EFFECT OF OXYGEN AND OXIDATION ON TENSILE BEHAVIOR OF V-5Cr-5Ti*

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OBJECTIVE

The objectives of this task are to (a) evaluate the oxygen uptake of V-Cr-Ti alloys as a function of temperature and oxygen partial pressure in the exposure environment, (b) examine the microstructural characteristics of oxide scales and oxygen entrapped at the grain boundaries in the substrate alloys, (c) evaluate the influence of oxygen uptake on the tensile properties of the alloys at room and elevated temperatures, (d) evaluate oxidation kinetics of the alloys with aluminum-enriched surface layers, and (e) determine the effect of oxygen uptake on the tensile behavior of the alloys.

SUMMARY

Oxidation studies were conducted on V-5Cr-5Ti alloy specimens in an air environment to evaluate the oxygen uptake behavior of the alloy as a function of temperature and exposure time. The oxidation rates calculated from parabolic kinetic measurements of thermogravimetric testing and confirmed by microscopic analyses of cross sections of exposed specimens were 5, 17, and 27 μm per year after exposure at 300, 400, and 500°C, respectively. Uniaxial tensile tests were conducted at room temperature and at 500°C on preoxidized specimens of the alloy to examine the effects of oxidation and oxygen migration on tensile strength and ductility. Microstructural characteristics of several of the tested specimens were determined by electron optics techniques. Correlations were developed between tensile strength and ductility of the oxidized alloy and microstructural characteristics such as oxide thickness, depth of hardened layer, depth of intergranular fracture zone, and transverse crack length.

EXPERIMENTAL PROGRAM

The heat of vanadium alloy selected for the study had a nominal composition of V-5 wt.%Cr -5 wt.%Ti and was designated as BL-63. A sheet material of the alloy was annealed for 1 h at 1050°C prior to its use in oxidation and tensile testing. Coupon specimens measuring ≈15 x 7.5 x 1 mm were used for the oxidation studies. Oxidation experiments were conducted in air in a thermogravimetric test apparatus. The test temperatures ranged between 300 and 650°C.

Tensile specimens were fabricated according to ASTM specifications and had a gauge length of ≈19 mm and a gauge width of ≈4.5 mm. The grain size of the specimens was ≈32 μm. The specimens were preoxidized in air at 500°C for 24, 250, 600, 1000, and 2060 h prior to tensile testing in air at 500°C. Similar specimens preoxidized up to 1000 h at 500°C in air were also tensile tested at room temperature. As-annealed (control) specimens were tensile tested on an Instron machine at constant crosshead speeds between 0.0005 and 0.2 cm/min. These speeds correspond to initial strain rates in the range of 4.4×10^{-6} to $1.8 \times 10^{-3}\ s^{-1}$. The preoxidized specimens were tested at a strain rate of $1.8 \times 10^{-4}\ s^{-1}$. The test temperature was maintained within 2°C in all tests performed in air at 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 by using load/elongation chart records. The fracture surfaces and longitudinal and axial cross sections of tested specimens were examined by scanning electron microscopy (SEM). In addition, Vickers hardness was measured on several of the tested specimens. Coupon specimens of the alloy that were oxidized with the tensile specimens were analyzed for bulk oxygen content by a vacuum-fusion technique.

RESULTS AND DISCUSSION

Oxidation Behavior

Oxidation of the alloy followed parabolic kinetics with time. Detailed SEM analysis (with both energy-dispersive and wavelength-dispersive analysis) of the oxidized samples showed that the outer layer was predominantly vanadium-rich oxide and the inner layer was (V,Ti) oxide. Further, X-ray diffraction of the

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oxides showed the outer oxide to be V_2O_5; no nitrogen or nitride phases were detected. The thickness of the oxide scale of the specimens calculated from a parabolic rate equation was in good agreement with the values determined by microscopy. The results were discussed in earlier reports (1, 2). Secondary ion mass spectrometry is presently used to obtain depth profiles for oxygen in the tested specimens.

Effect of Oxidation on Tensile Properties

To evaluate the effect of oxide scale formation and oxygen penetration into the substrate alloy, tensile behavior of the alloy was examined as a function of oxygen ingress and oxide scale formation. Specimens were exposed to air for 24-2060 h in air at 500°C and then tensile tested in air at either room temperature or 500°C at a strain rate of $1.8 \times 10^{-4}\ s^{-1}$.

Figure 1 shows the engineering stress/engineering strain curves at 500°C for specimens after oxidation for several exposure times up to 2060 h. Stress/strain behavior of the alloy is virtually unaffected by 24 h exposure in air at 500°C. As exposure time increases to 250 h, alloy strength increases, with some loss in tensile ductility. In the exposure period of 250-1000 h, the alloy essentially has the same ultimate tensile strength but with reductions in tensile ductility from 0.21 at 24 h exposure to 0.14 at 1000 h exposure. Further exposure of the alloy to air at 500°C causes loss of strength and tensile ductility, as evidenced by the stress/strain curve for the specimen preoxidized for 2060 h.

Figure 2 shows engineering stress/engineering strain curves obtained at room temperature for specimens after oxidation for several exposure times up to 1000 h. A significant increase in tensile strength of the alloy is seen after 24 h of oxidation. The rupture strain of the specimen decreased from ≈0.32 to 0.265 after 24 h of oxidation at 500°C. However, the effect of oxidation on ductility was more severe as oxidation time increased to 260 h and beyond. The rupture strain values were, respectively 0.07, 0.032, and 0.032 after 260, 600, and 1000 h of oxidation. Figures 3 and 4 show the variations in maximum engineering stress and rupture strain as a function of preoxidation time in air at 500°C for tests conducted at room temperature and 500°C.

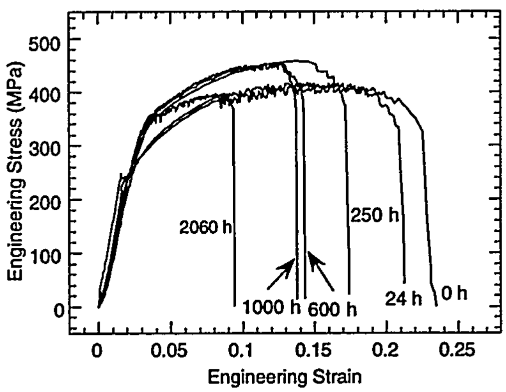

Figure 1. Effect of preoxidation at 500°C on stress-strain behavior of V-5Cr-5Ti alloy tested at 500°C in air at a strain rate of $1.75 \times 10^{-4}\ s^{-1}$.

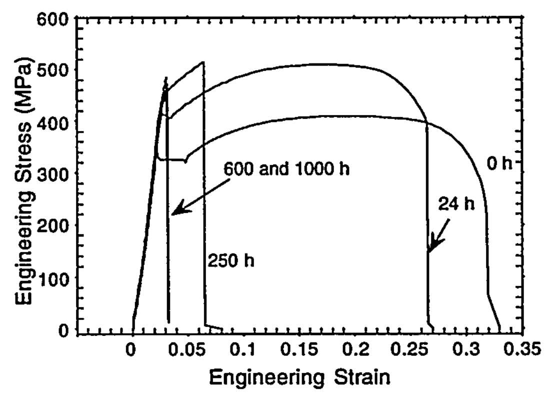

Figure 2. Effect of preoxidation at 500°C on stress-strain behavior of V-5Cr-5Ti alloy tested at room temperature in air at a strain rate of $1.75 \times 10^{-4}\ s^{-1}$.

Microstructural Observations

Axial cross sections of several tested specimens were examined by SEM. Figure 5 shows specimen sections tested in as-annealed condition and after oxidation for 24, 250, 1000, 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 oxidation time increases, the specimen undergoes little necking of the gauge section during the tensile test. It is evident, especially from specimens exposed for 1000 and 2060 h, that fracture occurred by propagation of an axial crack 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°.

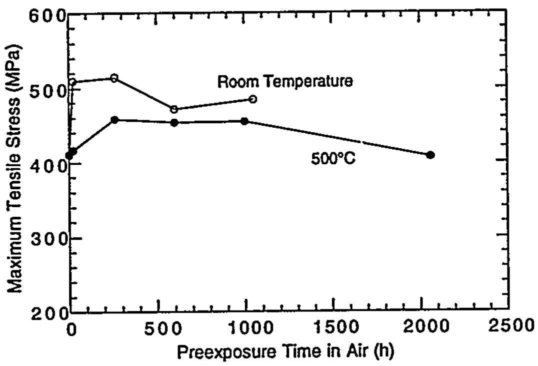

Figure 3. Maximum tensile stress as a function of preexposure time in air for specimens of V-5Cr-5Ti alloy tested in air.

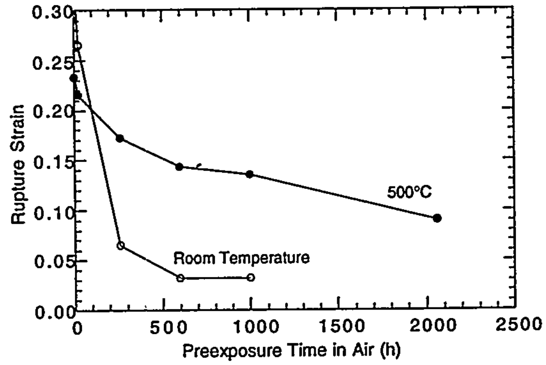

Figure 4. Rupture strain as a function of pre–exposure time in air for specimens of V-5Cr-5Ti alloy tested in air.

Figure 5. Scanning electron photomicrographs of axial sections of V-5Cr-5Ti specimens tensile tested at 500°C in as-annealed condition and after oxidation in air at 500°C for several exposure times

Figure 6 shows photomicrographs of as-annealed and preoxidized specimens tested at room temperature. The as-annealed specimen exhibited significant necking prior to fracture. The specimen preoxidized for 24 h exhibited numerous cracks, but less necking than in the as-annealed specimen. The specimen preoxidized for 260 h exhibited several small cracks but virtually no necking prior to fracture. The specimens preoxidized for 600 and 1000 h showed no surface cracks but were fully embrittled.

Table 1 lists the calculated and measured thickness of oxide layers, depths of hardened layers (from Vickers hardness measurements), thickness of intergranular fracture zones, and transverse crack lengths for specimens as-annealed, preoxidized, and tensile tested at room temperature and 500°C. The data in Table 1 show that the oxide layer is fairly thin even after exposure to air at 500°C for 2060 h. However, oxygen diffusion into the substrate alloy and its enrichment in the surface regions of the specimens alter the fracture mode, changing it from ductile to cleavage. Furthermore, the thickness of the intergranular fracture zone is in good agreement with the crack lengths measured in the transverse direction. The difference in the intergranular fracture zone thickness and the crack length can be attributed to a subsurface oxygen-enriched layer that is not fully brittle. The results also indicate a threshold oxygen concentration in the alloy for embrittlement to ensue, and this is presently being investigated.

Figure 6. Scanning electron photomicrographs of axial sections of V-5Cr-5Ti specimens tensile tested at room temperature in as-annealed condition and after oxidation in air at 500°C for several exposure time

Table 1. Oxidation, hardness, and fracture data for V-5Cr-5Ti alloy

Exposure time (h)	Calculated oxide thickness[a] (μm)	Measured oxide thickness (μm)	Depth of hardened layer after exposure at 500°C (μm)	Intergranular-fracture zone (μm)		Measured crack length (μm)		Rupture strain	
				RT[b]	500°C	RT[b]	500°C	RT[b]	500°C
0	0	0	0	0	0	0	10	0.322	0.233
24	1.4	1.2	<25	c	25	24	22	0.265	0.215
250[d]	4.6	5.0	45	>500	65	e	50	0.065	0.172
600	7.1	7.1	68	>500	100	e	90	0.032	0.143
1000[d]	9.1	9.0	80	>500	120	e	110	0.032	0.135
2060	13.1	14.0	120	NT[f]	165	NT[f]	160	NT[f]	0.090

[a]Values were calculated with an equation developed from a parabolic fit of all the oxidation data .

[b]RT=room temperature.

[c]Fracture is partially ductile and no transition is noted from ductile to brittle fracture.

[d]Exposure times were 260 and 1050 h for the samples tested at room temperature.

[e]Specimen fully embrittled.

[f]NT=not tested.

Additional exposures as a function of oxygen partial pressure in the exposure environment, as well as tensile tests at other temperatures on oxidized specimens, are underway to establish alloy performance in an oxygen-containing environment.

REFERENCES

1. K. Natesan and W. K. Soppet, "Effect of Oxidation on Tensile Behavior of V-Cr-Ti Alloys," Fusion Reactor Materials Progress Report for the Period Ending March 31, 1995, Argonne National Laboratory.

2. K. Natesan and W. K. Soppet, "Effect of Oxidation on Tensile Behavior of a V-Cr-Ti Alloy," Proc. 2nd Intl. Conf. Heat Resistant Materials, Gatlinburg, TN, Sept. 11-14, 1995, ASM International, p. 375 (1995).