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IRRADIATED AT <430°C***

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TENSILE AND IMPACT PROPERTIES OF VANADIUM-BASE ALLOYS IRRADIATED AT <430°C*

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ABSTRACT

Tensile and impact properties were investigated at <430°C on V-Cr-Ti, V-Ti-Si, and V-Ti alloys after irradiation to \approx 2-46 dpa at 205-430°C in lithium or helium in the Fast Flux Test Facility (FFTF), High Flux Isotope Reactor (HFIR), Experimental Breeder Reactor II (EBR-II), and Advanced Test Reactor (ATR). A 500-kg heat of V-4Cr-4Ti exhibited high ductile-brittle transition temperature and minimal uniform elongation as a result of irradiation-induced loss of work-hardening capability. Work-hardening capabilities of 30- and 100-kg heats of V-4Cr-4Ti varied significantly with irradiation conditions, although the 30-kg heat exhibited excellent impact properties after irradiation at \approx 390-430°C. The origin of the significant variations in the work-hardening capability of V-4Cr-4Ti is not understood, although fabrication variables, annealing history, and contamination from the irradiation environment are believed to play important roles. A 15-kg heat of V-3Ti-1Si exhibited good work-hardening capability and excellent impact properties after irradiation at \approx 390-430°C. Helium atoms, either charged dynamically or produced via transmutation of boron in the alloys, promote work-hardening capability in V-4Cr-4Ti and V-3Ti-1Si.

KEY WORDS: low-temperature tensile and impact properties, neutron irradiation, work hardening, helium, V-4Cr-4Ti, V-3Ti-1Si

INTRODUCTION

Recent attention to vanadium alloys has focused on low-temperature irradiation performance of V-(4-5)Cr-(4-5)Ti, especially tensile and impact properties after irradiation at <430°C. From several irradiation experiments at 80-430°C, it has been reported that a 500-kg (ID #832665) and a 30-kg (BL-47) heat of V-4Cr-4Ti¹⁻⁵ and an 80-kg heat (BL-63) of V-5Cr-5Ti⁶ exhibited low or negligible uniform elongation as a result of irradiation-induced loss of work-hardening capability. In contrast, similar loss of work-hardening capability (LWHC) of vanadium alloys has been not observed after irradiation at \geq 500°C.⁷ Recent data also indicated that work-hardening capability in V-4Cr-4Ti is dependent not only on individual heat but also on irradiation variables at <430°C.²⁻⁵ Because understanding the LWHC phenomenon is considered to be important in governing the minimum operating temperature of a fusion reactor, a more systematic evaluation of the phenomenon was conducted in this work on a wider variety of alloys, including V-5Ti and V-3Ti-1Si, that were irradiated at <430°C in several conventional and dynamic-helium-charging experiments (DHCE) in FFTF, HFIR, and EBR-II. The 500-kg heat of V-4Cr-4Ti also exhibited severe embrittlement manifested by very low impact energy and high ductile-brittle transition temperature (DBTT) after irradiation either at 100-275°C in a helium environment in HFBR¹ or at \approx 390°C in an Li environment in EBR-II.⁶ This was a major surprise in view of a previous report that the 30-kg heat of V-4Cr-4Ti (BL-47) exhibited excellent impact properties after conventional irradiation (i.e., "non-DHCE") at >430°C in FFTF⁸ and that the 500-kg heat exhibited excellent impact properties in the nonirradiated state.⁷ To confirm the excellent resistance of the 30-kg heat of V-4Cr-4Ti to irradiation embrittlement and to investigate this heat's impact properties under more severe conditions (i.e., in the cold-worked state at lower irradiation temperatures), unannealed Charpy impact specimens of the heat were irradiated at \leq 400°C, and the results are also reported in this paper.

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

EXPERIMENTAL PROCEDURES

The elemental composition of the alloys, determined prior to irradiation, is given in Table 1. Tensile properties were measured at temperatures equal to or lower than the irradiation temperature in flowing argon at a strain rate of 0.0011 s^{-1} . Tensile specimens with a gauge length of 7.62 mm and a gauge width of 1.52 mm were machined from $\approx 1.0\text{-mm-thick}$ sheets that had been produced by rolling a $\approx 3.8\text{-mm-thick}$ plate at 25 or 400°C . Specimens from the V-Cr-Ti alloys were annealed at $950\text{-}1125^\circ\text{C}$ for 1 h in an ion-pumped vacuum system, whereas specimens from V-Ti and V-Ti-Si alloys were annealed at $1000\text{-}1050^\circ\text{C}$ for 1 h. Charpy impact specimens were machined from $\approx 3.8\text{-mm-thick}$ plates and were inserted in the irradiation capsules in either the annealed or cold-worked state. Some of the Charpy specimens of a 500-kg heat of V-4Cr-4Ti, irradiated at $\approx 390^\circ\text{C}$ in EBR-II, were annealed at $>430^\circ\text{C}$ to investigate the effect of postirradiation annealing on toughness recovery.

Table 1. Chemical composition of vanadium alloys

Heat ID	Nominal Comp. (wt. %)	Impurity Concentration (wt. ppm)			
		O	N	C	Si
BL-50	1.0Ti	230	130	235	1050
BL-62	3.1Ti	320	86	109	660
BL-52	3.1Ti	210	310	300	500
BL-46	4.6Ti	305	53	85	160
BL-12	9.8Ti	1670	390	450	245
BL-15	17.7Ti	830	160	380	480
BL-10	7.2Cr-14.5Ti	1110	250	400	400
BL-24	13.5Cr-5.2Ti	1190	360	500	390
BL-40	10.9Cr-5.0Ti	470	80	90	270
BL-41	14.5Cr-5.0Ti	450	120	93	390
BL-43	9.2Cr-4.9Ti	230	31	100	340
BL-49	7.9Cr-5.7Ti	400	150	127	360
BL-63 ^a	4.6Cr-5.1Ti	440	28	73	310
BL-27	3.1Ti-0.25Si	210	310	310	2500
BL-45	2.5Ti-1Si	345	125	90	9900
QN74 ^b	4.0Cr-4.1Ti	480	79	54	350
BL-47	4.1Cr-4.3Ti	350	220	200	870
VX-8 ^c	3.73Cr-3.93Ti	350	70	300	500
832665 ^d	3.8Cr-3.9Ti	310	85	80	783

^a80-kg heat fabricated with sponge Ti.

^bContains ≈ 250 appm B¹⁰.

^c100-kg heat, contains (in wppm) 1120 Al, 280 Fe, 500 Co, 270 Mo, 1280 Nb, and 19 Zr.

^d500-kg production-scale heat.

^eAll others 15- to 30-kg laboratory heats.

Details of the conventional and dynamic-helium-charging irradiation experiments (i.e., the FFTF-MOTA,^{5,7} HFIR 200J and 400J,⁹ EBR-II COBRA-1A2,¹⁰ EBR-II X-530,¹¹ and ATR-A1¹² experiments) are summarized in Table 2. In the DHCE, helium atoms were produced during irradiation in the range of 14-76 appm He,⁵ whereas in the non-DHCE helium generation was negligible except in the boron-doped heat QN74.

RESULTS AND DISCUSSION

Tensile Properties

Uniform plastic strains measured after irradiation at $<430^\circ\text{C}$ are summarized in Table 3, while those of the laboratory heats of V-4Cr-4Ti (BL-47) and V-3Ti-1Si (BL-45), irradiated in Li environment at $\approx 430^\circ\text{C}$ in the DHCE and non-DHCE in the FFTF,⁵ are plotted in Fig. 1 as a function of tensile test temperature. Similar results measured on specimens irradiated at 500-600 are also plotted in Fig. 1 for comparison. After conventional irradiation (non-DHCE)

at $\approx 427^\circ\text{C}$, the 30-kg heat of V-4Cr-4Ti (BL-47) exhibited uniform elongation $< 0.5\%$, manifesting a nearly complete LWHC. In contrast, a 15-kg heat of V-3Ti-1Si (BL-45) exhibited relatively high uniform elongation ($\approx 3.7\%$), showing that the heat exhibits good work-hardening capability.

Table 2. Summary of irradiation experiments

Experiment ID	Subcapsule	Environment	Temperature ($^\circ\text{C}$)	dpa	He/dpa Ratio
FFTF-nonDHCE	many	Li	427-600	33-53	-
FFTF-DHCE	many	Li	430-600	13-27	0.4-4.2
HFIR	200J	He	200	10	-
	400J	He	400	10	-
COBRA-1A2	V499	Li	395	36	-
	V495	Li	379	31	-
EBR-II X530	S8	Li	394	4	-
	S9	Li	390	4	-
ATR-A1	many	Li	138-285	1.5-4.3	-

Table 3. Uniform plastic strain of vanadium-base alloys irradiated at $<430^\circ\text{C}$ (test temperature same as irradiation temperature except for experiments in FFTF^a)

Heat ID	Composition (wt.%)	HFIR-200J 300°C, 10 dpa	HFIR-400J 400°C, 10 dpa	COBRA-1A2/V499 395°C, 36 dpa	COBRA-1A2/V495 379°C, 31 dpa	EBRII-X530-S8 394, 4 dpa	EBRII-X530-S9 390, 4 dpa	FFTF-nonDHCE ^a 427°C, 33-53 dpa	FFTF-DHCE ^a 430°C, 13-27 dpa
BL-50	1.0Ti	0.56							
BL-62	3.1Ti				0.65				
BL-46	4.6Ti	0.66		1.44		0.75, 0.95			1.8, 1.4, 1.4 (425°C) 3.0 (200); 2.4 (100); 4.5 (23)
BL-12	9.8Ti	0.79							
BL-15	17.7Ti	2.42						3.7, 4.1 (420°C) 2.8 (23°C)	
BL-10	7.2Cr-14.5Ti							2.6, 4.9, 3.0 (420) 2.8 (23)	
BL-24	13.5Cr-5.2Ti							0.9, 1.8 (420) 1.3 (23)	
BL-40	10.9Cr-5.0Ti			1.43					
BL-41	14.5Cr-5.0Ti		0.94						
BL-43	9.2Cr-4.9Ti	0.66		1.32				1.2 (420)	1.0 (425); 3.5 (200); 6.3 (23)
BL-49	7.9Cr-5.7Ti	0.52		1.35	1.64		0.65	1.2 (420)	1.8, 1.0 (425); 4.2 (23)
BL-63	4.6Cr-5.1Ti					0.80, 0.52			
BL-27	3.1Ti-0.25Si							1.8, 1.5, 4.0 (420)	
BL-45	2.5Ti-1Si	0.22		4.55		1.68, 1.56		3.7 (420); 4.1 (23)	5.1, 6.3 (425); 9.4 (25)
QN74	4.0Cr-4.1Ti-B					1.07, 1.31			
BL-47	4.1Cr-4.3Ti	0.4, 0.79	2.91		0.92, 2.23 (CW) ^b			0.3 (420); 0.5 (23)	1.9, 1.3 (425); 3.2 (200) 2.6 (100); 4.4, 4.3 (23)
VX-8	3.7Cr-3.9Ti					2.62, 3.93	0.82, 0.59		
832665	3.8Cr-3.9Ti						0.65, 0.45, 0.34, 0.57, 0.65		

^aTensile test temperature given in $^\circ\text{C}$ in parenthesis.

^b0.92 and 2.23%, respectively, for annealed and cold-worked specimens.

Helium atoms produced at $\approx 430^\circ\text{C}$ during the DHCE seem to be conducive to higher ductility (compared to that under non-DHCE) and lower yield strength. Satou et al.¹³ reported that the ductility of V-5Cr-5Ti-1Si, doped with Al and Y, was higher after irradiation in the same DHCE than in non-DHCE in the FFTF. Results in Fig. 1 are consistent with this, indicating that helium atoms suppress susceptibility to LWHC at $\approx 430^\circ\text{C}$. When irradiated in the same S9 subcapsule in the X-530 experiment, uniform elongation was higher in V-4Cr-4Ti doped with ≈ 250 appm B¹⁰ (Heat QN74) than in two heats of V-4Cr-4Ti (VX-8 and #832665), i.e., 1.1-1.3% vs. 0.3-0.8%. This observation also indicates that helium produced from transmutation of B¹⁰ promoted work-hardening capability in QN74. This seemingly beneficial effect of helium is believed to be important in evaluating the performance of V-4Cr-4Ti and V-3Ti-1Si, because LWHC at $<430^\circ\text{C}$ under helium-generating reactor conditions is considered to be a major factor in governing the minimum operating temperature. From the standpoint of susceptibility to LWHC, V-3Ti-1Si appears to be more advantageous than V-4Cr-4Ti, although other important factors such as creep strength could be inferior.⁷

Uniform elongation of four heats of V-4Cr-4Ti and one heat of V-3Ti-1Si (from Table 3) is plotted in Fig. 2. For irradiation temperatures at 380-430°C, effect of dpa level appears to be secondary. Work-hardening capability, and hence uniform elongation, of V-4Cr-4Ti appear to be influenced significantly by subtle irradiation conditions. The 500-kg heat (#832665) exhibited the lowest work-hardening capability after irradiation in Li in the X-530 experiment (uniform elongation 0.3-0.7%). The specimens from the 100-kg heat (VX-8), irradiated in the S8 subcapsule of X-530, showed good work-hardening capability (uniform elongation 2.6-3.9%), whereas the specimens from the same heat irradiated in the S9 subcapsule exhibited uniform elongation of 0.6-0.8% only. The 30-kg heat (BL-47) exhibited uniform elongation of 0.3-2.9% after irradiation at \approx 380-430°C. Irradiation in either Li in the FFTF-DHCE or helium in the HFIR-400J experiment resulted in relatively higher uniform elongation in BL-47. This heat appears to exhibit uniform elongation that is somewhat higher than that of #832665, but not as good as that of VX-8, although data that will allow a direct comparison are not yet available. In the COBRA-1A2 experiment, annealed and cold-worked specimens of BL-47 were irradiated to 379°C in an identical subcapsule (V495). Interestingly, uniform elongation of the latter material was significantly higher than that of the former (i.e., 2.23 vs. 0.92%).

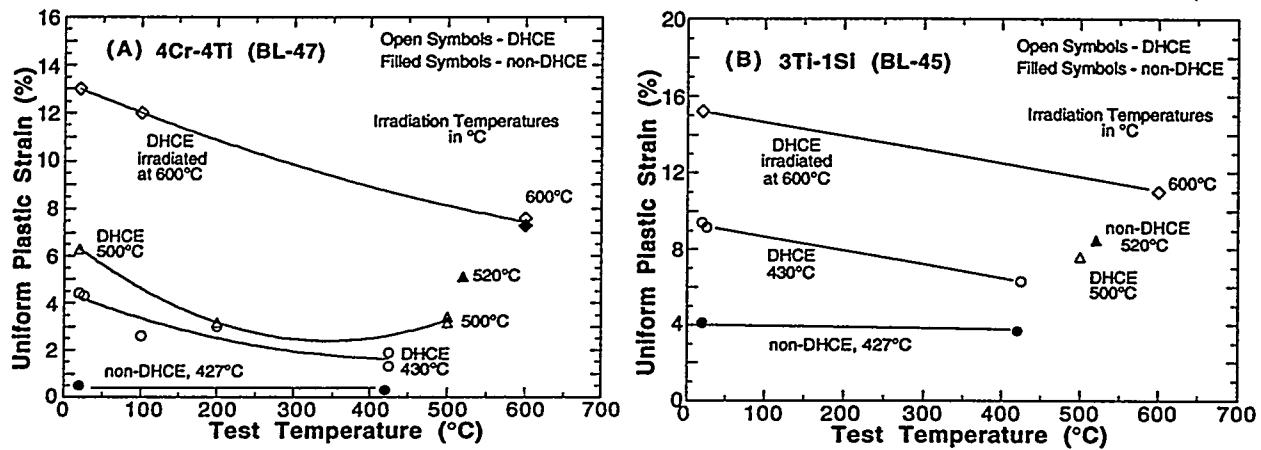


Fig. 1. Uniform elongation of annealed specimens of V-4Cr-4Ti (BL-47, left) and V-3Ti-1Si (BL-45, right) irradiated in the DHCE (13-27 dpa, 4-75 appm He) and in non-DHCE (14-33 dpa) in FFTF.

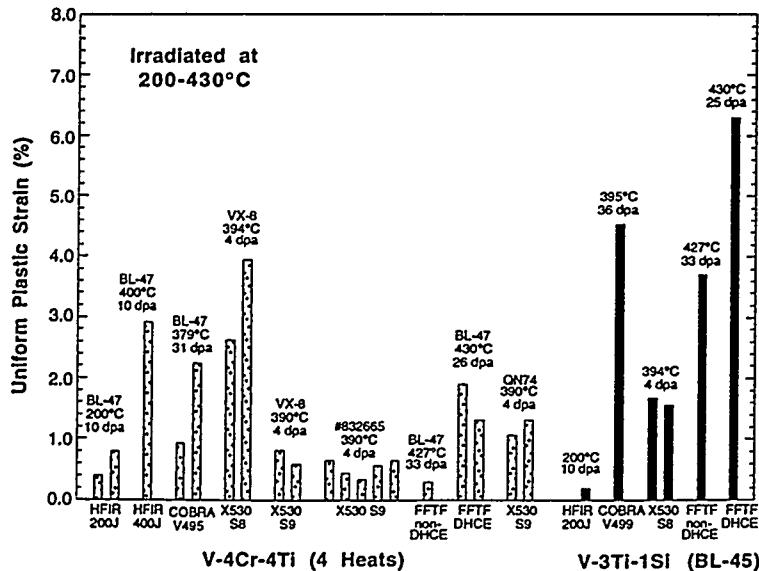


Fig. 2.

Uniform plastic strain of four heats of V-4Cr-4Ti and one heat of V-3Ti-1Si irradiated at 200-430°C to 4-46 dpa in several experiments.

A 15-kg heat of V-3Ti-1Si (BL-45), fabricated in parallel with BL-47 of V-4Cr-4Ti, exhibited good work-hardening capability when irradiated at 390-430°C (uniform elongation of 1.6-9.4%). This heat, like BL-47, exhibited a very low DBTT of <-190°C after conventional irradiation at 420-600°C to 14-33 dpa.^{7,8} Compared to V-3Ti-1Si (BL-45), a similar alloy V-3Ti-0.25Si (BL-27) exhibited significantly less work-hardening capability (uniform elongation of 1.5-1.8%).

The cause of the significant variation in work-hardening capability is not understood at this time, although fabrication variables (e.g., coldwork, density of titanium oxycarbonitride precipitates), annealing history, minor impurities, and contamination from the environment of the irradiation capsules (e.g., N transferred from sodium to Li through a Type 304 stainless steel subcapsule) are believed to play important roles. Based on the observation that irradiation-induced loss of work-hardening capability is sensitive not only to heats of similar composition (e.g., QN74, VX-8, and #832665 irradiated in S9 subcapsule in the X-530 experiment and BL-45 and BL-27 irradiated in the FFTF non-DHCE) but also to subtle irradiation conditions, it is likely that one or more alloying or impurity elements are involved in the process, most likely in association with a synergistic interaction with irradiation-induced defects.

Uniform elongation of V-4Cr-4Ti and V-3Ti-1Si alloys is plotted in Fig. 3 as a function of irradiation/test temperature. Although significant uncertainty is obvious, these plots provide an estimate of the approximate threshold temperature above which uniform elongation of the alloys is higher than a threshold level, e.g., 2%. It appears that the threshold temperature to meet a minimum uniform elongation of 2% could be anywhere between ≈320 and ≈470°C, depending on alloy type, heat, and basis of extrapolation. A possible advantage of a heat resistant to LWHC, such as VX-8, is obvious. One heat of V-3Ti-1Si (BL-45) exhibits relatively good work-hardening capability for irradiation at ≈390°C to ≈600°C, although data that support a similar behavior for <390°C are not yet available.

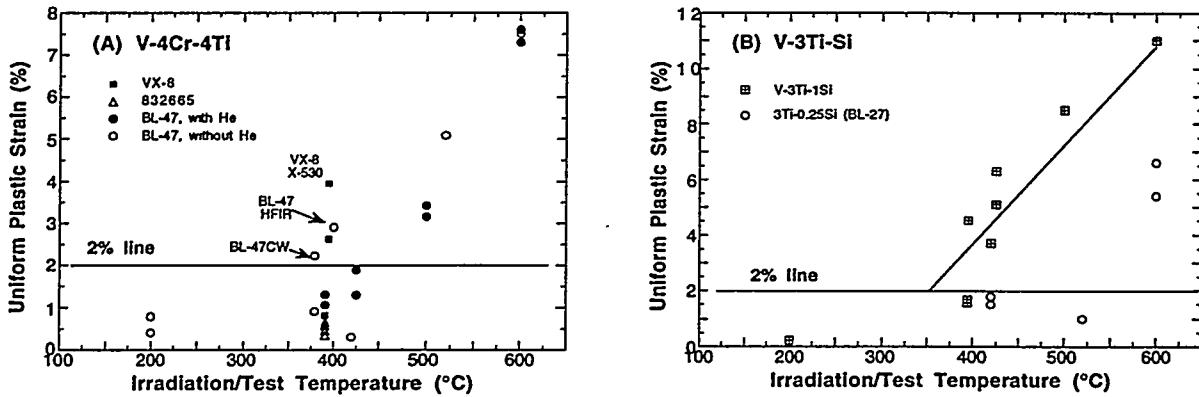


Fig. 3. Uniform plastic strain as function of irradiation temperature (same as test temperature) of V-4Cr-4Ti (left) and V-3Ti-Si (right).

RESULTS AND DISCUSSION

Impact Properties

Impact specimens were irradiated at ≈430°C in the FFTF⁷ or at <390°C in EBR-II and ATR. Details of the specimen orientation, annealing history, and irradiation parameters are summarized in Table 4. Impact properties of the cold-worked specimens of V-4Cr-4Ti (BL-47) and V-3Ti-1Si (BL-45), irradiated to ≈4 dpa in the X-530 experiment, are shown in Fig. 4. Similar results obtained from annealed specimens of the same heats, irradiated to ≈34 dpa at ≈430°C in non-DHCE in the FFTF,⁸ are also shown for comparison. In contrast to that of the annealed material, the effect of specimen orientation in the cold-worked material was very significant. Cold-worked V-4Cr-4Ti (BL-47) specimens machined in L-S orientation exhibited good impact properties even at -150°C after irradiation to ≈4 dpa at ≈390°C in the X-530

experiment. However, cold-worked specimens machined in L-T orientation exhibited inherently lower impact energies even before irradiation (Fig. 4), although outright brittle fracture was not observed even in L-T-oriented cold-worked specimens after irradiation in the X-530 experiment.

Table 4. Summary of Charpy impact specimen orientation, annealing history, and irradiation parameters

Experiment ID	Subcapsule	Environment	Temperature (°C)	dpa	He/dpa Ratio
FFTF-nonDHCE	many	Li	427-600	33-53	-
FFTF-DHCE	many	Li	430-600	13-27	0.4-4.2
HFIR	200J	He	200	10	-
	400J	He	400	10	-
COBRA-1A2	V499	Li	395	36	-
	V495	Li	379	31	-
EBR-II X530	S8	Li	394	4	-
	S9	Li	390	4	-
ATR-A1	many	Li	138-285	1.5-4.3	-

^aAnnealed in laboratory in ion-pumped vacuum of 2×10^{-6} Pa.

^bAnnealed in factory in diffusion-pumped vacuum for 2 h at a nominal temperature between 1050-1070°C, followed by annealing in laboratory in ion-pumped vacuum for 1 h at 1050 or 1125°C.

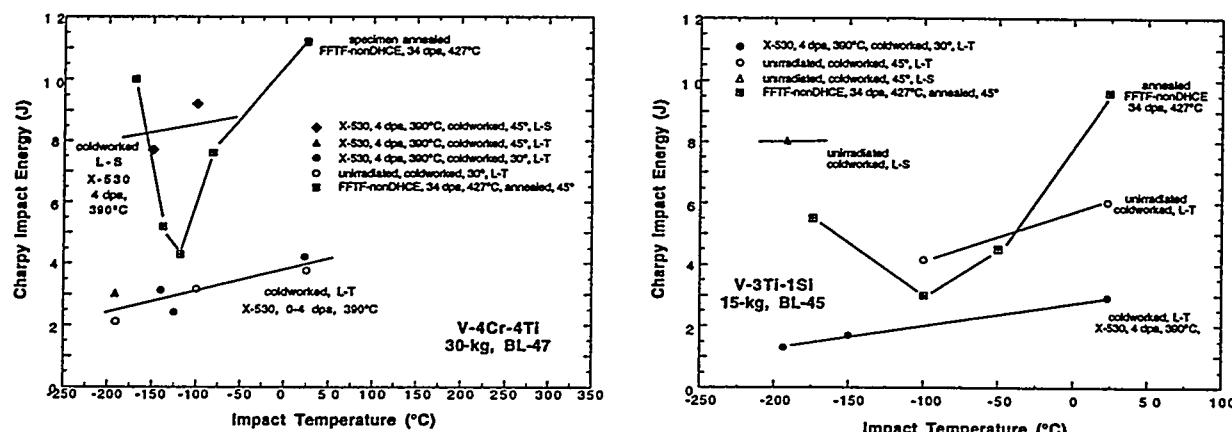


Fig. 4. Impact properties of cold-worked specimens of V-4Cr-4Ti (BL-47, left) and V-3Ti-1Si (BL-45, right) after irradiation to ≈ 4 dpa at $\approx 390^\circ\text{C}$ in X-530 experiment. Similar results from annealed FFTF-irradiated specimens are also shown.

In contrast to these laboratory heats, two-stage-annealed specimens of the 500-kg heat of V-4Cr-4Ti (#832665) exhibited brittle cleavage and intergranular fracture after irradiation to ≈ 4 dpa at $\approx 390^\circ\text{C}$ in the X-530 experiment (Fig. 5). Impact energies of six specimens, machined in L-S orientation from a 3.8-mm factory-annealed plate and annealed again at 1050-1125°C in ion-pumped vacuum in the laboratory (Table 4), were < 0.3 J when tested at -150 to +300°C. Ductile behavior (at 23°C) could be restored in the material only after postirradiation reannealing at $> 650^\circ\text{C}$ for ≈ 20 min in ion-pumped vacuum; this is shown in Fig. 6.

Charpy specimens, machined from cold-worked plates of the 500- (#832665) and 30-kg (BL-47) heat of V-4Cr-4Ti and annealed in ion-pumped vacuum at 1000°C for 1 h, were irradiated at 205-280°C in the ATR-A1 experiment. Results of impact tests are shown in Fig. 5. Specimens from the two heats, when annealed in ion-pumped high vacuum and irradiated at $< 280^\circ\text{C}$ in ATR-A1, exhibited relatively low upper-shelf energies of < 5 J and high DBTT of ≈ 60 -150°C. However, in contrast to the two-stage-annealed specimens of #832665 that were irradiated to ≈ 4 dpa at $\approx 390^\circ\text{C}$ in the X-530 experiment, outright brittle behavior at $> 23^\circ\text{C}$ was not observed in single-stage laboratory-annealed specimens of BL-47 and #832665 after irradiation to 1.5-4.3 dpa at 205-280°C in the ATR-A1 experiment.

CONCLUSIONS

1. Tensile properties of three heats of V-4Cr-4Ti (500-kg heat #832665, 100-kg VX-8, and 30-kg BL-47) were investigated after irradiation at 200-430°C in lithium or helium. All heats suffered significant loss of work-hardening capability, except for BL-47 irradiated to \approx 10 dpa in helium at \approx 400°C in HFIR and VX-8 irradiated to \approx 4 dpa in lithium at \approx 394°C in EBR-II. The latter heat, when irradiated in a similar sister subcapsule in the same EBR-II experiment, suffered significant loss of work-hardening capability. The origin of this apparent sensitivity to irradiation condition is not understood, although impurity contamination from the subcapsule environment is suspected. Although dislocation channeling appears to be responsible, the root cause of the susceptibility of V-4Cr-4Ti alloys to irradiation-induced loss of work-hardening capability at $<430^\circ\text{C}$ is not understood.
2. One heat of V-3Ti-1Si (BL-45) exhibited excellent impact and good tensile properties after irradiation at 390-430°C, indicating that the alloy retains good work-hardening capability.
3. Helium atoms, produced in V-4Cr-4Ti at \approx 390-430°C either during dynamic helium charging irradiation (Heat BL-47) or via transmutation of boron (Heat QN74), seem to promote work-hardening capability, thereby producing higher ductility (compared to that under conventional irradiation). Similar evidence was observed for V-3Ti-1Si (Heat BL-45).
4. The 30-kg heat of V-4Cr-4Ti (BL-47) exhibited excellent impact properties in either the annealed or cold-worked state after conventional irradiation at 390-430°C. However, for cold-worked material, the effect of specimen orientation was significant. Cold-worked specimens machined in L-S orientation exhibited excellent impact properties after irradiation to \approx 4 dpa at 390-430°C, whereas cold-worked specimens machined in L-T orientation exhibited inferior impact properties even before irradiation.

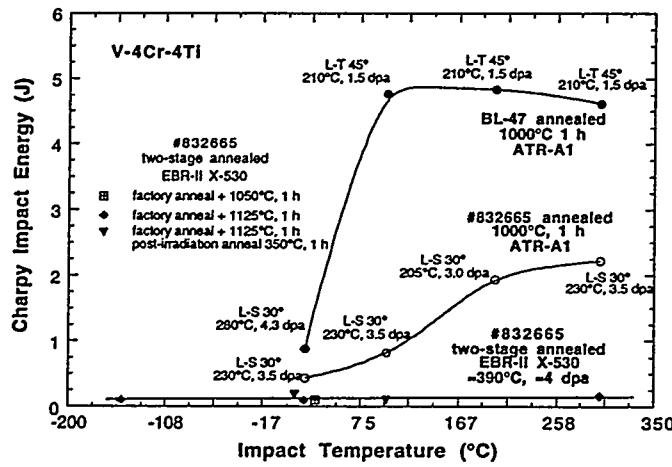


Fig. 5.

Impact properties of 500- and 30-kg heats of V-4Cr-4Ti irradiated in EBR-II X-530 and ATR-A1 experiments. Details of each specimen preparation and irradiation conditions are given.

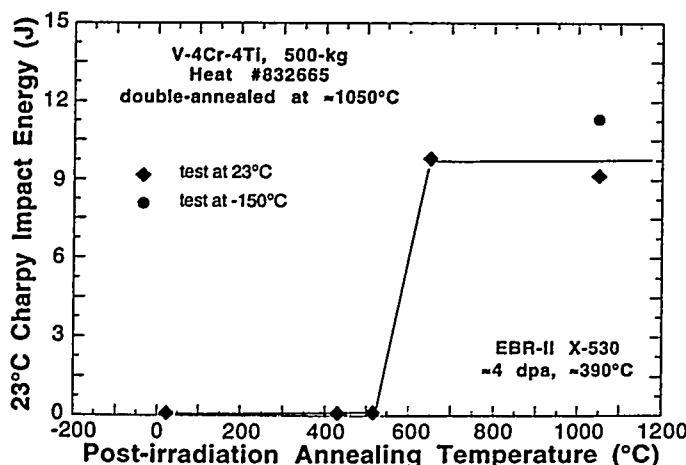


Fig. 6.

Impact energy measured on specimens of 500-kg heat of V-4Cr-4Ti (#832665) after irradiation to \approx 4 dpa at \approx 390°C and postirradiation annealing at 23-1000°C.

5. Two-stage-annealed specimens from the 500-kg heat of V-4Cr-4Ti (#832665) exhibited brittle impact characteristics after irradiation at $\sim 390^{\circ}\text{C}$ in EBR-II. Ductile behavior of this material could be restored only after postirradiation reannealing at $>600^{\circ}\text{C}$. In contrast to the two-stage-annealed material of #832665, outright brittle fracture (at $>23^{\circ}\text{C}$) was not observed in single-stage laboratory-annealed specimens of 500-kg #832665 and 30-kg BL-47 of V-4Cr-4Ti after irradiation to 1.5-4.3 dpa at 205-280°C in ATR. These observations indicate that irradiation at $<400^{\circ}\text{C}$ exacerbates the sensitivity of impact properties of V-4Cr-4Ti to thermomechanical and annealing treatments.

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