TENSILE PROPERTIES OF ALUMINIZED V-5Cr-5Ti ALLOY AFTER EXPOSURE IN AIR ENVIRONMENT*

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OBJECTIVE

The objectives of this task are to (a) develop procedures to modify surface regions of V-Cr-Ti alloys in order to minimize oxygen uptake by the alloys when exposed to environments that contain oxygen, (b) evaluate the oxygen uptake of the surface-modified V-Cr-Ti alloys as a function of temperature and oxygen partial pressure in the exposure environment, (c) characterize the microstructures of oxide scales and oxygen trapped at the grain boundaries of the substrate alloys, and (d) evaluate the influence of oxygen uptake on the tensile properties of the modified alloys at room and elevated temperatures.

SUMMARY

A pack diffusion process was used to enrich surface regions of V-5 wt.% Cr-5 wt.% Ti alloy specimens with aluminum, which is a more stable oxide former than vanadium; also, the oxide has a much slower growth rate than that of vanadium oxide. Oxidation studies were conducted on surface-modified 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. Uniaxial tensile tests were conducted at 500°C on several preoxidized specimens of the surface-modified alloy to examine the effects of oxidation and oxygen migration on tensile strength and ductility.

EXPERIMENTAL PROGRAM

Detailed studies have been conducted earlier on oxidation and on effect of oxygen ingress on tensile properties of V-Cr-Ti alloys; results have beed reported elsewhere [1-3]. For the development of a surface modification approach to minimize oxidation of the alloy and to retain the mechanical properties of the base alloy, a heat of vanadium alloy with a nominal composition of V-5 wt.% Cr -5 wt.% Ti was selected; this heat was designated BL-63. A sheet of the alloy was annealed for 1 h at 1050°C prior to pack diffusion, oxidation, and tensile testing. Coupon specimens that measured ≈15 x 7.5 x 1 mm were used for the oxidation studies. 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.

Aluminum oxide is thermodynamically more stable than the oxides of the constituent elements (V, Cr, and Ti) of the vanadium alloy (see Fig. 1). Further, the growth rates of aluminum oxide is much slower than that of the refractory metal oxides such as vanadium or titanium oxide. Therefore, surface enrichment of the alloy with Al to develop aluminum oxide scale in oxidizing environments can protect the underlying alloy from further oxidation.

Surface aluminization of the V alloy was done by a pack-diffusion process in which the V-alloy substrate material was brought into contact with a pack of powders and heated for 4-12 h at ≈900°C. The composition of such powders (e.g., 65 wt.% Al_2O_3, 33 wt.% Al, 2 wt.% NH_4Cl) provided the packing with metallic Al, alumina as filler material, and NH_4Cl as activator. The Al deposited on the substrate surface diffuses into the subsurface regions of the material, where it forms intermetallic phases consisting of Al and V. Because the substrate material is heated to near the annealing range for times sufficient to cause solution processes in the matrix, the material needs a final treatment to optimize the structure. The aluminide layers reach thicknesses of 0.02–0.04 mm, depending on the composition of the substrate material. The high temperature of the formation process creates layers that develop compressive stresses at lower temperatures; thus, the layers do not contain cracks after preparation is complete.

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Coupon specimens of bare and aluminized V-5Cr-5T alloy were oxidized in air at 400, 500, and 575°C in a thermogravimetric apparatus to evaluate their oxidation behavior. Surface-modified tensile specimens of V-5Cr-5Ti alloy were oxidized in air at 500°C for periods ranging from 164 to 2544 h. The preoxidized specimens were tensile-tested on an Instron machine at a constant crosshead speed of 0.02 cm/min, which corresponds to an initial 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 in the 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 the tested specimens were examined by scanning electron microscopy (SEM). In addition, Vickers hardness was examined in several tested specimens.

RESULTS

Oxidation Behavior

Figure 2 shows weight change versus time for both bare and aluminized specimens. Oxidation of the alloys followed parabolic kinetics with respect to time. Detailed SEM analysis (with both energy-dispersive and wavelength-dispersive analysis) of the oxidized samples of bare alloy showed that the outer layer was predominantly vanadium-rich oxide and the inner layer was (V,Ti) oxide. Furthermore, X-ray diffraction of the oxides showed the outer oxide to be V_2O_5; no nitrogen or nitride phases were detected. The aluminized specimens exhibited much lower rates of oxidation at all temperatures in the present study. The oxidation rate at 575°C for the aluminized specimen was lower than that observed for the bare alloy at 575°C. Detailed microscopy and hardness measurements are in progress on the aluminized 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 aluminized alloy was examined as a function of oxygen ingress and oxide scale formation. Specimens were exposed to air for 164-2544 h at 500°C and then tensile-tested in air at 500°C at a strain rate of $1.8 \times 10^{-4}\ s^{-1}$. Figure 3 shows the engineering stress/engineering strain curves at 500°C for specimens after oxidation for several exposure times up to 2544 h. Stress/strain behavior of the alloy is virtually unaffected by oxidation in air at 500°C.

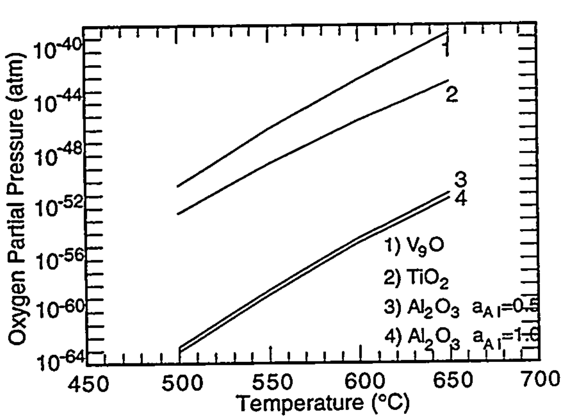

Figure 1. Stability of alumina compared with that of vanadium and titanium oxides. a_{Al} indicates aluminum activity in the alloy.

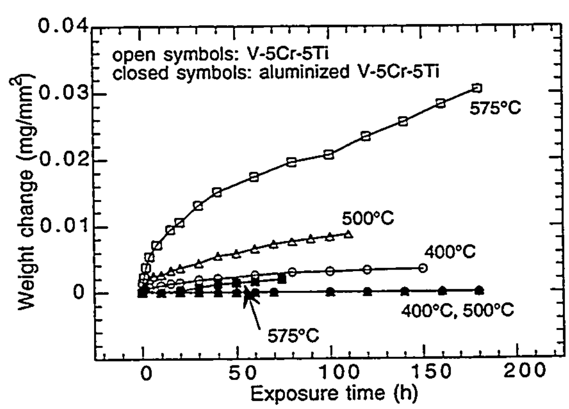

Figure 2. Weight change vs. time for bare and aluminized V-5Cr-5Ti specimens during oxidation in air

The load-displacement curves were analyzed by drawing lines parallel to the initial portion of the loading curve at the points of maximum load and rupture load. The intersects of these lines with the displacement axis are used to calculate the uniform and total elongation for the specimenssubjected to various oxidation treatments. This approach accounts for the stiffness, or lack of it, in the loading fixture of the tensile machine and, as a result, yields elongation values that are more representative of the gauge section of the tensile specimen.

Figures 4 and 5 show the variations in maximum engineering stress and uniform and total elongation as a function of preoxidation time in air at 500°C for tests conducted at 500°C on both bare and aluminized specimens. Figure 4 shows that the maximum tensile stress for the aluminized alloy was essentially the same over the oxidation period of 2544 h and is similar to the values observed for the oxidized V-5Cr-5Ti alloy. Figure 5 shows that total elongation for the

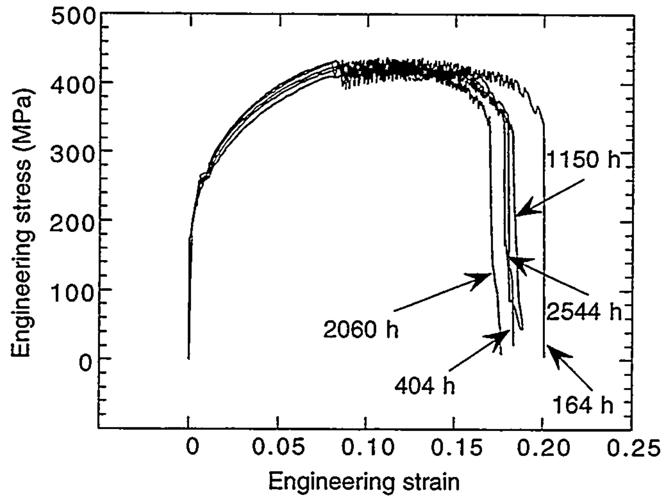

Figure 3. Effect of preoxidation at 500°C on stress/strain behavior of aluminized V-5Cr-5Ti alloy tested at 500°C in air at a strain rate of 1.8×10^{-4} s^{-1}.

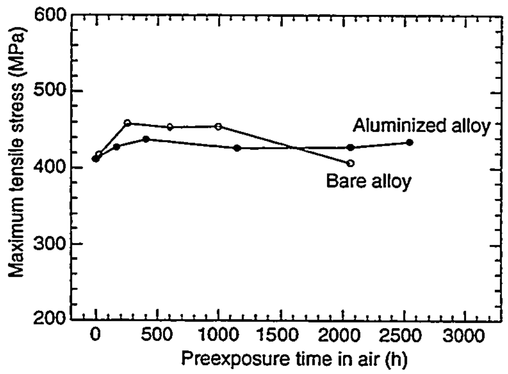

Figure 4. Maximum tensile stress as a function of preexposure time at 500°C in air for aluminized V-5Cr-5Ti alloy tensile-tested at 500°C in air.

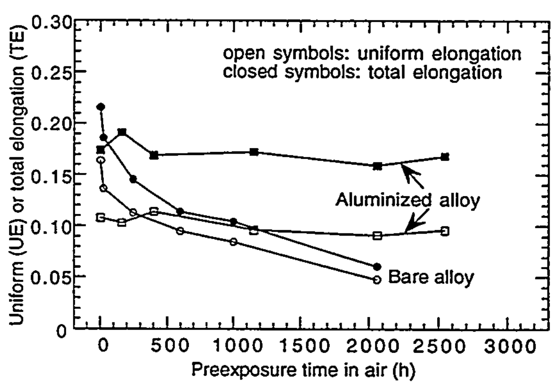

Figure 5. Uniform and total elongation as a function of preexposure time at 500°C in air for aluminized V-5Cr-5Ti alloy tensile- tested at 500°C in air.

aluminized alloys remains fairly high over the oxidation period of 2544 h, while that of the oxidizedbase alloy exhibited decreased total elongation with increase in oxidation time. The uniform elongation for the aluminized specimens was lower (≈0.11) but remained constant over theoxidation period. These results indicate that the drop in uniform elongation occurs during the aluminizing process, which is conducted at 900°C. We believe that the initial drop in uniform elongation can be minimized by optimizing the aluminizing process with regard to time and temperature.

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 Period Ending March 31, 1995, Argonne National Laboratory, DOE/ER-0313/18, p. 247, July 1995.

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

3. K. Natesan and W. K. Soppet, "Effect of Oxygen and Oxidation on Tensile Behavior of V-5Cr-5Ti," Fusion Reactor Materials Progress Report for the Period Ending Dec. 31, 1995, Argonne National Laboratory, DOE/ER-0313/19, p. 50, April 1996.