EFFECTS OF STRAIN RATE, TEST TEMPERATURE AND TEST ENVIRONMENT ON TENSILE PROPERTIES OF VANADIUM ALLOYS — A. N. Gubbi, A. F. Rowcliffe, W. S. Eatherly, and L. T. Gibson (Oak Ridge National Laboratory)

OBJECTIVE

The aim of this work is to examine the effects of test temperature, strain rate, and test environment on the tensile properties of the large heat of V-4Cr-4Ti and the small heats of compositional variants.

SUMMARY

Tensile testing was carried out on SS-3 tensile specimens punched from 0.762-mm-thick sheets of the large heat of V-4Cr-4Ti and small heats of V-3Cr-3Ti and V-6Cr-6Ti. The tensile specimens were annealed at 1000° for 2 h to obtain a fully recrystallized, fine grain microstructure with a grain size in the range of 10-19 µm . Room temperature tests at strain rates ranging from 10^{-3} to 5×10^{-1}/s were carried out in air; elevated temperature testing up to 700°C was conducted in a vacuum better than 1×10^{-5} torr ($<10^{-3}$ Pa). To study the effect of atomic hydrogen on ductility, tensile tests were conducted at room temperature in an ultra high vacuum chamber (UHV) with a hydrogen leak system.

Tensile properties of V-3Cr-3Ti, V-4Cr-4Ti, and V-6Cr-6Ti were measured at room temperature and 100–700°C at a strain rate of 1.1×10^{-3}/s. The ultimate tensile strength of all the alloys exhibited a minima at 300°C, whereas the 0.2% yield strength was relatively independent of temperature between 400° and 700°C. The total and uniform elongations were relatively insensitive to variation in test temperature above 400°C. All the alloys exhibited good ductility (e.g., uniform elongation >15%) and a large amount of work hardening ability. A yield point was typically obtained at all test temperatures. Serrations, indicative of dynamic strain aging, were observed in the stress-strain curves of all the alloys at test temperatures above 300°C.

V-6Cr-6Ti is the strongest of the three alloys with the highest values of 0.2% yield strength (YS) and the ultimate tensile strength (UTS), and V-3Cr-3Ti is the weakest showing the lowest values at all strain rates; V-4Cr-4Ti possesses intermediate strength. Both YS and UTS showed a similar trend of incremental increase with strain rate for the three alloys. All three alloys exhibited almost no change in uniform and total elongations up to a strain rate of 10^{-1}/s followed by a decrease with further increase in strain rate. The room temperature tensile behavior of V-4Cr-4Ti was unaffected by the introduction of a significant partial pressure of atomic hydrogen into the testing environment.

INTRODUCTION

In the early stages of the program on the development of alloys for fusion reactor applications, vanadium alloys with 3 to 6 wt.% Cr and Ti were investigated.[1-4] This composition range was subsequently narrowed down to vanadium alloys with 4 wt.% each of Cr and Ti based on the thermal creep properties, low DBTT under Charpy impact testing, resistance to swelling, and also resistance to helium– and irradiation-induced embrittlement exhibited by a laboratory-scale heat of this alloy.[5] A production-scale heat (~500–kg, heat 832665) of V-4Cr-4Ti alloy was fabricated by Teledyne Wah Chang, Albany, Oregon (TWCA). Impact data have been reported from the testing conducted on the samples machined from a warm-worked plate[6] as well as from an annealed plate.[7] Also, recovery and recrystallization behavior of this heat has been documented.[8] Impact data for compositional variants with Ti and Cr contents ranging from 3 to 6 wt.% have been presented earlier[7] as well as their recovery and recrystallization behavior.[8] The present study reports the results from the tensile testing of the large heat of V-4Cr-4Ti and the small heats of compositional variants with Ti and Cr contents ranging from 3 to 6 wt.%. These alloys will help to define a window for permissible ranges of Cr and Ti concentrations for consistent properties. Any candidate material for first wall of fusion reactor is exposed to both molecular and atomic hydrogen in service; tensile tests were carried out in a controlled atmosphere of hydrogen to examine the effect of hydrogen on the tensile behavior of the V-4Cr-4Ti alloy.

EXPERIMENTAL PROCEDURE

Two small heats (~15-kg melt), V-3Cr-3Ti (heat T91) and V-6Cr-6Ti (heat T90), were fabricated by TWCA according to the specifications set by Oak Ridge National Laboratory. The chemical compositions of the small heats of compositional variants have been presented elsewhere.[8] Tensile specimens (SS-3s) of these two alloys, with gage dimensions of $0.76 \times 1.52 \times 7.6$ mm, were punched from the as-received sheets of ~0.76 mm thickness from TWCA. Tensile specimens of the large heat of V-4Cr-4Ti (heat 832665), the chemical composition for which is given elsewhere[8], were punched from a 0.76 mm sheet of which was cold rolled from 1.05-mm-thick sheet received from TWCA. The punched specimens from the three alloys were recrystallized by annealing for 2 h at 1000°and 1100°C in a vacuum better than 1×10^{-6} torr ($<10^{-4}$ Pa). In order to study the effect of strain rate, tensile testing was carried out in air at room temperature on 1000°C–annealed specimens of all three alloys at strain rates ranging from 1.1×10^{-3} to 5×10^{-1}/s. The effects of test temperature and grain size on tensile properties were investigated on the 1000°C– and 1100°C–annealed specimens of the three alloys by testing them at room temperature in air, and 100–700°C in a vacuum better than 1×10^{-5} torr ($<10^{-3}$ Pa), and at a strain rate of 1.1×10^{-3}/s.

To analyze the effect of hydrogen on tensile behavior, tensile tests were conducted on SS-3 specimens of V-4Cr-4Ti (annealed at 1000°C for 2 h) in an UHV chamber which had a provision for bleeding in hydrogen so that a desired partial pressure of hydrogen could be maintained in the test chamber. The details of the testing setup are given elsewhere.[9] The pumping system of the UHV test chamber consisted of two liquid-nitrogen-cooled sorption pumps and an ion pump, wherein the sorption pumps were used for roughing, and the ion pump was used for bake out and maintaining subsequent UHV in the chamber. High purity (99.9999%) hydrogen was supplied to the chamber through a leak valve. For each test, after loading the specimen, the chamber was baked out for ~18 h at ~200°C in dynamic vacuum. The vacuum in the chamber was better than 5×10^{-10} torr (5×10^{-8} Pa) after baking out and cooling to room temperature. The chamber was then backfilled with pure, dry hydrogen (dried by flowing it through a liquid nitrogen trap) to a partial pressure of 30 to 50 torr (3 to 5 KPa). Hydrogen pressures were measured with a bare ionization gage located in the UHV chamber. The tensile tests were conducted at room temperature, 30 minute after back-filling with hydrogen, at a strain rate of ~10^{-3}/s. By assuming that the specimen strain occurred completely in the gage length, the total elongations were determined by measuring the specimen lengths before and after fracture.

RESULTS AND DISCUSSION

Effect of Strain Rate

Figure 1 shows the variation of 0.2% yield strength with strain rate and Figure 2 shows the ultimate tensile strength (UTS) as a function of strain rate for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys. There is a general tendency for yield strength and UTS to increase incrementally with strain rate for all three alloys. This strain rate sensitivity for vanadium alloys is similar to the observed results for many other bcc metals e.g., niobium and high–purity iron.[10] At all strain rates, V-3Cr-3Ti showed the lowest strength properties for all strain rates with V-6Cr-6Ti being the strongest of the three alloys. V-6Cr-6Ti is stronger than V-4Cr-4Ti both at 0.2% strain and in UTS at all strain rates except between 1.1×10^{-3} and 1.1×10^{-2}/s where it appears that the two alloys have similar yield strength. It is useful to recall here the results of the microhardness testing from an earlier study[8] which showed that in both the recovered and fully recrystallized states, V-6Cr-6Ti was the hardest alloy (160-185 DPH) and V-3Cr-3Ti was the weakest alloy (130-140 DPH), with V-4Cr-4Ti being in the intermediate range (140-160 DPH). Hence, the results of tensile testing for strain rate sensitivity in the present study in conjunction with the previous results from microhardness testing reveal that the V-6Cr-6Ti alloy is an intrinsically stronger alloy (possibly due mainly to solid solution strengthening from higher Cr and Ti contents) compared to both V-3Cr-3Ti and

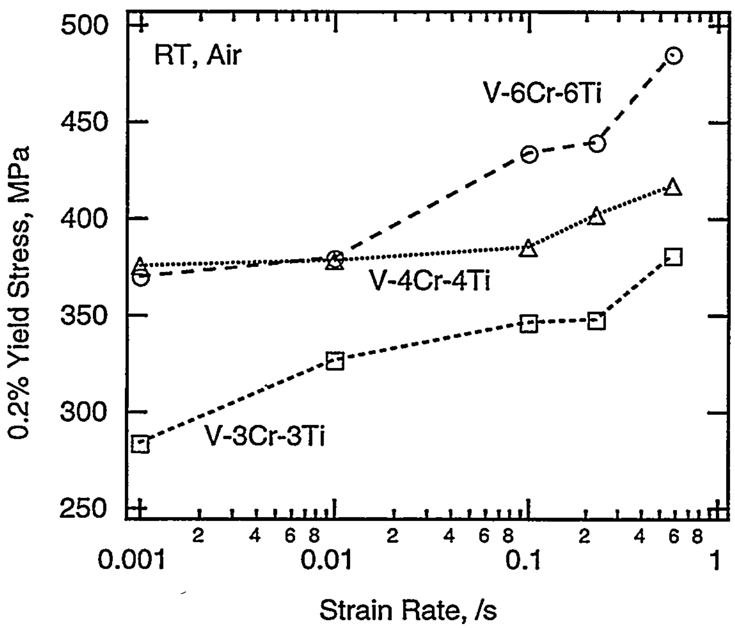

Figure 1. Variation of 0.2% yield strength with strain rate for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

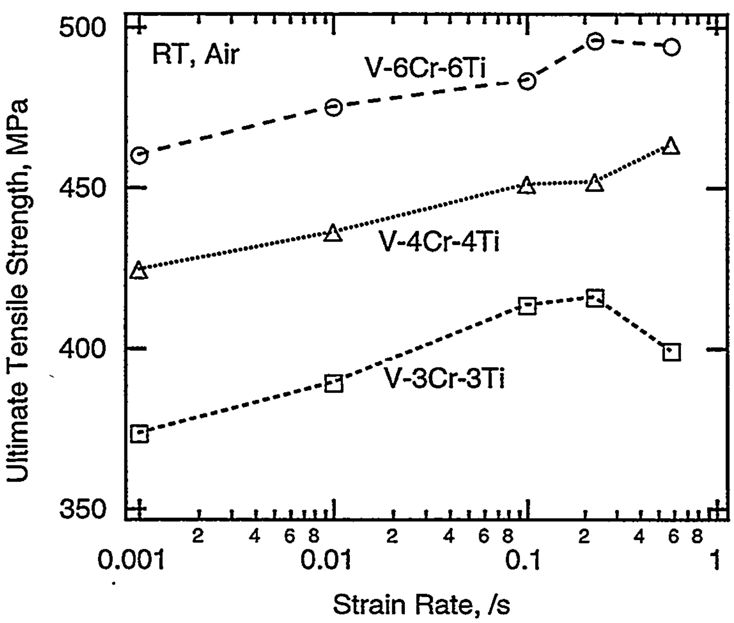

Figure 2. The ultimate tensile strength as a function of strain rate for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

V-4Cr-4Ti alloys.

The effect of strain rate on the uniform and total elongations of the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys is shown in Figure 3 and Figure 4, respectively. For all the three alloys, both uniform and total elongations were nearly independent of strain rate up to 0.1/s, but showed a significant decrease at strain rates above 0.1/s.

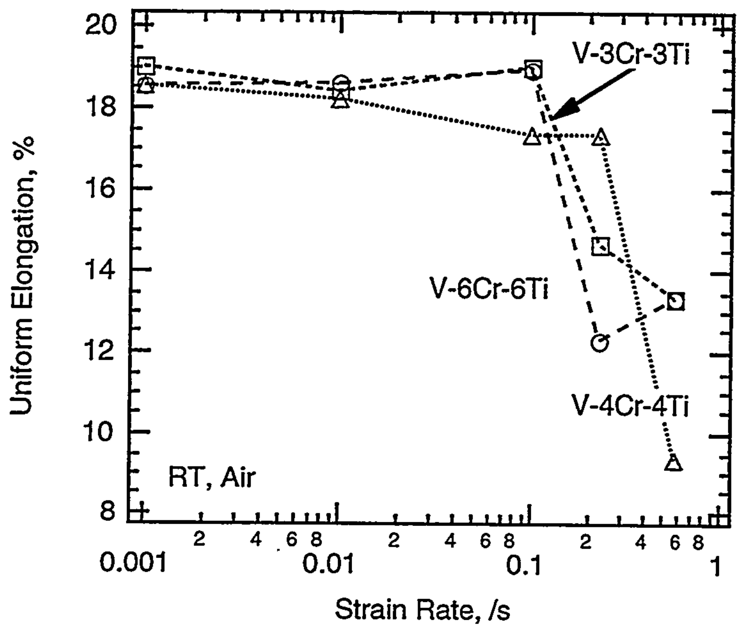

Figure 3. The uniform elongation as a function of strain rate for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

The general relationship between flow stress σ and strain rate Φ (at constant temperature T and strain ε) is given as[11]

$$\sigma = C(\Phi)^m \Big|_{\varepsilon,T} \qquad (1)$$

where C is a constant and m is the strain–rate sensitivity parameter. If one plots logarithm of stress as a function of logarithm of strain rate (at a constant temperature and strain), the slope of the line of fit gives the value of exponent m. In general, for most metals, the value of m is less than 0.1.[11] Figure 5 shows a plot of logarithm of stress as a function of logarithm of strain rate which contains data for the three alloys, V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti, at a nominal strain of 8% and tested at room temperature. It is interesting to note that, for all the three alloys, the exponent m is almost identical and falls in the range of 0.022 to 0.024. Thus the strain–rate sensitivity of the three alloys is similar at room temperature for strain rates ranging from 1.1×10^{-3} to 5×10^{-1}/s. The range of values for strain–rate sensitivity for the alloys studied in the present work compares favorably with the value of 0.024 for unalloyed vanadium (containing 265 wppm oxygen) determined by Bradford and Carlson.[12] Hence, it can be inferred here that vanadium has similar strain–rate sensitivity in both unalloyed and alloyed (with Ti and Cr) states.

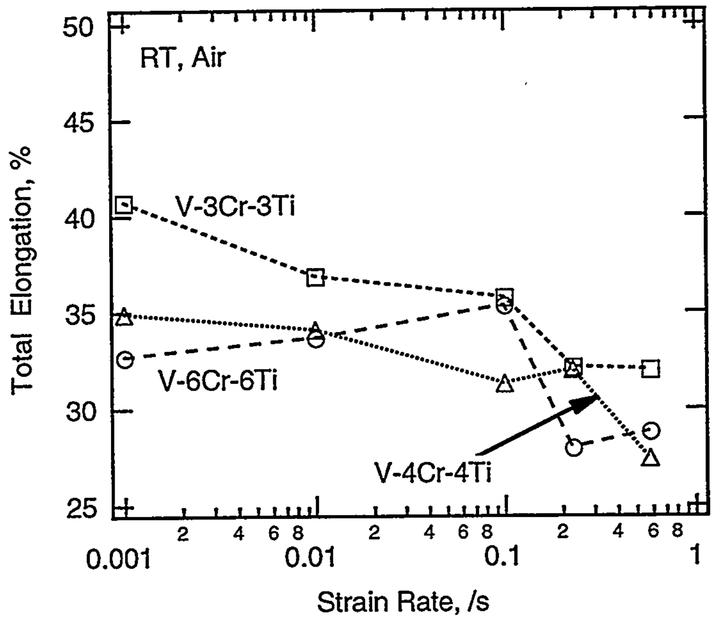

Figure 4. The total elongation as a function of strain rate for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

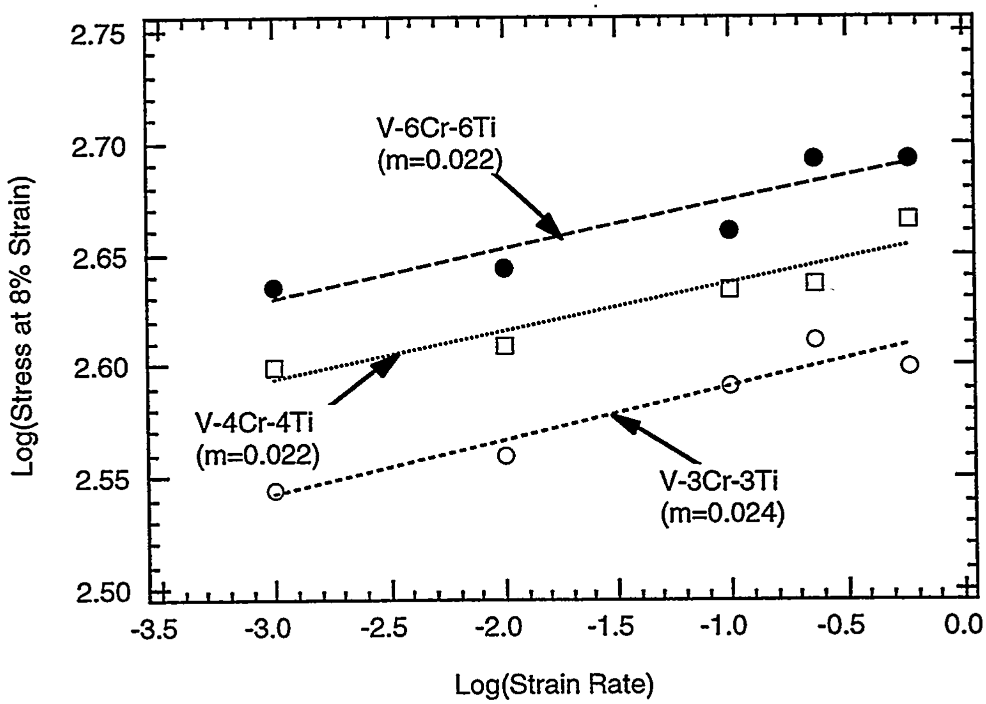

Figure 5. Logarithm of stress as a function of logarithm of strain rate at a nominal strain of 8%, and at room temperature for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

Effect of Test Temperature

Figures 6, 7, and 8 show, respectively, for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys, the variation of 0.2% yield strength and ultimate tensile strength with test temperature. The ultimate tensile strength of all the alloys decreases with increase in temperature from room temperature and passes through a minimum at 300°C, before increasing with further increase in temperature up to 700°C. For all the three alloys, the yield strength also decreases with increase in temperature up to 300–400°C and then becomes relatively independent of temperature up to 700°C.

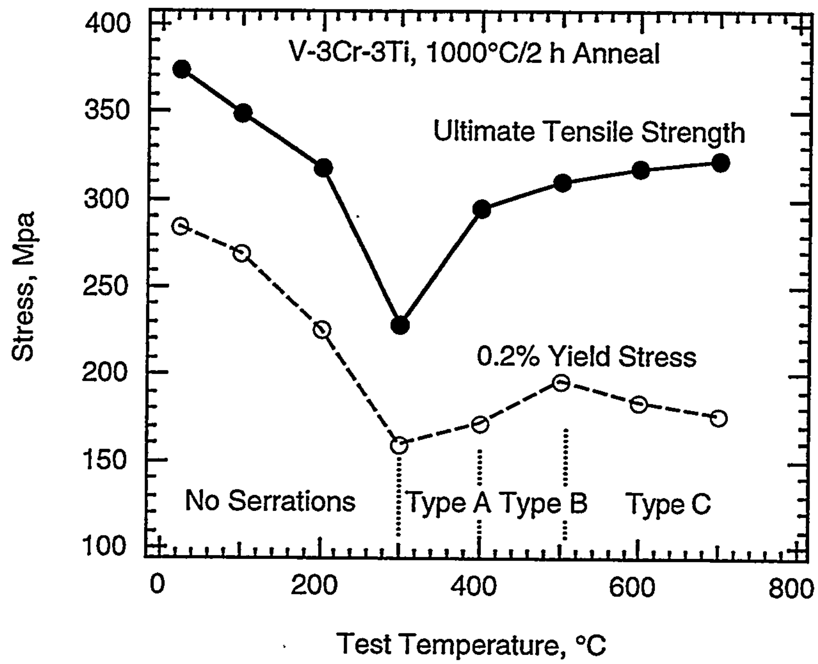

Figure 6. Variation of 0.2% yield strength and ultimate tensile strength with test temperature for the V-3Cr-3Ti alloy.

The effect of test temperature on the uniform and total elongations of the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys is shown in Figures 9, 10 and 11, respectively. Both uniform and total elongations are relatively unaffected by the temperature for all the alloys except V-3Cr-3Ti which shows much higher ductility at room temperature than the other two alloys.

***Discontinuous Yielding*:**

The phenomenon of discontinuous yielding (also called jerky flow or Portevin–Le Chatelier effect),[13] has been seen in solid solution alloys by many researchers. To cite a few, Russel[14] on his work on tin bronze alloys, Soler–Gomez and McG.Tegart[15] on their work on gold–indium, Brindley and Worthington[16] on their work on aluminum–3% magnesium, Keh et al[17] on their study on iron and steel, and Bradford and Carlson[12] on vanadium, observed serrations in the flow curves during tensile testing. In general, there are three types of serrations,[13-15] *Type A, Type B,* and *Type C.* The serrations which are periodic in nature and rise above the general level of stress–strain curve are termed as *Type A*. The serrations which occur in rapid succession and oscillate about the general level of stress–strain curve are termed as *Type B*, and serrations which fall always below the general level of the stress–strain curve are *Type C*.

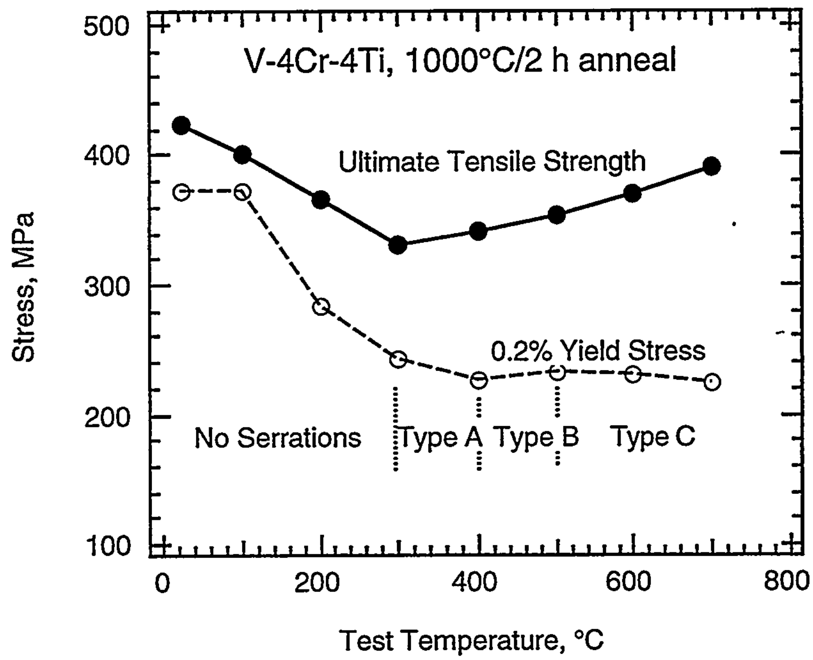

Figure 7. Variation of 0.2% yield strength and ultimate tensile strength with test temperature for the V-4Cr-4Ti alloy.

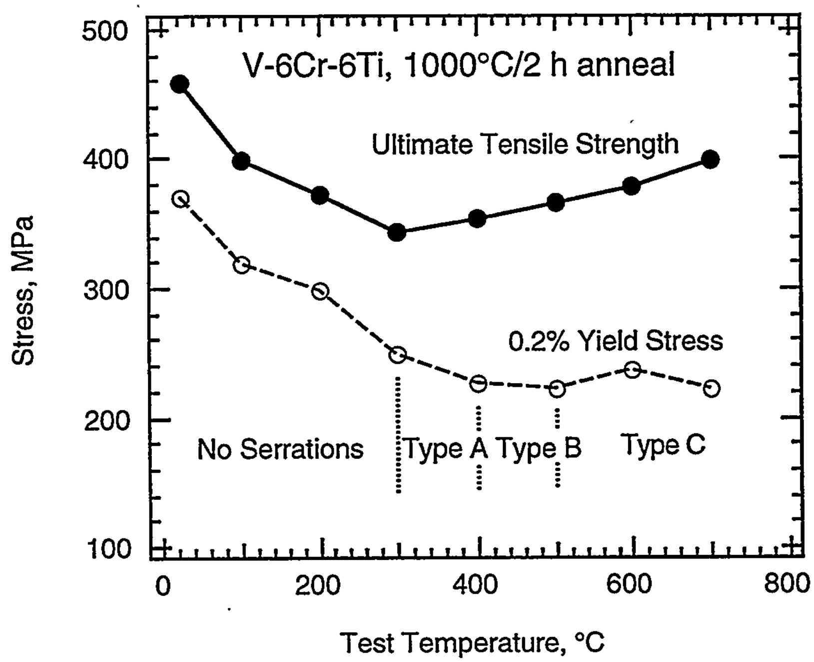

Figure 8. Variation of 0.2% yield strength and ultimate tensile strength with test temperature for the V-6Cr-6Ti alloy.

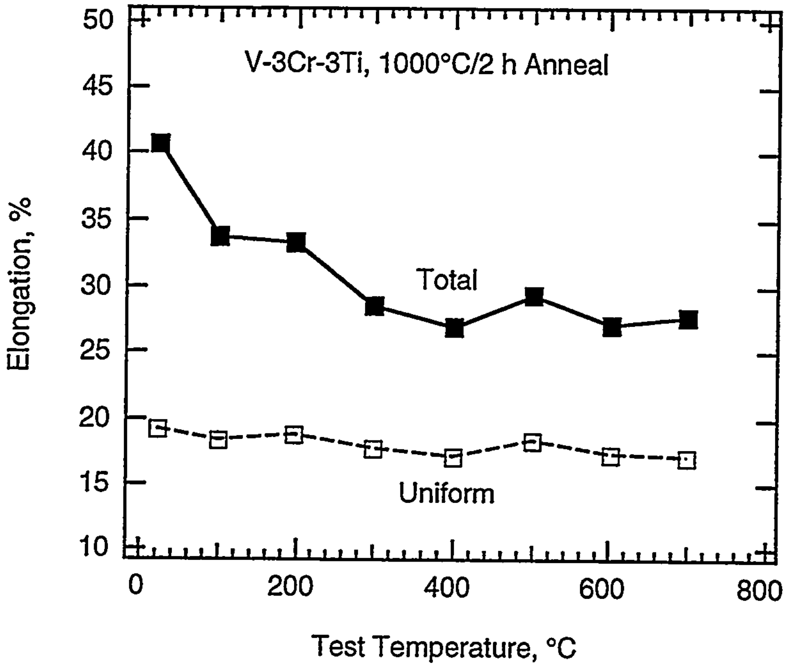

Figure 9. Variation of uniform and total elongations with test temperature for the V-3Cr-3Ti alloy.

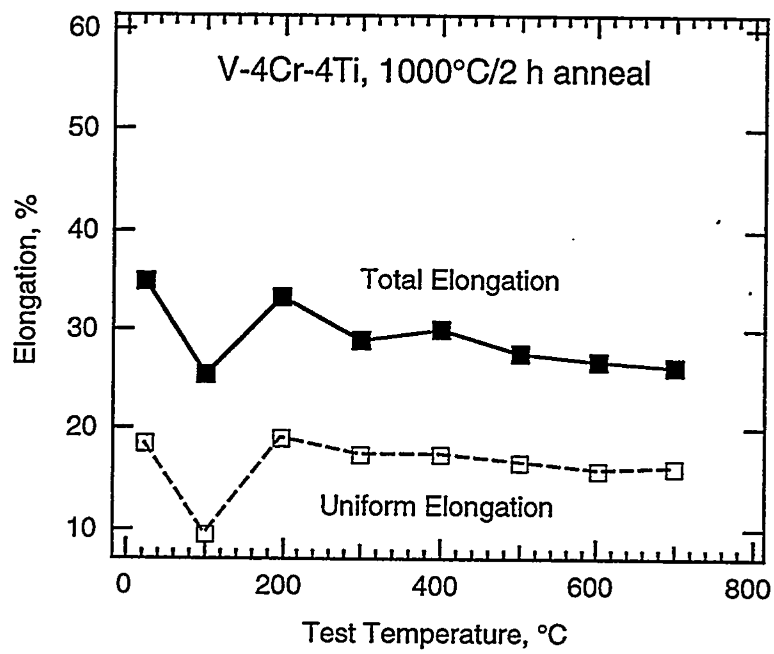

Figure 10. Variation of uniform and total elongations with test temperature for the V-4Cr-4Ti alloy.

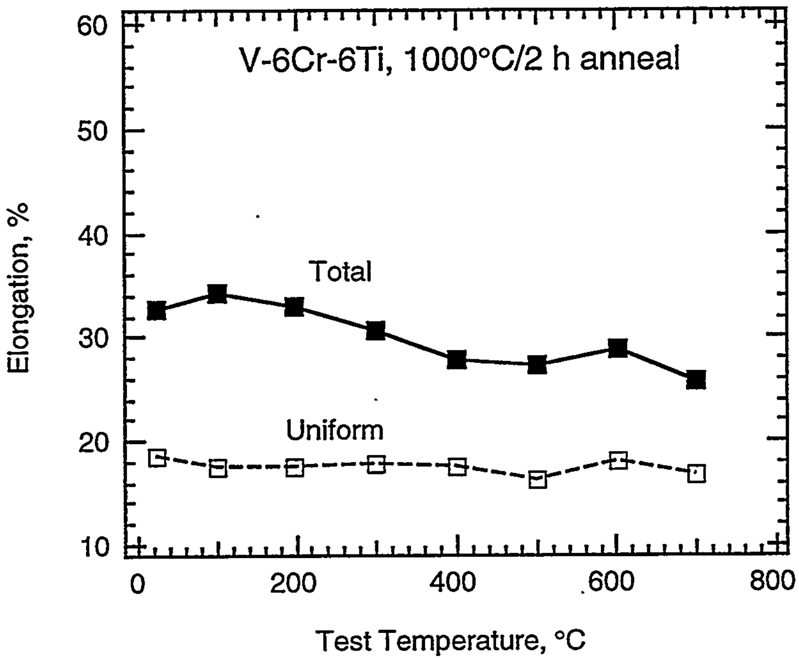

Figure 11. Variation of uniform and total elongations with test temperature for the V-6Cr-6Ti alloy.

The stress–strain curves for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys are smooth with no serrations in the temperatures ranging from room temperature to 300°C. For temperatures above 300°C, discontinuous yielding is observed in all three alloys. Figures 12, 13, and 14 show flow curves (partial curves are shown for temperatures from 300 to 700°C for the V-4Cr-4Ti and V-6Cr-6Ti alloys due to the failure of a computer used in data acquisition during testing) at various test temperatures. At 400°C, *Type A* (periodic in nature) appear after a small strain beyond the lower yield point in all the three alloys. In addition to *Type A* serrations in the beginning of the stress–strain curve, V-6Cr-6Ti showed *Type B* (oscillating) after some strain. Figure 15 shows the variation of serration magnitude with test temperature for the three alloys. The magnitude of the *Type A* serrations is similar in these alloys in the range of 8–10 MPa. With an increase in temperature to 500°C, *Type B* serrations appear immediately following the lower yield point for all the three alloys. At this temperature, the magnitude of *Type B* serrations is around 10 MPa in V-3Cr-3Ti and much higher in the range of 16–18 MPa in V-4Cr-4Ti and V-6Cr-6Ti, see Figure 15. A further increase in temperature to 600°C causes the mode of serrations to change to *Type C* (always below the general stress–strain level) in all the three alloys. These serrations appear generally after some strain and their magnitude is similar for all three alloys around 22–25 MPa, see Figure 15. At 700°C, *Type C* serrations appear in the three alloys after an appreciable amount of strain. The frequency of the serrations decreases whereas their magnitude increases (except in V-3Cr-3Ti where it remains the same as at 600°C) to around 28–33 MPa, see Fig. 15. In addition to the discontinuous yielding or serrations in the flow curves, the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys exhibit a general trend of increasing work hardening ability with increase in temperature which is quite evident from the stress–strain curves in Figures 12, 13 and 14. Another observation is that all the three alloys showed minima in both yield and ultimate tensile strengths at 300°C, the highest temperature at which no serrations were seen in the stress–strain curves. With the increase in temperature above 300°C, the serrations start appearing, and the ultimate tensile strength increases monotonically with increase in the height of serrations with temperature, compare Figures 6, 7, 8, with Figure 15.

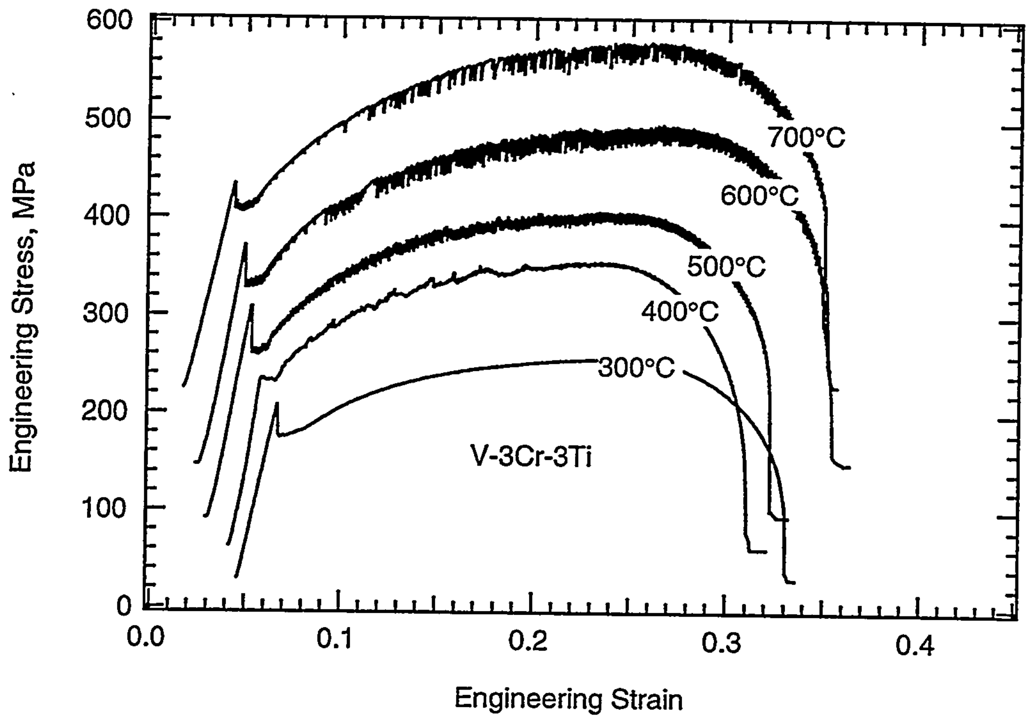

Figure 12. The stress–strain curves as a function of test temperature for the V-3Cr-3Ti alloy.

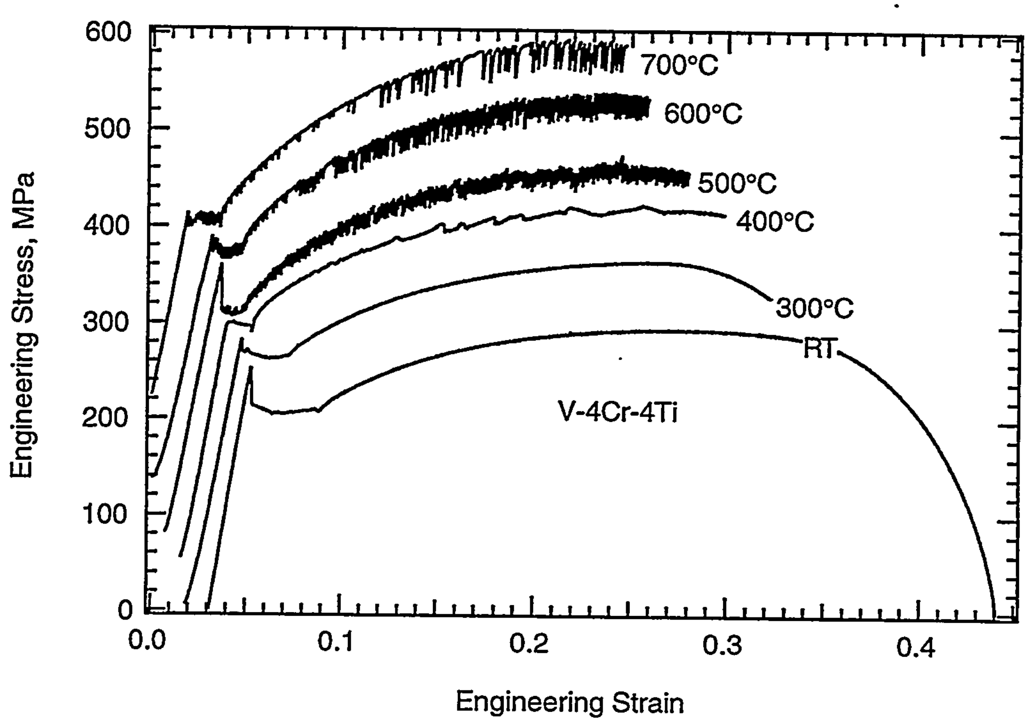

Figure 13. The stress–strain curves as a function of test temperature for the V-4Cr-4Ti alloy.

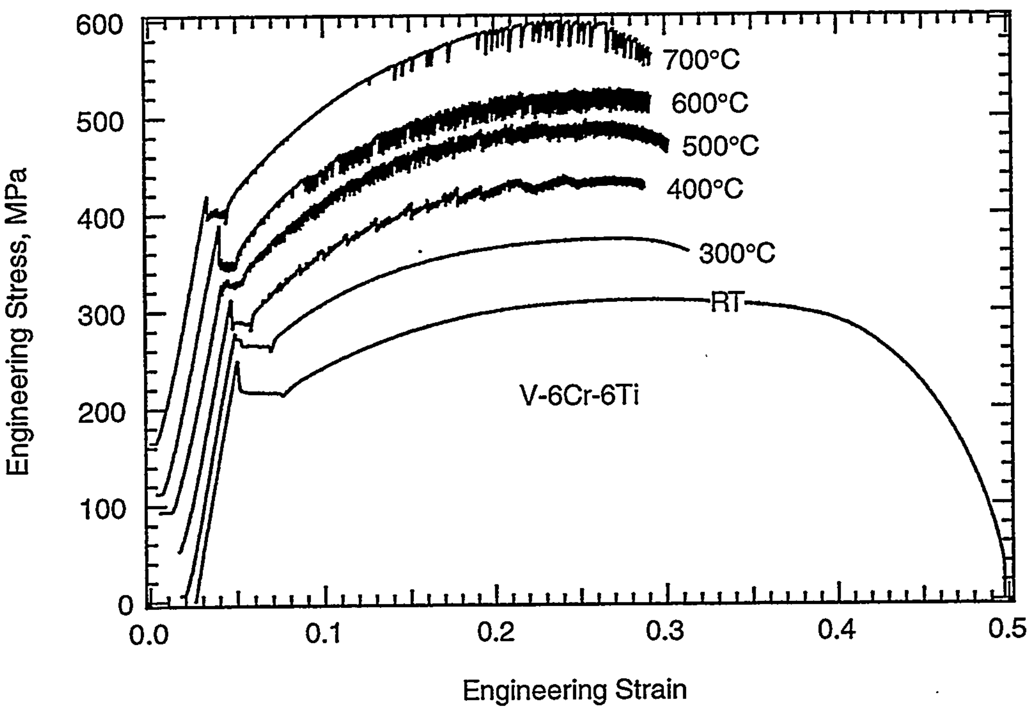

Figure 14. The stress–strain curves as a function of test temperature for the V-6Cr-6Ti alloy.

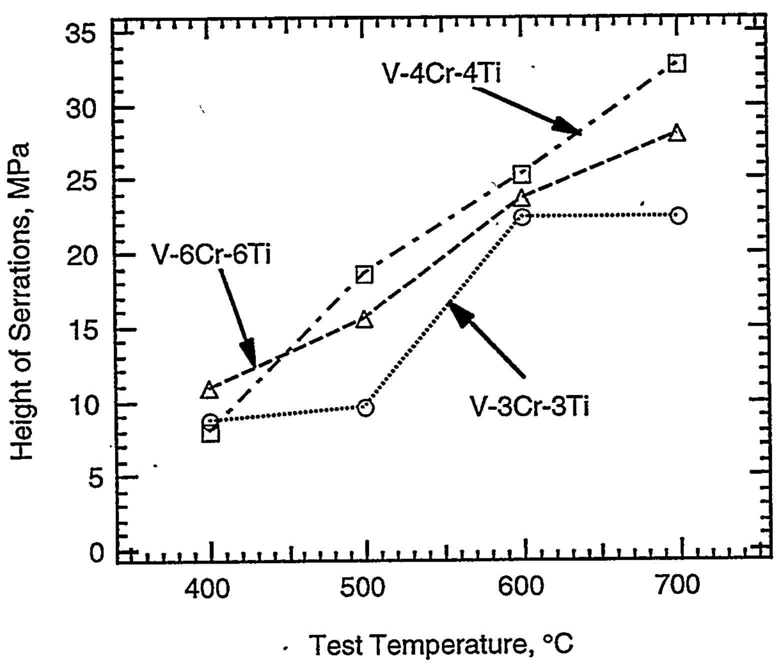

Figure 15. Plot of the variation in height of serrations with test temperature for the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys.

Dynamic strain aging due to solute atoms has been identified as the probable cause for the appearance of serrations in the stress–strain curves of many alloys.[12-18] The vanadium alloys examined in the present study contain the interstitial impurities, oxygen (around 250 wppm), carbon (around 100 wppm), and nitrogen (around 100 wppm). At low temperatures, these interstitials are relatively immobile compared to dislocations, and hence, no serrations are observed for test temperatures up to 300°C. At temperatures of 400°C and above, the interstitials atoms become sufficiently mobile so that they can diffuse fast enough to form a solute atmosphere around the freely moving dislocations after the initial yielding, reducing their velocity and eventually pinning them. Rapid dislocation multiplication takes place with further increase in load, the overall effect of which is to cause serrations in the flow curves and an increase in yield and tensile strengths. This enhanced multiplication of dislocations, rather than unlocking of existing ones, is quite evident from the increased work hardening ability (which indicates continuous creation of new dislocations). Cottrell,[19] in his work on interstitial strain-aging in iron, established a relationship between strain rate Φ and diffusivity of nitrogen atoms D as

$$\Phi = 10^9 D \qquad (2)$$

for calculating the minimum temperature for serrated yielding. In equation (2), D is given by

$$D = D_o \exp\left[\frac{-Q}{RT}\right] \qquad (3)$$

where D_o is the diffusion coefficient, Q is the activation energy, with R and T having the usual meaning. Equation (2) can be used to estimate the minimum temperatures for serrated yielding for the three interstitials, oxygen, carbon, and nitrogen in the present study. By using the values for diffusion coefficient and activation energy for oxygen, carbon, and nitrogen in vanadium[18] in Table 1, one can estimate the temperatures for each interstitial element.

By using the strain rate value of 1.1×10^{-3}/s used in the tensile testing in this study in equation (2), the temperatures so determined for oxygen, carbon, and nitrogen are 370°C, 350°C, and 480°C, respectively. These calculations suggest that the serrations in the stress–strain curves of the V-3Cr-3Ti, V-4Cr-4Ti and V-6Cr-6Ti alloys at ~400°C are most probably related to the mobility of either oxygen or carbon, or both.

Table 1. Diffusion coefficient and activation energy for various interstitial elements

Diffusing Element	D_o cm^2/s	Q kJ/mole
Oxygen	0.0025	115
Carbon	0.0047	114
Nitrogen	0.0018	147

For temperatures of 500°C and above, the strain-aging phenomenon is probably controlled by the diffusion of nitrogen.

Effect of Hydrogen

Cohron et al[9] found in their study on the effect of hydrogen on the intermetallic Ni_3Al alloy that the tensile elongation was affected by whether the ionization gage, which is used to measure the hydrogen pressure, was on or off. There was a significant reduction in the elongation of Ni_3Al when the ion gage was on as opposed to the much higher elongations obtained with the gage off. They proposed that when the gage is on, molecular hydrogen is converted into atomic hydrogen by the tungsten filament, and this atomic hydrogen embrittles the Ni_3Al specimens. The present work was limited to determining whether or not the V-4Cr-4Ti alloy would be similarly embrittled by exposure to an atmosphere containing atomic hydrogen. Exposure and tensile testing were carried out under hydrogen partial pressures of 30 and 50 torr

(3 and 5 KPa). Cohron et al[9] have determined the true temperature of the ion gage filament, with corrections made for emissivity of tungsten and absorption by the Pyrex viewport, to be around 1677°C (with the temperature measured with an optical pyrometer being ~1552°C). They estimated that at this temperature and 1 torr (1.3×10^2 Pa) pressure of hydrogen, approximately 10% of the hydrogen gas is in the form of atomic hydrogen, with the degree of hydrogen dissociation decreasing with increase in pressure. As shown in Figure 16, the ductility of the V-4Cr-4Ti alloy remains the same under high vacuum (~5×10^{-10} torr or 5×10^{-8} Pa) conditions and under the conditions which create atomic hydrogen. The proposed mechanism[9] for embrittlement of Ni_3Al involves the diffusion of hydrogen to the crack tip. In the case of V-4Cr-4Ti studied in the present work, this type of hydrogen embrittlement was not observed and the alloy remained ductile for hydrogen partial pressures up to 50 torr (5 KPa).

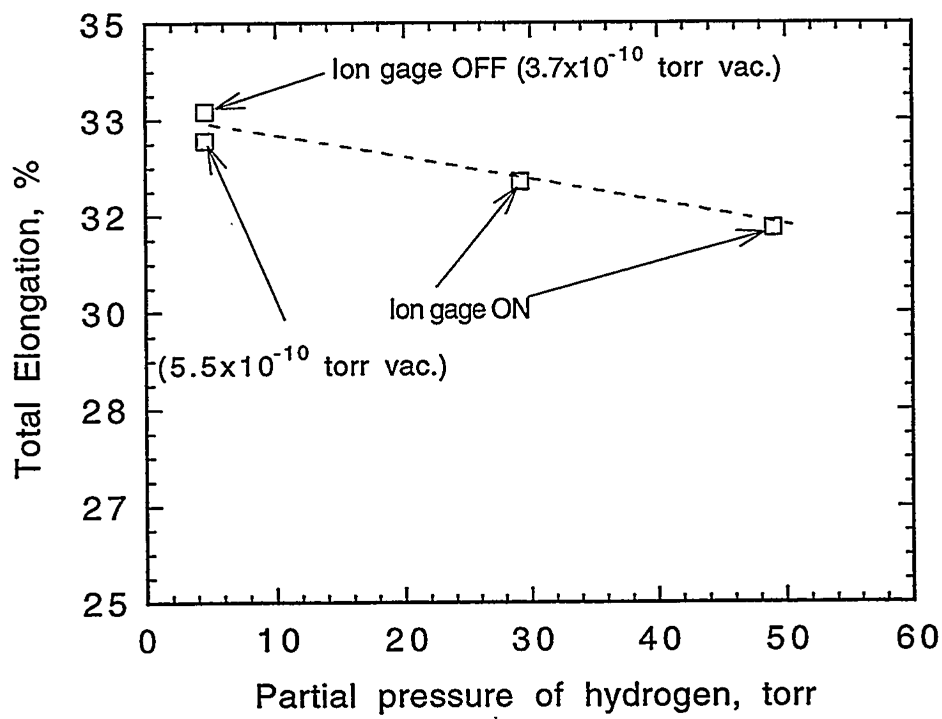

Figure 16. The total elongation as a function of the partial pressure of hydrogen for the V-4Cr-4Ti alloy.

SUMMARY AND CONCLUSIONS

The results from this investigation reveal that:

(1) Of the three vanadium alloys tested, V-6Cr-6Ti was the strongest and V-3Cr-3Ti was the weakest with V-4Cr-4Ti showing intermediate yield and ultimate tensile strengths at all strain rates. The strain–rate sensitivity of the vanadium alloys was found to be similar to that of unalloyed vanadium.

(2) All the three alloys exhibited almost no change in uniform and total elongations up to a strain rate of 10^{-1}/s, followed by a decrease with a further increase in strain rate.

(3) The ultimate tensile strengths of all the alloys exhibited minima at 300°C, whereas the yield strength, total, and uniform elongations were relatively insensitive to temperatures between 400 and 700°C.

(4) Serrated yielding (indicative of dynamic strain aging) was observed in the stress-strain curves of all the alloys at test temperatures above 300°C.

(5) At room temperature, the test environment (vacuum, air and atomic hydrogen) showed no significant influence on the tensile behavior of the V-4Cr-4Ti alloy.

ACKNOWLEDGMENTS

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