	2,000	1	Ig, <1, C and L	F, R, Z
Ta-10W(1)	1800	110	1	Ig, 16, W	-------
	2200	8	1	Ig, 16, W	-------
Ta-10W(2)	1800	128	1	Ig, 16, W	-------
	2200	8	1	Ig, 17, W(inv.)	-------
T-111	2200	4,000	1	None	F, R
	2300	2,000	2	None	F, R
	2400	2,000	1	Ig, <1, C and L	F, R
T-222	1800	4,000	1	None	----
	2200	4,000	1	None	R, F($K_2Ta_4O_{11}WO_3$, by X-ray diffraction)
	2400	4,000	1	Ig, 3,(e) L	R, Z, F($K_2Ta_4O_{11}WO_3$, by X-ray diffraction)

[a] Corrosion results are presented in the following order: (1) Type: Ig, intergranular; sol., solution. (2) Maximum depth in mils. (3) Location: C, vapor condensation region; L, liquid pool; B, region between C and L; W, weld. Note that normal capsule test position was with weld in region C. Some tests were run with capsule inverted, inv., so that weld was in region L.

[b] The following code is used: D, crystalline deposit at region B; F, film mainly in region B; G, accelerated grain growth, mainly in region C; P, partial recrystallization; R, complete recrystallization; Z, zone at inner edge, mainly in region C, deficient in second-phase precipitates.

[c] One capsule of pair tested for stress corrosion effects.

[d] Large capsule: 11 inches by 1.0-inch diameter by 0.08-inch wall. The initial oxygen content of the Nb-1Zr capsule was <50 ppm.

[e] Penetration was in one highly localized area of bottom corner; no other penetration was found.

TABLE III. - CORROSION AND Zr CONTENT OF B-33 ALLOYS AT 2200° F

Heat	Test time, h	Corrosion (a)	Zr_A, %	Zr_{th}, %	
				Case 1	Case 2
1	1000	Sol., 3.5, C	0.02	0.12	0.04
2	2000	Sol., 1, C	0.05	0.12	0.03
3	2000	None	0.19	0.18	0.05

[a] Corrosion results are presented in the following order: (1) Type: sol., solution. (2) Maximum depth in mils. (3) Location: C, vapor condensation region.

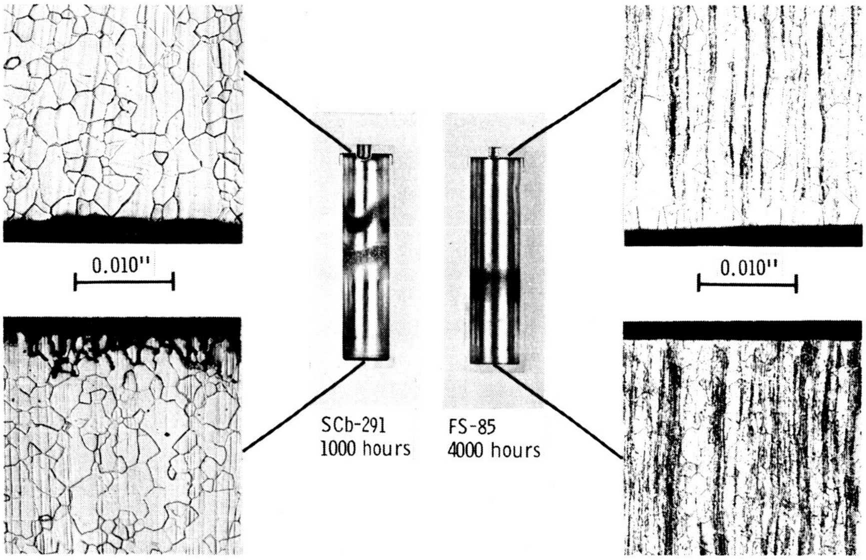


Figure 1. - Comparison of ungettered (SCb-291) and gettered (FS-85) niobium-base alloys tested at 2200° F.

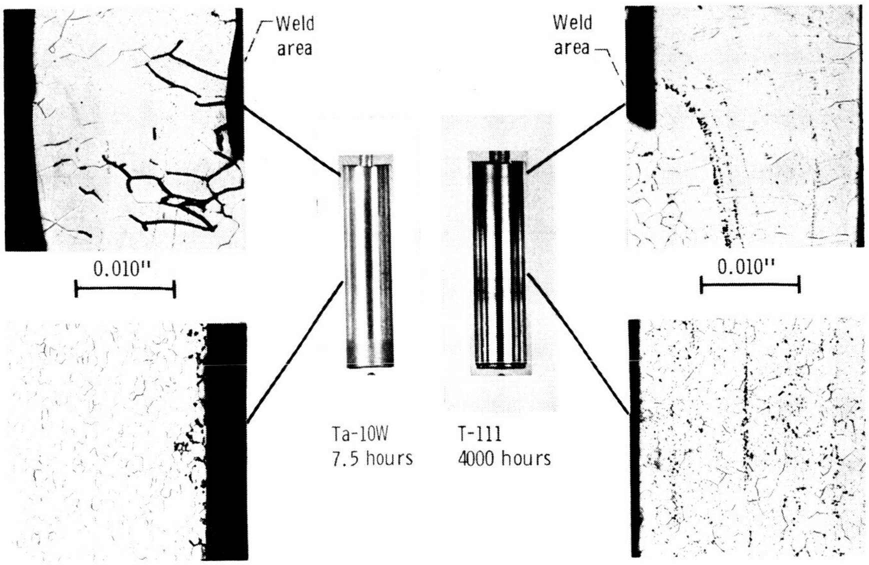
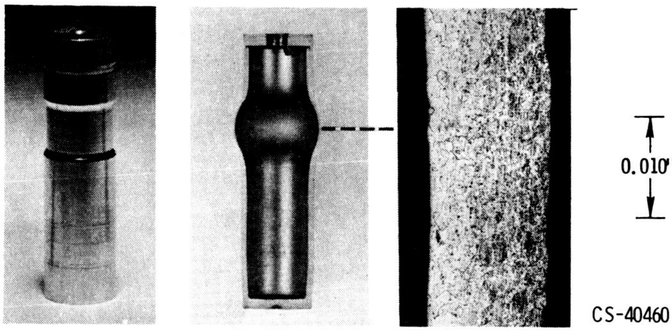


Figure 2. - Comparison on ungettered (Ta-10W) and gettered (T-111) tantalum-base alloys tested at 2200° F.



CS-40460

Before test

Sectioned capsule, post test

Photomicrograph of high-stressed regicn

Figure 3. - Test for stress corrosion in niobium alloy D-43 at 2300° F for 1000 hours.

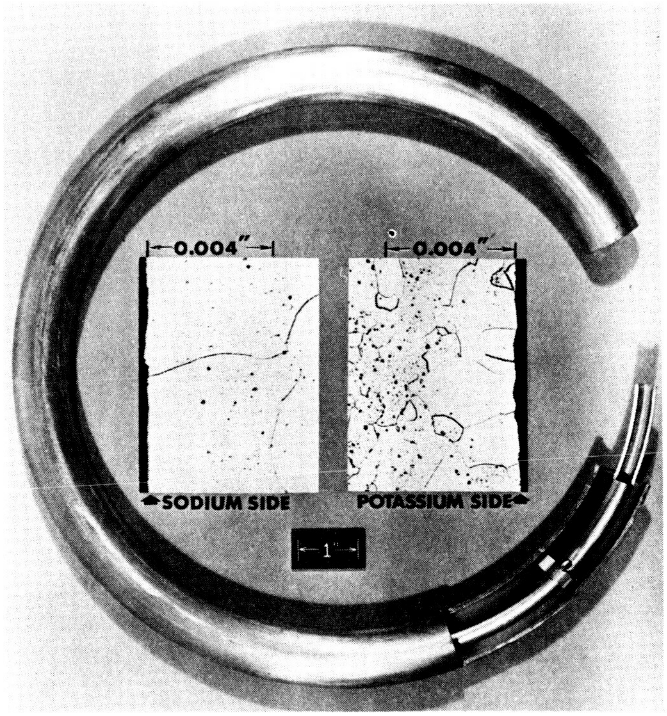


Figure 4. - Post-test view of Nb-1Zr tube-in-tube boiler.