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

K. Natesan and W. K. Soppet (Argonne National Laboratory)

OBJECTIVE

The objectives of this task are to (a) evaluate the oxygen uptake of V-5Cr-5Ti alloy as a function of temperature and oxygen partial pressure in the exposure environment, (b) examine the microstructural characteristics of oxide scales and oxygen trapped at the grain boundaries in the substrate alloy, (c) evaluate the influence of oxygen uptake on the tensile properties of the alloy at room and elevated temperatures, (d) evaluate oxidation kinetics of the alloy with aluminum-enriched surface layers, and (e) determine the effect of oxygen uptake on the tensile behavior of the alloy.

SUMMARY

Oxidation studies were conducted on V-5Cr-5Ti alloy specimens at 500°C in air to evaluate oxygen uptake of the alloy as a function of temperature and exposure time. The oxidation rates derived from thermogravimetric testing are 5, 17, and 27 μm after one year of exposure at 300, 400 and 500°C, respectively. Uniaxial tensile tests were conducted 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 characterized by electron optic techniques. Correlations have been developed between tensile strength and ductility of the oxidized alloy and microstructural characteristics such as oxide thickness, depths of hardened layers, depths of intergranular fracture zones, and lengths of transverse cracks.

INTRODUCTION

Refractory alloys in general and vanadium alloys in particular are susceptible to pickup of interstitials such as oxygen, carbon, and nitrogen, which can affect the short- and long-term mechanical properties of the materials. The vanadium alloy with a composition of V-5Cr-5Ti contains 5 wt.% Ti (a much more stable oxide-former than V and Cr), which can have an even stronger effect on mechanical properties, especially on tensile and creep ductility. The degree of influence of interstitials such as oxygen on the alloy's properties will be dictated by alloy grain size (the amount of grain-boundary areas), amount and distribution of oxygen in the alloy, amount and size of second-phase oxide precipitates (such as Ti oxide), service temperature and time of exposure. The purpose of this study is to examine the role of oxygen and oxidation rate on tensile properties of the alloy.

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. Actual composition of the alloy is given in Table 1. A sheet 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 from 300 to 650°C.

Tensile specimens were fabricated according to ASTM specifications with a gauge length of ≈19 mm and a gauge width of ≈4.5 mm. 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. As-annealed 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 4.3×10^{-6} and 1.8×10^{-3} s^{-1}. The preoxidized specimens were tested at a strain rate of 1.75×10^{-4} s^{-1}. All tests were performed in air at 500°C and the test temperature was maintained within ±2°C. The specimens were loaded by means of pins

*This work has been supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

that pass through holes in the enlarged end sections, thus minimizing misalignment. Total elongation was measured by vernier caliper on the tested specimens and from load/elongation chart records. The fracture surfaces and longitudinal and axial cross sections of tested specimens were examined by scanning electron microscopy (SEM) and optical metallography. In addition, Vickers hardness measurements were made on several of the tested specimens. Coupon specimens of the alloy that were oxidized along with the tensile specimens were analyzed for their bulk oxygen content by the vacuum-fusion technique.

RESULTS AND DISCUSSION

Oxidation Behavior. Oxidation of the alloy followed parabolic kinetics with time. Detailed SEM analysis of the oxidized samples showed that the outer layer was predominantly vanadium-rich oxide and the inner layer was (V,Ti) oxide. A parabolic rate equation was used to calculate oxide scale thicknesses, which were in agreement with the values determined by metallography; the results are discussed in an earlier report (1).

Effect of Oxidation on Tensile Properties. To evaluate the effects of oxidation and oxide penetration into the substrate alloy, several tests were conducted to examine the tensile behavior of the alloy as a function of oxygen ingress and oxide scale formation. Tensile specimens were exposed to air for 24-2060 h in air at 500°C and then tensile tested in air at the same temperature. Most of the tests were conducted at a strain rate of 1.8×10^{-4} s^{-1}.

Figure 1 shows the engineering stress/engineering strain curves for specimens after oxidation for several exposure times in a range 0-2060 h. The data indicate that the stress/strain behavior of the alloy is virtually unaffected by 24-h exposure in air at 500°C. As the 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 has essentially the same ultimate tensile strength, but tensile ductility is reduced from 0.21 at 24 h exposure to 0.14 at 1000 h. 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 the specimen preoxidized for 2060 h.

Microstructural Observations. Axial cross sections of several tested specimens were examined SEM. Figure 2 shows sections of specimen tested in as-annealed condition and after oxidation for 24, 250, 1000, and 2060 h in air at 500°C. Crack depths in the transverse direction increase as the oxidation time increases. Further, crack spacing in the axial direction increases as oxidation time is increased. As oxidation time increases, the alloy undergoes little necking in the gauge section of the specimen during the tensile test. It is evident, especially from the 1000 and 2060 h specimens, that fracture occurred by propagation of one of these axial cracks and that because the core of the alloy is somewhat ductile, the crack propagation direction in the core region is ≈45°.

Figure 3 shows SEM photomicrographs of fracture surfaces of specimens tested in as-annealed condition and after oxidation for several time periods. The fracture mode was predominantly ductile in the as-annealed specimen. The specimen exposed for 24 h to air at 500°C showed a layer of grain-boundary or cleavage morphology to a depth of ≈25 µm, beyond which a ductile fracture mode was observed. With increases in oxidation time, the zone of intergranular fracture increased, and for the 2060 h oxidized specimen the depth of this zone was 165 µm.

Table 1 lists the calculated and measured thicknesses of oxide layers, depths of hardened layers (from Vickers hardness measurements), thicknesses of intergranular fracture zone, and lengths of transverse cracks for as-annealed/preoxidized specimens that were tensile tested at 500°C. The data in Table 1 show that the oxide layer is fairly thin even after 2060 h exposure to air at 500°C. However, oxygen diffusion into the substrate alloy and its enrichment in the surface regions of the specimens alter the fracture mode from ductile to cleavage. Further, thicknesses of the zones of intergranular fracture are in agreement with the crack lengths measured in the transverse direction. The difference in intergranular fracture zone thickness and crack length can be attributed to a subsurface oxygen-enriched layer that is not fully brittle. There is also a threshold oxygen concentration in the alloy for embrittlement to ensue, and this aspect is presently being investigated. Figure 4 shows variations in hardened layer thickness, intergranular fracture zone thickness, and transverse crack length as a function of oxidation time. Tensile rupture strain values are also shown in the figure. Results to date indicate that the alloy is not subject to catastrophic embrittlement due to oxygen ingress into the material. Additional exposures as a function of oxygen partial pressure in the exposure environment, as well as tensile tests at lower temperatures, are in progress to establish the performance envelope for the alloy in an oxygenated environment.

REFERENCES

1. K. Natesan and W. K. Soppet, "Effect of Oxidation on Tensile Behavior of V-5Cr-5Ti Alloy," Argonne National Laboratory, Fusion Reactor Materials Semiannual Progress Report for the Period Ending September 30, 1994, Argonne National Laboratory.

Table 1. Oxidation, hardness, and fracture data for V-5Cr-5Ti alloy at 500°C

Exposure time (h)	Calculated oxide thickness (μm)	Measured oxide thickness (μm)	Depth of hardened layer (μm)	Thickness of intergranular-fracture zone (μm)	Measured crack length (μm)	Rupture strain
0	0	0	0	0	10	0.233
24	1.4	1.2	<25	25	22	0.215
250	4.6	5.0	45	65	50	0.172
600	7.1	7.1	68	100	90	0.143
1000	9.1	9.0	80	120	110	0.135
2060	13.1	14.0	120	165	160	0.090

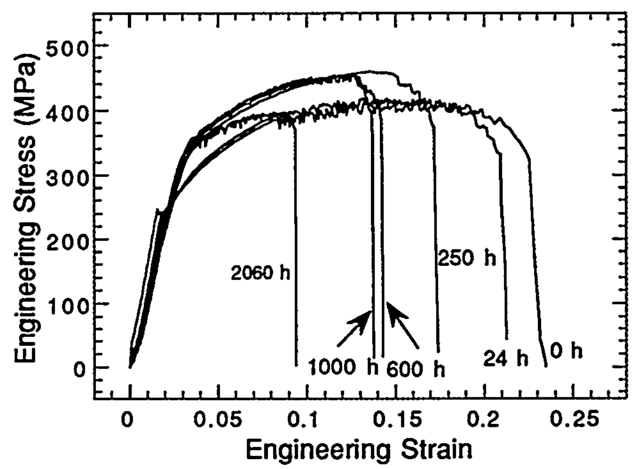

Figure 1. Effect of preoxidation at 500°C on stress-strain behavior of V-5Cr-5Ti alloy tested at 500°C in air at a strain rate of 1.75 x 10^{-4} s^{-1}.

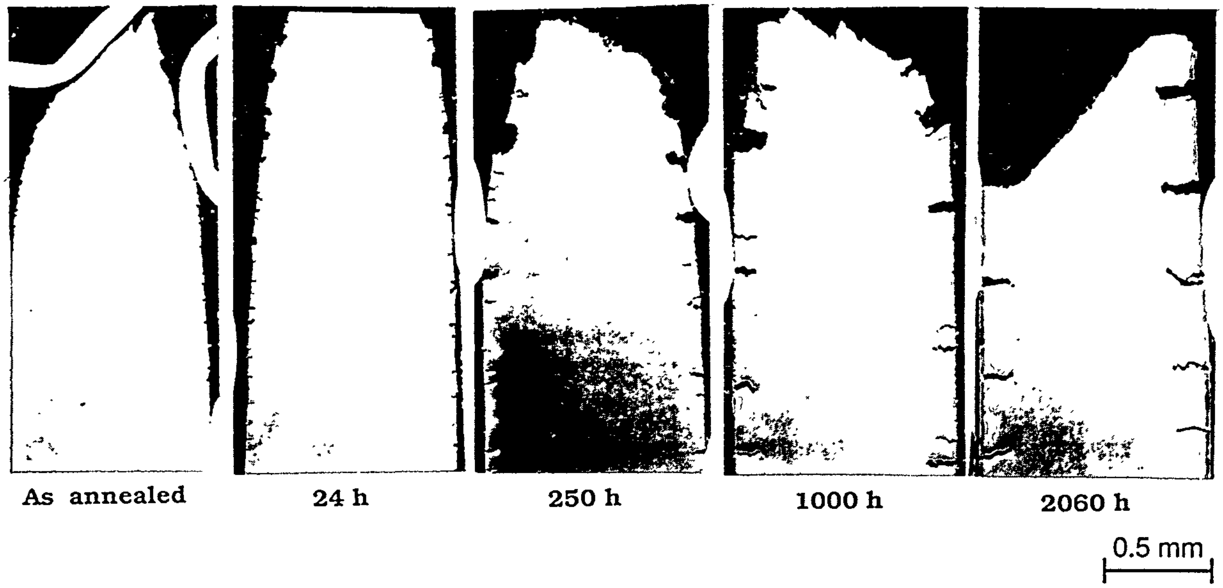

Figure 2. SEM photomicrographs of axial sections of V-5Cr-5Ti specimens tensile tested in as-annealed condition and after oxidation in air at 500°C for several exposure times

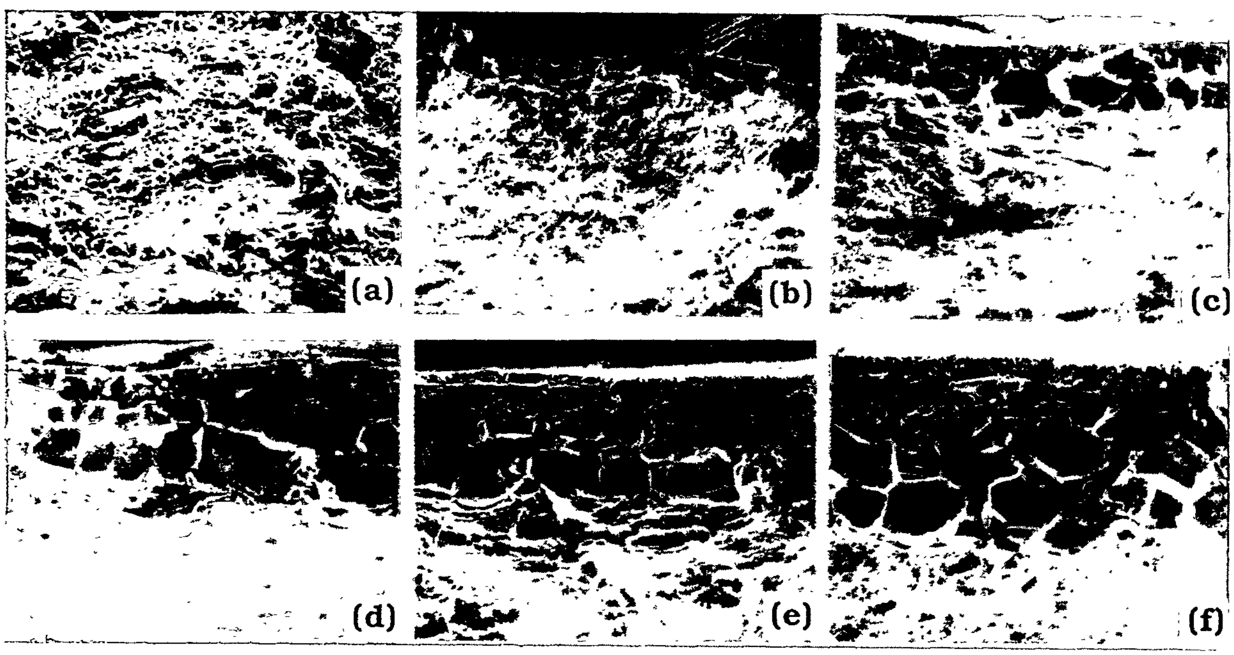

Figure 3. SEM photomicrographs of fracture surfaces of V-5Cr-5Ti specimens tensile tested (a) in as-annealed condition and after oxidation in air at 500°C for (b) 24 h, (c) 250 h, (d) 600 h, (e) 1000 h, and (f) 2060 h.

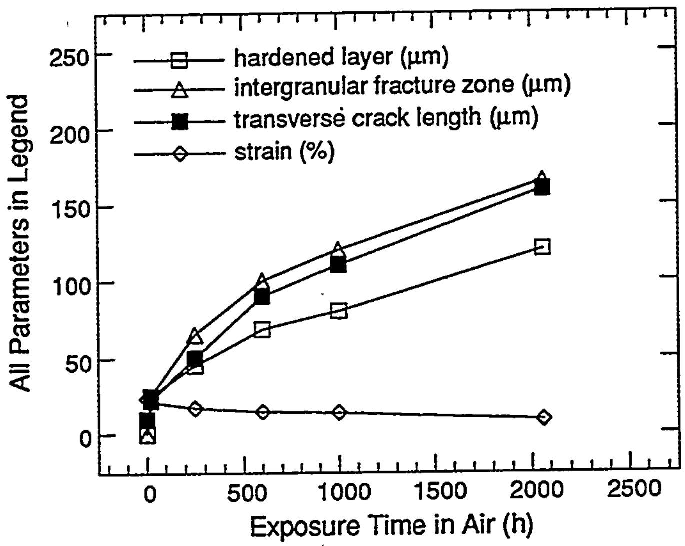

Figure 4. Variation in hardened-layer thickness, intergranular fracture zone depth, transverse crack length, and rupture strain as a function of exposure time at 500°C for V-5Cr-5Ti alloy.