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by

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Abstract

Vanadium-base alloys are promising candidate materials for application in fusion reactor first-wall and blanket structures because they offer several important advantages, i.e., inherently low irradiation-induced activity, good mechanical properties, good compatibility with lithium, high thermal conductivity, and good resistance to irradiation-induced swelling and damage. As part of a program to screen candidate alloys and develop an optimized vanadium-base alloy, extensive investigations of various V-Ti, V-Cr-Ti, and V-Ti-Si alloys have been conducted after irradiation in lithium in fission reactors. From these investigations, V-4 wt.%Cr-4 wt.%Ti was identified as the most promising alloy. The alloy exhibited attractive mechanical and physical properties that are prerequisites for first-wall and blanket structures, i.e., high tensile strength, high ductility, good creep properties, high impact energy, low ductile-brittle transition temperature before and after irradiation, excellent resistance to irradiation-induced swelling and microstructural instability, and good resistance to corrosion in lithium. In particular, the alloy is virtually immune to irradiation-induced embrittlement, a remarkable property compared to other candidate materials being investigated in the fusion-reactor-materials community. Effects of helium, charged dynamically in simulation of realistic fusion reactor conditions, on tensile, ductile-brittle transition, and swelling properties were insignificant. Thermal creep behavior of the alloy was significantly superior to that of austenitic and ferritic/martensitic steels.

1. 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 magnetic fusion reactors. These advantages include intrinsically lower levels of long-term activation, irradiation afterheat, neutron-induced helium- and hydrogen-transmutation rates, biological hazard potential, and thermal stress factor.[1–5] However, to make use of these favorable neutronic and physical properties of structural materials in fusion systems, the alloys must be resistant to neutron-induced swelling, creep, and embrittlement, and they must also be compatible with the reactor coolant and environment.

As part of a program to screen candidate alloys and develop an optimal alloy, extensive investigations have been conducted on the swelling behavior, tensile properties, impact toughness, and microstructural evolution of V, V-Ti, V-Cr, V-Cr-Ti, and V-Ti-Si alloys after

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irradiation by fast neutrons at 420, 520, and 600°C. These investigations revealed that V-Cr-Ti alloys containing 4–5 wt.% Cr, 3-5 at.% Ti, 400-1000 wt. ppm Si, and <1000 wt. ppm O+N+C were most desirable because they exhibit superior resistance to swelling, embrittlement, and hydrogen-induced effects during fast-neutron irradiation in lithium.[6-10] As a result, recent attention has focused primarily on the ternary alloys V-(4–5)Cr-(3–5)Ti, and on the binary alloy V-5Ti.

Subsequently, a heat of V-4Cr-4Ti alloy containing ≈800 wppm silicone and ≈800 wppm O+N+C has been fabricated and was selected for comprehensive testing and examination, particularly for the effects of fusion-relevant high-dose irradiation and dynamically charged helium on the tensile, ductile-brittle-transition, irradiation-induced density change and microstructural evolution, and thermal creep behavior. Results of this comprehensive investigation, as well as baseline properties of the alloy, are presented in this paper to provide insights and a database that are necessary to evaluate the alloy for application to fusion reactor first-wall and blanket structures.

2. Materials and Irradiation

The elemental composition of the V–4Cr–4Ti alloy, determined prior to irradiation, is given in Table 1. The 30-kg alloy ingot, melted from low-chlorine titanium and low-impurity vanadium, was extruded at 1150°C and annealed at 1050°C several times after 8–10 passes of warm rolling between the anneal. Final forms of the product were annealed plates and sheets 3.8-, 1.0-, and 0.5-mm in thickness. Test specimens, machined from the annealed plates and sheets to investigate impact, tensile, and swelling behaviors and microstructural characteristics, were ≈95% recrystallized and exhibited an average grain size of ≈14 μm. The only secondary phase present in the as-annealed specimen was Ti(O,N,C), which is normally observed in titanium-containing vanadium alloys with O+N+C < 400 wppm.[11] Grain structure and precipitate distribution of the unirradiated material have been reported elsewhere.[12]

Recently, a production-scale ingot of V–4Cr–4Ti weighing ≈500 kg has been melted and extruded successfully using essentially the same fabrication procedures. Initial tests on specimens machined from plates and sheets of this production heat indicated that Charpy-impact and tensile properties are as good as those of the 30-kg heat.

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

The alloy specimens were irradiated in the Fast Flux Test Facility (FFTF), a fission test reactor located in Richland, Washington, at 420, 520, and 600°C to neutron fluences (E > 0.1 MeV) ranging from 3 x 10^{22} n/cm^2 (17 displacements per atom, or dpa) to 1.9 x 10^{23} n/cm^2 (114 dpa). The specimens were sealed in TZM/Mo capsules filled with 99.99%-enriched ^7Li during irradiation to prevent contamination by oxygen, nitrogen, and carbon impurities dissolved in the sodium coolant of the FFTF. In this type of irradiation test, designed to simulate and investigate the effects of displacement damage on the physical and mechanical properties of the alloy, formation of any appreciable levels of helium and tritium from ^6Li was avoided.

In contrast to the above type of irradiation test, the dynamic helium charging experiment (DHCE) was designed to simulate and investigate the effects of simultaneous displacement damage and fusion-relevant helium generation (at a ratio of ≈5 appm helium/dpa) on the properties of the alloy. During irradiation in the DHCE, the fusion-relevant helium-to-dpa ratio is simulated realistically by utilizing transmutation of controlled amounts of ^6Li and predetermined amount of tritium-doped vanadium mother alloy immersed in ^6Li + ^7Li.[13,14] Table 2 summarizes the irradiation temperature, dose, tritium inventory charged at the beginning of irradiation, and measured helium and tritium contents at the time of post-irradiation tests for V–4Cr–4Ti specimens irradiated in seven DHCE capsules.

The retrieved specimens were cleaned ultrasonically in alcohol prior to density measurement, microstructural analysis, Charpy-impact, tensile, and bending tests. Bending tests were conducted on transmission electron microscopy (TEM) disks and pieces of fractured tensile specimens, irradiated in the DHCE, to determine ductile-brittle transition behavior. Density change was determined from specimen weights measured in air and in research grade CCl_4. Disk specimens were jet-thinned for analysis by TEM in a solution of 15% sulfuric acid-72% methanol-13% butyl cellosolve maintained at -5°C.

Table 2.. Irradiation Parameters of Dynamic Helium Charging Experiment and Helium and Tritium Contents Measured in V-4Cr-4Ti Specimens

Capsule ID No.	Irradiation Temp.	Initial Tritium Charge	Total Damage	Calculated Helium (appm) to dpa Ratio[a] at EOI[b] (Assumed k_a or k_w)[c]	Measured Helium Content[d]	Actual Helium to dpa Ratio	Measured Tritium Content[e]
	(°C)	(Ci)	(dpa)	k_a=0.073 (k_w=0.01)	(appm)	(appm/dpa)	(appm)
4D1	425	99	31	3.8	11.2-13.3	0.39	27
4D2	425	70	31	2.8	22.4-22.7	0.73	39
5E2	425	26	18	2.1	3.3-3.7	0.11	2
5D1	500	73.5	18	4.4	14.8-15.0	0.83	4.5
5E1	500	57	18	3.1	6.4-6.5	0.36	1.7
5C1	600	16.4	18	1.1	8.4-11.0	0.54	20
5C2	600	18	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.

3. Tensile Properties

Tensile properties of the unirradiated material, measured at 25°C in flowing argon at a strain rate of 0.0011 s^{-1}, are summarized in Fig. 1. Yield and ultimate tensile strengths (Fig. 1A) were ≈402 and ≈462 MPa, respectively, whereas uniform and total elongations (Fig. 1B) were ≈24 and ≈33%, respectively. Yield strength of the unirradiated material measured at 420-600°C was ≈260 MPa. In Fig. 2, the yield strengths of the alloy are presented with room- and high-temperature yield strengths of other V-Ti binary and V-Cr-Ti ternary alloys.[15] The hydrogen content of the alloys shown in Fig. 2 was <30 appm.

Effects of displacement damage (measured on specimens irradiated in the non-DHCE) and combined effects of simultaneous displacement damage and helium generation (measured on specimens irradiated in the DHCE) are summarized in Figs. 3A-3D. In the figures, yield and ultimate tensile strengths and uniform and total elongations, measured on specimens irradiated at 425°C-600°C to 18-34 dpa in the two types of experiments, have been plotted as function of irradiation temperature.

After irradiation to ≈30 dpa in either a DHCE or a non-DHCE, ductility of the alloy remained significantly high, i.e., >8% uniform elongation and >10% total elongation. Low-temperature (≤400°C) ductilities of the DHCE specimens were higher than those of the

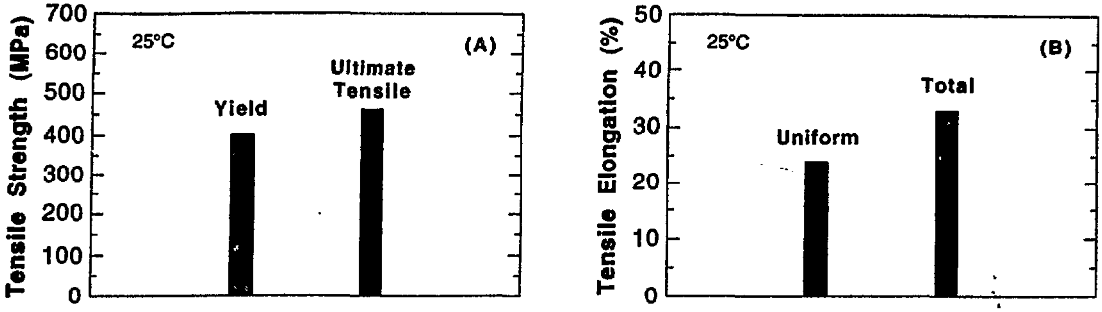


Fig. 1 Tensile strength (A) and elongation (B) of unirradiated V-4Cr-4Ti at 25°C

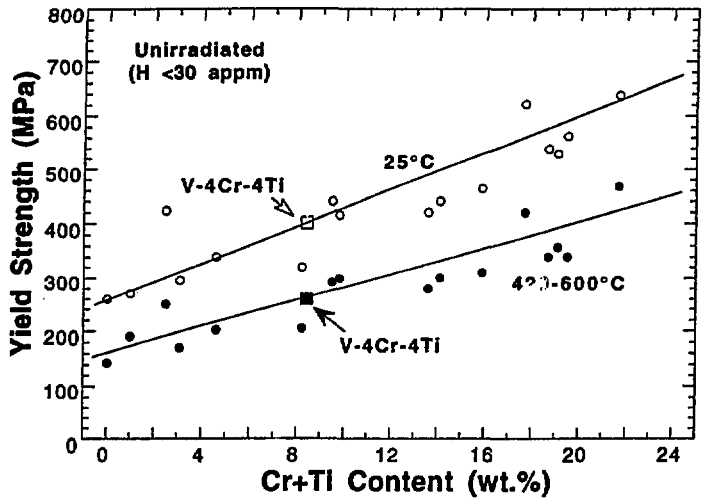


Fig. 2.

Yield strength of V-Ti and V-Cr-Ti alloys as a function of Cr+Ti content

non-DHCE specimens, whereas strengths were lower. Although the mechanisms leading to the higher ductility and lower strength of the DHCE specimens are not understood at this time, the consistent observations indicate that different types of hardening centers are produced during the DHCE and non-DHCE irradiation.

4. Fracture Behavior

Fracture behavior of the alloy was investigated by Charpy-impact tests on one-third-size (3.3-mm-thick) V-notched (45°) specimens and by bend tests on irradiated miniature specimens. In the latter test, a TEM disk or a broken tensile specimen submerged in a low-temperature bath was bent repeatedly until fracture, and cleavage morphology was characterized quantitatively to determine ductile-brittle transition behavior.

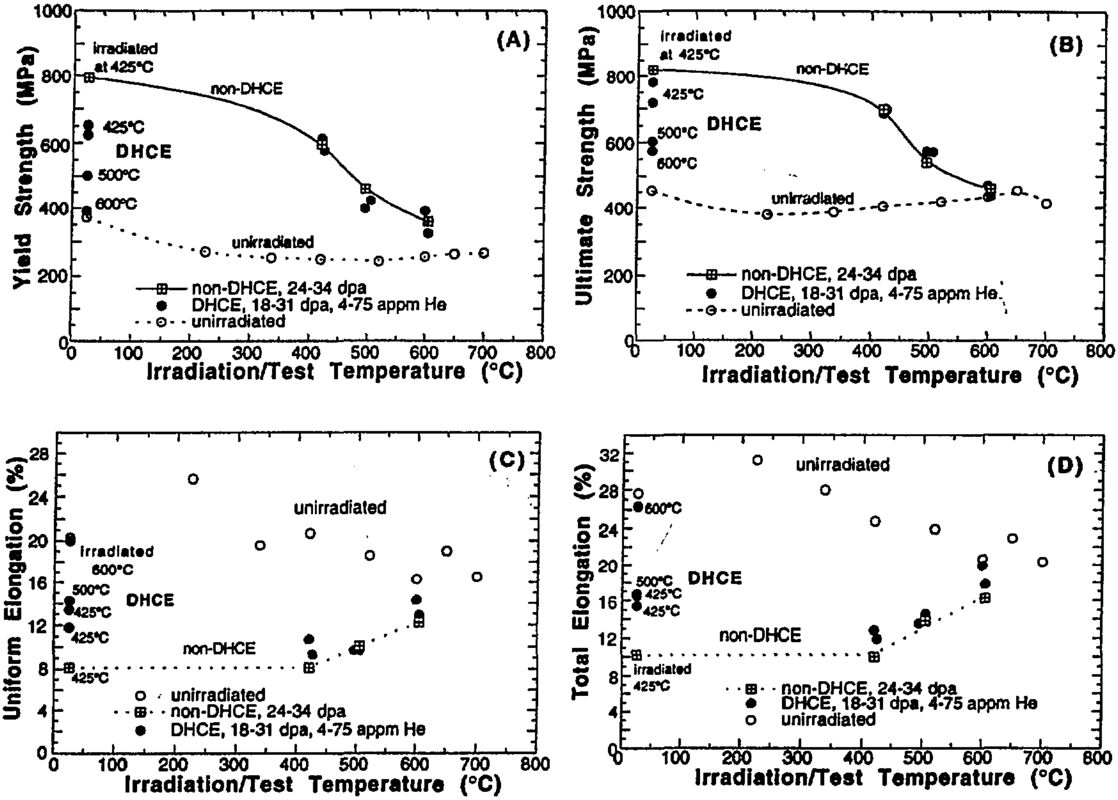


Fig. 3. Yield strength (A), ultimate tensile strength (B), uniform elongation (C), and total elongation (D) of V–4Cr–4Ti after irradiation at 420–600°C to 18–34 dpa in the DHCE and in non-DHCE conventional irradiation (negligible helium generation).

Charpy-impact energy and percentage of ductile fracture surface morphology determined on unirradiated specimens from the two types of tests are plotted as a function of test temperature in Fig. 4. As shown in the figure, the alloy remained ductile for all temperatures >–196°C, indicating that ductile-brittle transition temperature (DBTT) is <–200°C. Upper-shelf Charpy energy of the alloy is ≈140 J/cm^2.

Effects of neutron displacement damage, determined on specimens irradiated at 420–600°C to 24–34 dpa in the non-DHCE (helium generation negligible), are shown in Fig. 5.[16] The alloy remained ductile at >–196°C, even after the high-dose irradiation, exhibiting a virtual immunity to dpa damage. This is a truly remarkable property considering the bcc structure of the alloy. The energy minimum observed at ≈–120°C (surface temperature measured with spot-welded thermocouples) is believed to be associated with hydrogen concentrated in the interior of the Charpy specimen at a colder temperature.

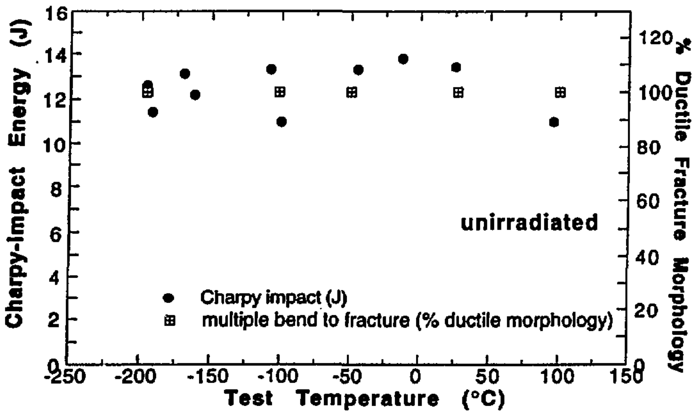


Fig. 4.

Charpy-impact energy (one-third-size) 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.

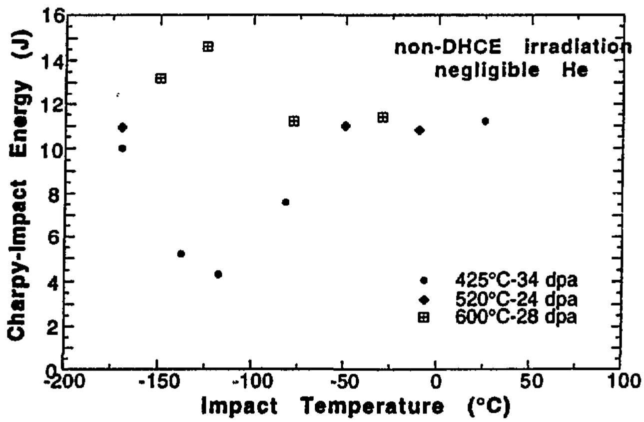
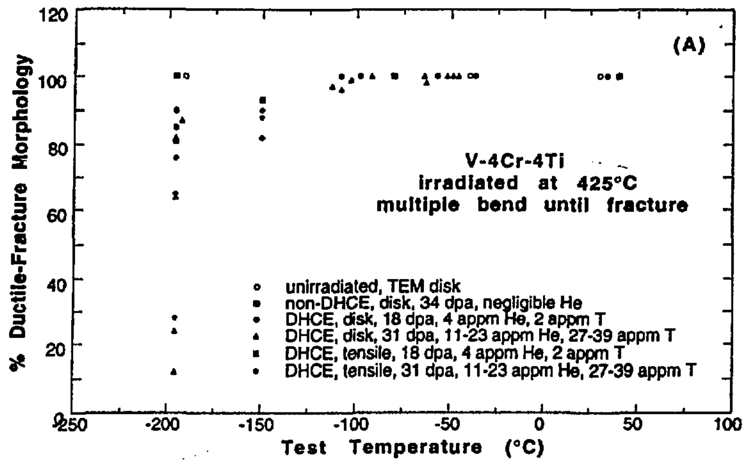


Fig. 5.

Charpy energy vs. impact temperature measured on one-third-size specimens irradiated in the non-DHCE (negligible helium generation) to 24–34 dpa at 420–600°C.

Effects of simultaneous displacement damage (18–31 dpa), helium (4–75 appm, Table 2) generation, and tritium (2–63 appm) uptake, determined on specimens irradiated at 420–600°C in the DHCE, are shown in Fig. 6.[17] No Charpy-impact specimens were irradiated during the experiment. Therefore, the ductile-brittle transition behavior was determined from results of quantitative fractography of TEM disks (0.3-mm-thick) and broken pieces of tensile specimens (1-mm-thick) that were fractured by repeated bending in a bath of liquid nitrogen or a mixture of acetone and dry ice.



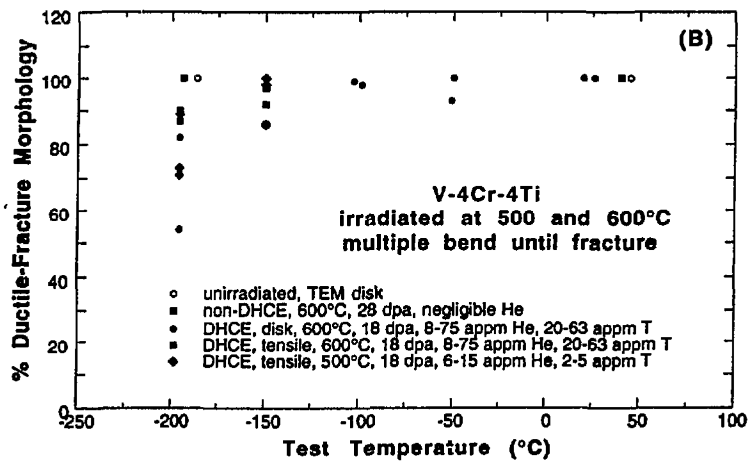


Fig. 6. Percent ductile-fracture morphology vs. test temperature of V-4Cr-4Ti TEM disks and broken tensile specimens irradiated at (A) 425°C and (B) 600°C and fractured by multiple bending.

As in irradiation during the non-DHCE, no brittle behavior was observed at temperatures >-150°C for DHCE specimens in which helium generation rate was ≈0.3-4.2 appm helium/dpa (Table 2). Brittle-fracture surface morphology was not observed at >-120°C, regardless of the level of dpa damage or helium and tritium content in the alloy. Predominantly brittle-cleavage fracture morphologies were observed only at -196°C in some specimens that were irradiated to 31 dpa at 425°C during DHCE. No intergranular fracture was observed in any tensile (Fig. 3) or bending-test (Fig. 6) specimens.

One of the important findings from the DHCE was the actual measured contents of helium and tritium in the V–4Cr–4Ti specimens were significantly lower than those calculated based on the assumed equilibrium ratio ($k_w = 0.01$) of tritium in the alloy to that in the liquid lithium (Table 2). This indicates that tritium level in lithium-cooled V–4Cr–4Ti first wall/blanket structure, and hence, the effect of tritium on the fracture toughness, will be significantly less than previously assumed. However, a more comprehensive database for the effects of higher helium–dpa ratio is needed from a further investigation.

5. Creep Properties

Stress-rupture life and steady-state creep rate of V-4Cr-4Ti and V-10Cr-5Ti have been determined in a carefully controlled vacuum environment at 600°C.[18] In Fig. 7, stress-rupture behavior of V–4Cr–4Ti is shown in Larsen-Miller plots. For comparison, similar properties of ferritic/martensitic and austenitic steels are also included in the figure. From the figure, it is obvious that the creep strength of V-4Cr-4Ti is substantially superior to that of HT-9, Type 316 stainless steel, and V-20Ti. In particular, this difference in creep strength is more pronounced when Larsen-Miller parameters are higher, i.e., at higher temperatures or longer times.

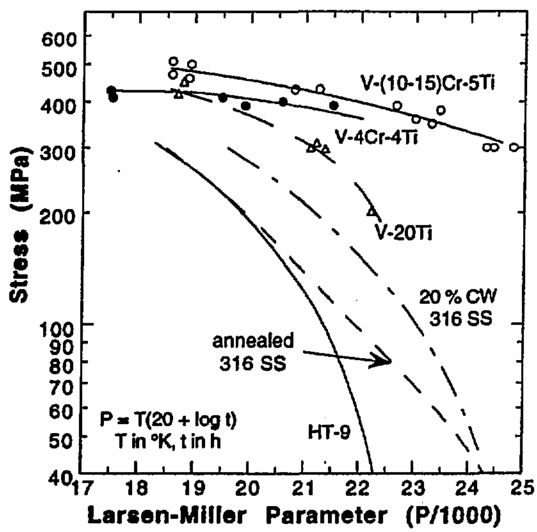


Fig. 7.

Larsen-Miller plots of creep strength of unirradiated V-4Cr–4Ti and ferritic and austenitic steels.

6. Density Change under Irradiation

Irradiation-induced density change and swelling behavior of V–4Cr–4Ti have been investigated after irradiation in conventional non-DHCEs.[19] V–Cr–Ti ternary alloys exhibited swelling (density change) maxima in the damage range of 30-70 dpa, as shown in Fig. 8, and swelling decreased on irradiation to higher dpa. The swelling resistance of the alloys was associated with high-density precipitation of ultrafine Ti_5Si_3, and it was concluded that >4 wt.% Ti and 400-1000 wppm Si are desirable to effectively suppress swelling.[20] Swelling resistance of V-4Cr-4Ti was excellent (Fig. 8), and has been associated with ultrafine Ti_5Si_3 for irradiation at 520-600°C and with dense formation of dislocation loops for irradiation at <420°C.[20]

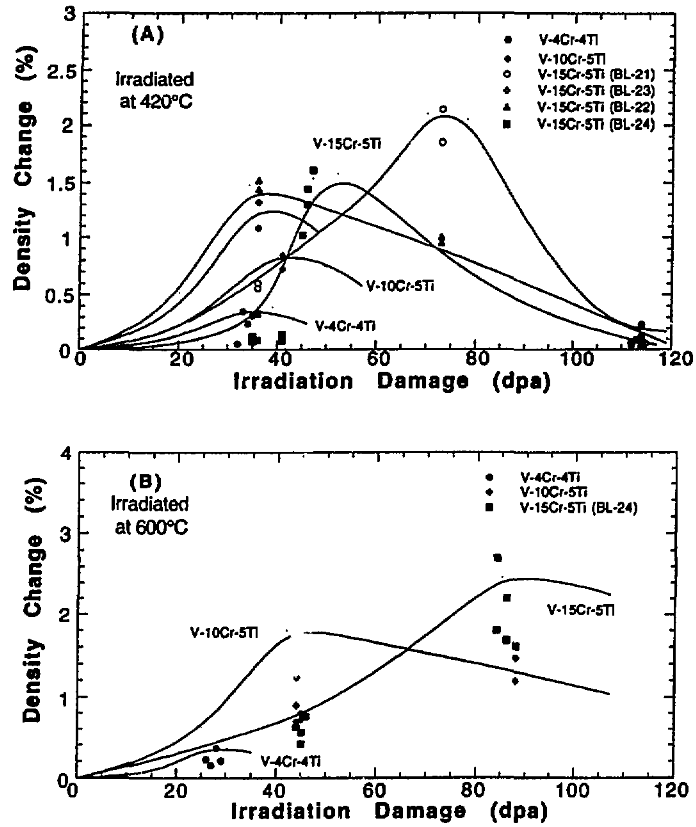


Fig. 8. Density change of V-Cr-Ti alloys as function of dose after irradiation at (A) 420 and (B) 600°C. Note the excellent resistance to swelling of V–4Cr–4Ti.

Combined effects of dynamically charged helium and neutron damage on density change, void distribution, and microstructural evolution of V–4Cr–4Ti have been determined after irradiation to 18–31 dpa at 425–600°C in a DHCE.[20] Results of density measurements for DHCE specimens are given in Fig. 9, along with the similar results from non-DHCE specimens.

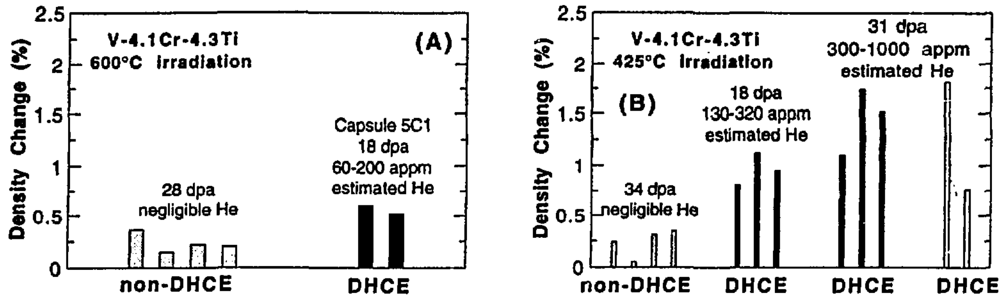


Fig. 9. Comparison of density changes of V–4Cr–4Ti during DHCE and non-DHCEs: (A) 600°C and (B) 425°C.

7. Microstructural Stability

The primary feature of microstructural evolution during non-DHCE (negligible helium generation) irradiation at 520 and 600°C was high-density formation of ultrafine Ti_5Si_3 precipitates, dislocation loops, and short dislocations.[11] For irradiation at 420°C, precipitation of Ti_5Si_3 was negligible, and "black-dot" defects and dislocations were observed in significantly higher densities. Despite their extremely high densities, neither black-dot defects nor Ti_5Si_3 precipitates are overly detrimental to ductility and toughness of the alloy, yet they effectively suppress irradiation-induced swelling.[9]

Thermally formed Ti(O,N,C) precipitates and irradiation-induced Ti_5Si_3 precipitates, normally observed in vanadium-base alloys containing titanium, are stable during irradiation at 420–600°C. Unstable microstructural modifications that are likely to degrade mechanical properties significantly were not observed, e.g., irradiation-induced formation of fine oxides, carbides, nitrides, or chromium-rich clusters. As in the non-DHCE, Ti_5Si_3 did not precipitate during irradiation at 420°C but precipitated in similar density at 500–600°C in the DHCE.[20]

No microvoids were observed in any of the specimens irradiated at 420–600°C to 28–34 dpa in the non-DHCE. Microvoids were observed only in DHCE specimens irradiated to ≈31 dpa at 425°C in high-tritium capsules (4D1 and 4D2, Table 2). In these specimens, moderate number densities of diffuse helium bubbles were observed in localized grain matrix and near limited fraction (≈15–20%) of grain boundaries. 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.[21-27] Microvoids were either absent or negligible in V–4Cr–4Ti specimens irradiated in the DHCE capsules other than 4D1 and 4D2, including Capsule 5C2 (4.2 appm helium/dpa). It seems that most of the dynamically produced helium atoms were trapped in the grain matrix without significant bubble nucleation or growth in these specimens.

8. Conclusions

V-4 wt.%Cr-4 wt.%Ti has been identified as the most promising vanadium–base alloy for application in fusion reactor first-wall and blanket structures. A comprehensive tests have been conducted on this alloy because it exhibited the most attractive combination of the mechanical and physical properties that are prerequisites for first-wall and blanket structures, i.e., high tensile strength, high ductility, good creep properties, high impact energy, low ductile-brittle transition temperature before and after irradiation, microstructural stability, excellent resistance to irradiation-induced swelling, and good resistance to corrosion in lithium.

In particular, the alloy was found to be virtually immune to neutron displacement damage at 420–600°C, a remarkable property compared to other candidate materials being investigated by the fusion–reactor–materials community. Effects of helium, charged dynamically in simulation of fusion reactor conditions, on tensile, ductile–brittle transition, and swelling properties were insignificant. Ductile–brittle transition temperatures of the alloy were below −150°C after irradiation to 18–31 dpa at 425°C–600°C either in the dynamic helium charging experiment or in conventional irradiation (negligible helium generation). Results of analysis of tritium and helium in specimens irradiated in the dynamic helium charging experiment indicate that tritium level in lithium–cooled V–4Cr–4Ti first wall/blanket structure, and hence, the effect of tritium on the fracture toughness of the structure, will be significantly less than previously assumed. Thermal creep behavior of the alloy was also significantly superior to that of austenitic and ferritic/martensitic steels.

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