

TENSILE PROPERTIES OF V-Cr-Ti ALLOYS AFTER EXPOSURE IN HELIUM AND LOW-PARTIAL-PRESSURE OXYGEN ENVIRONMENTS*

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OBJECTIVE

The objectives of this task are to (a) quantify the oxygen partial pressure (pO_2) in argon and helium environments of different purity, (b) determine the oxygen uptake of V-Cr-Ti alloys as a function of temperature and 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, (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 test program is in progress to evaluate the effect of oxygen at low pO_2 on the tensile properties of V-(4-5)wt.% Cr-(4-5)wt.% Ti alloys. Some of the tensile specimens were precharged with oxygen at low pO_2 at 500°C and reannealed in vacuum at 500°C prior to tensile testing. In another series of tests, specimens were exposed for 250-275 h at 500°C in environments with various pO_2 levels and subsequently tensile tested at room temperature. The preliminary results indicate that both approaches are appropriate for evaluating the effect of oxygen uptake on the tensile properties of the alloys. The data showed that in the relatively short-time tests conducted thus far, the maximum engineering stress slightly increased after oxygen exposure but the uniform and total elongation values exhibited significant decrease after exposure in oxygen-containing environments. The data for a specimen exposed to a helium environment were similar to those obtained in low pO_2 environments.

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 $\approx 15 \times 7.5 \times 1$ 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 an earlier report (1).

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 $32 \mu\text{m}$, respectively. Three types of exposures were used to evaluate the oxygen effect on the properties. In the first approach, specimens were exposed to a low- pO_2 environment for 4 to 24 h at 500°C, and subsequently the surface-charged specimens were annealed under vacuum at 500°C for 100 h to diffuse the surface oxygen into the interior of the specimens. In the second approach, specimens were exposed at 500-700°C to high-purity oxygen gas at low partial pressures, maintained by a feed/bleed system. In the third approach, specimens were exposed to helium environments of various purity grades at temperatures of 500 to 700°C.

Pretreated specimens from the above-described approaches were tensile-tested in air at room and elevated temperatures. The crosshead speed in an Instron machine was set to yield a strain rate of $1.8 \times 10^{-4} \text{ s}^{-1}$ for all the tests. 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 of several tested specimens was determined. 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

Measurement of Oxygen in Environment

The pO_2 in the exposure environment was characterized by using a solid electrolyte oxygen sensor. The electrolyte was a stabilized zirconia tube containing 8 wt.% yttria. Based on conduction domains reported in the literature for solid electrolytes (2), the yttria-stabilized zirconia electrolyte should exhibit fully ionic conductivity at temperatures above 500°C in the low- pO_2 environments of interest in the present study. The sensor used in the present investigation had the following approximate dimensions: ID, 6.3 mm, OD, 9.5 mm, and length 610 mm. A porous platinum coating ≈ 40 mm long was applied at the closed end of the electrolyte tube and used for the electrode leads. Air was used as the reference gas mixture and continuously flowed inside the zirconia tube. The outside electrode was exposed to the low pO_2 environment of the desired pressure. Pure gases such as argon and helium, and premixed gases such as CO-CO_2 and $\text{O}_2\text{-N}_2$ were used in the study.

The equilibrium voltage E of the cell with respect to an air reference electrode is given by the Nernst equation:

$$E \text{ (in volts)} = 2.15 \times 10^{-5} \times T \times \ln(-0.21/pO_2),$$

where pO_2 is the oxygen partial pressure in the low-pressure side of the cell, 0.21 is the partial pressure of oxygen in air at the reference electrode, and T is the absolute temperature.

Figure 1 shows the response of the cell, after conversion into oxygen partial pressure for the calibration gases CO-CO_2 and $\text{O}_2\text{-N}_2$ gases and for 99.999% pure argon and helium. The cell response for the calibration gases was in agreement with values calculated on the basis of thermodynamic equilibrium and gas composition. Figure 2 shows an expanded version of the data on the pO_2 , in torr, for the argon and helium gases. In addition, the argon gas was further purified to eliminate moisture, which resulted in substantial reduction in pO_2 relative to that of unpurified gas. The data indicate that the pO_2 in 99.999% He is $\approx 0.1\text{-}0.3$ torr over the temperature range $400\text{-}700^\circ\text{C}$.

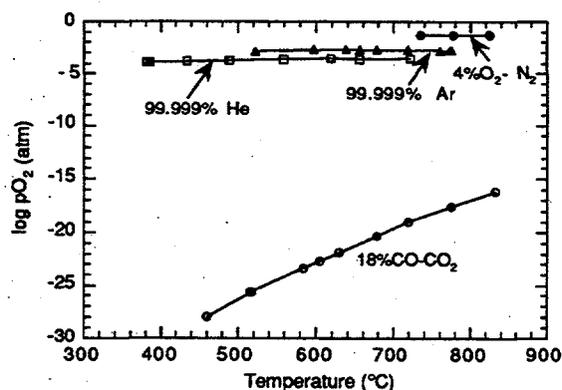


Figure 1. Variation in oxygen partial pressure as a function of temperature for calibration gases $\text{O}_2\text{-N}_2$ and CO-CO_2 and for argon and helium.

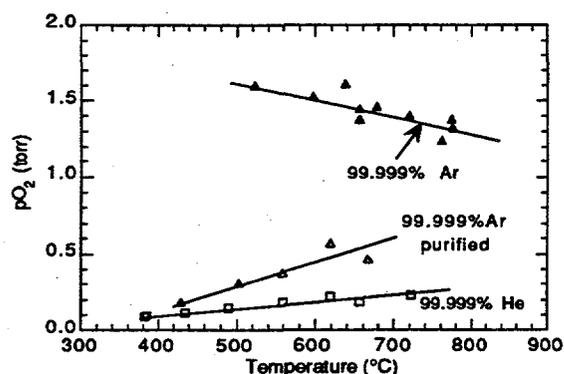


Figure 2. Expanded plot of variation in oxygen partial pressure as a function of temperature for argon and helium.

Effect of Oxygen Exposure on Tensile Properties

Figure 3 shows the engineering stress/engineering strain curves at room temperature for specimens of V-5Cr-5Ti alloy in as-rolled condition, after annealing for 1 h at 1050°C, after a second annealing for 100 h at 500°C, and after oxygen charging for 4, 8, and 24 h at 500°C in an environment with $pO_2 = 1.0 \times 10^{-6}$ torr followed by a 100 h annealing at 500°C in vacuum. The purpose of a 100-h annealing at 500°C is to diffuse the oxygen at the surface of the specimens obtained by charging for 4, 8, and 24 h in a low- pO_2 environment.

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 specimens subjected to various oxidation treatments. Table 1 lists the maximum engineering stress and uniform and total elongation for the specimens with various pretreatments tensile-tested at room temperature. Maximum engineering stress is ≈ 505 -537 MPa after oxygen charging and annealing at 500°C. The uniform elongation values are ≈ 0.131 -0.155 and are slightly less than the 0.165 observed for the specimen with only an initial annealing treatment of 1 h at 1050°C. Total elongation for the specimens annealed for 100 h at 500°C decreased

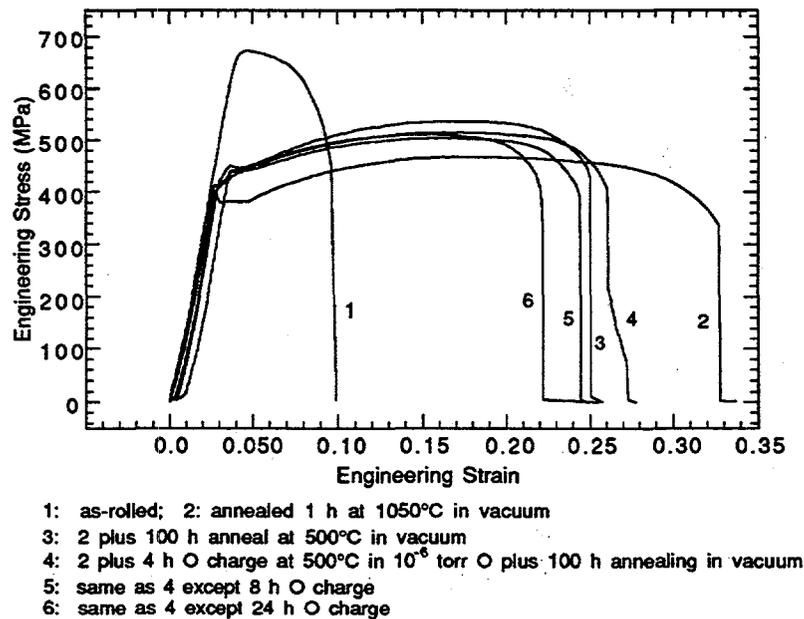
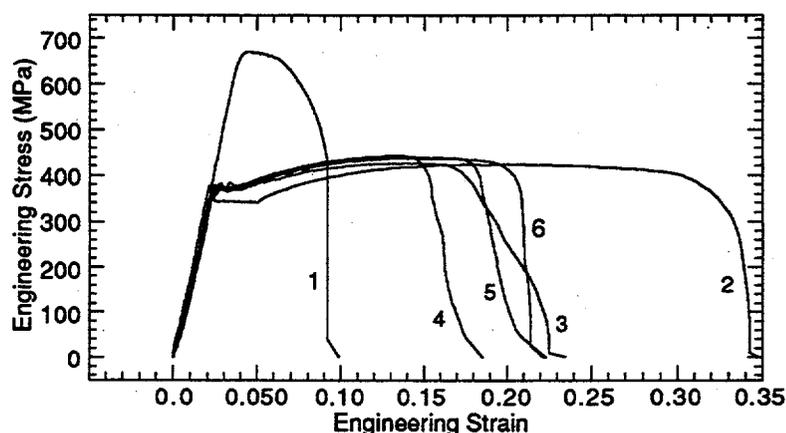


Figure 3. Engineering stress/engineering strain curves for V-5Cr-5Ti alloy with several pretreatments tested at room temperature.

Table 1. Tensile test data at room temperature for V-5Cr-5Ti alloy after several pretreatments

Specimen pretreatment	Maximum engineering stress (MPa)	Uniform elongation	Total elongation
As-rolled	673.7	0.011	0.074
Vacuum annealing, 1 h at 1050°C (a)	468.7	0.165	0.303
(a) + vacuum annealing, 100 h at 500°C (b)	537.3	0.148	0.214
(a) + 4 h O charge + (b)	515.1	0.155	0.232
(a) + 8 h O charge + (b)	505.0	0.146	0.221
(a) + 24 h O charge + (b)	511.4	0.131	0.198

substantially from an initial value of 0.303 to values of ≈ 0.198 - 0.232 . Examination of the surfaces and cross sections of the specimens exposed to different oxygenated treatments showed significant surface cracks spaced uniformly but appearing to become blunted after a certain depth indicating that the extent of oxygen diffusion and the local concentration of oxygen in the surface regions of the specimens will determine the cracking depth. Figure 4 shows the engineering stress/engineering strain curves at room temperature for specimens of V-4Cr-4Ti alloy in as-rolled condition, after annealing for 1 h at 1050°C , after exposure in environments with different $p\text{O}_2$ at 500°C , and in 99.999% He environment. Table 2 lists the maximum engineering stress and uniform and total elongation for the specimens with various treatments tensile-tested at room temperature. The maximum engineering stress for this alloy in as-rolled condition is similar to that of V-5Cr-5Ti alloy. However, upon annealing for 1 h at 1050°C , the strength falls to 423.7 MPa, which is somewhat lower than that for V-5Cr-5Ti alloy. Correspondingly, the uniform and total elongation values were slightly higher for the V-4Cr-4Ti alloy than for the V-5Cr-5Ti alloy. The effect of 250 h exposure in oxygen at three different levels is to slightly increase the maximum stress, especially at higher



- 1: as-rolled; 2: annealed 1 h at 1050°C in vacuum
- 3: 250 h exposure at $p\text{O}_2 = 10^{-6}$ torr at 500°C
- 4: same as 3 except $p\text{O}_2 = 7.6 \times 10^{-4}$ torr at 500°C
- 5: same as 3 except $p\text{O}_2 = 0.1$ torr at 500°C
- 6: 275 h exposure in 99.999% He environment at 500°C

Figure 4. Engineering stress/engineering strain curves for V-4Cr-4Ti alloy exposed to several $p\text{O}_2$ environments and tested at room temperature.

Table 2. Tensile test data at room temperature for V-4Cr-4Ti alloy exposed to low- $p\text{O}_2$ environments

Specimen pretreatment	Maximum engineering stress (MPa)	Uniform elongation	Total elongation
As-rolled	670.1	0.010	0.067
Vacuum annealing, 1 h at 1050°C (a)	423.7	0.186	0.322
(a) + 250 h in $p\text{O}_2 = 1 \times 10^{-6}$ torr at 500°C	426.3	0.127	0.217
(a) + 250 h in $p\text{O}_2 = 7.6 \times 10^{-4}$ torr at 500°C	443.0	0.110	0.145
(a) + 250 h in $p\text{O}_2 = 0.1$ torr at 500°C	440.6	0.133	0.202
(a) + 275 h in 99.999% He at 500°C	437.8	0.140	0.191

pO₂ levels; however, the uniform and total elongation values decreased substantially, even though exposure time was only 250 h and exposure temperature was relatively modest at 500°C. The specimen tested in 99.999% He gas at 500°C exhibited properties similar to those tested in oxygen environments. Microstructural characteristics of the tested specimens are presently being examined by several electro-optical techniques.

REFERENCES

1. K. Natesan and M. Uz, "Oxidation Kinetics and Microstructure of V-(4-5)wt.% Cr-(4-5)wt.% Ti Alloys Exposed to Air at 300-650°C," Fusion Reactor Materials Progress Report for the Period Ending June 30, 1996, Argonne National Laboratory, DOE/ER-0313/20, p. 105, Oct. 1996.
2. J. W. Patterson, "Conduction Domains for Solid Electrolytes," J. Electrochem. Soc. 118, p. 1033-1039, 1971.