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CORROSION AND OXIDATION OF VANADIUM-BASE ALLOYS*

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The corrosion of several V-base alloys on exposure at elevated temperatures to helium environments containing hydrogen and/or water vapor are presented. These results are utilized to discuss the consequences of the selection of certain radiation-damage resistant, V-base alloys for structural materials applications in a fusion reactor.

1. INTRODUCTION

Vanadium-base alloys offer a potentially significant advantage over other candidate alloys for structural materials applications in fusion reactors. High-fluence neutron irradiations under LMFBR conditions and high-damage-level ion irradiations[1] have demonstrated an inherent resistance of certain V-base alloys, e.g., the V-15Cr-5Ti alloy, to void and cavity swelling. A major concern regarding the use of V alloys for this application is the chemical reactivity of the alloys at elevated temperatures with the constituents in air during an accidental exposure. The chemical reactivity of these alloys might result in unacceptable corrosion, oxidation, and embrittlement effects when exposed to even fairly low partial pressures of either oxygen, nitrogen, hydrogen or water vapor.

In this paper, the corrosion and the increase in weight of several V-base alloys on exposure at elevated temperatures to helium environments containing hydrogen and/or water vapor are presented. The increase in weight data for the V-base alloys are compared with the data determined for Path A-PCA austenitic and HT-9 ferritic steels in the same environments. The increase in weight data are utilized to discuss the consequences of selection of certain radiation-damage resistant, V-base alloys for structural materials applications in a fusion reactor.

2. MATERIALS AND PROCEDURE

The materials for this investigation were unalloyed V, V-5 Ti, V-15Cr, V-15Cr-5Ti, Path A-PCA (nominal Fe-16Ni-14Cr composition) steel, and HT-9 (nominal Fe-12Cr-0.5Ni composition) steel.+ Specimens of unalloyed V, V-5Ti and V-15Cr were prepared from V stock that contained ~150 ppm oxygen. Rolled sheets of these materials were annealed for one hour at 1475 K in a vacuum of ~10^{-6}Pa to produce a recrystallized structure with an average grain diameter of ~0.02 cm. The V-15Cr-5Ti alloy was obtained from the Fusion Materials Program Inventory (MFE Heat No. CAM 834-6) at Oak Ridge National Laboratory. Specimens of this material were also annealed for one hour at 1475 K. The Path A-PCA and HT-9 materials were supplied by the Fusion Materials Program Inventory from MFE Heats No. K-280 and No. XAA-3587, respectively. The Path A-PCA material was aged for 8 hours at 1075 K in argon, 23%-cold worked, and aged for 2 hours at 1025 K

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+The nominal compositions of the materials are expressed in weight percent of solute.

in argon. The HT-9 alloy was normalized for one hour at 1325 K in argon, air cooled from 1325 K, tempered for 2.5 hours at 1025 K, and air cooled from 1025 K.

Specimens of each of the materials with dimensions of 1 cm x 1 cm x 0.08 cm and a 0.003-cm surface finish were inserted in furnaces at temperatures of 725, 825 and 925 K. The specimens were exposed to flowing helium that contained either 10 ppm by volume of water or 1 ppm hydrogen and 1 ppm water for increasing periods of time ranging up to 1000 hours. The flow rate of the gas was 50 cc/hour. The increase in weight of the specimens was determined with an accuracy of 2 $\times 10^{-6}$g at time intervals of ~140 hours.

3. EXPERIMENTAL RESULTS

The increase in weight of a specimen on exposure to the He environments utilized in this study was accurately described (R^2 correlation coefficient > 0.9) by the equation

$$W = K(E)^n. \qquad (1)$$

In Eq. (1), W is the increase in weight per unit of surface area ($\mu g/cm^2$); E is the cumulative exposure time (hours); K and n are constants for a material and a set of experimental parameters. The values of the K and n for each of the materials and exposure conditions utilized in this study are listed in Table 1. An example of the experimental data obtained in this study is shown in Fig. 1.

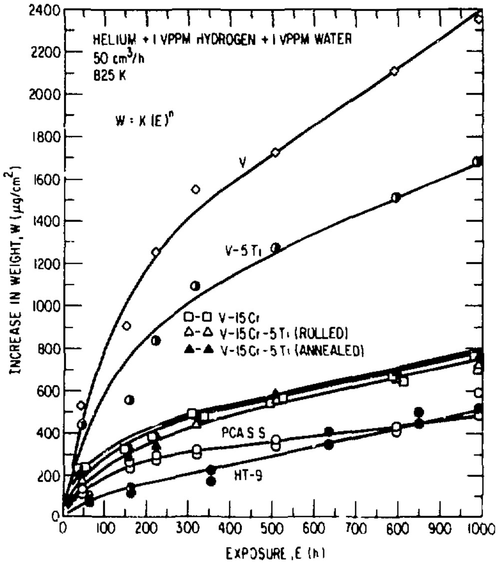

Fig. 1. Increase in weight of unalloyed V, V-5Ti, V-15Cr, V-15Cr-5Ti, PCA and HT-9 materials on exposure at 825 K to He + 1 ppm H_2 + 1 ppm H_2O.

The observation of the microstructure in cross-sections of the specimens after 1000-hour exposure to the He environments revealed, in general, contiguous surface and near-surface layers (Fig. 2). We shall denote the outermost layer as the "corrosion" layer and the near-surface layer as the "internal oxidation" layer. The thickness of these layers in the specimens are listed in Table 1. In the case of the V and V-5Ti specimens, the corrosion layer tended to spall with a maximum tendency at 825 K. In contrast to the corrosion layer

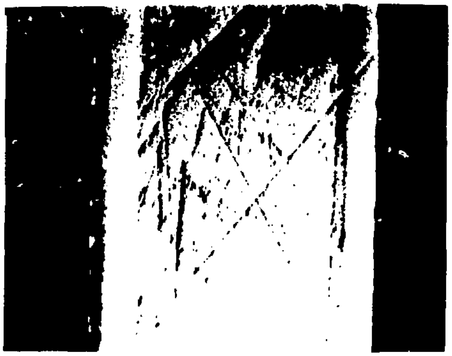

Fig. 2. Corrosion and diffusion layers in the V-15Cr-5Ti alloy after 1033-hour exposure at 925 K to He + 10ppm H_2O.

Table 1. Parameters for the increase in weight of V-base alloys, PCA stainless steel and HT-9 alloy at 725, 825, and 925 K in He.

Material	Gas (vppm)[a]	Temperature (K)	Eq.(1) Parameters		Layer Thickness at 1000 Hours		Rate of Weight Change[b] ($\mu g/cm^2 \cdot hr$)
			K ($\mu g/cm^2 \cdot hr$)	n	Corrosion (10^{-4}cm)	Oxidation (10^{-4}cm)	
V	He+10H_2O	725	19.23	0.54	11[c]	180	0.504
V-5Ti	"	"	8.62	0.59	13[c]	35	0.344
V-15Cr	"	"	14.32	0.39	6	~10	0.106
V-15Cr-5Ti[d]	"	"	6.37	0.55	6	20	0.186
V-15Cr-5Ti[e]	"	"	7.57	0.49	6	20	0.131
PCA	"	"	0.13	0.55	3	n.v.[f]	0.004
V	He+10H_2O	825	32.90	0.76	64[c]	280	5.075
V-5Ti	"	"	22.42	0.75	31[c]	75	3.325
V-15Cr	"	"	4.94	0.83	14	~20	1.374
V-15Cr-5Ti[d]	"	"	6.65	0.81	18	28	1.549
V-15Cr-5Ti[e]	"	"	8.40	0.75	16	36	1.227
PCA	"	"	31.36	0.28	8	n.v.	0.082
V	He+10H_2O	925	96.90	0.66	16[c]	> 400	7.104
V-5Ti	"	"	24.29	0.77	9[c]	220	4.180
V-15Cr	"	"	18.17	0.72	17	~15	2.146
V-15Cr-5Ti[d]	"	"	10.15	0.84	29	110	3.051
V-15Cr-5Ti[e]	"	"	12.92	0.75	18	128	1.902
PCA	"	"	18.32	0.37	9	n.v.	0.118
HT-9	"	"	11.20	0.26	5	n.v.	0.024
V	He+1H_2+1H_2O	825	76.67	0.50	25[c]	200	1.533
V-5Ti	"	"	66.12	0.47	27[c]	60	0.971
V-15Cr	"	"	51.23	0.38	10	~15	0.349
V-15Cr-5Ti[d]	"	"	42.20	0.42	11	60	0.397
V-15Cr-5Ti[e]	"	"	29.21	0.47	12	50	0.426
PCA	"	"	24.47	0.43	11	n.v.	0.267
HT-9	"	"	4.65	0.68	10	n.v.	0.319

(a) Volume parts per million of H_2 or H_2O in He.
(b) 400 to 1000 hours exposure.
(c) Layer tends to spall.
(d) As-received, rolled material.
(e) Annealed material.
(f) Not visible.

on the V and V-5Ti specimens, the corrosion layer on the V-15Cr and V-15Cr-5Ti materials did not show a tendency to spall for these experimental conditions. The near surface, internal oxidation layer was readily distinguishable in the microstructure of the V-5Ti and V-15Cr-5Ti specimens and generally less apparent in the microstructure of the V and V-15Cr specimens. The response of the diffusion layer to abrasion during the preparation of the specimen cross-sections for microstructural observation clearly indicated that the internal oxidation layer was extremely hard (Fig. 2).

The thickness of the corrosion layer on the V-15Cr, V-15Cr-5Ti and the PCA materials approached a limiting value for temperatures above 825 K (Table 1). However in the case of the V and V-5Ti materials, the corrosion layer thickness attained a maximum value at

825 K. The thickness of the internal oxidation layer in the V and V-base mateials increased substantially with an increase of temperature.

The increase in weight of the specimens for exposure times beyond 400 hours obtained a nearly constant rate. For a comparison of the increase in weight for the unalloyed V, V-base alloys and Fe-base alloys, the approximately linear rate of weight change (400 to 1000 hours) for these materials is presented in Table 1.

4. DISCUSSION OF RESULTS

The data obtained in this experimental study on the increase in weight of unalloyed V, V-base alloys and Fe-base alloys during exposure at temperatures ranging from 725 to 925 K in He environments containing H_2 (1 ppm) and/or H_2O (1-10 ppm) suggest the following conclusions.

1. The presence of Cr in a V alloy is beneficial for a minimum increase in weight.
2. The presence of Ti in a V alloy is not necessary for a minimum increase in weight.
3. The presence of Ti in a V alloy may result in unacceptable mechanical properties because of formation of an internal oxidation layer.
4. The rate of increase in weight (400 to 1000 hours) for the V-base alloys is substantially greater than the rate for the Fe-base alloys on exposure to He environments containing 10 ppm H_2O.
5. The rate of increase in weight for the V-base alloys is comparable to the rate for Fe-base alloys on exposure to He environments containing 1 ppm H_2 and 1 ppm H_2O.

The swelling of unalloyed V[2-4] and V-base alloys[1,2,5] by the formation of voids at ~975 K during Ni^+- or V^+-ion irradiation has been investigated for displacement damage levels up to ~60 dpa. These investigations have shown that the addition of 15 w/o Cr to V causes a substantial increase of the V swelling, and, therefore, exacerbates the swelling resistance of V.[5] However, the results of the present study show that Cr is a beneficial solute for improving the corrosion resistance of V in He containing low (< 10 ppm) concentrations of water vapor. The swelling studies have also shown that the addition of 1 w/o Ti to V or 5 w/o Ti to a V-15Cr alloy effectively suppresses void formation and growth.[1,2] The present study has shown that the addition of 5 w/o Ti to a V-15Cr alloy does not significantly alter the corrosion resistance of the V-15Cr alloy. Moreover, the presence of the Ti solute in unalloyed V or a V-15Cr alloy causes the formation of an internal oxidation layer that may result in embrittlement of the material. Loomis and Gerber[6] have investigated the effects of substitutional-atom solutes on the void swelling of Nb. These studies revealed that Ti solute was effective in minimizing void swelling. However, the most effective solutes for suppression of void swelling in Nb were solutes that diffuse relatively slowly in Nb. These results suggest that slowly diffusing solutes in V might be equally effective for the suppression of void swelling, and thus the deleterious internal oxidation effects of the Ti solute might be avoided. The slowly diffusing solutes which might be effective in the suppression of swelling of V are Nb, Ta, Mo and W.[7] However, the impact of these solutes on the relatively low induced radioactivity of V will need to be considered.[8]

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3. EXPERIMENTAL RESULTS

The increase in weight of a specimen on exposure to the He environments utilized in this study was accurately described (R^2 correlation coefficient > 0.9) by the equation

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In Eq. (1), W is the increase in weight per unit of surface area ($\mu g/cm^2$); E is the cumulative exposure time (hours); K and n are constants for a material and a set of experimental parameters. The values of the K and n for each of the materials and exposure conditions utilized in this study are listed in Table 1. An example of the experimental data obtained in this study is shown in Fig. 1.

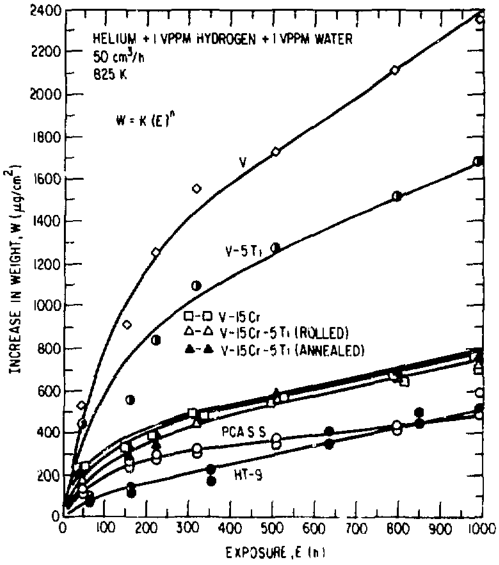
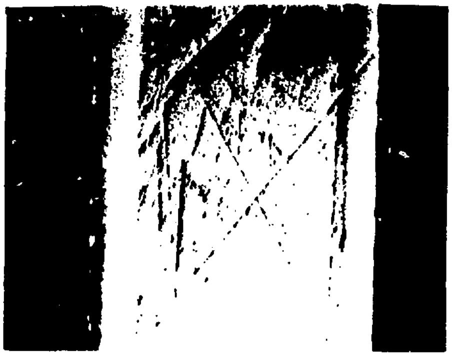


Fig. 1. Increase in weight of unalloyed V, V-5Ti, V-15Cr, V-15Cr-5Ti, PCA and HT-9 materials on exposure at 825 K to He + 1 ppm H_2 + 1 ppm H_2O.

The observation of the microstructure in cross-sections of the specimens after 1000-hour exposure to the He environments revealed, in general, contiguous surface and near-surface layers (Fig. 2). We shall denote the outermost layer as the "corrosion" layer and the near-surface layer as the "internal oxidation" layer. The thickness of these layers in the specimens are listed in Table 1. In the case of the V and V-5Ti specimens, the corrosion layer tended to spall with a maximum tendency at 825 K. In contrast to the corrosion layer



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2. The presence of Ti in a V alloy is not necessary for a minimum increase in weight.
3. The presence of Ti in a V alloy may result in unacceptable mechanical properties because of formation of an internal oxidation layer.
4. The rate of increase in weight (400 to 1000 hours) for the V-base alloys is substantially greater than the rate for the Fe-base alloys on exposure to He environments containing 10 ppm H_2O.
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