



ELSEVIER

Journal of Nuclear Materials 273 (1999) 102–110

Journal of
nuclear
materials

www.elsevier.nl/locate/jnucmat

Reactions of hydrogen with V–Cr–Ti alloys¹

J.R. DiStefano^{a,*}, J.H. De Van^a, D.H. Röhrig^b, L.D. Chitwood^a

^a Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6157, USA

^b University of Karlsruhe, D76000 Karlsruhe, Germany

Received 8 July 1998; accepted 12 November 1998

Abstract

The interest in vanadium alloys for use as a first-wall material in fusion reactor applications has led to a study to determine the effects of hydrogen on the mechanical properties of certain V–Cr–Ti alloys. Hydrogen uptake by the alloys is a function of temperature and pressure. In the absence of increases in oxygen concentration, additions of up to 400 wppm hydrogen to V–4Cr–4Ti did not result in any significant embrittlement. However, when hydrogen approached 500 wppm, rapid embrittlement occurred at 325°C that was suggested to result from formation of a hydride phase upon cooling. When oxygen was added as well, either during or prior to hydrogen exposure, synergistic effects depending on grain size occurred that led to significant embrittlement by 35–130 wppm hydrogen. Because of its synergism with hydrogen and ubiquitous presence in most environments, oxygen pick-up remains as one of the major concerns for use of V–Cr–Ti alloys in fusion reactor applications. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

Vanadium alloys have attractive properties as a first-wall material in fusion reactor blanket applications, and designs have been proposed with liquid lithium as the coolant/tritium breeding material and a vanadium alloy as the structural/containment material [1,2]. A major concern is the affinity of vanadium alloys for gases such as hydrogen and oxygen. The purpose of this study was to assess the effects of hydrogen (and, by analogy, deuterium and tritium) on the mechanical properties of V–Cr–Ti alloys. Although effects of oxygen contamination were not included in the original objectives of this study, its ubiquitous presence during initial testing profoundly affected the results that were obtained and ultimately led to a companion study of oxygen effects on V–Cr–Ti alloys [3].

Given the fusion neutron energy spectrum (up to 14 MeV), hydrogen is produced as a transmutation product in situ in vanadium alloys, the concentration being determined by the neutron fluence and the diffusivity of hydrogen. Additionally, deuterium and tritium comprise the primary fuel components and can also contact the first-wall, albeit at relatively low pressure. To the extent that water is used as a coolant, corrosion reactions portend another potential source of hydrogen.

As yet there are very little data on hydrogen effects on the vanadium alloys considered as candidates for fusion application. However, given the relatively high vanadium content of the alloys (generally 90 wt% or greater), it is useful to review hydrogen effects reported for pure vanadium. Vanadium, although relatively ductile when tested in air, may exhibit a significant reduction in ductility when tested in a hydrogen environment [4–8]. Embrittlement in hydrogen depends on temperature, strain rate, and the impurity content of the metal. Although high purity vanadium shows no susceptibility to embrittlement when tested at room temperature in air or hydrogen at 10⁵ Pa (1 atm), vanadium containing increased amounts of the interstitials oxygen or nitrogen can exhibit significant embrittlement at room temperature in hydrogen, even though there is little effect in air [4]. This increased susceptibility to

* Corresponding author. Tel.: +1-423 574 4462; fax: +1-423 241 0215; e-mail: distefanojr@ornl.gov.

¹ Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

embrittlement due to interstitial alloying is believed to be related to an increase in strength, because cold work and associated grain size, which also increase strength, similarly reduce ductility in hydrogen. For any given alloy and test condition, there is a threshold hydrogen pressure below which embrittlement does not occur and above which the fracture stress decreases linearly with the square root of hydrogen pressure [5]. If hydrogen is present in vanadium prior to testing, it can exhibit brittle fracture near ambient temperature and below. This internal hydrogen embrittlement does not require hydrogen to be present in the test atmosphere. Since the solubility of hydrogen in vanadium increases at temperatures above ambient, significant hydrogen pickup can occur by exposure to hydrogen-containing environments at higher temperatures, e.g., above 300°C. On cooling the hydrogen-containing metal to room temperature, hydrogen in solid solution becomes unstable when the temperature falls below the hydrogen solus, and a hydride (β -phase) may precipitate, resulting in a significant reduction in ductility [6–8]. Higher hydrogen contents shift the ductile-to-brittle transition to higher temperatures, consistent with the temperature dependence of the hydrogen solvus.

2. Experimental

Two slightly different alloys with the following nominal compositions were investigated: V–5Cr–5Ti and V–4Cr–4Ti. (Concentrations are in weight percent.) Two heats of V–5Cr–5Ti were tested, and the respective chemistries are shown in Table 1. The heat identified as 832394 was produced commercially by Teledyne–Wah Chang, and the other, identified as ORNL, was a 500-g heat produced at Oak Ridge National Laboratory. The composition of the single V–4Cr–4Ti heat, produced by Teledyne–Wah Chang, is also shown in Table 1. Note that the ORNL heat contains a significantly higher concentration of nitrogen and carbon than the other two heats. The alloys were tested in the form of small tensile specimens, nominally 0.76 mm thick with 1.5×7.6 mm long gage sections, that were either machined or stamped from warm-reduced sheet stock. Following fabrication, the specimens were cleaned by acid-etching in a mixture of HF–HNO₃. Samples were given various heat treatments before and after exposure to hydrogen

to evaluate different microstructural features or phases within the alloy. These treatments were conducted in vacuum furnaces at total pressures $\leq 10^{-4}$ Pa.

When exposed to low-pressure hydrogen, the vanadium alloys were placed in an ultra-high vacuum apparatus consisting of a gas supply, an alumina reaction tube coupled to a vacuum system, and a high temperature furnace around the reaction tube. Prior to introducing hydrogen, the system was outgassed at 500°C until a vacuum of 10^{-6} Pa (10^{-8} torr) was established. High purity hydrogen gas (99.9999%) was admitted through a controllable leak valve while the system was being evacuated by a turbomolecular pump. The pressure at the specimen was determined from the pumping speed, conductance of the system, and ion-gage pressure measurements at the entrance and exit of the reaction tube. At 10 Pa, a variable capacitance diaphragm sensor (Baratron) was used to monitor pressure instead of an ion-gage. The temperatures of the exposures were in the range 325–500°C. The amounts of hydrogen picked up by the specimens were monitored by weight changes and by selective chemical analyses after the exposures. During the course of the investigation, the gas supply system was modified by the addition of a hydrogen purifier that contained a Pd–Ag alloy membrane to exclude oxygen and water vapor from the test environment.

3. Results

3.1. Initial exposures in low-pressure hydrogen

Initial tests in hydrogen were conducted on the two V–5Cr–5Ti heats. Hydrogen pressures were fixed in the range 10^{-2} – 10 Pa (10^{-4} – 10^{-1} torr), and exposures were generally for 24 and 100 h, respectively. Following the exposures, specimens were either left in the furnace in the hydrogen atmosphere to cool to room temperature (termed slow cooling) or were immediately pulled from the hot zone to an unheated region (termed fast cooling).

Weight gains generally exceeded those attributable to hydrogen alone. In all cases the additional weight gains correlated closely with increases in oxygen content. For any given exposure condition, the uptake of hydrogen was similar for the two heats. However, the uptake did depend on the rate at which the specimens were cooled

Table 1
Compositions of V–Cr–Ti alloys

Nominal composition (wt%)	Heat ID	Concentration (wt%)			Concentration (wppm)			
		Cr	Ti	Fe	O	N	C	Si
V–5Cr–5Ti	ORNL	4.0	5.6	0.11	324	512	204	1100
V–5Cr–5Ti	832394	4.2	5.4	<0.045	427	52	40	<310
V–4Cr–4Ti	8326	3.1	4.1	0.022	310	85	86	780

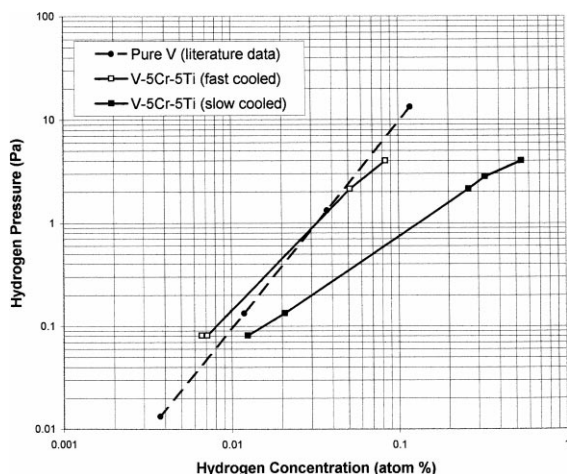


Fig. 1. Hydrogen concentrations in V-5Cr-5Ti and unalloyed vanadium [9,10] after exposure to low-pressure hydrogen at 500°C.

from the test temperature while in the hydrogen environment. As shown in Fig. 1, specimens slowly cooled under the hydrogen exposure pressure showed significantly higher hydrogen concentrations than companion specimens moved quickly to a relatively cool region of the furnace. Note that the hydrogen concentrations of the fast cooled specimens were similar to those reported previously for pure vanadium at a comparable exposure temperature [9,10].

The effect of hydrogen on room temperature ductility differed significantly between the two V-5Cr-5Ti heats and between the same heat when samples were pre-annealed at 1125°C. As shown in Fig. 2(a), the ORNL heat showed little effect of hydrogen on ductility until the concentration approached 80 ppm, where there was also a significant uptake of oxygen. For the same weight change (hydrogen plus oxygen), a much greater decrease in ductility for a given hydrogen exposure occurred in the case of heat 832394 (Fig. 2(a)). The grain size of heat 832394 was much coarser (ASTM 5–6) than the ORNL heat (ASTM 9). Note that a difference in both grain size and ductility for heat 832394 resulted even though it received ostensibly the same annealing treatment (1 h/1125°C). Although this effect was not clarified, it was possibly due to slightly different fabrication histories. In all cases, the coarser the grain size of the specimen, the greater the effect of the hydrogen exposure on the ductility. For example, a specimen with ASTM grain size 5, with 20 ppm hydrogen and a very minor uptake of oxygen, exhibited an elongation of only 3.1%, while a specimen with ASTM grain size 6 and 47 ppm hydrogen showed an elongation of 6.8% (Fig. 2(a)). As shown in Fig. 2(b), the tensile strengths of the two V-5Cr-Ti heats were increased by the uptake of hydrogen, the

increases being roughly proportional to the hydrogen contents (up to the point of brittle fracture).

Since grain size depends upon the temperature of the annealing heat treatment, specimens of heat 832394 were annealed at 1050°C or 1125°C, respectively, and then exposed side-by-side in 10 Pa (0.1 torr) hydrogen at 450°C for times of 24 and 100 h, respectively. Prior to hydrogen exposure, elongations for this group of specimens were in the range of 27–29% irrespective of the annealing temperature. As shown in Table 2, after hydrogen exposure room temperature ductilities of specimens annealed at 1050°C were significantly higher than the corresponding ductilities of specimens annealed at 1125°C. While specimens annealed at 1050°C showed a slight drop in elongation, specimens annealed at 1125°C showed elongations as low as 2%.

Specimens of heat 832394 annealed at 1125°C exhibited significantly less embrittlement when the hydrogen pressure was reduced to 1 Pa (0.01 torr) than for exposures at 10 Pa (0.1 torr). In contrast to elongations of 2–5% for most exposures at 10 Pa, elongations after exposure at 1 Pa were all above 14%. However, the detrimental effects on ductility at the higher hydrogen pressure (10 Pa) were clearly augmented by oxygen contamination, which increased as the hydrogen pressure increased. Fig. 3 shows a typical fracture cross section of the coarse-grained V-5Cr-5Ti specimens that underwent simultaneous ingress of oxygen and hydrogen at 500°C. The fractures were predominately intergranular except for a shallow cleavage zone near the exposed surface. Post-exposure heat treating the V-5Cr-5Ti alloy in vacuum for 100 h at 500°C was effective in removing hydrogen from the alloy as shown in Table 3. Although this treatment recovered most of the ductility in the finer-grained ORNL heat, heat 832394 remained brittle. Based on previous studies that showed this latter heat was more easily embrittled by oxygen as well, this was an indication that oxygen contamination was a significant factor in the embrittlement of the coarse-grained heat [3].

3.2. Exposures to pure hydrogen

As described above, the hydrogen environment in the initial exposures contained sufficient oxygen such that both gases were introduced into the V-5Cr-5Ti alloy specimens. Subsequently, the test system was modified by the addition of a hydrogen purifier (diffusive barrier) and a quadrupole residual gas analyzer. The Pd-Ag alloy membrane in the purifier permitted only hydrogen to pass through, effectively eliminating oxygen as an impurity in the hydrogen environment. The effectiveness of the purifier was demonstrated by the residual gas analyzer which indicated that, except for a small amount of residual water vapor in the system, hydrogen was the only gas that could be measured. Furthermore, chemical

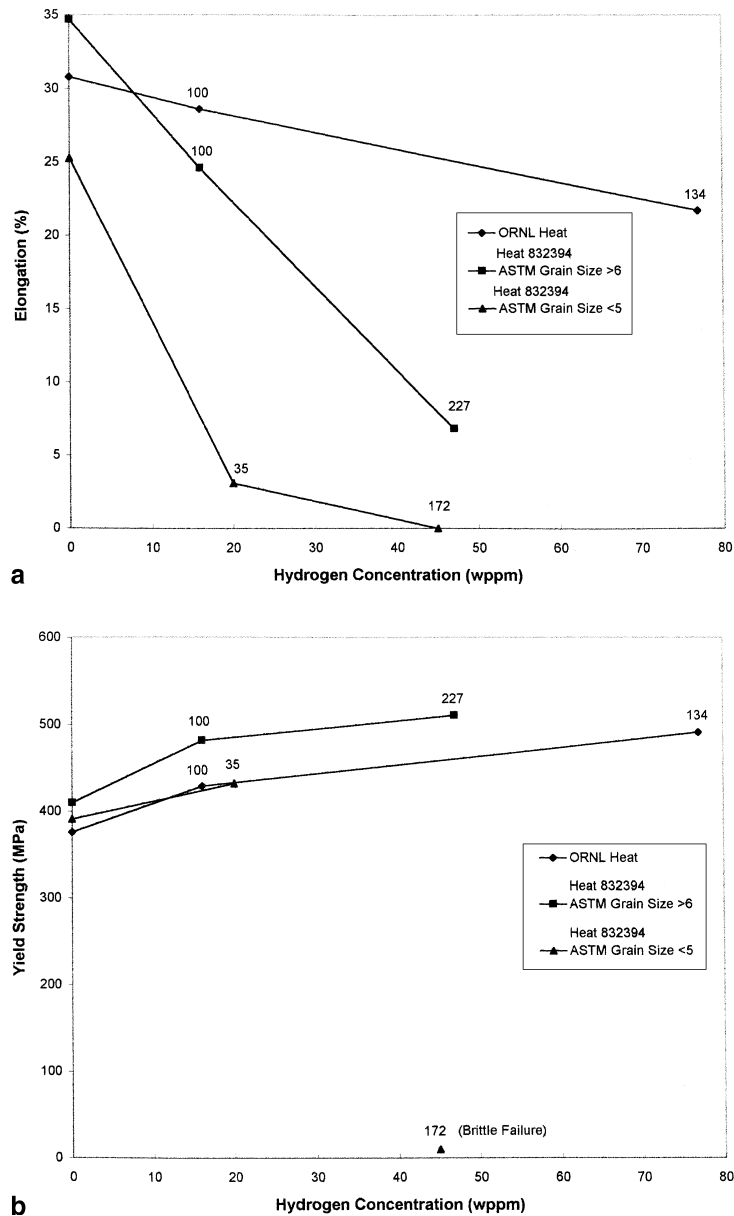


Fig. 2. Room temperature elongation (a) and yield strength (b) of two heats of V–5Cr–5Ti annealed at 1125°C prior to exposure to high purity hydrogen gas at 500°C. Numbers above data points show increases in oxygen concentration (ppm) accompanying hydrogen exposures.

analyses of selected specimens did not indicate any significant oxygen increase as a result of the hydrogen exposures.

Hydrogen uptake by V–4Cr–4Ti was determined at 10 Pa ($\sim 10^{-1}$ torr) at 450°C and between 25 and 250 Pa (0.2–2 torr) at 325°C, as shown in Fig. 4. As reference, two V–H isotherms for 450 and 325°C have also been drawn based on the phase diagram of pure V–H [10], and these isotherms generally subscribe to the V–4Cr–

4Ti pressure–temperature measurements. With these conditions, we were initially able to achieve hydrogen concentrations in V–4Cr–4Ti up to 400 ppm and ~ 700 ppm, but intermediate concentrations were not achieved. However, modification of the cooling rate/pressure reduction schedule subsequently did allow these intermediate levels to be achieved.

Results of room temperature tensile tests conducted on V–4Cr–4Ti after exposure to hydrogen at 325°C are

Table 2

Effect of annealing temperature and hydrogen pressure on room temperature properties of V–5Cr–5Ti heat 832394 (Hydrogen exposure at 450°C)

Annealing temperature (°C)	Hydrogen pressure (torr)	Exposure time (h)	Weight change (ppm)	Yield strength (MPa)	Ultimate strength (MPa)	Elongation (%)
1050		As-received ^a		344	423	29.0
1050	0.1	24	38 ^a	404	494	21.3
1050	0.1	100	423 ^a	414	499	19.2
1125		As-received ^a		423	503	27.3
1125	0.1	24	308 ^a	430	494	10.0
1125	0.1	24	348 ^a	441	476	5.6
1125	0.1	100	231 ^a	451	451	1.6
1125	0.01	24	59 ^b	396	469	20.7
1125	0.01	100	58 ^b	395	474	21.3

^aSpecimen thickness = 0.61 mm.

^bSpecimen thickness = 0.76 mm.

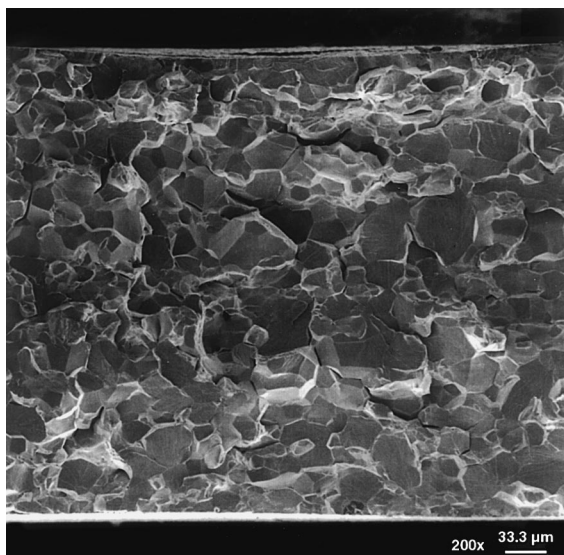


Fig. 3. Fracture cross-section of coarse-grained V–5Cr–5Ti specimen (annealed at 1125°C) showing predominately intergranular cracking. Specimen was exposed to H₂ at 1 Pa (10^{–2} torr) at 500°C for 166 h and picked up 28 ppm H and 960 ppm O.

summarized in Fig. 5. Without a concomitant pickup of oxygen, hydrogen concentrations >500 ppm were required before significant embrittlement of the V–4Cr–4Ti specimens occurred. Increasing the grain size of the samples by increasing their pre-test annealing temperature reduced their ductility at hydrogen levels up to 400 ppm (Fig. 5), but it was not as large an effect as in the previous case of V–5Cr–5Ti, where oxygen was also present.

The synergistic effect of oxygen in combination with hydrogen was also determined for V–4Cr–4Ti (Fig. 5).

An initial addition of ~850 ppm oxygen at 500°C followed by an aging treatment for 25 h at 800°C in vacuum reduced its room temperature elongation from approximately 30% to 20%. However, after the same oxidation treatment, subsequent exposure to hydrogen drastically reduced its elongation to 5% after only 100 ppm hydrogen had been added. Without the addition of oxygen, >500 ppm hydrogen was required before room temperature ductility fell precipitously. Fracture cross-sections at the higher and lower hydrogen concentrations are compared in Fig. 6.

4. Discussion

The data in Fig. 4 for hydrogen concentration in V–4Cr–4Ti as a function of pressure at 325°C can be described by a Sievert's law relation and correlate with data for fast-cooled V–5Cr–5Ti specimens at 500°C, shown in Fig. 1. The agreement of the present data with isotherms derived from the literature for the V–H system demonstrates that the V–Cr–Ti alloy behaves similarly to pure vanadium with respect to hydrogen uptake.

In general, the room temperature properties of all the V–Cr–Ti test heats prior to hydrogen exposure were similar, and fracture morphologies were classic cup/cone with relatively large reductions in area. Aging treatments in ultra-high vacuum for long times (3000 h) at temperatures of 400, 500, and 600°C did not change ductility or the mode of fracture. When oxygen was excluded, the addition of up to 400 ppm hydrogen increased the room temperature tensile strength slightly with only a small decrease in tensile elongation (Fig. 5). The fractures tended to be of the cup/cone type, similar to that of the as-received alloys. However, a further increase in hydrogen concentration to 700 ppm resulted in complete embrittlement (Fig. 5). As shown in Fig. 7,

Table 3

Effect of hydrogen exposure at 3×10^{-2} torr on V-5Cr-5Ti at 500°C before and after vacuum heat treating at 500°C^a (Exposure and heat treating times were 100 h, respectively)

Heat	Cooling rate in hydrogen	Vacuum heat treat	Concentration (ppm)		Yield strength (MPa)	Ultimate strength (MPa)	Elongation (%)
			Hydrogen	Oxygen uptake			
832394	Fast	No	45	172	FBY ^b	FBY ^b	0.0
832394	Slow	No	107	245	FBY ^b	FBY ^b	0.0
832394	Slow	Yes	2.4	185	506	515	0.3
ORNL	Fast	No	59	284	476	515	18.0
ORNL	Slow	No	150	332	493	533	7.0
ORNL	Slow	Yes	0.8	246	453	561	22.6

^aSamples annealed at 1125°C prior to exposure to hydrogen.

^bFailed before yield.

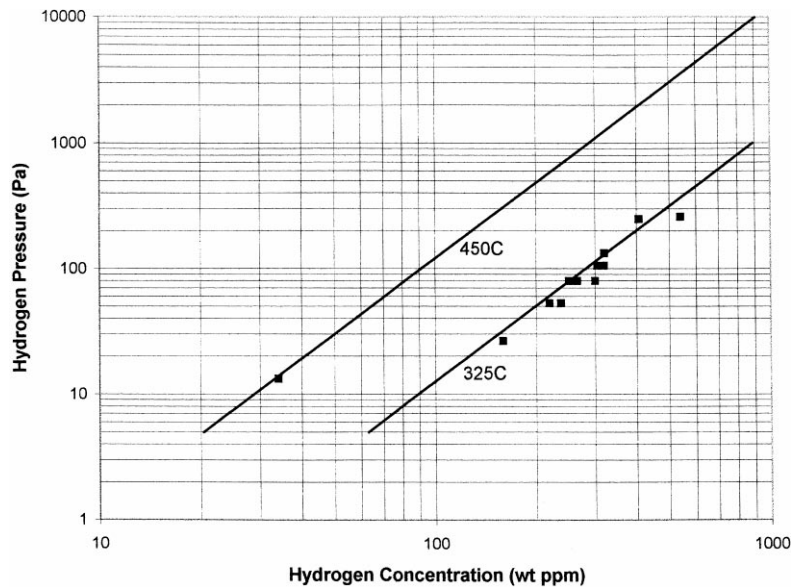


Fig. 4. Comparison of hydrogen concentrations in V-4Cr-4Ti with calculated concentrations in unalloyed vanadium at 450 and 300°C [9,10].

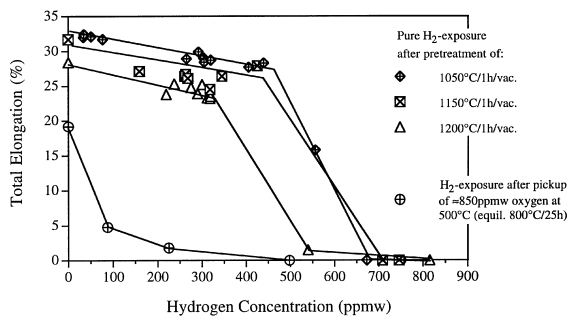


Fig. 5. Effect of hydrogen on room temperature elongation of V-4Cr-4Ti.

this hydrogen concentration corresponds to the solubility threshold for hydride formation in pure vanadium below 80°C and is indicative that the ductile–brittle transition for specimens containing more than 400 ppm hydrogen is most likely associated with hydride formation.

The technique of hydrogen doping at 325°C was straightforward in achieving room temperature hydrogen concentrations up to 400 ppm and over 700 ppm, but intermediate concentrations were more difficult to establish. After the exposure at 325°C, depending on the rate of cooling and hydrogen pressure in our tests, hydrogen was either lost as excess hydrogen diffused out of

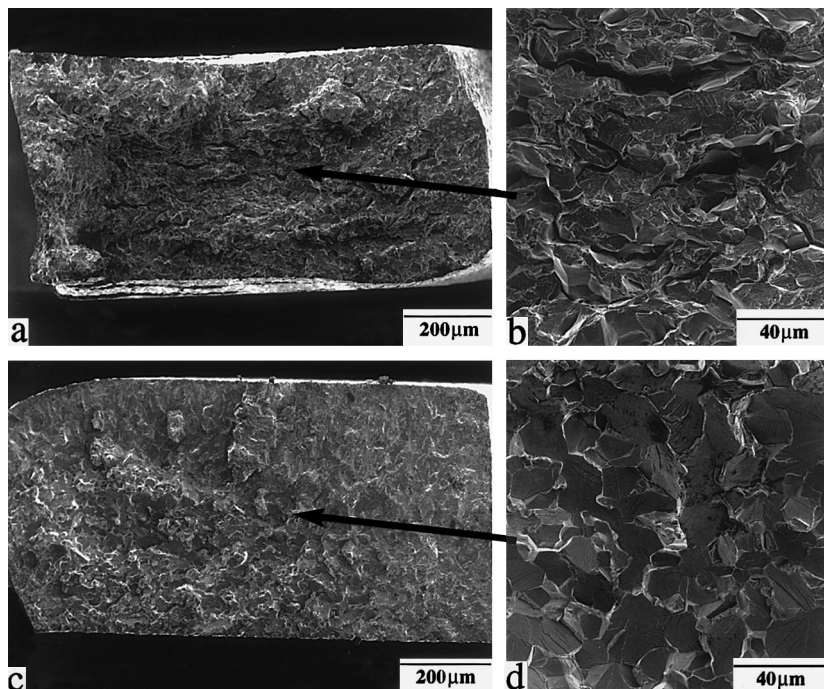


Fig. 6. SEM secondary electron images of V-4Cr-4Ti tensile fracture sections. Specimens were pre-exposed to oxygen (≈ 850 ppm) and aged at 800°C prior to hydrogen exposure at 325°C . The region of brittle cleavage and intergranular cracking increased in depth as H concentration increased from (a,b) 900 ppm to (c,d) 500 ppm.

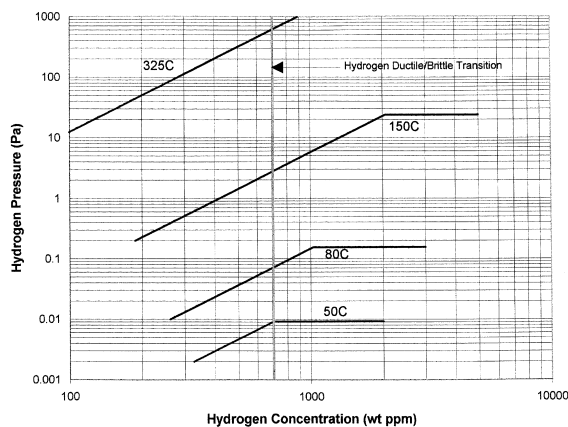


Fig. 7. Hydrogen concentrations in unalloyed vanadium at 50– 325°C [10]. Horizontal portions represent two-phase regions (hydride + solid solution).

the specimen as the pressure and temperature were lowered, or additional hydrogen was absorbed when the hydrogen pressure was maintained while the specimen was cooled. Only by applying a sophisticated cool down/pressure reduction strategy in which hydrogen pressure

was carefully controlled (reduced) as the specimen cooled were we able to achieve a few intermediate hydrogen levels (500–600 ppm) in the alloy specimen. Although not completely embrittled, the latter specimens showed properties more typical of 700 ppm than specimens containing 400 ppm hydrogen, a result pointing to the presence of hydrides in these specimens.

Despite the use of high purity hydrogen and an ultra high vacuum containment system, concomitant oxygen contamination was a persistent problem in the initial hydrogen exposures, and it was found that the greater the oxygen content the less hydrogen that was needed to cause embrittlement. Such an effect has also been previously reported in a niobium–hydrogen study [11]. For the V–Cr–Ti alloys, the combined effect of oxygen and hydrogen on reducing ductility at room temperature was much greater than the sum of individual effects. When oxygen contamination occurred, the annealing treatment and, therefore, the grain size had a significant effect on the ductility of V-5Cr-5Ti heat 832394. Heat treatment of the latter heat at 1125°C resulted in an ASTM grain sizes from 5–6, and this heat was seriously embrittled by relatively small concentrations of hydrogen and oxygen. In contrast, when annealed at 1050°C (ASTM grain size 9), this same heat showed ductilities above 19% at similar contamination levels (Table 2).

The other V–5Cr–5Ti heat retained a grain size of ASTM 9 even when annealed at 1125°C and also showed a much smaller decrease in ductility at these contamination levels. When embrittled by hydrogen/oxygen, the coarser-grained specimens exhibited primarily intergranular fractures as illustrated in Fig. 3, while the finer-grained specimens were less embrittled and showed mainly ductile cleavage. This fracture morphology was similar to that induced by oxygen alone, but the relative depth of the intergranular region appears to increase when hydrogen is added. Although there were some differences in chemistry among the three heats, grain size was the only consistent parameter that correlated with embrittlement effects.

In a previous paper [3] which discussed the effect of oxygen on the V–Cr–Ti alloys, a model was proposed to account for the intergranular nature of fractures produced by oxygen alone. The model attributed the intergranular weakness to a depletion of titanium near the grain boundary, induced by oxygen diffusing along the boundary. Assuming such a model to be operative, the strengthening of the grain matrix by hydrogen would exacerbate the relative weakening of the near-boundary region by titanium depletion and could account for the present findings. Alternative explanations could involve a shifting of the hydride solvus by oxygen to lower hydrogen concentrations [12], or an effect of hydrogen on the extent and morphology of oxide precipitates on the boundary. Each of these potential effects is presently being examined using transmission electron microscopy.

In the case of a fine-grained V–4Cr–4Ti specimen containing ~850 ppm oxygen, ductility showed a sharp decrease with as little as 100 ppm hydrogen added, but the ductility did not fall to zero until the calculated hydrogen solubility limit was reached. This result can be explained by observations of the fracture cross sections. As shown in Fig. 6, the alloy retained a relatively ductile core until the hydrogen concentration approached its solubility limit. Oxygen apparently was confined to a fixed depth immediately below the surface, and embrittlement was associated with the combined effects of hydrogen and oxygen in this outer case. However, the initiation of cracks in this outer case could also have affected the fracture toughness of the more ductile but hydrogen-strengthened core, which further contributed to a reduction in ductility.

If the hydrogen pickup is below that for hydride formation, its effect on mechanical properties can be reversed by vacuum annealing, a process that desorbs the hydrogen and effectively restores the as-received condition. This was demonstrated by exposing a group of V–5Cr–5Ti fine-grained specimens to hydrogen for 100 h at 3 Pa (3×10^{-2} torr) at 500°C and then vacuum annealing one of the specimens at 500°C for 100 h at 10^{-6} Pa (10^{-8} torr). The vacuum heat treatment reduced the hydrogen content from 150 to 0.8 ppm, and the

room temperature elongation increased from 7% to 23%. We did not investigate the effect of desorbing hydrogen from specimens that had been embrittled by relatively large amounts of hydrogen (500 ppm). However, if in the latter case the embrittlement was due primarily to hydride formation, the damaging effects on mechanical properties may not be completely reversed by vacuum annealing, based on related studies of other refractory metals [13].

Where embrittlement is due to the combined effects of oxygen and hydrogen, desorbing hydrogen by vacuum heat treating will generally improve the ductility, although embrittlement induced by oxygen is not reversed by heat treatments in the range normally used to remove hydrogen (500°C). In fact, embrittlement by oxygen is exacerbated by heat treating in the 400–500°C temperature range [3], and the desorption of hydrogen advisedly should be conducted above or below this range. The heat treatment needed to counter embrittlement by oxygen, when it is absorbed at 400–500°C, was shown in an earlier report [3] to be in the range of 950°C and higher, where the oxygen is precipitated from the matrix in the form of TiO₂.

Based on the present results, one can speculate that serious embrittlement of the reference V–Cr–Ti alloys by hydrogen isotopes should not occur under nominal fusion reactor operating conditions, providing that oxygen pickup is carefully controlled. Given present estimates of plasma leakage to the first-wall, our calculations of the deuterium–tritium pressures behind the currently proposed light element coatings (i.e., in cracks or crevices) indicate that pressures should not exceed about 10^{-1} Pa (10^{-3} torr). Hydrogen concentrations in V–Cr–Ti alloys at the latter pressures are well within the solid solution range, and the principal mechanical property effect would be limited to slight hardening. One caveat is a possible additive effect with radiation damage, where protium produced in the alloys by transmutation reactions and radiation hardening may lead to an increase in the ductile-to-brittle transition temperature. Also, depending on design, hydrogen isotopes could affect the properties of V–Cr–Ti alloys if used for components on which plasma directly impinges, such as the divertor.

One of the major concerns in the application of V–Cr–Ti alloys to fusion reactors will be oxygen pickup, particularly in welds during reactor construction and during system bake-out prior to operation. As shown in an earlier paper [3], oxidation that occurs internally at lower temperatures (e.g., 500°C) can of itself cause serious embrittlement, and, as shown here, the uptake of relatively small concentrations of hydrogen further increases the degree of embrittlement for a given level of oxygen. Accordingly, the permissible level of oxygen contamination will be strongly impacted by the uptake of hydrogen isotopes expected during reactor operation.

5. Conclusions

Hydrogen in V–Cr–Ti alloys affects its room temperature tensile properties. In the absence of an increase in oxygen concentration, matrix hardening and a slight effect of grain size on total elongation were noted up to ~400 ppm (~2.2 at.%) hydrogen in the alloys. At higher concentrations, brittle behavior rapidly occurred, which can be attributed to the formation of a hydride phase that forms upon cooling. When oxygen was added (either intentionally or unintentionally) at 500°C, tensile behavior was very strongly affected. It is hypothesized that the combined effect of intergranular embrittlement by oxygen and matrix hardening by hydrogen dramatically reduces the ductility of V–5Cr–5Ti or V–4Cr–4Ti to a greater extent than would be expected based on the effects of the individual gases.

Oxygen pick-up remains as one of the major concerns in the use of V–Cr–Ti alloys for fusion reactor applications, particularly in the case of welds. The uptake of hydrogen isotopes during reactor operation will be an important factor in setting the permissible level of oxygen contamination.

Acknowledgements

The submitted manuscript has been authored by a contractor of the US Government under contract DE-ACO5-96OR22464. Accordingly, the US Government retains a non-exclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for US Government purposes.

References

- [1] J. Bethin, A. Tobin, *J. Nucl. Mater.* 122&123 (1984) 864.
- [2] E.E. Bloom, R.W. Conn, J.W. Davis, R.E. Gold, R. Little, K.R. Schultz, D.L. Smith, F.W. Wiffen, *J. Nucl. Mater.* 122&123 (1984) 17.
- [3] J.R. DiStefano, J.H. DeVan, *J. Nucl. Mater.* 249 (1997) 150.
- [4] A. Clauss, R. Kieger, J. Schmuck, Room Temperature Behavior of Va Metals (V, Nb, Ta) under Hydrogen Exposure, Colloque sur l'Hydrogene des les Metaux, Valduc, France, Commissariat à l'Energie Atomique, 1967.
- [5] J. Schmuck, A. Clauss, Influence of various factors on embrittlement of tantalum in hydrogen atmosphere, Hydrogen in Metals, Editions Science et Industrie, Paris, France, 2 (1972).
- [6] H.K. Birnbaum, et al., The effects of hydrogen on the mechanical properties and fracture of Zr and refractory metals, Hydrogen in Metals, American Society for Metals, Metals Park, Ohio, 303 (1974).
- [7] C.V. Owen, E. Scott, *Metall. Trans.* 3 (1972) 1715.
- [8] S. Takano, T. Suzuki, *Acta Metall.* 22 (1974) 265.
- [9] L.A. Charlot, A.B. Johnson, R.E. Westerman, Solubilities and diffusivities of hydrogen isotopes in niobium, niobium–zirconium alloys, and vanadium, technology of controlled thermonuclear fusion experiments and the engineering aspects of fusion reactors, CONF-721111-23, 1973.
- [10] E. Fromm, E. Gebhardt, *Gase und Kohlenstoff in Metallen*, Springer, Berlin, 1976, pp. 441–460.
- [11] B. Longson, The hydrogen embrittlement of niobium, TRG Report 1035(C), United Kingdom Atomic Energy Authority, The Reactor Group, Risley, Warrington, Lancs, 1966.
- [12] J. Mareche, J. Rat, A. Herold, *J. Chim. Phys.* 72 (1) (1976) 1.
- [13] R.J. Walter, J.A. Ytterhus, R.D. Lloyd, W.T. Chandler, Effect of water vapor/hydrogen environments in columbium alloys, Technical Report AFML-TR-66-322, 1966.