FRACTURE TOUGHNESS TESTING OF V-4Cr-4Ti AT 25 °C AND -196 °C - H-X (Huaxin) Li, and R. J. Kurtz (Pacific Northwest National Laboratory)[1]

OBJECTIVE

To determine the effect of temperature on the fracture toughness of the production-scale heat (#832665) of V-4Cr-4Ti using compact tension specimens and to compare the results with data obtained from one-third scale Charpy impact specimens.

SUMMARY

Measurements of the fracture toughness of the production-scale heat (#832665) of V-4Cr-4Ti have been performed at 25°C and -196°C using compact tension (CT) specimens. Test specimens were vacuum annealed at either 1000°C for 1 hour (HT1) or 1050°C for two hours (HT2). Specimens given the HT1 treatment were annealed after final machining, whereas the HT2 specimens received the 1050°C anneal at Teledyne Wah Chang prior to final machining. Following machining HT2 specimens were then vacuum annealed at 180°C for two hours to remove hydrogen. Specimens treated using HT1 had a partially recrystallized microstructure and those treated using HT2 had a fully recrystallized microstructure. The fracture toughness at 25°C was determined by J-integral tests and at -196°C by ASTM E 399 type tests. Toughness values obtained at -196°C were converted to J-integral values for comparison to the 25°C data. The 25°C fracture toughness was very high with none of the specimens giving valid results per ASTM criteria. Specimens fractured by microvoid coalescence. The fracture toughness at -196°C was much lower than that at 25°C and the fracture surface showed predominantly cleavage features. The present results show a transition from ductile to brittle behavior with decreasing test temperature which is not observed from one-third scale Charpy impact tests. The fracture toughness at -196°C was still quite high, however, at about 75 kJ/m^2.

Delaminations in planes normal to the thickness direction were seen at both test temperatures. Fracture surfaces inside the delaminations exhibited nearly 100% cleavage facets. The cause of the brittle delaminations was not determined, but will be a subject for further investigation.

PROGRESS AND STATUS

1. Material and Experimental Method

The production-scale heat (#832665) of V-4Cr-4Ti was used for this study. CT specimens were machined from 6.35 mm (0.25 in.) thick plates. One plate was received in the warm-rolled condition and was heat treated at 1000°C for 1 hour in a vacuum of 10^{-7} torr (HT1) at PNNL following machining of specimens. The other plate had been warm-rolled and heat treated at 1050°C for two hours (HT2) by Teledyne Wah Chang, Albany, OR. After machining HT2 specimens were vacuum annealed at 180°C for two hours to remove hydrogen.

CT specimens were used for all tests and all specimens had the T-L orientation. Two specimen

[1] Operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

widths, 30.5 mm (1.2 in.) and 50.8 mm (2 in.) were used for the HT2 material. HT2 specimens were tested only at 25°C. All HT1 specimens were 30.5 mm (1.2 in.) wide. Fracture toughness values at 25°C were determined using the J-integral test procedure given in ASTM E 813 and those at -196°C were determined using the ASTM E 399 procedure. For the purpose of comparison, K-values determined from the -196°C tests were converted to J-values using Equation 1:

$$J = \frac{K^2}{E}(1-\nu^2) \qquad (1)$$

where E is Young's modulus and n is Poison's ratio. Values for E and n at -196°C for V-4Cr-4Ti were not available so estimates were obtained from data on pure vanadium. E and G (shear modulus) for pure vanadium at -196 °C can be determined from Equations 2 and 3 [1]:

$$E\,(GPa) = 0.1*(1.28 - 9.61x10^{-5}*T) \qquad (2)$$

$$G\,(GPa) = 0.1*(0.488 - 8.43x10^{-5}*T) \qquad (3)$$

where T is temperature in Kelvin. At -196 °C the estimated value of E is 127.3 GPa and G is 48.2 GPa. Assuming isotropic behavior, n value can be determined from Equation 4.

$$G = \frac{E}{2(1+\upsilon)} \qquad (4)$$

Substituting the E and G values into Equation 4 gives a value of 0.32 for n at -196 °C.

Fracture surfaces were examined in a scanning electron microscope (SEM) to determine the effect of temperature on the failure mode and to determine the failure mechanism inside the delaminations.

2. Results And Discussion

The fracture toughness test results for both temperatures are plotted in Figure 1. The toughness of specimens tested at 25°C was very high such that ASTM E 813 validity criteria were not satisfied for the specimen dimensions utilized. The 6.35 mm specimen thickness gives a valid toughness up to about 250 kJ/m^2 which suggests a minimum value for the toughness of V-4Cr-4Ti at 25°C. Toughness values determined from 30.5 mm and 50.8 mm wide specimens were nearly same, indicating little effect of specimen width within this range. Similar high fracture toughness values have also been measured on samples prepared from heat BL-63 heat treated at 1100°C for one hour plus an additional anneal at 890°C for 24 hours. J-integral tests for specimens given the HT1 anneal are in progress. It is anticipated that the 25°C fracture toughness behavior of HT1 specimens will be similar to HT2 specimens since Charpy impact data shows HT1 material exhibits upper shelf properties at 25°C similar to HT2 material. Thus, 25°C fracture toughness values from HT2 specimens are compared to -196°C fracture toughness values from HT1 specimens.

The results plotted in Figure 1 show that temperature has a significant effect on the fracture toughness of V-4Cr-4Ti. Specimens tested at -196°C yielded fracture toughness values of about 103 MPa$\sqrt{m}$. This is equal to about 75 kJ/m^2, which is significantly lower than the 25°C fracture toughness. The present results are in contrast to recent data obtained from one-third scale pre-cracked Charpy tests [3]. The Charpy data does not display a transition in absorbed energy with decreasing temperature. The absorbed energy was found to increase gradually with decreasing test temperature, reaching a peak at around -150°C. Below -150°C the absorbed energy decreased with temperature, but even at -196°C it was still larger than that at room temperature [3]. In addition, Charpy specimens failed by microvoid coalescence at all temperatures.

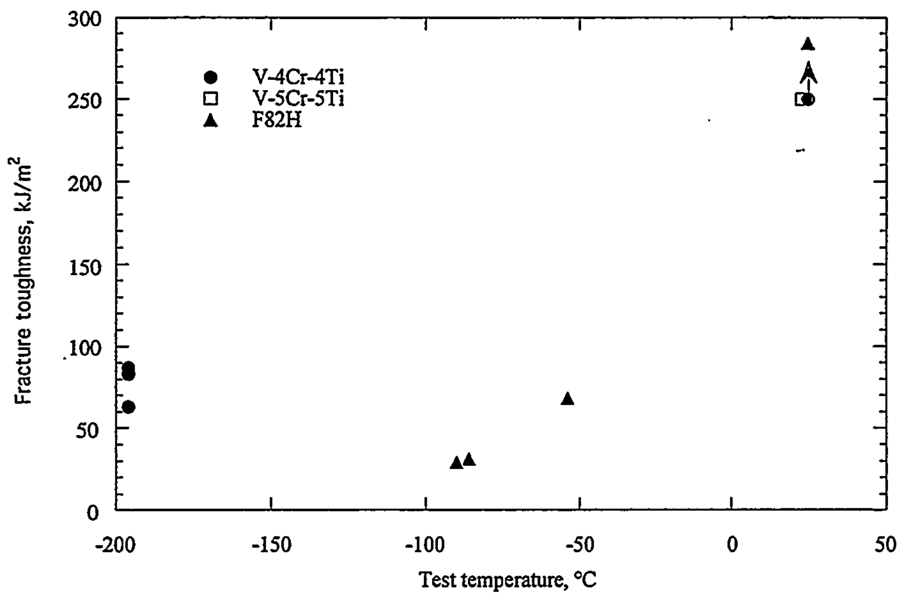

Figure 1. Effect of temperature on the fracture toughness of a production scale heat of V-4Cr-4Ti. Data from a V-5Cr-5Ti alloy and F-82H steel are included for comparison.

SEM examination of fracture surfaces showed that the 25°C specimens failed by microvoid coalescence, as depicted in Figure 2. At -196°C the fracture surface was predominantly cleavage with some microvoid coalescence, as displayed in Figure 3. Taken together, the fractographic results and fracture toughness data clearly shows that V-4Cr-4Ti undergoes a ductile-to-brittle transition at a temperature higher than -196°C. This result differs from Charpy impact data, where no ductile-to-brittle transition was found at temperatures above -196°C [3]. The reason for this difference may be due to differences in constraint between the two types of specimens. The state of stress at the crack tip for the larger CT specimen will be more triaxial than for the small Charpy specimen. This favors crack extension in the CT specimen more than for the Charpy specimen.

It was also found that V-4Cr-4Ti was prone to delaminate in planes normal to the thickness direction, regardless of test temperature, as shown in Figure 4. More significantly, the fracture surfaces inside the delaminations were largely cleavage (see Figure 5). 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 systems. 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.

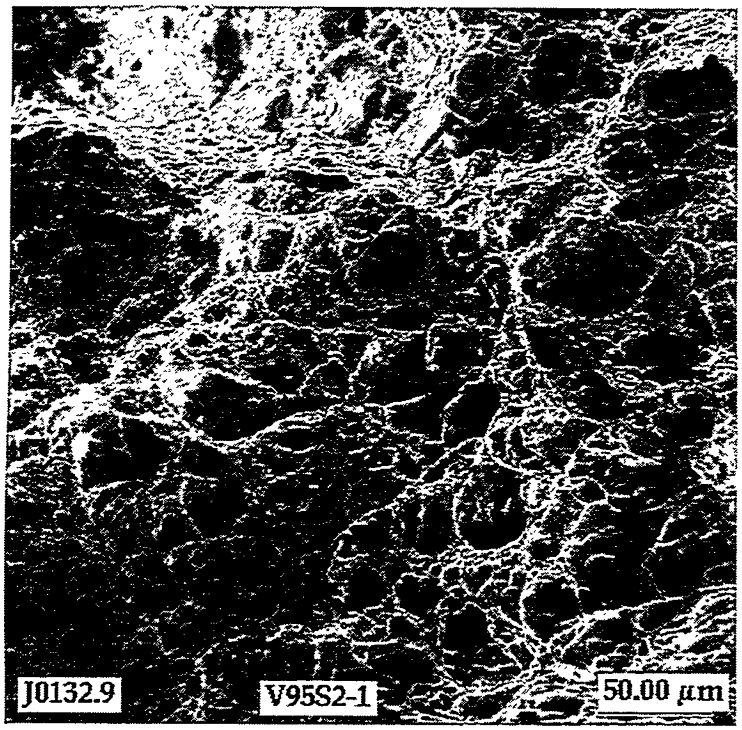

Figure 2. SEM photograph showing microvoid coalescence fracture of V-4Cr-4Ti alloy at 25°C.

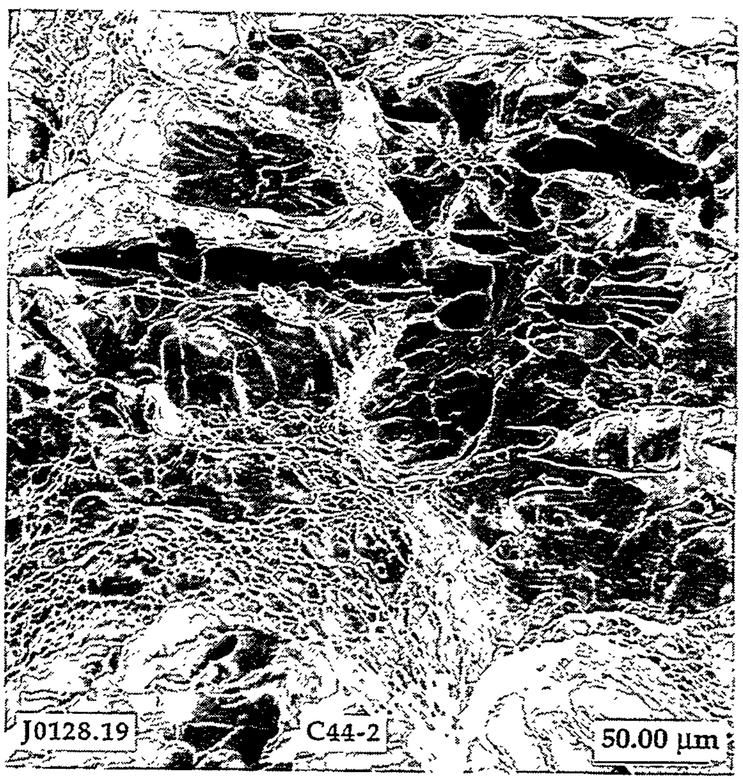

Figure 3. SEM photograph shows predominantly cleavage fracture of V-4Cr-4Ti alloy at -196°C.

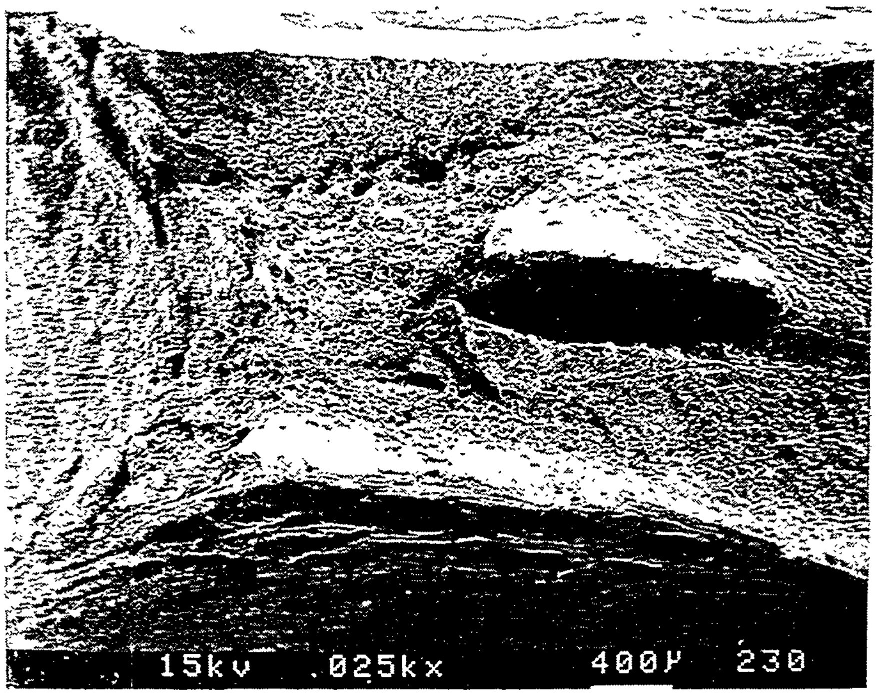

Figure 4. SEM photograph of delaminations in V-4Cr-4Ti specimen tested at 25°C.

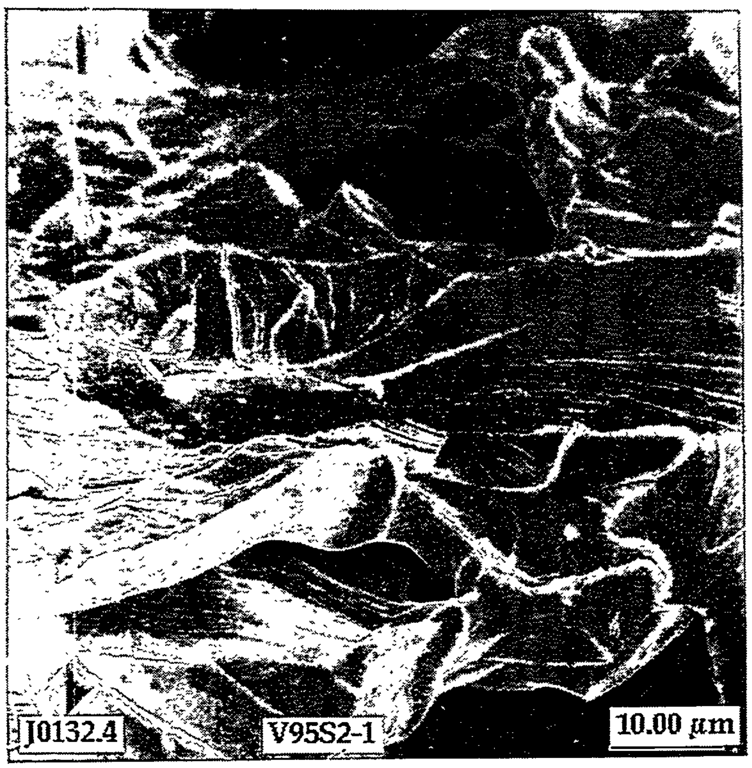

Figure 5. SEM photograph of cleavage facets inside delamination in V-4Cr-4Ti tested at 25°C.

FUTURE WORK

Detailed examination by electron microprobe will be performed to search for micro-chemical segregation effects which may be responsible for the delaminations. The effect of mixed-mode I/III loading on the fracture toughness of V-4Cr-4Ti alloy heat treated at 1000°C for 1 hour is being studied. Preliminary results indicate that mixed-mode loading enhances crack initiation and propagation.

REFERENCES

1. R. J. Farraro and R. B. McLellan, "High Temperature Elastic Properties of Polycrystalline Niobium, Tantalum, and Vanadium", Met. Trans. A, 10A (1979), p 1699.

2. Huaxin Li, M. L. Hamilton and R. H. Jones, "Effect of Heat Treatment and Test Method on DBTT of a V-5Cr-5Ti Alloy", Fusion Reactor Materials Semiannual Progress Report. DOE/ER-0313/18, Oak Ridge National Lab., Oak Ridge, TN (March 1995), p. 215.

3. H. M. Chung, L. Nowicki and D. L. Smith, "Impact Properties of Precracked V-4Cr-4Ti Charpy Specimens", Fusion Reactor Materials Semiannual Progress Report. DOE/ER-0313/18, Oak Ridge National Lab., Oak Ridge, TN (March 1995), p. 253.