TENSILE PROPERTIES OF V-Cr-Ti ALLOYS AFTER EXPOSURE IN OXYGEN-CONTAINING ENVIRONMENTS*

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OBJECTIVE

The objectives of this task are to (a) determine the oxygen uptake of V-Cr-Ti alloys as a function of temperature and oxygen partial pressure (pO_2) in the exposure environment, (b) examine the microstructural characteristics of oxide scales and oxygen trapped at the grain boundaries in the substrate alloys, and (c) evaluate the influence of oxygen uptake in low-pO_2 environments (which include oxygen and helium of various purities) on the tensile properties and cracking propensity of the alloys at room and elevated temperatures.

SUMMARY

A systematic study was conducted to evaluate the oxidation kinetics of V-4Cr-4Ti ("44 alloy") and V-5Cr-5Ti alloys ("55 alloy") and to establish the role of oxygen ingress on the tensile behavior of the alloys at room temperature and at 500°C. The oxidation rate of the 44 alloy is slightly higher than that of the 55 alloy. The oxidation process followed parabolic kinetics. Maximum engineering stress for 55 alloy increased with an increase in oxidation time at 500°C. The maximum stress values for 55 alloy were higher at room temperature than at 500°C for the same oxidation treatment. Maximum engineering stresses for 44 alloy were substantially lower than those for 55 alloy in the same oxidation treatment. Uniform and total elongation values for 55 alloy were almost zero at room temperature after ≈500 h exposure in air at 500°C; the same values were 4.8 and 6.1%, respectively, at 500°C after ≈2060 h oxidation in air at 500°C Uniform and total elongation values for 44 alloy were 1.6 and 3.6% at 500°C after 2060 h oxidation in air at 500°C.

Maximum engineering stress for 44 alloy at room temperature was 421.6-440.6 MPa after ≈250 h exposure at 500°C in environments with a pO_2 range of 1 x 10^{-6} to 760 torr. The corresponding uniform and total elongation values were 11-14.4% and 14.5-21.7%, respectively. Measurements of crack depths in various specimens showed that depth is independent of pO_2 in the preexposure environment and was of 70-95 μm after 250-275 h exposure at 500°C.

EXPERIMENTAL PROGRAM

The heats of vanadium alloy selected for the study had nominal compositions of V-5 wt.%Cr-5 wt.%Ti (designated BL-63) and V-4 wt.%Cr-4 wt.%Ti (designated BL-71). Sheets of the alloys were annealed for 1 h at 1050°C prior to oxidation and tensile testing. Coupon specimens that measured ≈15 x 7.5 x 1 mm were used for the oxidation studies. Oxidation experiments were conducted in air in a thermogravimetric test apparatus at temperatures of 300 to 650°C; results were discussed in earlier publications (1,2).

Tensile specimens were fabricated according to ASTM Standard E8-69 specifications and had a gauge length of ≈19 mm and a gauge width of ≈4.5 mm. Grain sizes of the V-4Cr-4Ti and V-5Cr-5Ti specimens were ≈18 and 32 μm, respectively. Tensile samples of the two alloys were exposed to several environments with pO_2 of 1 x 10^{-6} to 760 torr and to 99.999% pure He for 250-275 h at 500°C; they were subsequently tensile-tested at a strain rate of 1.8 x 10^{-4} s^{-1} in air at room temperature or 500°C. The specimens were loaded by means of pins that pass through holes in the grips and enlarged end sections of the specimen, thus minimizing misalignment. Total elongation was measured with a vernier caliper and load/elongation chart records. The fracture surfaces and longitudinal and axial cross sections of tested specimens were examined by scanning electron microscopy (SEM). Oxide scales on the samples were identified by X-ray diffraction (XRD) analysis on the surface of several samples, as well as on the oxides scraped from their surfaces. In addition, the Vickers hardness of several tested specimens was determined.

RESULTS AND DISCUSSION

To evaluate the effect of oxide scale formation and oxygen penetration into the substrate alloy, the tensile behavior of the alloys was examined as a function of oxygen ingress and oxide scale formation after exposure. Tensile-test data were reported earlier for the 55 alloy specimens exposed to air for 24-2060 h at 500°C and then tensile-tested in air at either room temperature or 500°C [1,3]. Similar exposures to air were made for tensile specimens of 44 alloy that were tensile-tested at 500°C in air.

Figure 1 shows the engineering stress/engineering strain curves at 500°C for specimens after oxidation for several exposure times in the range of 0-2060 h. The maximum engineering stress, uniform elongation, and total elongation for specimens with various treatments are listed in Table 1. The data indicate that the stress/strain behavior of both alloys is virtually unaffected by 24 h exposure in air at 500°C. As the exposure time increases to ≈250 h, the strength of the 55 alloy (but not that of the 44 alloy) increases but with some loss in tensile ductility in both alloys. The ductility reduction continues as the exposure time increases further to 600, ≈1000, and 2060 h at 500°C. Further exposure of the alloy to air at 500°C results in loss of strength and tensile ductility, as evidenced by the stress/strain curve for both alloys preoxidized for 2060 h. Similar data were obtained for the 55 alloy specimens preoxidized at 500°C in air and tensile-tested at room temperature. The results from these tests are also listed in Table 1.

Table 1. Effects of oxidation in air on tensile properties of 44 and 55 alloys

Exposure time (h)	V-5 wt.% Cr-5 wt.% Ti alloy								V-4 wt.% Cr-4 wt.% Ti alloy							
	Maximum engg. stress (MPa)		Uniform elongation		Total elongation		Measured crack length (µm)		Maximum engg. stress (MPa)		Uniform elongation		Total elongation		Measured crack length (µm)	
	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C
0	468.7	411.3	0.165	0.164	0.303	0.216	0	10	423.7	378.7	0.186	0.107	0.322	0.179	0	6.7
24	509.5	416.7	0.153	0.136	0.241	0.186	24	22	-	360.5	-	0.103	-	0.186	-	17
250[b]	513.9	458.5	0.035	0.113	0.035	0.145	d	50	-	367.2	-	0.099	-	0.135	-	85
600	471.4	453.3	0.003	0.095	0.003	0.114	d	90	-	362.4	-	0.082	-	0.108	-	93
1000[b]	483.9	454.4	0.001	0.085	0.001	0.104	d	110	-	340.6	-	0.059	-	0.075	-	137
2060[c]	433.6	406.4	0.002	0.048	0.002	0.061	d	160	-	329.2	-	0.016	-	0.036	-	210

[a]RT = room temperature.
[b]Exposure times were 260 and 1050 h for 55 samples tested at room temperature and for 44 samples tested at 500°C.
[c]Exposure time was 2110 h for 55 sample tested at room temperature.
[d]Specimen fully embrittled.

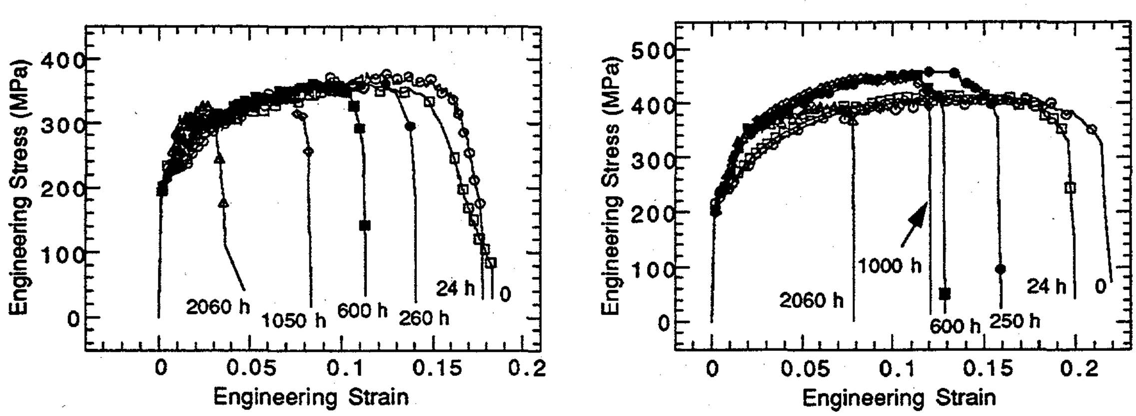

Figure 1. Effect of preoxidation at 500°C on stress-strain behavior of 44 (left) and 55 (right) alloys tested at 500°C in air at strain rate of 1.75×10^{-4} s^{-1}.

A comparison of the tensile properties of the 44 and 55 alloys shows that for the same pretreatment, the 55 alloy exhibits 10 to 25% higher ultimate tensile strength at both 500°C and at room temperature. For the same pretreatment, the uniform and total elongation values of the 44 alloys were lower than those of the 55 alloy. For example, the uniform elongation values at 500°C were 0.048 and 0.016 for 55 and 44 alloy specimens after 2060-h exposure to air at 500°C. The effect of oxidation on elongation values at room temperature for 55 alloy is even more substantial in that the specimens were fully embrittled. Even though corresponding tests were not conducted at room temperature for the 44 alloy oxidized in air, similar behavior is expected on the basis of the similarities in the oxidation characteristics of both alloys.

A test program is underway to evaluate the effect of partial pressure of oxygen in the exposure environment on oxygen ingress into the V alloys and its effect on tensile properties. Specimens of 44 alloy, initially in annealed condition, were exposed for 250-275 h at 500°C in environments with various pO_2 levels and subsequently tensile-tested at either room temperature or 500°C. Figure 2 shows the engineering stress/engineering strain curves at 500°C and at room temperature for 44 alloy specimens after oxidation for 250-275 h in environments with pO_2 of 1×10^{-6} to 760 torr. The maximum engineering stress, uniform elongation, and total elongation for specimens with various treatments are listed in Table 2. The exposure environments included pO_2 values of 760, 160, 0.15, 0.1, 7.6×10^{-4}, and 1×10^{-6} torr. Among these, 160 and 0.15 torr correspond to air and 99.999% He environments, respectively.

The stress/strain curves indicate that for a given exposure time and test temperature (500°C or room temperature), the pO_2 value in the preexposure environment has very little effect on maximum engineering stress and uniform and total elongation. The results also indicate that the alloy with identical pretreatment exhibits lower values of maximum engineering stress and uniform and total elongation at 500°C than those at room temperature. For example, the maximum engineering stress values for He-exposed specimens were 437.8 and 358.8 MPa at room temperature and 500°C, respectively. The corresponding uniform elongation values were 0.140 and 0.089 while the total elongation values were 0.191 and 0.119. A similar trend was observed in specimens exposed to pure oxygen (see Table 2).

Figure 3 shows the variations in maximum engineering stress and uniform and total elongation as a function of pO_2 value in the preexposure environment for 44 and 55 alloy specimens tested at room temperature and at 500°C. The plots indicate that the pO_2 value in the preexposure environment has virtually no effect on the any of the tensile properties reported here.

Table 2. Effects of oxygen partial pressure on tensile properties of 44 alloy

pO_2 in exposure environment (torr)	Maximum engg. stress (MPa)		Uniform elongation		Total elongation		Measured crack length (μm)	
	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C	RT[a]	500°C
1×10^{-6}	426.3	-	0.127	-	0.217	-	80	-
7.6×10^{-4}	443.0	-	0.110	-	0.145	-	90	-
0.1	440.6	-	0.133	-	0.202	-	95	-
0.15 (He)	437.8	358.8	0.140	0.089	0.191	0.119	80	70
160	-	367.2	-	.099	-	0.135	-	85
760	421.6	347.4	0.148	0.097	0.208	0.135	80	80

[a]RT = room temperature.

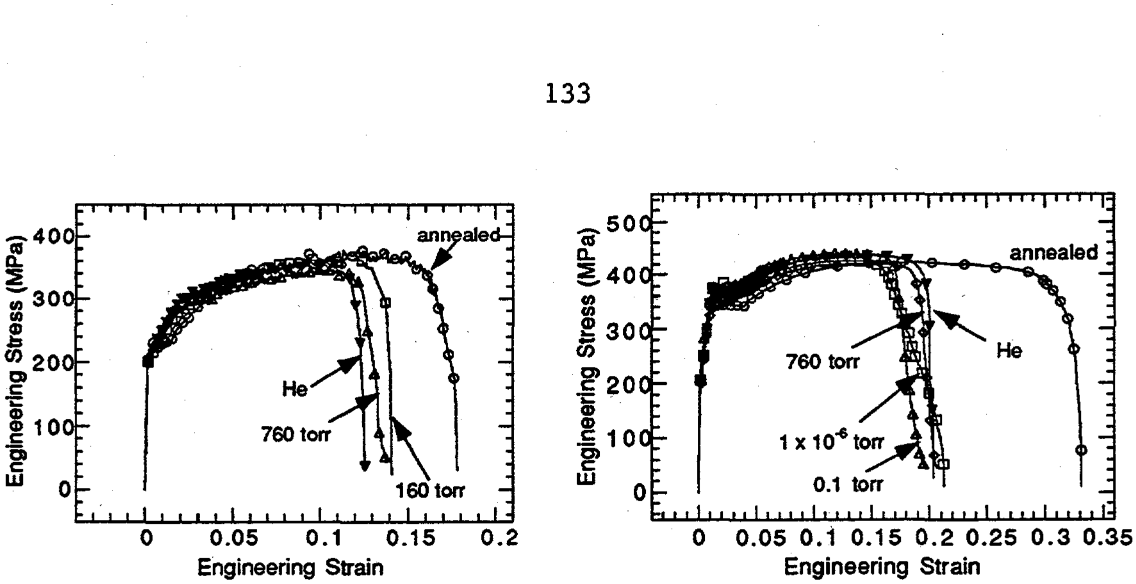

Figure 2. Effect of oxygen partial pressure in preexposure environment on stress-strain behavior of 44 alloy oxidized at 500°C for 250-275 h and tested at 500°C (left) and room temperature (right) in air at strain rate of $1.75 \times 10^{-4}\ s^{-1}$.

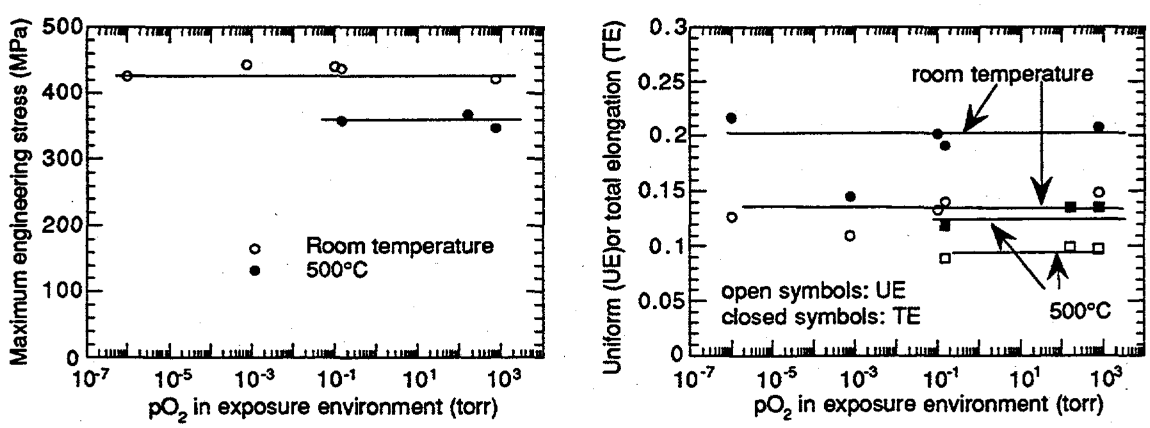

Figure 3. Effect of oxygen partial pressure in preexposure environment on maximum engineering stress (left) and uniform and total elongation (right) for 44 alloy oxidized at 500°C for 250-275 h and tested at room temperature and at 500°C at strain rate of $1.8 \times 10^{-4}\ s^{-1}$.

Microstructural Observations

Axial cross sections of several of the tested specimens were examined by SEM. Figure 4 shows 44 alloy specimen sections tested in as-annealed condition and after oxidation in air for 24, 260, 600, 1050, and 2060 h in air at 500°C. The photomicrographs show that as oxidation time increases, both the cracks in the transverse direction and the crack spacing in the axial direction increase. Furthermore, as the oxidation time increases, the specimen undergoes little necking of the gage section during the tensile test. It is evident, especially from specimens exposed for 1000 and 2060 h, that fracture occurred by propagation of one of the axial cracks and that because the core of the alloy was somewhat ductile, the crack-propagation direction in the core region was at an angle of ≈45°. Similar observations were reported earlier for 55 alloy specimens exposed to air for different time periods [3,4].

Figure 5 shows 44 alloy specimen sections tested in as-annealed condition and after oxidation at 500°C in environments with pO_2 values of 1×10^{-6}, 7.6×10^{-4}, 0.1, and 760 torr, and in 99.999% He at a pO_2 value of 0.15 torr. Exposure time for specimens in 760 torr O_2 and He was 275 h, while for the others it was 250 h. The photomicrographs show that transverse cracking is observed in all except the annealed specimens and that the crack spacing was almost the same except for the specimen exposed to pure oxygen (760 torr). The photomicrographs also indicate that the specimen surfaces

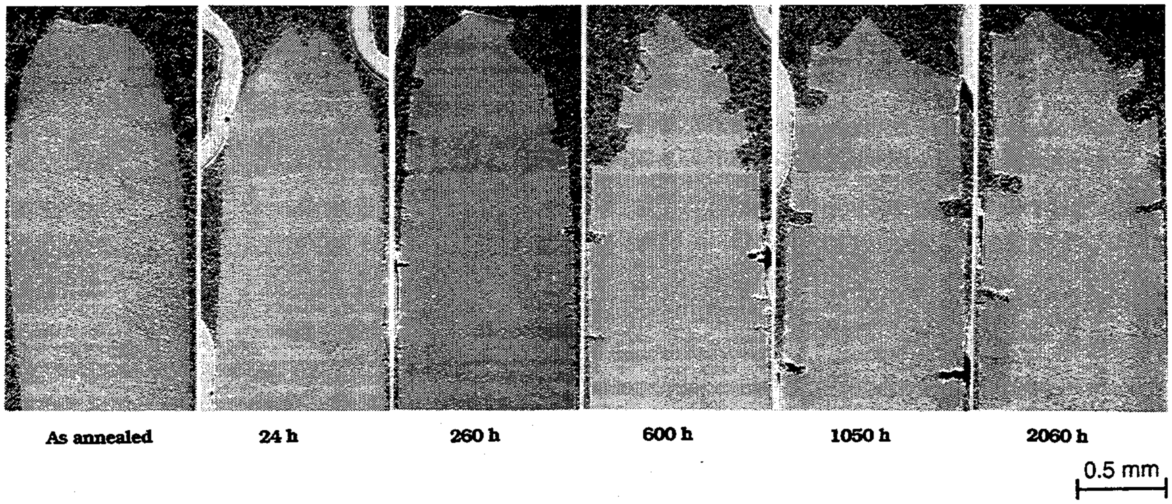

Figure 4. Scanning electron photomicrographs of axial sections of V-4Cr-4Ti specimens tensile-tested at 500°C in as-annealed condition and after oxidation in air at 500°C for several exposure times.

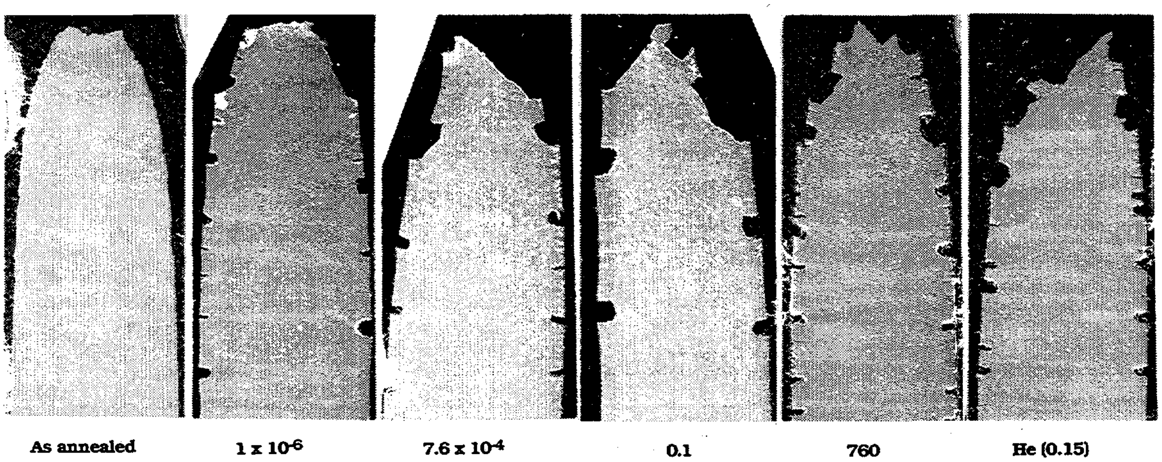

Figure 5. Scanning electron photomicrographs of axial sections of V-4Cr-4Ti specimens tensile-tested at room temperature in as-annealed condition and after oxidation at 500°C in environments with several oxygen partial pressures. Numbers in photographs are pO_2 values in torr.

equilibrate with oxygen in the environment and the increased oxygen content at the surface diffuses into the alloy with time. Even at the lowest pO_2 value of 1×10^{-6} torr, the oxygen concentration at the surface must be high enough to initiate cracks on axial loading of the specimen. The depth of the crack is determined by oxygen diffusion, which in turn is dictated by the time and temperature of preexposure.

Figure 6 shows a plot of measured crack depths for specimens exposed to environments with different pO_2 values. Over the entire range of pO_2 values used in this study, ≈250 h exposure at 500°C resulted in crack depths of 70-95 μm. These results, even though obtained from a relatively short exposure time of ≈250 h, indicate similar cracking propensity for 44 and 55 alloys at room temperature and at 500°C, and this propensity is independent of oxygen pressure in the exposure environment. This observation, coupled with embrittlement of the alloys after ≈2000 h exposure in air (discussed earlier), makes one infer that the alloys can embrittle even in environments with low pO_2 values, if exposed for times comparable to those of the air exposures conducted in this program. The ongoing experiments will examine this issue and develop models for life prediction of alloys as functions of time, temperature, and oxygen level.

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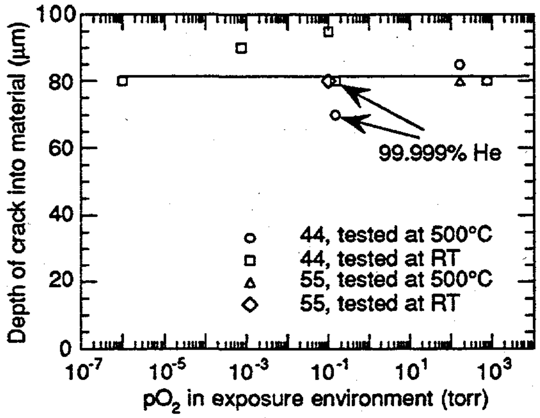

Figure 6. Measured crack depths in 44 and 55 alloy specimens after exposure for 250-275 h in various oxygen-containing environments.