

Journal of Nuclear Materials 258-263 (1998) 1386-1391



Effect of thickness and loading mode on the fracture properties of V-4Cr-4Ti at room temperature

H. Li, R.J. Kurtz *, R.H. Jones

Pacific Northwest National Laboratory, P.O. box 999, Richland, WA 99352, USA

Abstract

The effect of thickness on the room temperature (RT) mode I fracture behavior of V-4Cr-4Ti has been investigated. Mode I fracture properties were measured from *J*-integral tests of compact tension (CT) specimens ranging in thickness from 6.4 to 25.4 mm. All specimens were machined in the T-L orientation and vacuum annealed following final machining. Two heats of V-4Cr-4Ti were tested. Specimens 6.4 and 12.7 mm thick were taken from Wah Chang Heat No. 832665. The 25.4 mm thick specimens were obtained from Wah Chang Heat No. 832864. The effect of loading mode on fracture of V-4Cr-4Ti at RT was also studied using material from Heat No. 832665. Mode I fracture behavior was compared to mixed-mode (I/III) fracture properties obtained from modified CT specimens. Crack angles of 0° and 25° were used to vary the ratio of mode II to mode I loading. *J*-*R* curves were generated as the basis for determining the affect of loading mode. The specimen loaded in mixed-mode exhibited lower resistance to crack initiation and propagation than pure mode I specimens. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Development of vanadium alloys for the first-wall and blanket structures of fusion power systems requires detailed information on the fracture properties in both the unirradiated and irradiated conditions. Irradiation volumes for typical neutron sources limit the in-plane dimensions of fracture toughness of specimens to a few millimeters. Small-scale disk compact tension specimens are being used [1,2] to determine the fracture toughness of irradiated V-4Cr-4Ti. A recent study [3] of the fracture toughness behavior of V-5Cr-5Ti (Heat No. 832394) annealed at 1125°C for 1 h and tested at RT and 100°C indicated that the mode I toughness was about 60 and 470 kJ/m², respectively. ASTM validity requirements were satisfied for the RT tests, but not for the 100°C tests. Recent developments in vanadium alloy processing indicate the unirradiated fracture toughness will be high at RT and above. Thus, a study of the effect of specimen thickness on the mode I static fracture behavior of unirradiated V-4Cr-4Ti was performed to aid

the development and interpretation of results from small-scale specimens used in irradiation experiments.

Traditionally, mode I loading has been used to characterize the linear-elastic and elastic-plastic fracture behavior of structural materials. An inherent assumption has been that mode I loading conditions produce the lowest possible fracture toughness for a given material. In recent years it has become apparent that ductile, low-strength materials exhibit lower toughness when tested under mixed-mode conditions (I/III) [4-11]. The total energy required to initiate and propagate a crack under mode I/III loading conditions passes through a minimum at a position between mode I and mode III toughness values [3]. Recent work on the mixed-mode fracture behavior of V-5Cr-5Ti (Heat No. 832394) suggests that vanadium alloys also show lower fracture toughness when tested in mixed-mode. The purpose of the present study was to test the production-scale heat of V-4Cr-4Ti (Heat No. 832665) to determine if this material follows the same trend as Heat No. 832394.

2. Experimental procedure

Two types of fracture toughness tests were performed in this investigation. Conventional compact tension

^{*}Corresponding author. Tel.: 1 509 373 7515; fax: 1 509 376 0418; e-mail: rj.kurtz@pnl.gov.

(CT) specimens were utilized to study the effect of thickness on mode I fracture behavior. Specimens were machined in the T–L orientation and J-R curves were generated by the single specimen unload-compliance test technique. Testing was conducted in accordance with ASTM E813. All tests were performed in laboratory air at 25°C. Specimens were fatigue cracked before and after *J*-testing to mark the extent of ductile crack growth.

A modified CT specimen was used to study the effect of loading mode on fracture toughness. The essential modification of the standard CT specimen is the slanted crack plane. As shown in Fig. 1 the crack plane is slanted rather than normal to the loading axis of the specimen. This geometry produces a combination opening (mode I) and out-of-plane shear (mode III) loading condition. Varying the crack angle varies the ratio of mode I to mode III loading. Details of the experimental procedure for testing mixed-mode specimens has been reported previously [12,13]. A modified CT specimen with a crack angle of 25° was used in this study. This gives a ratio of resolved mode III to mode I load components of 0.47.

The V-4Cr-4Ti used in this study was produced by Wah Chang (formerly Teledyne Wah Chang) of Albany, Oregon. Two heats of V-4Cr-4Ti were tested. Mode I specimens 6.4 and 12.7 mm thick were taken from Wah Chang Heat No. 832665. Details on the fabrication history of this particular heat have been presented elsewhere [14]. The 25.4 mm thick mode I specimens were obtained from Wah Chang Heat No. 832864. Complete details of the processing of this heat of V-4Cr-4Ti have been reported previously [15–17]. Mode I specimens were annealed at either 1050°C for 2 h in a vacuum higher than $\sim 1.3 \times 10^{-3}$ Pa by Wah Chang or at 1000°C for 1 h in a vacuum higher than $\sim 1.3 \times 10^{-5}$ Pa at PNNL to recrystallize the warm-worked microstructure. The mixed-mode specimen was vacuum annealed at 1000°C for 1 h at PNNL. Table 1 gives pertinent fabrication information for the specimens employed in this study.

3. Results

Plots of the *J*–*R* curves for the 6.4 mm thick mode I specimens are shown in Fig. 2 (A and B in Table 1). These specimens had the same thickness but different inplane dimensions. It is clear from Fig. 2 that there is little difference in the fracture behavior for the two specimens. Fig. 2 shows *J*-values approaching 1500 kJ/m² for both specimens. ASTM validity criteria for J_{max} and $\triangle a_{max}$ were violated for these tests so critical *J*-values for crack extension were not computed from the data. Similar high fracture toughness values have also been measured on samples from Heat No. 832394 annealed at 1100°C for 1 h plus an additional anneal at 890°C for 24 h [3].

Test results for the 12.7 and 25.4 mm thick specimens (C and D, Table 1) indicated that no ductile crack initiation and growth occurred. Post-test inspection of the fracture surfaces confirmed this finding. Normally



Fig. 1. Schematic of modified compact tension specimen for mixed-mode (I/III) fracture testing.



Fig. 2. J-R curves for mode I loading of 6.4 mm thick compact tension specimens of V-4Cr-4Ti (Heat 832665).

fracture toughness should decrease with increasing thickness until plane-strain conditions are reached. Under plane-strain conditions the fracture toughness will not depend on specimen thickness and therefore, constitutes a fundamental material property. Since the present tests were not conducted under plane-strain conditions the fracture toughness should decrease with increasing thickness. The lack of ductile crack extension for the thicker samples suggests the toughness of these specimens is higher than that for the thinner ones. This result is contrary to expectation, but may be due to the tendency of this material to delaminate during testing.

It was found that V–4Cr–4Ti was prone to delaminate in planes normal to the thickness direction as shown in Fig. 3. More significantly, the fracture mode inside the delaminations was predominantly cleavage. The severity of the delaminations increased as specimen thickness increased. The delaminations were caused, in part, by development of tensile stresses in the thickness direction due to the constraining effect of the material surrounding the crack tip plastic zone, which limits through thickness deformation. Brittle delaminations are significant, from an operational viewpoint, because local triaxial states of stress will likely exist in actual power system structures. The cause of the delaminations in this material is not known yet. Inclusions could act as stress concentrators to promote cleavage fracture, but examination by optical metallography did not reveal inclusions that could produce such an effect. Weakening of grain boundaries by impurity segregation might cause intergranular separations which could trigger cleavage fracture, but no evidence for this mechanism has been obtained at the present time.

J-integral values versus crack extension for both mode I and mixed-mode I/III specimens (E and F, Table 1) are plotted in Fig. 4. Note the mode I results in Fig. 4 were obtained from a specimen heat treated differently than the mode I data presented in Fig. 2 (see Table 1). From Fig. 4, it can be seen that V–4Cr-4Ti is very tough in the unirradiated condition when tested at RT. No crack initiation took place in the mode I specimen. The crack extension plotted in Fig. 4 resulted strictly from severe crack tip blunting due to plastic deformation. This result differs from the results given in Fig. 2 which involved specimens heat treated at 1050°C for 2 h in lower quality vacuum.

Table 1

Specimen details for mode I and mixed-mode (I/III) J-R curve testing

Specimen ID	Width (mm)	Thickness (mm)	Side groove depth (%)	Crack angle°	Heat no.	Annealing conditions
A	30.5	6.4	10	0	832665	1050°C/2h
В	50.8	6.4	10	0	832665	1050°C/2h
С	50.8	12.7	0	0	832665	1050°C/2h
D	50.8	25.4	10	0	832864	1000°C/1h
E	30.5	6.4	10	0	832665	1000°C/1h
F	30.5	6.4	10	25	832665	1000°C/1h



Fig. 3. SEM photograph of delaminations in V–4Cr–4Ti specimen tested at 25° C.

The mixed-mode specimen behaved differently from the mode I specimen. During fatigue precracking the crack plane angle rotated from 25° to about 23° . However, during *J*-testing, the crack plane angle increased from 23° to about 30° when the crack started to grow. After crack initiation, the crack plane angle remained at about 30° until the end of the test. Total crack growth was 5.2 mm. The mixed-mode specimen also experienced extensive plastic deformation similar to the mode I specimen. The high *J*-values reached indicated that crack initiation was difficult. The reasons for this behavior are not fully understood. Crack plane rotation prior to crack initiation was one of the factors which contributed to the high *J*-value for initiation. While the mixed-mode crack was difficult to initiate, it propagated easily. The slope of the mixed-mode I/III J-R curve beyond the exclusion line is only 140 kJ/m²/mm, or about one-third of that for the mode I specimen.

Because no crack initiation occurred in the mode I specimen, SEM examination of fracture surfaces was only done on the mixed-mode specimen. The mixed-mode specimen failed by microvoid coalescence in which the microvoids were elongated in the direction of the shear loading component.

4. Discussion

Elastic-plastic fracture mechanics using the J-integral is based on the concept of J dominance, where the stress and strain states near the crack tip are established by the J-level. The applicability of the J-integral is limited to high constraint crack geometries. For J to be the relevant fracture mechanics parameter controlling ductile crack initiation the region of intense plastic deformation near the crack tip must be small relative to certain specimen dimensions such as the thickness and uncracked ligament. ASTM test procedure E 1152-87 [18] describes a standard method for determining valid J-R curves for metallic materials. This standard puts limits on the J-level and amount of ductile crack extension permitted for a measured J-R curve to be considered valid. The maximum J-integral capacity for a particular specimen is given by the smaller value of



Fig. 4. Effect of loading mode on J-R curves of a production scale heat of V-4Cr-4Ti.

1390

$$J_{\rm max} = b\sigma_{\rm Y}/20,\tag{1}$$

or

$$J_{\rm max} = B\sigma_{\rm Y}/20,\tag{2}$$

where $\sigma_{\rm Y}$ is the material flow stress (i.e., the average of the 0.2% offset tensile yield strength and the ultimate tensile strength), *b* the uncracked ligament and *B* the specimen thickness. The maximum crack extension, $\Delta a_{\rm max}$, capacity for a specimen is given by

$$\Delta a_{\max} = 0.1b_0 \tag{3}$$

where b_0 is the size of the initial uncracked ligament. Table 2 gives the maximum *J*-integral and crack extension capacities for the specimens used in this study. The RT value of $\sigma_{\rm Y}$ used to generate Table 2 was 420 MPa for Heat No. 832665 [19] and 349 MPa for Heat No. 832864 [20]. Note the *J*-integral measurement capacity of the specimens used in this study are limited by their thickness dimensions. Clearly the results presented in Figs. 2 and 4 are not valid *J*–*R* curves per ASTM criteria.

Thus, a critical *J*-value for ductile crack initiation can not be determined from these curves. Larger test specimens or alternative analysis techniques such as those recently advanced by Edsinger et al. [21] may be needed to obtain relevant estimates of the fracture toughness of V-4Cr-4Ti over the temperature range of interest in a fusion power system.

Fracture of V-4Cr-4Ti under mixed-mode loading conditions follows the same trend observed previously for V-5Cr-5Ti and for other ductile, low-strength materials [12]. The addition of a mode III loading component causes shear localization in the trajectory of the crack [22]. For tough materials which fail by a microvoid coalescence mechanism, the development of shear localization can cause large stress concentrations at second phase particles because of strain incompatibility. Particle/matrix decohesion or particle fracture can result, which leads to void formation that limits the mode I plastic flow field. Other factors, such as the presence of hydrogen, can lower fracture toughness by enhancing void nucleation which contributes to greater shear lo-

 Table 2

 Maximum J-integral measurement capacities for various specimen geometries

$J_{max}\;(kJ/m^2)\;{}^a$	$J_{max} \ (kJ/m^2) \ ^b$	$\Delta a_{\rm max}$ (mm)
134	320	1.5
134	533	2.5
267	533	2.5
443	443	2.5
	J _{max} (kJ/m ²) ^a 134 134 267 443	J _{max} (kJ/m²) ^a J _{max} (kJ/m²) ^b 134 320 134 533 267 533 443 443

^a Based on thickness dimension.

^b Based on uncracked ligament dimension.

calization [8]. The mode I specimens tested in this study, which exhibited crack initiation and growth, failed by a microvoid coalescence mechanism. Thus, it is reasonable to expect this material to be sensitive to mixed-mode loading conditions. The present results clearly demonstrate that fracture of V-4Cr-4Ti is sensitive to the addition of shear loading components and that mode I fracture toughness tests may not give the most conservative measure of this material's resistance to ductile fracture.

5. Conclusions

(1) The RT mode I fracture toughness of V-4Cr-4Ti (Heat 832665) is very high. ASTM validity criteria for J-R curve determination were not satisfied for any of the specimens tested. Ductile crack growth was observed for 6.4 mm thick specimens heat treated at 1050°C for 2 h but no ductile crack extension was found for 12.7 mm (heat treated at 1050°C for 2 h) or 25.4 mm (heat treated at 1000°C for 1 h) thick specimens.

(2) The RT crack initiation toughness of V-4Cr-4Ti tested under mixed-mode loading conditions was very high, but lower than that for a comparably heat treated mode I specimen. The limited results of this study follow the same trend found for V-5Cr-5Ti. Once a crack was initiated under mixed-mode loading it was relatively easy for it to grow. The present results demonstrate that for tough materials, mode I loading may not be the most severe stress state for crack initiation and growth. In a complex engineering structure, it is expected that cracks will exist at a variety of angles relative to the principal stresses. Therefore, it may be necessary to determine the mixed-mode toughness in order to ensure conservative design.

Acknowledgements

Work supported by the Office of Fusion Energy Sciences, US Department of Energy under Contract DE-AC06-76RLO 1830.

References

- D.J. Alexander, in: W.R. Corwin, F.M. Hagag, W.L. Server (Eds.), Small Specimen Test Techniques Applied to Nuclear Reactor Vessel Thermal Annealing and Plant Life Extension, ASTM-STP 1204, ASTM, Philadelphia, 1993, pp. 130–142.
- [2] C.M. Elliott, M. Enmark, G.E. Lucas, G.R. Odette, A. Rowcliffe, J. Nucl. Mater. 179–181 (1991) 434.
- [3] H. Li, M.L. Hamilton, R.H. Jones, in: Fusion Materials: Semiannual Progress Report for Period Ending 30 September 1994, DOE/ER-0313/17 (1995) pp. 165–177.

- [4] M.T. Miglin, J.P. Hirth, A.R. Rosenfield, Res. Mech. 11 (1984) 85.
- [5] J.G. Schroth, J.P. Hirth, R.G. Hoagland, A.R. Rosenfield, Metall. Trans. A 18A (1987) 1061.
- [6] M. Monoharan, J.P. Hirth, A.R. Rosenfield, J. Test. Eval. 18 (2) (1990) 106.
- [7] S. Raghavachary, A.R. Rosenfield, J.P. Hirth, Metall. Trans. A 21A (1990) 2539.
- [8] J.A. Gordon, J.P. Hirth, A.M. Kumar, N.R. Moody, Jr., Metall. Trans. A 23A (1991) 1013.
- [9] M. Monoharan, S. Raghavachary, J.P. Hirth, A.R. Rosenfield, J. Eng. Mater. Technol. 111 (1989) 440.
- [10] A.M. Kumar, J.P. Hirth, Scripta Metall. Mater. 25 (1991) 985.
- [11] M. Manoharan, Scripta Metall. Mater. 26 (1992) 1187.
- [12] H. Li, R.H. Jones, J.P. Hirth, D.S. Gelles, J. Nucl. Mater. 212–215 (1994) 741.
- [13] A. Kumar, J.P. Hirth, R.G. Hoagland, X. Feng, J. Test. Eval. 22 (4) (1994) 327.
- [14] H.M. Chung, H.-C. Tsai, D.L. Smith, R. Peterson, C. Curtis, C. Wojcik, R. Kinney, in: Fusion Materials: Semiannual Progress Report for Period Ending 30 September 1994, DOE/ER-0313/17 (1995) pp. 178–182.

- [15] W.R. Johnson, J.P. Smith, R.D. Stambaugh, in: Fusion Materials: Semiannual Progress Report for Period Ending 31 December 1995, DOE/ER-0313/19 (1995) pp. 5–11.
- [16] W.R. Johnson, J.P. Smith, R.D. Stambaugh, in: Fusion Materials: Semiannual Progress Report for Period Ending 30 June 1996, DOE/ER-0313/20 (1996) pp. 3–10.
- [17] W.R. Johnson, J.P. Smith, R.D. Stambaugh, in: Fusion Materials: Semiannual Progress Report for Period Ending 31 December 1996, DOE/ER-0313/21 (1997) pp. 3–9.
- [18] Annual Book of ASTM Standards, vol. 03.01, 1997, pp. 763–773.
- [19] B.L. Loomis, L.J. Nowicki, D.L. Smith, in: Fusion Materials: Semiannual Progress Report for the Period Ending 31 March 1991, DOE/ER-0313/10 (1991) pp. 145– 155.
- [20] W.R. Johnson, P. Trester, private communication.
- [21] K. Edsinger, G.R. Odette, G.E. Lucas, in: Proceedings of the IEA International Symposium on Miniaturized Specimens for Testing of Irradiated Materials, Jülich, Germany, 1994, p. 150.
- [22] S.V. Kamat, J.P. Hirth, J. Eng. Mater. Technol. 117 (1995) 391.