DUCTILE–BRITTLE TRANSITION BEHAVIOR OF V–4Cr–4Ti IRRADIATED IN THE DYNAMIC HELIUM CHARGING EXPERIMENT*

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OBJECTIVE

The objective of this work is to determine the effect of simultaneous displacement damage and dynamically charged helium on the ductile–brittle transition behavior of V–4Cr–4Ti specimens irradiated to 18–31 dpa at 425–600°C in the Dynamic Helium Charging Experiment (DHCE).

SUMMARY

One property of vanadium-base alloys that is not well understood in terms of their potential use as fusion reactor structural materials is the effect of simultaneous generation of helium and neutron damage under conditions relevant to fusion reactor operation. In the present DHCE, helium was produced uniformly in the specimen at linear rates ranging from ≈0.4 to 4.2 appm helium/dpa by the decay of tritium during irradiation to 18–31 dpa at 425–600°C in Li-filled DHCE capsules in the Fast Flux Test Facility. Ductile–brittle transition behavior of V–4Cr–4Ti, recently identified as the most promising vanadium–base alloy for fusion reactor use, was determined from multiple–bending tests (at –196°C to 50°C) and quantitative SEM fractography on TEM disks (0.3–mm thick) and broken tensile specimens (1.0–mm thick). No brittle behavior was observed at temperatures >–150°C, and predominantly brittle–cleavage fracture morphologies were observed only at –196°C in some specimens irradiated to 31 dpa at 425°C during DHCE. Ductile–brittle transition temperatures (DBTTs) were –200°C to –175°C for both types of specimens. In strong contrast to tritium–trick experiments in which dense coalescence of helium bubbles is produced on grain boundaries in the absence of displacement damage, no intergranular fracture was observed in the bend–tested specimens irradiated in the DHCE.

INTRODUCTION

Vanadium-base alloys have significant advantages over other candidate alloys (such as austenitic and ferritic steels) for use as structural materials in fusion devices, e.g., the International Thermonuclear Experimental Reactor (ITER) and DEMO reactor. These advantages include intrinsically low long-term activation, low decay heat, low neutron-induced helium- and hydrogen-transmutation rates, low biological hazard, and high thermal stress factor. Recently, V-4Cr-4Ti has been identified as the most promising vanadium–base alloy for fusion reactor structural components because of its excellent combination of mechanical and physical properties before and after irradiation.[1–6] One property of the alloy that is not well understood is the effect of helium on ductile–brittle transition behavior; no data on impact properties or ductile–brittle transition behavior have been reported on effects of simultaneous generation of helium and neutron displacement damage under fusion–relevant conditions (i.e., ≈5 appm He/dpa ratio), although helium effects on other vanadium alloys have been investigated by less–than–prototypical simulation techniques such as tritium-trick,[7–11] cyclotron-injection,[12–16] and boron-doping.[16–19] In the DHCE,[20–22] the fusion–relevant helium–to–dpa damage ratio is closely simulated by utilizing slow transmutation of controlled amounts of ^{6}Li and a tritium–doped mother alloy immersed in ^{6}Li + ^{7}Li. Effects of the fusion–relevant generation of helium on tensile properties have been investigated on specimens irradiated in the DHCE to 18–31 dpa at 425°C–600°C and are reported elsewhere.[23] However, no Charpy–impact specimens were included in the DHCE capsules because of severe limitations on irradiation space. Therefore, the important information on ductile–brittle transition behavior of the alloy

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could be extracted only from miniature specimens such as TEM disks and broken tensile specimens. This paper presents results of multiple–bending tests of V-4Cr-4Ti alloy and quantitative SEM fractography of the fracture surface morphologies conducted subsequently to determine ductile–brittle transition behavior.

MATERIALS AND PROCEDURES

The elemental composition of the V–4Cr–4Ti alloy, determined prior to irradiation, is given in Table 1. Information on fabrication, phase structure, grain size, and other microstructural characteristics of the alloy has been given elsewhere.[4,5,23] The tensile and TEM disk specimens were irradiated in the Fast Flux Test Facility (FFTF) at 420, 520, and 600°C to neutron fluences (E > 0.1 MeV) ranging from 3.7 x 10^{22} n/cm^2 (≈18 displacements per atom, or dpa) to 6.4 x 10^{23} n/cm^2 (≈31 dpa). Helium in the specimens was produced by utilizing transmutation of controlled amounts of ^{6}Li and predetermined amounts of tritium–doped vanadium mother alloy immersed in ^{6}Li + ^{7}Li.[20–22]

Table 1. Chemical composition of V–4Cr–4Ti (ANL ID BL–47)

ANL ID	Nominal Composition (wt.%)	Impurity Composition (wppm)							
		O	N	C	Si	S	P	Nb	Mo
BL-47	V–4.1Cr–4.3Ti	350	220	200	870	20	<40	<100	<100

Table 2 summarizes actual postirradiation parameters determined from tensile and TEM disk specimens of the V–4Cr–4Ti alloy, i.e., fast neutron fluence, dose, and helium and tritium contents measured shortly (≈20–25 days) after the post–irradiation tests. Helium and tritium contents were determined by mass spectrometry at Rockwell International Inc., Canoga Park, California. Two TEM disk or broken tensile specimens were selected from each capsule after multiple–bending (at –196°C to 50°C) or tensile tests (at room–temperature) and analyzed to determine helium and tritium contents. For each disk or broken tensile specimen, four separate analyses of ^{3}He and ^{4}He were conducted. The tritium contents were determined on the basis of analysis of ^{3}He decay measured on the same specimens ≈50 days apart.

Table 2. Summary of irradiation parameters of DHCE and helium and tritium contents measured in V–4Cr–4Ti specimens

Capsule ID No.	Irradiation Temp. (°C)	Total Damage (dpa)	Calculated Helium (appm) to dpa Ratio[a] at EOI[b] (Assumed k_a or k_w)[c] k_a=0.073 (k_w=0.01)	Measured Helium Content[d] (appm)	Actual Helium to dpa Ratio (appm/dpa)	Measured Tritium Content[e] (appm)
4D1	425	31	3.8	11.2–13.3	0.39	27
4D2	425	31	2.8	22.4–22.7	0.73	39
5E2	425	18	2.1	3.3–3.7	0.11	2
5D1	500	18	4.4	14.8–15.0	0.83	4.5
5E1	500	18	3.1	6.4–6.5	0.36	1.7
5C1	600	18	1.1	8.4–11.0	0.54	20
5C2	600	18	1.1	74.9–75.3	4.17	63

[a] L. R. Greenwood "Revised Calculations for the DHCE," April 30, 1993.

[b] Beginning of irradiation (BOI) May 27, 1991; end of irradiation (EOI) March 19, 1992; 203.3 effective full power days (EFPD), hot standby at ≈220°C until November 1992.

[c] Equilibrium ratio (k_a by atom, k_w by weight) of tritium in V alloy to that in the surrounding liquid lithium.

[d] Measured June 1994.

[e] Measured August 1994.

Fracture behavior was determined by repeatedly bending a TEM disk (thickness 0.25–0.3 mm) or a piece of the shoulder region of a fractured tensile (thickness 1.0 mm) specimen[23] in low–temperature baths of liquid nitrogen or mixtures of dry ice and acetone. Tensile specimens fractured in gauge section only at room temperature[23] were selected for this bending tests. The tensile specimens, irradiated at 425°C, 500°C, and 600°C and recovered from four capsules, were not heat treated (at 400°C for 1 h), which has been a customary procedure to expel hydrogen or tritium. Temperature of the surrounding liquid was measured with a calibrated thermocouple. Approximately one–third of each side of a bend specimen was firmly held by two grips in the test bath and the middle portion of the disk was bent repeatedly until fracture. Thus, specimen constraint was similar to that in a four–point bend test. Depending on irradiation conditions and test temperature, 3 to 62 bends were required to produce fracture in the specimens at –196 to 50°C. At a given test temperature, cold–work accumulated in the specimen as the number of bends increased, thereby hardening the material near the bend progressively. After fracture, morphology of the fracture surface was examined quantitatively by SEM. A few dozen SEM fractographs were taken at ≈300 X, and a composite of the whole fracture surface was made. Four types of fracture morphology were observed: cleavage, quasicleavage, ductile-dimple, and fibrous ductile fracture. From the fracture surface composite, the percentage of ductile-fracture morphology was measured for each specimen with a planimeter.

RESULTS AND DISCUSSION

As reported previously,[2,5] brittle fracture of V–4Cr–4Ti (ANL ID BL–47) did not occur at temperatures >–196°C in impact tests on one–third–size Charpy specimens either in the nonirradiated condition or after irradiation to 24–34 dpa at 425–600°C in non–DHCE (i.e., negligible helium/dpa ratio). Consistent with this behavior, only ductile fracture was observed from the present multiple–bend tests in a similar temperature range on disk and broken tensile specimens of the alloy either unirradiated or irradiated in non–DHCE. This is shown in Figs. 1 and 2, respectively.

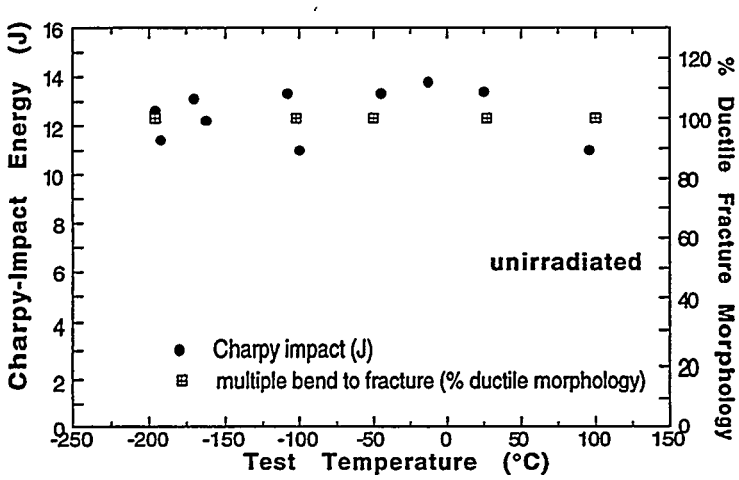

Figure 1. Comparison of Charpy–impact energy and percentage of ductile fracture surface morphology of TEM disks (determined after multiple bend until fracture) of unirradiated V–4Cr–4Ti, plotted as a function of test temperature.

The percentage of fracture surface of disk and broken tensile specimens (irradiated at 425–600°C to 18–31 dpa in the DHCE) with ductile–fracture morphology is plotted as a function of test temperature in Fig. 3. The figure shows combined effects of simultaneous displacement damage (18–31 dpa, Table 2), helium generation (at rates of 0.4–4.2 appm helium/dpa), and tritium (2–63 appm, Table 2) uptake on ductile–brittle transition behavior. For comparison, similar results obtained for nonirradiated and irradiated non–DHCE specimens (containing negligible amounts of helium, tritium, and hydrogen) are also shown in Fig. 3. Despite the thickness difference, ductile–brittle transition behaviors of the disk and broken tensile specimens were similar.

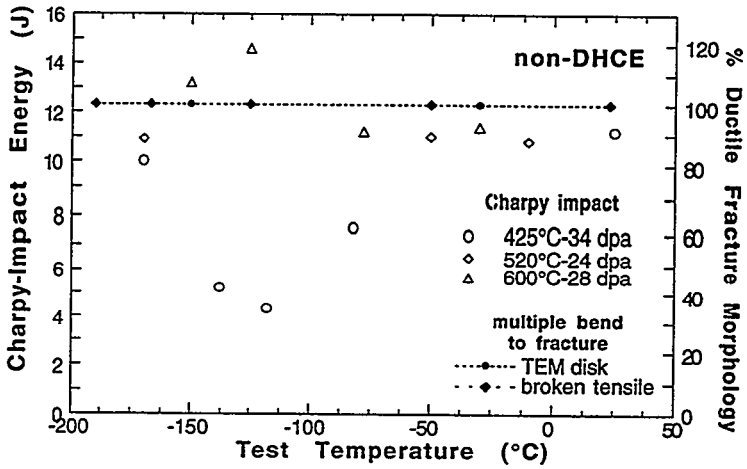

Figure 2. Comparison of Charpy–impact energy and percentage of ductile fracture surface morphology of TEM disks and broken pieces of tensile specimens (determined after multiple bend until fracture) of V–4Cr–4Ti irradiated in non–DHCE, plotted as a function of test temperature.

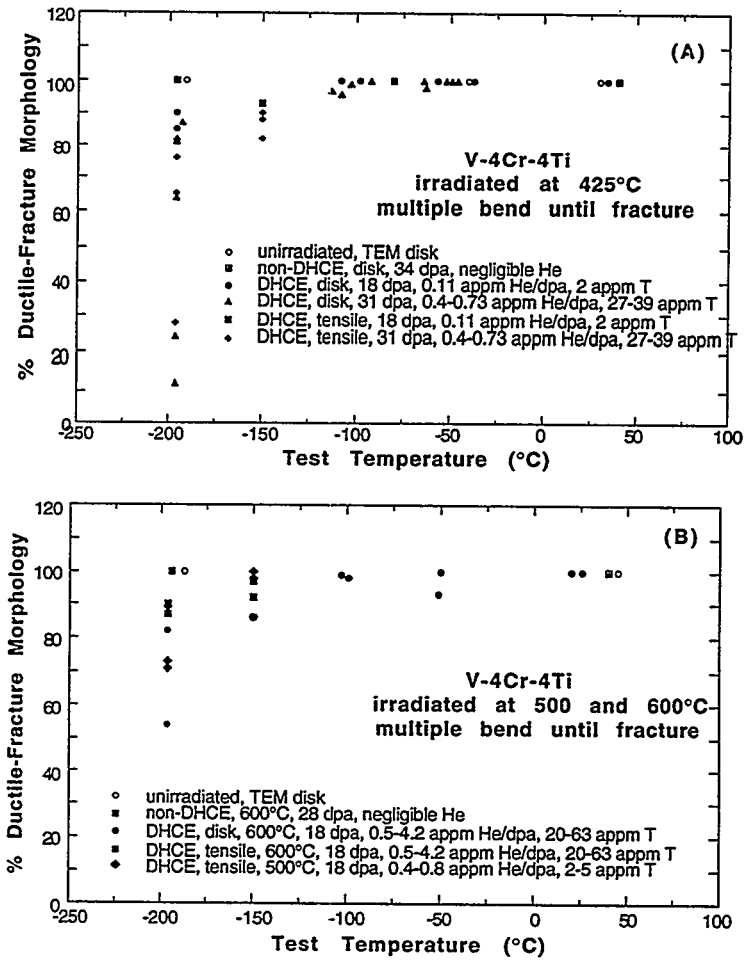

Figure 3. Percent ductile–fracture morphology vs. test temperature of V–4Cr–4Ti disks and tensile specimens irradiated at (A) 425°C and (B) 500 and 600°C and fractured by multiple bending.

The results in Fig. 3 show that the percentage of brittle cleavage measured from fracture surfaces produced near –190°C was more pronounced in specimens irradiated at 425°C and retrieved from high–tritium capsules (e.g., 4D1 and 4D2) than in specimens irradiated at 500–600°C and retrieved from low–tritium capsules (e.g., 5E1 and 5C2). As in non–DHCE irradiation, no brittle behavior was observed at temperatures >–150°C in DHCE specimens in which the helium generation rate was ≈0.4–4.2 appm helium/dpa (Table 2). Predominantly brittle–cleavage fracture morphologies were observed only at –196°C in some specimens irradiated to 31 dpa at 425°C during DHCE. Figure 3 indicates that DBTTs of specimens irradiated at 425°C and 500–600°C are –175°C and –200°C, respectively. As in the tensile tests at 25–600°C,[23] no intergranular fracture was observed in any specimens fractured at –196 to 50°C by multiple bending.

As described in a separate article,[24] helium microvoids were negligible in all specimens irradiated in DHCE except those irradiated at 425°C and retrieved from Capsules 4D1 and 4D2; only a few helium bubbles were observed at the interface between the grain matrix and some Ti(O,N,C) precipitates that are normally present in V–Ti and V–Cr–Ti alloys. Even in specimens irradiated at 600°C at the highest helium generation rate of ≈4.2 appm helium/dpa (Capsule 5C2), no microvoids could be detected in either grain matrix or grain boundaries. In specimens irradiated to 31 dpa at 425°C (retrieved from Capsules 4D1, ≈0.4 appm helium/dpa and 4D2, ≈0.73 appm helium/dpa), moderate number densities of diffuse helium bubbles were observed in localized grain matrix and near a limited fraction (≈15%) of grain boundaries.[24] The number density of helium bubbles, observed near the limited region of grain boundaries, was significantly lower than those in other alloys tested in the tritium–trick experiments, where extensive coalescence of helium bubbles occurred on all grain boundaries.[7–11] The absence of intergranular fracture morphology in any of the present specimens seems consistent with the negligible or insignificant coalescence of helium bubbles on grain boundaries.

CONCLUSIONS

1. Effects of dynamically charged helium, neutron damage, and retained tritium on DBTT of V–4Cr–4Ti alloy were determined after irradiation in the Dynamic Helium Charging Experiment (DHCE). TEM disks and broken pieces of tensile specimens, irradiated at 425–600°C to 18–31 dpa and at helium generation rates of 0.4–4.2 appm helium/dpa, were fractured by repeated bending while submerged in low–temperature baths. Ductile–brittle transition behaviors of both types of specimens were similar. Ductile–brittle transition temperatures, based on quantitative SEM fractography of ductile and brittle fracture surface morphologies, were –175°C and –200°C, respectively, in specimens irradiated at 425°C and 500°C–600°C.

2. Neither partial nor predominantly intergranular fracture was observed in multiple–bending tests at –196 to 50°C. Predominantly brittle–cleavage fracture morphologies were observed only at –196°C in some specimens irradiated to 31 dpa at 425°C in the DHCE. Cleavage fracture was negligible during tests at >–150°C regardless of irradiation temperature, dose, and helium generation rate.

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