DENSITY DECREASE IN VANADIUM-BASE ALLOYS IRRADIATED IN THE DYNAMIC HELIUM CHARGING EXPERIMENT*

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SUMMARY

Combined effects of dynamically charged helium and neutron damage on density decrease (swelling) of V–4Cr–4Ti, V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti alloys have been determined after irradiation to 18–31 dpa at 425–600°C in the Dynamic Helium Charging Experiment (DHCE). To ensure better accuracy in density measurement, broken pieces of tensile specimens ≈10 times heavier than a transmission electron microscopy (TEM) disk were used. Density decreases of the four alloys irradiated in the DHCE were <0.5%. This small change seems to be consistent with the negligible number density of microcavities characterized by TEM. Most of the dynamically produced helium atoms seem to have been trapped in the grain matrix without significant cavity nucleation or growth.

INTRODUCTION

Recent attention in the development of vanadium–base alloys for application in fusion reactor first–wall and blanket structure has focused on the findings of excellent impact toughness of V–4Cr–4Ti[1,2] and V–5Cr–5Ti[3] and virtual immunity of V-4Cr-4Ti, V–5Ti, and V–3Ti–1Si to embrittlement by fast neutron displacement damage.[4] Excellent resistance of these alloys to neutron–irradiation–induced (negligible helium generation) swelling has also been reported.[5] One unresolved issue in the performance of these alloys, however, has been the effect of fusion–relevant simultaneous generation of helium and neutron damage (at a ratio of 4–5 appm helium/displacement per atom [dpa]) on swelling (i.e., density decrease). In the unique DHCE,[6] the fusion–relevant helium–to–dpa ratio is simulated realistically by utilizing transmutation of controlled amounts of ^{6}Li and a predetermined amount of tritium–doped mother alloy immersed in $^{6}Li + ^{7}Li$. This report describes results of density measurements on V–4Cr–4Ti, V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti alloys irradiated to 18–31 dpa at 425–600°C in the DHCE. The results were compared to similar data obtained from non–DHCE specimens (negligible helium generation) irradiated to 18–34 dpa at 420–600°C. Earlier measurements of density change that utilized small (≈10 mg) TEM disks indicated significant data scattering.[5,7] Therefore, heavier (70–110 mg) broken pieces of tensile specimens were used in this study to determine density decrease more accurately.

MATERIALS AND PROCEDURES

The elemental compositions of the V–4Cr–4Ti, V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti are given Table 1. Tensile specimens, 0.7–1.0 mm thick, were polished and annealed at 1050–1125°C in high vacuum. Microstructures of V–4Cr–4Ti, characterized before and after irradiation in a non–DHCE (negligible helium generation) and a DHCE (0.4–4.2 appm helium/dpa), have been described elsewhere.[7] The only secondary phase in the as–annealed specimens was Ti(O,N,C), 200–500 nm in size, which is normally observed in titanium–containing vanadium alloys with O+N+C > 400 wppm. After irradiation in a non–DHCE or a DHCE, Ti_5Si_3 precipitates <20 nm in size were observed in high density.

Table 1. Composition of vanadium–base alloys

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
BL-46	V–4.6Ti	305	53	85	160	10	<100	<100	–
BL-45	V–2.5Ti–1.0Si	345	125	90	9900	30	–	200	140
BL-49	V–7.9Cr–5.7Ti	400	150	127	360	20	–	<100	170

Sheet tensile specimens were irradiated in the Fast Flux Test Facility (FFTF) Cycle 12 Materials Open Test Assembly (MOTA) 2B at 420, 500, and 600°C to neutron fluences (E > 0.1 MeV) ranging from 3.7 x 10^{22}

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n/cm^2 (≈18 dpa) to 6.4 x 10^{23} n/cm^2 (≈31 dpa). Counterpart tensile specimens were also irradiated to similar fluence at similar temperatures in non–DHCE (negligible helium generation) capsules in the MOTA–2B. Helium in the DHCE specimens was produced by utilizing transmutation of controlled amounts of ^{6}Li and a predetermined amount of tritium–doped vanadium mother alloy immersed in ^{6}Li + ^{7}Li. Table 2 summarizes the actual postirradiation parameters determined from tensile and disk specimens of the V–4Cr–4Ti alloy, i.e., fast neutron fluence, dose, and helium and tritium content measured shortly after the postirradiation tests. Helium and tritium were determined by mass spectrometry at Rockwell International Inc., Canoga Park, CA.

Table 2. Summary of irradiation parameters of Dynamic Helium Charging Experiment and helium and tritium content of V–4Cr–4Ti specimens

Capsule ID No.	Irradiation Temp. (°C)	Fluence (E >0.1 MeV) (10^{22} n cm^{-2})	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	6.4	31	3.8	11.2–13.3	0.39	27
4D2	425	6.4	31	2.8	22.4–22.7	0.73	39
5E2	425	3.7	18	2.1	3.3–3.7	0.11	2
5D1	500	3.7	18	4.4	14.8–15.0	0.83	4.5
5E1	500	3.7	18	3.1	6.4–6.5	0.36	1.7
5C1	600	3.7	18	1.1	8.4–11.0	0.54	20
5C2	600	3.7	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 Li.

[d] Measured June 1994.

[e] Measured August 1994.

Density decrease (swelling) was determined from weight of nonirradiated and irradiated (DHCE and non–DHCE) specimens, measured in air and in research–grade CCl_4. A typical specimen (70–110 mg in air) was a piece of the shoulder region of a 1.0–mm–thick tensile specimen, cut after a tensile test in an inert atmosphere at the same temperature as the irradiation temperature or at 20–200°C.[8]

RESULTS AND DISCUSSION

Density could be determined more accurately by measurements on broken tensile specimens than on 0.3–mm thick, ≈10–mg TEM disks. This is shown in Fig. 1, in which data scattering of baseline density of nonirradiated tensile and disk specimens of V–4Cr–4Ti is presented. The uncertainty limit of the density measured on tensile specimens is significantly smaller than that measured on small TEM disks.

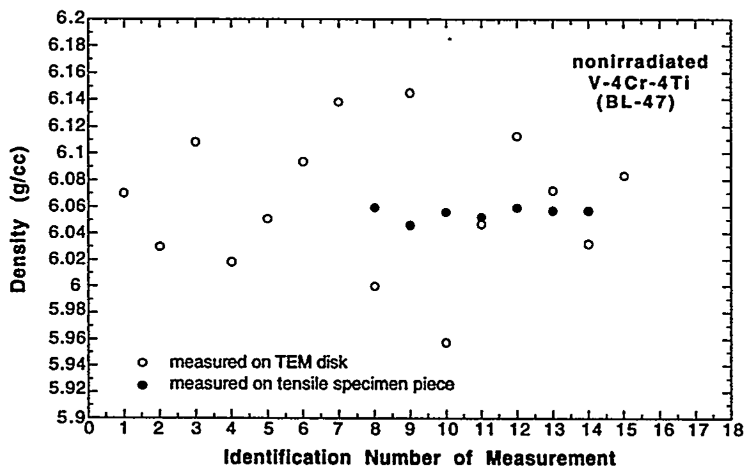

Figure 1.
Densities obtained from ≈10–mg TEM disks in weight) and ≈70–110–mg broken tensile specimens of nonirradiated V–4Cr–4Ti, showing significantly more accurate data from tensile specimens.

The decrease in density for V–4Cr–4Ti, V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti is given in Figs. 2–5, respectively. For comparison, density changes determined under similar irradiation conditions in a non–DHCE are shown in Figs. 2–4.

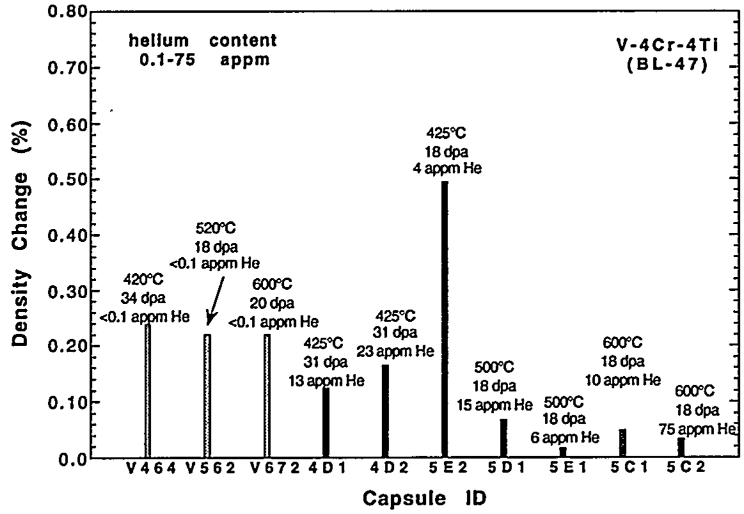

Figure 2.
Density changes of V–4Cr–4Ti (BL–47) after irradiation to 18–34 dpa at 420–600°C in the DHCE (0.4–4.2 appm He/dpa) (dark bars) and in a non–DHCE (negligible helium) (light bars). Each bar represents the average of densities measured on three separate pieces of broken tensile specimens.

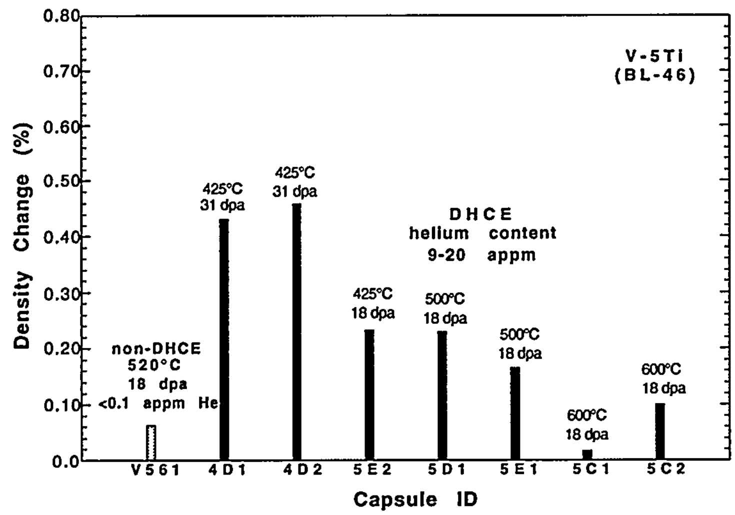

Figure 3.
Density changes of V–5Ti (BL–46) after irradiation to 18–31 dpa at 425–600°C in the DHCE (9–20 appm He) (dark bars) and in a non–DHCE (negligible helium) (light bars). Each bar represents the average of densities measured on three separate pieces of broken tensile specimens.

Density changes measured for the non–DHCE and DHCE specimens of the reference alloy V–4Cr–4Ti were low (<0.5 %). This small density change seems to be consistent with the negligible density of voids or helium bubbles in the DHCE specimens of the alloy. At least for helium generation rates in the range of 0.4–4.2 appm He/dpa, there was no evidence of a significant effect of dynamically charged helium on density change (Fig. 2); the reference alloy V–4Cr–4Ti seems to be inherently resistant to swelling under the present conditions of the DHCE and non–DHCE, indicating that swelling of the alloy under fusion reactor conditions is also low.

Although helium generation was rather low (<1.1 appm He/dpa), density changes measured for the DHCE specimens of V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti were also low (<0.5%), regardless of irradiation temperature and dose. There are indications that density changes measured for V–5Ti and V–3Ti–1Si irradiated at 425°C (to ≈31 dpa) are somewhat higher than those measured for specimens irradiated at 500–600°C (to ≈18 dpa).

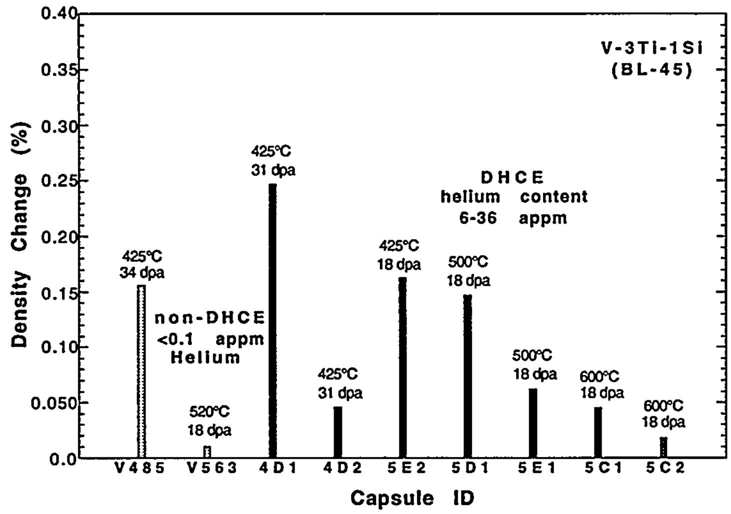

Figure 4.
Density changes of V–3Ti–1Si (BL–45) after irradiation to 18–34 dpa at 425–600°C in the DHCE (6–36 appm helium) (dark bars) and in a non–DHCE (negligible helium) (light bars). Each bar represents the average of densities measured on three separate pieces of broken tensile specimens.

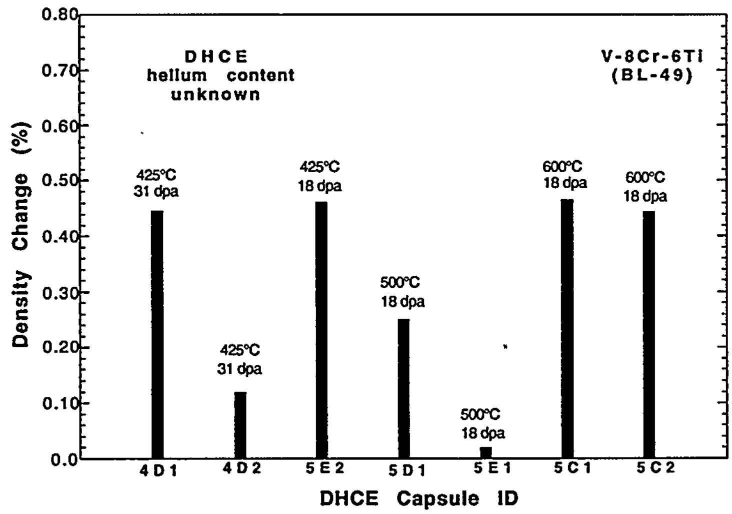

Figure 5.
Density changes of V–8Cr–6Ti (BL–49) after irradiation to 18–34 dpa at 420–600°C in the DHCE (helium content unknown). Each bar represents the average of densities measured on three separate pieces of broken tensile specimens.

CONCLUSIONS

1. For the reference alloy V–4Cr–4Ti irradiated to ≈18–31 dpa at 425–600°C in the Dynamic Helium Charging Experiment (DHCE) with helium generation rates of ≈0.4–4.2 appm He/dpa, density decrease (swelling) was low (<0.5%). Density changes measured for the DHCE and non–DHCE (negligible helium generation) specimens were similar, showing insignificant effect of dynamically charged helium. The reference alloy seems to be inherently resistant to swelling under the present conditions of the DHCE (helium and dpa damage) and non–DHCE (dpa damage only), indicating that swelling of the alloy under fusion reactor conditions is also low.

2. Although the dynamic helium generation rate was low (<1.1 appm He/dpa), density changes measured for the DHCE specimens of V–5Ti, V–3Ti–1Si, and V–8Cr–6Ti were also low (<0.5%), regardless of the irradiation temperature (420–600°C) and dose (18–31 dpa).

3. Density change can be determined significantly more accurately with heavier tensile specimens than with small TEM disks.

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