

## TENSILE PROPERTIES OF V-Cr-Ti ALLOYS AFTER EXPOSURE IN HYDROGEN-CONTAINING ENVIRONMENTS\*

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### OBJECTIVE

The objectives of this task are to (a) determine the hydrogen uptake of V-Cr-Ti alloys as a function of temperature and partial pressure of hydrogen ( $p_{H_2}$ ) in the exposure environment, (b) examine the microstructural characteristics of surfaces and cross sections of the alloys after exposure, (c) evaluate the influence of hydrogen uptake in low- $p_{H_2}$  environments on the tensile properties and cracking propensity of the alloys at room and elevated temperatures, and (d) determine the effects of oxygen/hydrogen interactions on the tensile properties of the alloys.

### SUMMARY

A systematic study has been initiated at Argonne National Laboratory to evaluate the performance of several V-Cr-Ti alloys after exposure to environments containing hydrogen at various partial pressures. The goal is to correlate the chemistry of the exposure environment with hydrogen uptake in the samples and its influence on the microstructure and tensile properties of the alloys. At present, the principal effort has focused on the V-4Cr-4Ti alloy of heat identified as BL-71; however other alloys (V-5Cr-5Ti alloy of heats BL-63, and T87, plus V-4Cr-4Ti alloy from General Atomics [GA]) are also being evaluated. Other variables of interest are the effect of initial grain size on hydrogen uptake and tensile properties, and the synergistic effects of oxygen and hydrogen on the tensile behavior of the alloys. Experiments conducted on specimens of various V-Cr-Ti alloys exposed to  $p_{H_2}$  levels of 0.01 and  $3 \times 10^{-6}$  torr showed negligible effect of  $H_2$  on either maximum engineering stress or uniform and total elongation. However, uniform and total elongation decreased substantially when the alloys were exposed to 1.0 torr  $H_2$  pressure. Preliminary data from sequential exposures of the materials to low- $p_{O_2}$  and several low- $p_{H_2}$  environments did not reveal an adverse effect on the maximum engineering stress or on uniform and total elongation. Further, tests in  $H_2$  environments on specimens annealed at different temperatures showed that grain-size variation by a factor of  $\approx 2$  had little or no effect on tensile properties.

### EXPERIMENTAL PROGRAM

The heats of vanadium alloy selected for the study had nominal compositions of V-5 wt.%Cr-5 wt.%Ti (designated as BL-63 and T87) and V-4 wt.%Cr-4 wt.%Ti (designated as BL-71 and 44 from the GA heat). Detailed chemical analyses of these heats were given in an earlier report (1). The tensile specimens were fabricated according to ASTM Standard E8-69 specifications and had a gauge length of  $\approx 19$  mm and a gauge width of  $\approx 4.5$  mm. Specimens were annealed for 1 h at  $1050^\circ\text{C}$  prior to hydrogen exposure and tensile testing. Some specimens of BL-63 and BL-71 heats were also annealed for 2 h at  $1200^\circ\text{C}$  to obtain a larger grain size before hydrogen exposure.

Tensile samples of the alloys were exposed for 100 h at  $500^\circ\text{C}$  to environments containing  $H_2$  at partial pressures of  $3 \times 10^{-6}$  and 1.0 torr and subsequently tensile-tested at a strain rate of  $1.8 \times 10^{-4} \text{ s}^{-1}$  in room-temperature air. 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 the tested specimens are being examined by scanning electron microscopy.

## RESULTS AND DISCUSSION

### Effect of Hydrogen Exposure

The engineering stress/engineering strain plots at room temperature for V-4Cr-4Ti and V-5Cr-5Ti materials in as-annealed condition and after exposure to H<sub>2</sub> at partial pressures of  $3 \times 10^{-6}$  and 0.01 torr were reported in Ref. 1. The results showed that in the pH<sub>2</sub> range of the present study, BL-63 exhibited a negligible effect of H<sub>2</sub> on tensile properties, while BL-71 exhibited a small decrease in uniform and total elongation after H<sub>2</sub> exposure. During this period, additional tensile specimens of the two alloys were exposed for 100 h at 500°C to H<sub>2</sub> gas at pressures of  $1 \times 10^{-4}$ ,  $1 \times 10^{-2}$ ,  $5 \times 10^{-2}$ , and 1 torr.

Engineering stress/engineering strain curves from different tests at room temperature were analyzed to evaluate several tensile properties for the alloys after several H<sub>2</sub> treatments. Table 1 shows the values for maximum engineering stress and uniform and total elongation in the two materials after exposure at different pH<sub>2</sub> levels. Figure 1 shows the maximum engineering stress values as a function of H concentration for both the alloys. The results show that H<sub>2</sub> pressures in the range of the present study have little or no effect on the maximum engineering stress for either alloy. The uniform and total elongation values for the two alloys are 0.14-0.19 and 0.18-0.31, respectively, at H<sub>2</sub> pressures of  $3 \times 10^{-6}$  to  $5 \times 10^{-2}$  torr. After exposure at an H<sub>2</sub> pressure of 1 torr, both alloys showed significant decrease in uniform and total elongation, indicating that this H<sub>2</sub> pressure may be the threshold for embrittlement of the alloys. Figure 2 is a plot of uniform and total elongation values as a function of H<sub>2</sub> concentration for both alloys. The hydrogen concentrations in the BL-63 and BL-71 alloys after exposure for 100 h at 500°C in 1 torr H<sub>2</sub> pressure were 330 and 358 wppm, respectively.

### Effect of Oxygen Pretreatment

To examine the synergistic effect, if any, of oxygen and hydrogen in the alloy on the tensile behavior of V-Cr-Ti alloys, tensile specimens of the two alloys were pretreated in several low-pO<sub>2</sub> environments for 100 h at 500°C. Subsequently, the exposure gas was changed from O<sub>2</sub> to H<sub>2</sub> at the same temperature and exposure of the specimens was continued for another 100 h. The preexposure of the specimens to the low-pO<sub>2</sub> environments resulted in different concentrations of O in the alloys, which were subsequently exposed to different H<sub>2</sub> pressures. The specimens exposed to the dual treatment were analyzed for O and H by the vacuum fusion technique. Table 2 lists the O and H concentrations for specimens with different treatments. The O concentrations ranged from 550 to 2230 wppm, while the H concentration was 6 to 16 wppm. It should be noted

Table 1. Effects of 100 h hydrogen exposure at 500°C on room-temperature tensile properties of V-Cr-Ti alloys, initially annealed for 1 h at 1050°C in vacuum

pH <sub>2</sub> in exposure environment (torr)	Maximum engineering stress (MPa)		Uniform elongation		Total elongation	
	BL-63	BL-71	BL-63	BL-71	BL-63	BL-71
-	469	424	0.165	0.186	0.303	0.322
$3 \times 10^{-6}$	437	440	0.189	0.174	0.313	0.263
$1 \times 10^{-4}$	523	467	0.157	0.154	0.182	0.227
$1 \times 10^{-4}$ (repeat)	487	491	0.169	0.148	0.249	0.206
$1 \times 10^{-2}$	445	459	0.194	0.169	0.313	0.263
$5 \times 10^{-2}$	483	477	0.143	0.142	0.223	0.191
1	481	468	0.0077	0.0016	0.020	0.0016

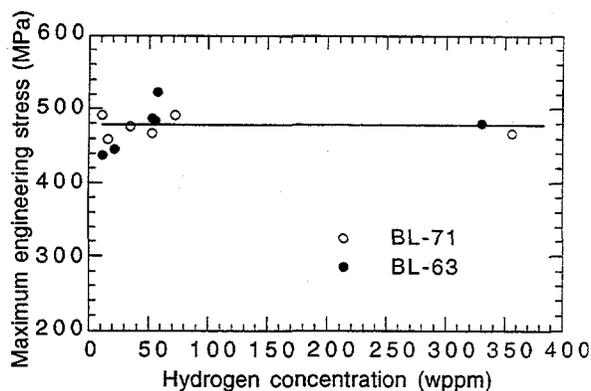


Figure 1. Maximum engineering stress as a function of H concentration for V-4Cr-4Ti and V-5Cr-5Ti alloys tested at room temperature

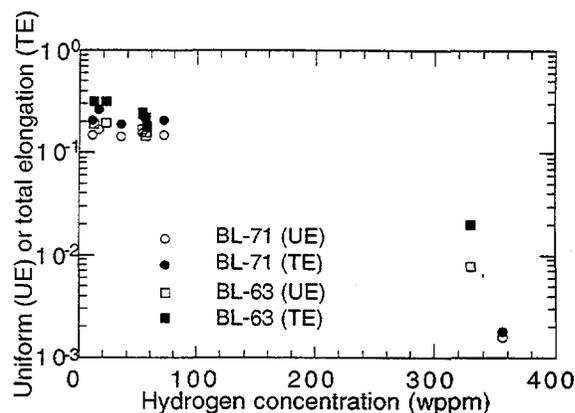


Figure 2. Uniform and total elongation as a function of H concentration for V-4Cr-4Ti and V-5Cr-5Ti alloys tested at room temperature

Table 2. Oxygen and hydrogen concentrations in pretreated tensile specimens

pO <sub>2</sub> in pre-exposure environment <sup>a</sup> (torr)	pH <sub>2</sub> in exposure environment <sup>b</sup> (torr)	Concentration (in wppm) of			
		Oxygen		Hydrogen	
		BL-63	BL-71	BL-63	BL-71
-	$3 \times 10^{-6}$	550	670	12	11
$3 \times 10^{-7}$	$1 \times 10^{-6}$	625	620	16	16
$1 \times 10^{-6}$	$1 \times 10^{-6}$	730	820	6	7
$1 \times 10^{-4}$	$3 \times 10^{-6}$	2230	2020	15	13

<sup>a</sup>Preexposure time was 100 h at 500°C.

<sup>b</sup>Exposure time in hydrogen was 100 h at 500°C.

that the diffusion coefficient for O is a few orders of magnitude lower than that for H in these alloys; as a result, after the 100-h exposures the measured H concentration will be uniformly distributed over the entire 1-mm-thick section of the specimens, while the O concentration will be confined to a depth of  $\approx 50 \mu\text{m}$  from the surface [2,3].

The pretreated tensile specimens were tensile-tested at a strain rate of  $1.8 \times 10^{-4} \text{ s}^{-1}$  in room-temperature air. Engineering stress/engineering strain curves from different tests were analyzed to evaluate several tensile properties for the alloys after the dual treatments. Table 3 shows the values for maximum engineering stress and uniform and total elongation for the two materials after the dual treatments. Figures 3 and 4 show the maximum engineering stress and the uniform and total elongation as a function of O concentration for both the alloys. It is evident that O concentration up to  $\approx 2300$  wppm, even though confined to the surface regions of the specimens, has a negligible effect on tensile properties; no synergistic effect of O and H on the properties is observed, based on the present study. Additional experiments are planned to examine this issue at higher O and H concentrations in the material, as well as at temperatures above room temperature.

#### Effect of Initial Grain Size

To examine the effect of initial grain size on subsequent H<sub>2</sub> uptake and tensile properties, specimens of BL-71 and BL-63 were annealed for 2 h at 1200°C; this increased the grain size by at

Table 3. Effects of 100 h hydrogen exposure at 500°C on room-temperature tensile properties of V-Cr-Ti alloys, initially annealed for 1 h at 1050°C in vacuum and pretreated in low-pO<sub>2</sub> environment

pO <sub>2</sub> in pre-exposure environment <sup>a</sup> (torr)	pH <sub>2</sub> in exposure environment (torr)	Maximum engineering stress (MPa)		Uniform elongation		Total elongation	
		BL-63	BL-71	BL-63	BL-71	BL-63	BL-71
-	-	469	424	0.165	0.186	0.303	0.322
-	3 x 10 <sup>-6</sup>	437	440	0.189	0.174	0.313	0.263
3 x 10 <sup>-7</sup>	1 x 10 <sup>-6</sup>	452	453	0.173	0.162	0.297	0.266
1 x 10 <sup>-6</sup>	1 x 10 <sup>-6</sup>	507	475	0.165	0.146	0.268	0.228
1 x 10 <sup>-4</sup>	3 x 10 <sup>-6</sup>	484	451	0.132	0.136	0.184	0.177

<sup>a</sup>Preexposure time was 100 h at 500°C.

Table 4. Effects of 100 h hydrogen exposure at 500°C on room-temperature tensile properties of enlarged-grain V-Cr-Ti alloys, initially annealed for 2 h at 1200°C in vacuum

pH <sub>2</sub> in exposure environment (torr)	Maximum engineering stress (MPa)		Uniform elongation		Total elongation	
	BL-63	BL-71	BL-63	BL-71	BL-63	BL-71
3 x 10 <sup>-6</sup>	515	472	0.144	0.159	0.250	0.225
1 x 10 <sup>-2</sup>	524	479	0.160	0.153	0.244	0.227
5 x 10 <sup>-2</sup>	544	509	0.129	0.082	0.188	0.082

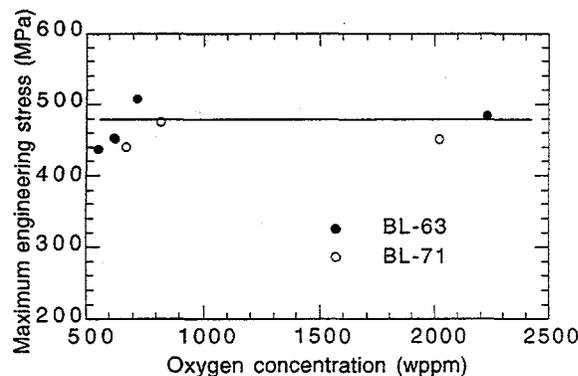


Figure 3. Maximum engineering stress as a function of O uptake during preoxidation and subsequent H<sub>2</sub> exposure for V-4Cr-4Ti and V-5Cr-5Ti alloys tested at room temperature

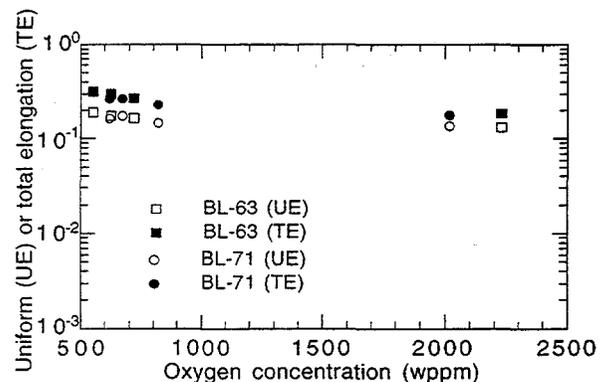


Figure 4. Uniform and total elongation as a function of O uptake during preoxidation and subsequent H<sub>2</sub> exposure for V-4Cr-4Ti and V-5Cr-5Ti alloys tested at room temperature

least a factor of 2 over those of alloys annealed for 1 h at 1050°C. Engineering stress/engineering strain curves for large- and small-grain materials after 100 h exposure at 500°C to a pH<sub>2</sub> of 0.01 torr were presented in the previous report [1]. During this period, additional tests were conducted on larger-grain-size materials after 100 h exposure in 5 x 10<sup>-2</sup> torr of H<sub>2</sub> pressure. With the increased grain size, both alloys exhibited a slight increase in strength and some decrease in elongation. Decrease in uniform and total elongation was much more pronounced at the higher H<sub>2</sub> pressure of 5 x 10<sup>-2</sup> torr. Examination of the fracture surfaces and specimen cross sections, and measurements of hardness profiles, are in progress and the results will be used to correlate the microstructure, H concentration, and hardness data with the tensile properties of the alloys.

## REFERENCES

1. K. Natesan and W. K. Soppet, "Tensile Properties of V-Cr-Ti Alloys After Exposure in Hydrogen-Containing Environments," Fusion Reactor Materials Progress Report for the Period Ending December 31, 1997, Argonne National Laboratory, DOE/ER-0313/23, p. 127, March 1998.
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