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Evaluation of Nb-Base Alloys for the Divertor Structure in Fusion Reactors*

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ABSTRACT

Niobium-base alloys are candidate materials for the divertor structure in fusion reactors. For this application, the materials should exhibit resistance to aqueous corrosion, hydrogen embrittlement, and radiation damage and should have high thermal conductivity and low thermal expansion. Results of corrosion and embrittlement screening tests of several binary and ternary alloys in high-temperature water indicated that Nb-1Zr, Nb-5Mo-1Zr and Nb-5V-1Zr (wt.%) showed sufficient promise for further investigation. These alloys, together with pure Nb and Zircaloy-4, have been exposed to high-purity water containing a low concentration of dissolved oxygen (<12 ppb) at 170, 230, and 300°C for up to ≈3200 h. Weight-change data, microstructural observations, and qualitative mechanical-property evaluations reveal that Nb-5V-1Zr is the most promising alloy at higher temperatures. Below ≈200°C the alloys exhibit similar corrosion behavior.

INTRODUCTION

The corrosion behavior of several binary and ternary Nb-base alloys was investigated previously to identify the most promising materials for possible use as the divertor structure in a fusion reactor with an aqueous coolant [1-3]. In this study, corrosion tests were conducted on selected alloys in high-purity deoxygenated water at 170, 230, and 300°C.

EXPERIMENTAL PROCEDURE

Details of the experimental procedures were described previously [4]. Thin sheets of pure Nb and several Nb-base alloys were sheared to approximate dimensions of 8 x 20 mm. The Nb-5V-1Zr alloy, formerly designated "Cb 753" by Cabot Corporation, was obtained from two sources. The thickness of the Nb and one lot of Nb-5V-1Zr alloy was 0.5 mm; that of the other materials was 0.8 mm. Impurity levels in the two heats of Nb-5V-1Zr differed by a factor of ≈2. The composition of the materials is given in Table 1.

The surface area of each specimen was measured by an image analysis technique. The specimens were annealed *in vacuo* at 1000°C for 2 h prior to exposure. The exposures were conducted in refreshed autoclave systems at 170, 230, and

300°C (±2°C) with ≈100 psi overpressure (above saturation) at each temperature to maintain liquid-phase water. Corrosion tests at 300°C were performed in a Type 304 stainless steel autoclave; a Hastelloy C276 autoclave (62%Ni-16%Cr-16%Mo) was used in experiments at 170 and 230°C. The dissolved-oxygen concentration of the inlet water was maintained at <12 ppb. Each ≈120-day immersion experiment was interrupted at approximately 30, 60, and 90 days for removal of specimens. Weight changes of the specimens were determined to ±1 µg. Ductility changes in the specimens were evaluated by a bend test. Zircaloy-4 was included in later tests at 170 and 230°C to compare its behavior with that of Nb-base alloys.

EXPERIMENTAL RESULTS

Weight-gain versus time-of-exposure results for pure Nb, Nb-base alloys, and Zircaloy-4 are shown in Figs. 1-4. The data were fitted to the expression $W = A \cdot t^n$, where W is the weight gain in $g \cdot m^{-2}$, t is the exposure time in hours, and A and n are empirical fitting parameters. The values of n in the power-law expression vary from ≈0.3-0.7 and are denoted by the slopes of the lines in Figs. 1-4. Most of the materials gained weight at 230 and 300°C; however, weight changes for all specimens at 170°C were relatively small (<0.3 mg) and did not increase with exposure time for Nb and the Nb-base alloys.

The absence of a time dependence at 230 and 170°C in Figs. 3 and 4 can be attributed primarily to deposition of a corrosion product from the Hastelloy autoclave system on the specimens. The contribution of corrosion-product deposition to weight gain becomes less significant at 300°C (Figs. 1 and 2) because corrosion rates of the Nb-base alloys are higher by an order of magnitude and deposition may be less in a stainless steel system. Figure 5 shows energy-dispersive-electron spectra (EDS) obtained from the surface of Nb-1Zr specimens after exposure to water at 170°C for ≈1000 and 3200 h, and from a platinum wire that was used to suspend test specimens in the autoclave for ≈3200 h at 230°C. The heights of the Fe, Ni, and O peaks are similar for all specimens, which is a qualitative indication that levels of corrosion product contamination on the surface of all specimens were similar.

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Table 1. Composition of Nb and Nb-base alloys^a

Alloy	wt.%					ppm					
	Mo	V	Zr	O	N	C	H	Si	Al	Fe	Cr
Nb ₁	<0.01	—	<0.01	<50	31	<20	<3	<20	—	<40	—
Nb ₂	<0.01	<0.01	<0.01	176	42	200	11.4	340	70	65	<20
Nb-1Zr ^a	<0.01	<0.01	0.82	153	57	70	4.0	<100	65	<50	<20
Nb-5Mo-1Zr ^a	4.86	<0.01	0.81	186	56	70	8.5	<100	70	<50	<20
Nb-5V-1Zr ₁ ^a	<0.01	5.76	1.11	720	75	300	10.3	150	155	100	150
Nb-5V-1Zr ₂ ^a	<0.01	5.69	0.84	310	95	130	4.1	260	65	35	25

^aConcentrations of Hf, W, Ta, and Ti are ≤100 ppm; Mn, Cu, and Ni ≤50 ppm; and Mg ≤5 ppm.

Table 2. Composition of Zircaloy^a

Alloy	wt.%			ppm						
	Fe	Cr	Sn	O	N	C	H	Si	Al	Hf
Zircaloy-4	0.23	0.11	1.55	1044	25	145	8.5	54	47	41

^aConcentrations of Mn, Ti, and W are <25 ppm; Cu and Ni <35 ppm.

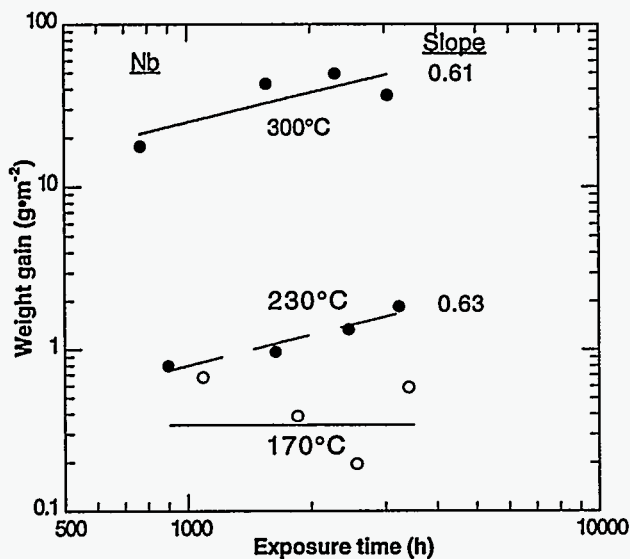


Figure 1. Weight gain versus time for Nb in high-purity, low-oxygen water at 170, 230, and 300°C

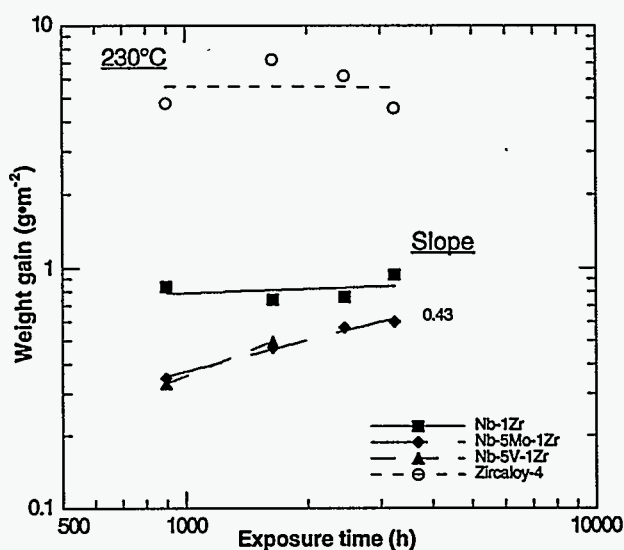


Figure 3. Weight gain versus time for Nb-base alloys and Zircaloy-4 in high-purity, low-oxygen water at 230°C

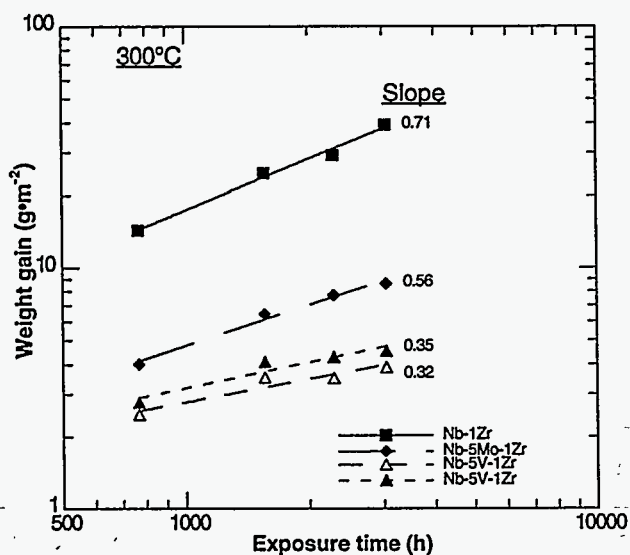


Figure 2. Weight gain versus time for Nb-base alloys in high-purity, low-oxygen water at 300°C

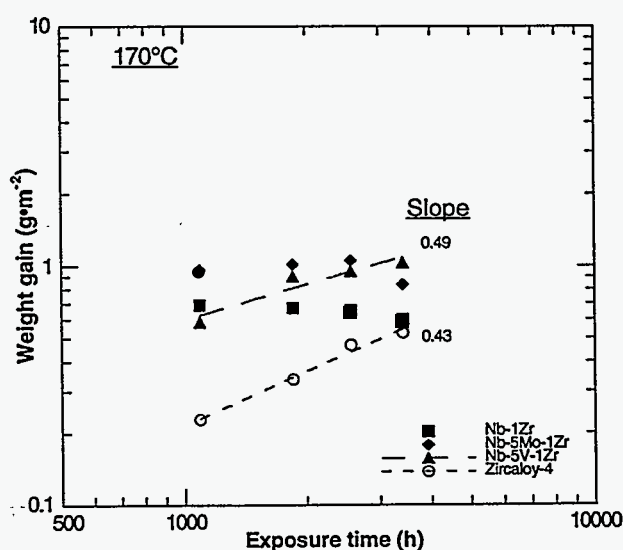
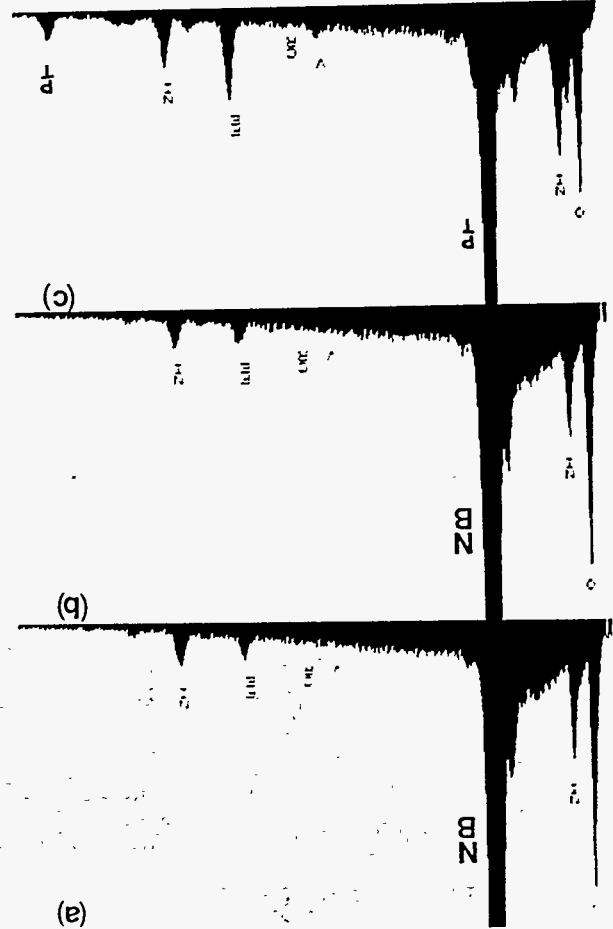


Figure 4. Weight gain versus time for Nb-base alloys and Zircaloy-4 in high-purity, low-oxygen water at 170°C

Metallographic sections of the specimens were prepared and examined by scanning electron microscopy (SEM). The SEM photomicrographs indicated that corrosion-product films were uniform in thickness across the specimens and tightly adherent to the underlying metal substrates. Cracks were observed in the oxide layer parallel to and just beneath the oxide/water interface in most of the specimens. Cracks in the oxide layer on Nb and Nb-1Zr specimens exposed to water at 300°C for >2000 h generated the underlying metal; however, the absence of corrosion on crack surfaces suggests that the cracks formed during metallographic preparation of the specimens (Fig. 6). EDS and wave length dispersive electron spectroscopy (WDS) were used to determine concentration profiles of Nb, V, and O, and Zr and Mo, respectively, across the specimens. Figure 7 shows line scans for Nb, Mo, Zr, and O in a Nb-5Mo-1Zr specimen after exposure to water at 300°C for ≈3200 h. X-ray diffraction results indicated that the corrosion product in this and other specimens is NbO but Nb₂O₅ also may be present. Alloying elements were incorporated into the corrosion-product layer as evidenced by no enrichment of Mo and Zr in the Nb-5Mo-1Zr alloy at the metal/oxide interface (Fig. 7). The weight-gain data at 300°C

Figure 5. EDS spectra for Nb, Fe, Cr, Ni, and O on the surface of Nb-1Zr specimens after (a) ≈1000 and (b) 3200 h at 170°C, and (c) on a Pt wire used to suspend specimens in a Hastelloy autoclave for ≈3200 h at 230°C

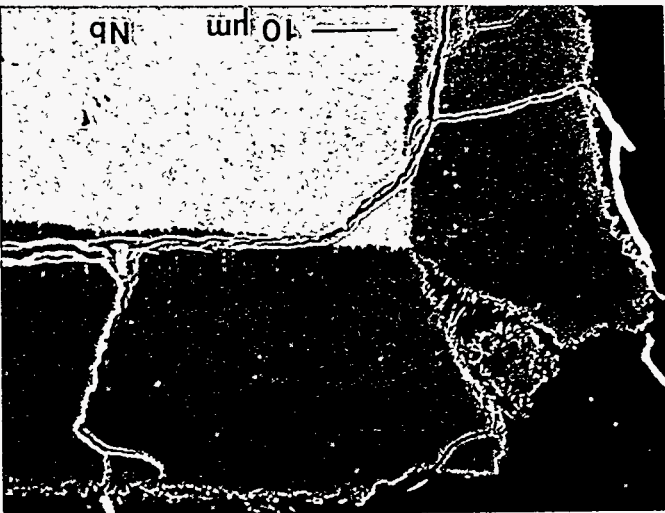


After a 30-day exposure at 300°C, none of the alloys fractured after a 180° bend and unfolding of a bend (FI=2); after 60- and 90-day exposures, Nb, Nb-5Mo-1Zr, and Nb-5V-1Zr specimens fractured during 180° bending (FI=4); and after a 120-day exposure, these materials fractured during a 90° bend (FI=5). Nb-1Zr specimens fractured during unbending (FI=3) after 90- and 120-day exposures at 300°C. At 230 and 170°C, the weight gains and film thicknesses for the alloys were relatively small and not dependent upon exposure time. Consequently, alloy ductility was independent of exposure time at these temperatures. Nb-1Zr showed the least ductility change, i.e., fracture occurred during unbending of the fold (FI=3) in specimens exposed to water at 230°C, whereas unbending did not produce fracture in specimens from corrosion tests at 170°C (FI=2). Nb-5Mo-1Zr and Nb-5V-1Zr specimens exposed to water at 230°C were somewhat less ductile and fractured during 180° bending (FI=4). Exposures at 170°C lead to less embrittlement; the Nb-1Zr, Nb-5V-1Zr, and Nb-5Mo-1Zr specimens exhibited FIs of 2, 3, and 4, respectively, after ≈120 days.

Bend tests were performed on the specimens at room temperature to detect changes in ductility after exposure to water. The results were characterized in terms of a fracture index (FI) that qualitatively indicates increasing degrees of embrittlement, i.e., an FI of 1 indicates neither fracture nor surface cracking at the conclusion of a 180° bend test; 2, fracture was not evident after unbending of the fold; 3, specimen fractured after unbending a fold; 4, fracture occurred after 180° of bending; 5, fracture at a ≈90° bend; 6, fracture or cracking was evident prior to the bend test [1].

Cracks in oxide layer on Nb after ≈3000 h at 300°C that formed during metallographic specimen preparation were consistent with measured film thicknesses based upon an NbO corrosion-product layer, which indicates that the corrosion product for the most part was retained on the specimens with minimal dissolution in water under low-flow-rate conditions (4 ml·min⁻¹) in these experiments.

Figure 6. Cracks in oxide layer on Nb after ≈3000 h at 300°C that formed during metallographic specimen preparation



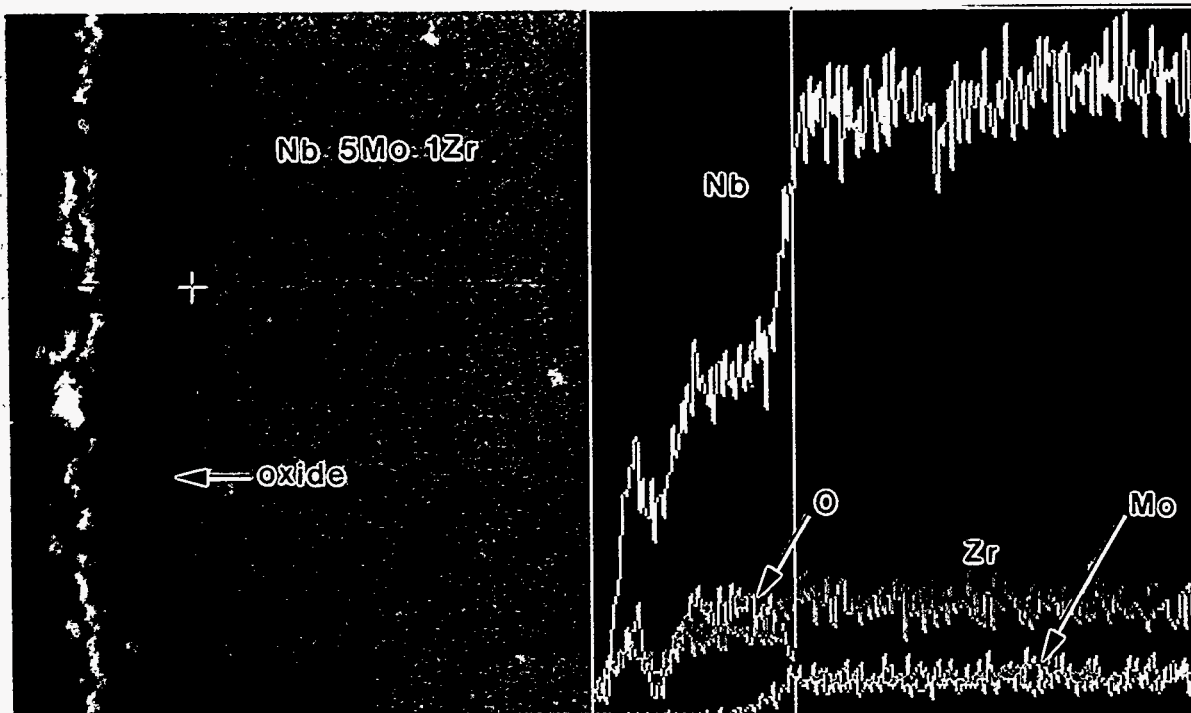


Figure 7. SEM photomicrograph of cross section of Nb-5Mo-1Zr specimen after ≈ 3000 h exposure to high-purity deoxygenated water at 300°C and EDS and WDS concentration profiles for Nb, Mo, Zr, and O across the specimen

CONCLUSIONS

The Nb-5V-1Zr and Nb-5Mo-1Zr alloys were more resistant to corrosion than pure Nb and Nb-1Zr at 230 and 300°C . Weight-gain data were consistent with measured thicknesses of NbO on the specimens, which indicates that dissolution of the oxide in water was not significant at a low flow rate in these experiments. At 170°C , corrosion of all materials in deoxygenated high-purity water was relatively low as indicated by weight-gain data and film thickness measurements. Deposition of corrosion products from the Hastelloy autoclave system contributed significantly to weight gain of the specimens at 170°C . At this temperature, the alloys could survive a 180° bend without fracture at room temperature, which indicates that embrittlement caused by pick up of hydrogen liberated by the corrosion reaction was minimal.

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