EFFECT OF HELIUM ON TENSILE PROPERTIES OF VANADIUM ALLOYS*

H. M. Chung, M. C. Billone, and D. L. Smith (Argonne National Laboratory)

SUMMARY

Tensile properties of V-4Cr-4Ti (Heat BL-47), 3Ti-1Si (BL-45), and V-5Ti (BL-46) alloys after irradiation in a conventional irradiation experiment and in the Dynamic Helium Charging Experiment (DHCE) were reported previously.[1] This paper presents revised tensile properties of these alloys, with a focus on the effects of dynamically generated helium on ductility and work-hardening capability at <500°C. After conventional irradiation (negligible helium generation) at ≈427°C, a 30-kg heat of V-4Cr-4Ti (BL-47) exhibited very low uniform elongation, manifesting a strong susceptibility to loss of work-hardening capability. In contrast, a 15-kg heat of V-3Ti-1Si (BL-45) exhibited relatively high uniform elongation (≈4%) during conventional irradiation at ≈427°C, showing that the heat is resistant to loss of work-hardening capability.

Helium atoms produced at ≈430°C in dynamic helium charging irradiation seem to be conducive to higher ductility (compared to that under conventional irradiation) and relatively lower yield strength. This seemingly beneficial effect of helium is believed to be important in evaluating the performance of V-4Cr-4Ti and V-3Ti-1Si alloys, because susceptibility to loss of work-hardening capability at low temperatures under fusion-relevant helium-generating conditions is considered to be a major factor in governing the minimum operating temperature for fusion applications. In this respect, V-3Ti-1Si appears to be more advantageous than V-4Cr-4Ti, although other factors such as creep strength could be inferior. Tensile data from conventional irradiation experiments (i.e., negligible helium generation), especially the data for <500°C, appear to differ significantly from the results obtained with simultaneous helium generated by the DHCE. Therefore, a dynamic helium charging irradiation experiment is strongly recommended with a focus on determining tensile and fracture properties of V-4Cr-4Ti and V-3Ti-1Si alloys at 300-470°C at doses of 10-20 dpa with high helium/dpa ratios of ≈4-5 appm He/dpa.

INTRODUCTION

An investigation of the effects of conventional neutron irradiation (at 420-600°C, 30-84 dpa in fast fission spectrum) on tensile properties of V-Ti, V-Ti-Si, and V-Cr-Ti alloys was reported previously.[1] Following that investigation, effects of simultaneous neutron irradiation and helium generation in these alloys were investigated in the DHCE, and results of postirradiation examination of swelling, tensile properties, and fracture behavior were reported for damage levels of up to ≈30 dpa and helium generation rates of 0.4-4.2 appm He/dpa.[2] To determine the effects of helium, tensile properties measured and analyzed for the same heats by the same procedure were compared for the conventional irradiation (negligible helium generated, referred to as "non-DHCE") and DHCE for similar irradiation temperature and damage level.

Subsequently, it was learned that values for uniform and total strains and yield strength reported in Refs. 1 and 2 were determined incorrectly. Also, revised data on neutron fluence and displacement damage accumulated in the DHCE have been since reported in Ref. 3. This paper presents revised tensile properties and irradiation parameters, with a focus on the effects of dynamically generated helium in V-4Cr-4Ti, V-3Ti-1Si, and V-5Ti alloys.

MATERIALS AND PROCEDURES

The elemental composition of the alloys, determined prior to irradiation, is given in Table 1. Tensile specimens, machined from ≈1-mm-thick cold-worked sheets, were annealed in high vacuum for 1 h at 1125°C (V-4Cr-4Ti) or 1050°C (V-3Ti-1Si and V-5Ti).

---

* Work supported by U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

Table 1. Chemical composition of alloys investigated

Alloy ANL ID	Ingot Size (kg)	Nominal Composition (wt.%)	Impurity Composition (wppm) O	N	C	Si
BL–45	15	V–2.5Ti–1Si	345	125	90	9900
BL–46	15	V–4.6Ti	305	53	85	160
BL-47	30	V–4.1Cr–4.3Ti	350	220	200	870

The specimens were irradiated in the DHCE in Li-bonded TZM capsules in the Fast Flux Test Facility (FFTF, in MOTA-2B), a sodium–cooled fast reactor, at 430, 500, and 600°C to neutron fluences (E > 0.1 MeV) ranging from 2.5 x 10^{22} to 4.8 x 10^{23} n/cm^2 which correspond to 14-27 dpa in vanadium. Helium in the alloy specimens was produced by utilizing transmutation of controlled amounts of ^{6}Li and predetermined amounts of tritium–doped vanadium mother vanadium immersed in ^{6}Li + ^{7}Li. Table 2 summarizes actual postirradiation parameters determined from tensile and disk specimens of the V–4Cr–4Ti alloy (Heat ID BL-47), i.e., fast neutron fluence, dose, and helium and tritium contents measured shortly after the postirradiation tests. Helium and tritium contents were determined by mass spectrometry at Rockwell International Inc., Canoga Park, CA.

Actual irradiation conditions of the seven DHCE subcapsules are described in Ref. 3, in which detailed information on original loading plan, history of canister exchange, tritium charge, and actual loading of subcapsules has been documented. Note that the actual irradiation conditions of Subcapsules 5E1 and 5E2 are different from those given in Ref. 4.

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

Capsule ID No.	Irradiation Temp. (°C)	Total Damage (dpa)	Measured Helium Content (appm)	Actual Helium to dpa Ratio (appm/dpa)	Measured Tritium Content (appm)
4D1	430	25	11.2–13.3	0.48	27
4D2	430	27	22.4–22.7	0.84	39
5E2	430	14	3.3–3.7	0.25	2
5D1	500	14	14.8–15.0	1.07	4.5
5E1	500	16	6.4–6.5	0.40	1.7
5C1	600	14	8.4–11.0	0.69	20
5C2	600	18	74.9–75.3	4.17	63

Tensile properties were measured at 23, 100, 200°C, and at the irradiation temperatures in flowing argon at a strain rate of 0.0011 s^{-1}. The same facility and the same procedures were used in testing the tensile specimens irradiated in the DHCE and non-DHCE.

RESULTS AND DISCUSSION

The 0.2%-offset yield strength, ultimate tensile strength, uniform plastic strain, and total plastic strain measured on tensile specimens of V–4Cr–4Ti irradiated in the DHCE at 430–600°C (14–27 dpa, 4–75 appm He) are summarized in Figs. 1A–1D, respectively. Similar properties measured on specimens irradiated at ≈427°C to 14-33 dpa in non–DHCE (revised from Ref. 1) are also plotted in the figure as function of tensile test temperature. Results measured on specimens of V–3Ti–1Si (14-27 dpa, 6–36 appm He) and V–5Ti (13-27 dpa, 9–20 appm He) are shown in Figs. 2 and 3, respectively.

Helium Effect in DHCE at 500-600°C

After irradiation to ≈13-27 dpa at 500-600°C in either a DHCE or a non–DHCE, the three alloys retained significantly high ductilities, i.e., 3.5-8% uniform elongation and 9-20% total elongation. For this range of irradiation temperature, effects of helium on tensile properties do not seem to be significant.

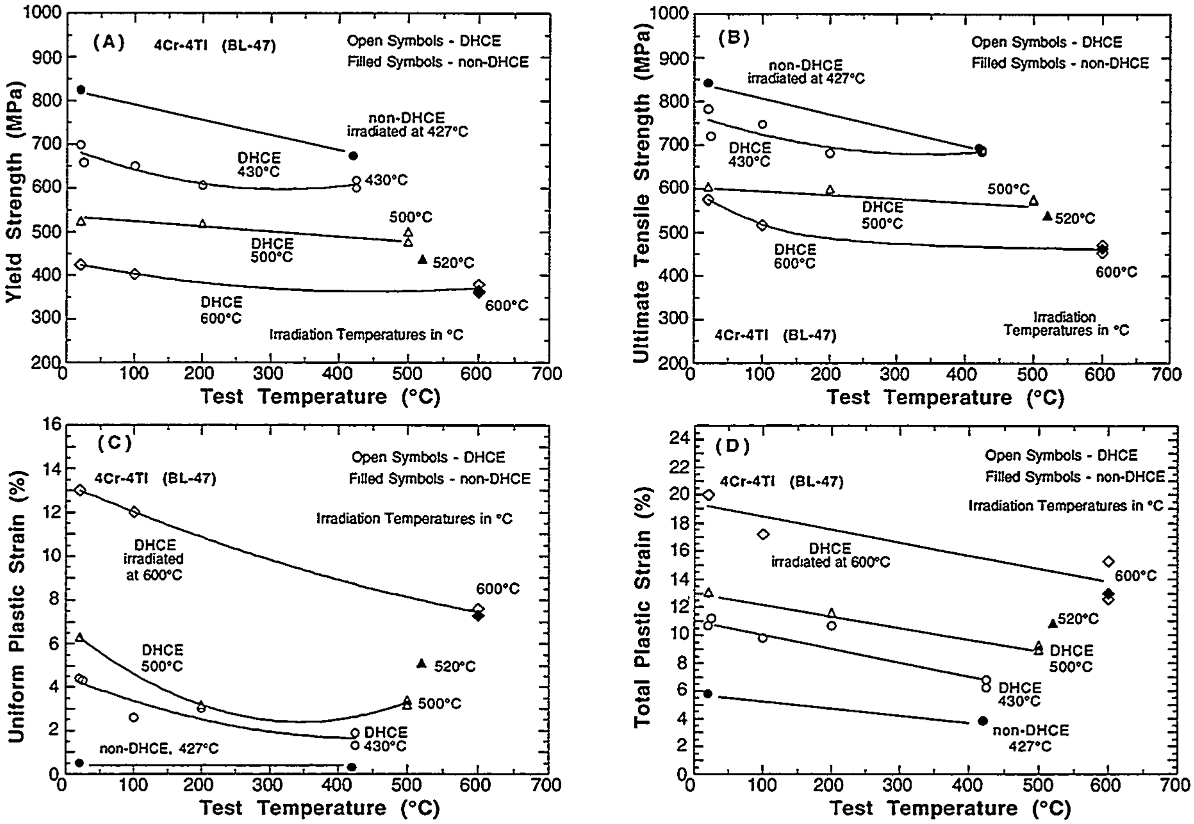

Fig. 1. Yield strength (A), ultimate tensile strength (B), uniform plastic strain (C), and total plastic strain (D) of V–4Cr–4Ti (BL-47) after irradiation at 427–600°C in the DHCE (14–27 dpa, 4–75 appm He) and in non–DHCE (14-33 dpa).

Effects of Test Temperature

Yield strength, ultimate tensile strength, uniform plastic strain, and total plastic strain of the specimens of the three alloys irradiated at 500-600°C appear to increase monotonically for decreasing test temperature (from 500-600°C to 23°C). However, for V-4Cr-4Ti and V-3Ti-1Si specimens irradiated at ≈427°C in a non–DHCE, uniform and total plastic strains appear to be more or less independent of test temperature at 23-420°C. In contrast, similar specimens of V-4Cr-4Ti and V-3Ti-1Si irradiated at ≈430°C in a DHCE exhibit a temperature dependency similar to that of the specimens irradiated at 500-600°C. That is, ductility increases significantly with decreasing test temperature.

Loss of Work-Hardening Capability in V-4Cr-4Ti in non-DHCE at ≈420°C

After irradiation in a non-DHCE at ≈420°C, the 30-kg heat of V-4Cr-4Ti (Heat ID BL-47) exhibited minimal uniform elongation (0.3-0.5%), which is a manifestation of a strong susceptibility to loss of work-hardening capability. The same heat did retain, however, good work-hardening capability with a uniform elongation of ≈3% in a non-DHCE in helium environment in a conventional irradiation experiment in the HFIR to ≈10 dpa at ≈400°C.[3]

A virtual loss of work-hardening capability was observed in a 500-kg heat of V-4Cr-4Ti (Heat ID #832665) in a conventional irradiation experiment (X-530 experiment) in Li-bonded stainless steel capsules in EBR-II to ≈4 dpa at ≈390°C.[4] However, in the same experiment, a 100-kg heat of V-4Cr-4Ti (Heat ID VX-8) exhibited a good work-hardening capability. These results indicate that

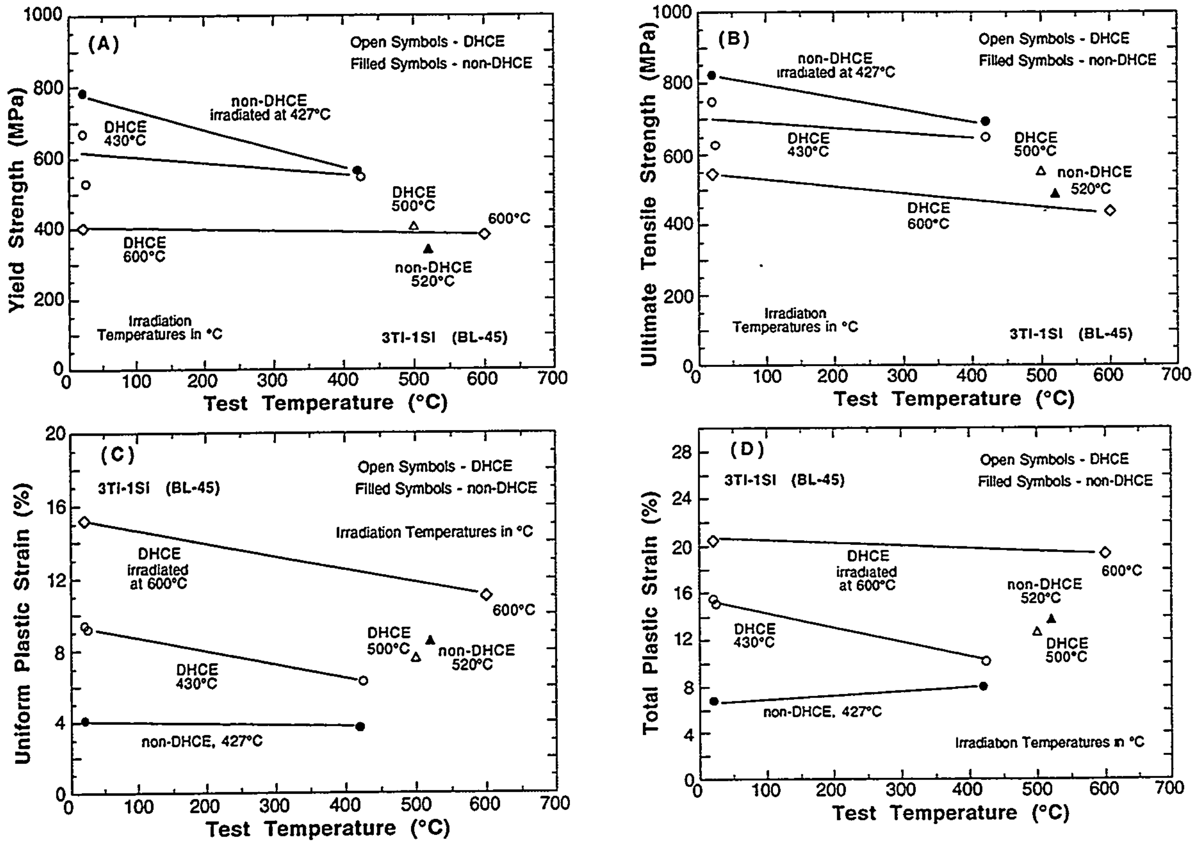

Fig. 2. Yield strength (A), ultimate tensile strength (B), uniform plastic strain (C), and total plastic strain (D) of V–3Ti-1Si (BL-45) after irradiation at 427–600°C in the DHCE (14–27 dpa, 6-36 appm He) and in non–DHCE (14-33 dpa).

loss of work-hardening capability, and hence uniform elongation, in V-4Cr-4Ti class during conventional irradiation experiments at <430°C varies strongly from heat to heat and is influenced by irradiation conditions.

Work-Hardening Capability in V-3Ti-1Si in non-DHCE at ≈430°C

In contrast to V-4Cr-4Ti, the 15-kg V-3Ti-1Si (Heat ID BL-45) exhibited relatively high uniform elongation (≈4 %) under a similar non-DHCE condition, showing that this heat retains good work-hardening capability under neutron irradiation at ≈430°C. Total elongation of the alloy was >7 % for under conditions tested in this study.

Helium Effect at ≈430°C

Interestingly, ductilities of DHCE specimens of V-4Cr-4Ti and V-3Ti-1Si alloys, irradiated at ≈430°C and measured at <430°C, were always higher than those of the similar non–DHCE specimens, whereas strengths were lower. Helium generated during irradiation at ≈430°C appears to promote work-hardening capability in the two alloys. As a result, helium generation seems to be conducive to higher uniform elongation (compared to that in a non-DHCE). This is summarized in Fig. 4. The trend is consistent for all capsules and specimens that were irradiated at ≈430°C, i.e., a total of 13 V-4Cr-4Ti and V-3Ti-1Si specimens irradiated in 3 DHCE and 2 non-DHCE capsules. The trend cannot be explained on the basis of the slightly higher dose level in non-DHCE than in DHCE, i.e., 14-33 versus 14-27 dpa.

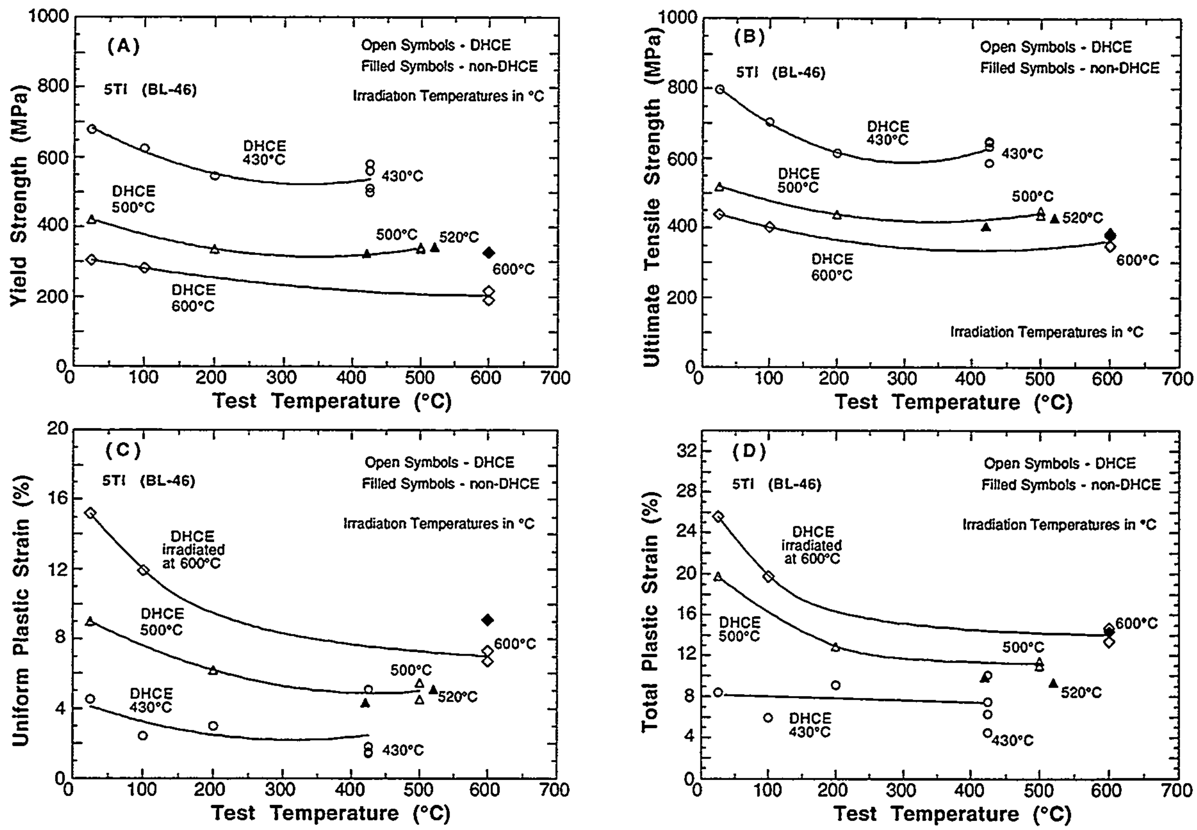

Fig. 3. Yield strength (A), ultimate tensile strength (B), uniform plastic strain (C), and total plastic strain (D) of V–5Ti (BL-46) after irradiation at 427–600°C n the DHCE (14-27 dpa, 9-20 appm He) and non–DHCE.

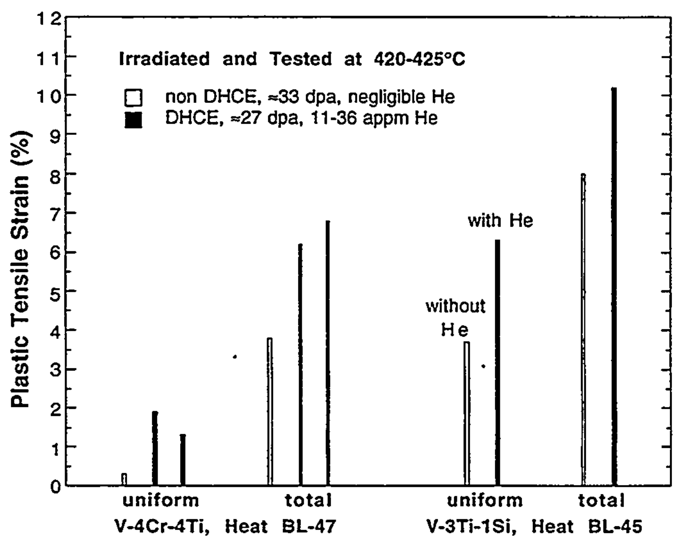

Fig. 4. Effect of helium on uniform and total plastic strains of V-4Cr-4Ti (BL-47) and V-3Ti-1Si irradiated at ≈430°C.

The seemingly beneficial effect of helium indicates that different type of hardening centers are produced during DHCE. This effect, also reported by Satou et al. for V-5Ti-5Cr-1Si-Y,Al alloy (irradiated at ≈430°C in DHCE to ≈24 dpa, 177 appm He),[5] is believed to be important in evaluating the performance of V-4Cr-4Ti and V-3Ti-1Si alloys, because susceptibility to loss of work-hardening capability at low temperatures under fusion-relevant helium-generating conditions is considered to be a major factor in governing the minimum operating temperature of a fusion reactor. In this respect, V-3Ti-1Si appears to be more advantageous than V-4Cr-4Ti, although other factors such as creep strength could be disadvantageous.

This finding indicates also that tensile data obtained with negligible helium produced in conventional irradiation experiments are not likely to be applicable, or may even be misleading, for designing and evaluating the performance of first wall structure. Therefore, a dynamic helium charging irradiation experiment is recommended which will focus on determining tensile and fracture properties of V-4Cr-4Ti and V-3Ti-1Si alloys at 300-470°C at higher dose (>30 dpa) and higher helium/dpa ratio (≈4-5 appm He/dpa).

Absence of Intergranular Fracture

No intergranular fracture surface morphology was observed in the present tensile specimens irradiated in the DHCE and non-DHCE and tested at 23–600°C. Even the fracture surface morphology of the specimens of V-4Cr-4Ti irradiated at ≈420°C in non-DHCE and exhibiting uniform elongation of only 0.3-0.5 % was ductile dimple fracture. That is, the low uniform elongation was a result of strong localization of plastic deformation rather than a brittleness. This is in sharp contrast to results reported from tritium-trick experiments, in which dense helium bubbles aggregated on grain boundaries produced intergranular fracture, leading to low uniform and total elongation in the specimen.

CONCLUSIONS

1. After irradiation to ≈14-30 dpa at 500-600°C with (helium generation rate ≈1 appm He/dpa) or without helium generation, V-4Cr-4Ti (Heat BL-47), 3Ti-1Si (BL-45), and V-5Ti (BL-46) retained significantly high ductilities, i.e., 3.5-8% uniform elongation and 9-20% total elongation. Results to date for this range of irradiation temperatures indicate that the effects of helium on tensile properties are insignificant in these alloys. Yield strength, ultimate tensile strength, uniform and total plastic strains of the alloys irradiated at 500-600°C in the DHCE increased monotonically for decreasing test temperature.

2. Uniform and total plastic strains of V-4Cr-4Ti and V-3Ti-1Si irradiated at ≈427°C in a non–DHCE were not influenced significantly by test temperatures at or below the irradiation temperature. In contrast, ductility increased significantly with decreasing test temperature when the specimens are irradiated at similar temperature in a DHCE. This indicates that different types of defect microstructure are produced at ≈430°C during irradiation with and without helium generation, most likely through interaction of impurities and helium atoms with defects or defect clusters.

3. After conventional irradiation in FFTF (negligible helium generation) at ≈427°C, a 30-kg heat of V-4Cr-4Ti (BL-47) exhibited minimal uniform elongation (0.3-0.5%), manifesting a strong susceptibility to loss of work-hardening capability. The same heat, however, did retain relatively good work-hardening capability with a uniform elongation of 1.3-1.9% after irradiation in the DHCE at ≈430°C at helium generation rates of 0.48-0.84 appm He/dpa.

4. No intergranular fracture surface morphology was observed in the tensile specimens after irradiation at 427-600°C either with or without helium generation. The fracture surface morphology of the specimens of V-4Cr-4Ti that exhibited low uniform elongation (0.3-0.5 %) after irradiation at ≈427°C, was ductile dimple fracture, indicating that the low uniform

elongation was a result of strong localization of plastic deformation rather than material brittleness. This is in sharp contrast to results reported from tritium-trick experiments, in which dense helium bubbles aggregated on grain boundaries produced intergranular fracture, leading to low uniform and total elongation in the specimen.

5. In contrast to V-4Cr-4Ti (BL-47), a 15-kg heat of V-3Ti-1Si (BL-45) exhibited relatively higher uniform elongations, i.e., ≈3.7% after conventional irradiation in FFTF at ≈427°C and ≈6.3 % after the dynamic helium charging irradiation in FFTF at ≈430°C. This indicates that this heat is resistant to loss of work-hardening capability.

6. Helium atoms produced at ≈430°C in the dynamic helium charging irradiation seem to be conducive to higher ductility (compared to that under conventional irradiation) and relatively lower tensile strength. This seemingly beneficial effect of helium is believed to be important in evaluating the performance of V-4Cr-4Ti and V-3Ti-1Si alloys, because susceptibility to loss of work-hardening capability at low irradiation temperatures under fusion-relevant helium-generating conditions is considered a major factor in governing the minimum operating temperature of a fusion reactor. In this respect, V-3Ti-1Si appears to be more advantageous than V-4Cr-4Ti, although other factors such as creep strength of the alloy could be inferior.

7. Tensile data obtained from conventional irradiation experiments (i.e., negligible helium generation), especially data for <500°C, appear to be not applicable, or may even be misleading, for designing and evaluating the performance of first wall structure. Therefore, a dynamic helium charging irradiation experiment is strongly recommended with a focus on determining tensile and fracture properties of V-4Cr-4Ti and V-3Ti-1Si alloys at 300-470°C to doses of 10-20 dpa with helium/dpa ratios of ≈4-5 appm He/dpa.

REFERENCES

[1] B. A. Loomis, L. Nowicki, and D. L. Smith, J. Nucl. Mater. 212–215 (1994), 790.

[2] H. M. Chung, B. A. Loomis, and D. L. Smith, J. Nucl. Mater. 233-237 (1996), 466-475.

[3] Memo, H. Matsui to D. L. Smith, "On Actual Loading of Tritium to DHCE Subcapsules," Nov. 22, 1993.

[4] A. M. Ermi, L. R. Greenwood, and H. L. Heinisch, "Irradiation Parameters for the FFTF Materials Open Test Assemblies from 1983 to 1992," WHC-SD-FF-TD-010, Westinghouse Hanford Company, Aug. 1994.

[5] H. M. Chung, L. Nowicki, and D. L. Smith, "Tensile Properties of Vanadium Alloys Irradiated at 200°C in the HFIR," in this report.

[6] H. M. Chung, H.-C. Tsai, L. Nowicki, and D. L. Smith, "Tensile Properties of Vanadium Alloys Irradiated at ≈390°C in EBR-II," in this report.

[7] M. Satou, H. Koide, A. Hasegawa, K. Abe, H. Kayano, H. Matsui, J. Nucl. Mater. 233-237 (1996), 447-451.