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Effects of low-temperature neutron irradiation on mechanical properties of vanadium-base alloys

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Abstract

Vanadium-base alloys were irradiated in three experiments to determine the effects of low-temperature neutron irradiation on their mechanical properties. The properties studied were tensile, Charpy impact and irradiation creep. The result of this study showed the alloys tested incurred significant hardening and embrittlement due to the irradiation. Heat-to-heat variations appeared to have little effect on hardening and embrittlement. Creep strains were measured from the specimens and a preliminary data set on creep strain rate was generated. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Vanadium-base alloys are attractive candidate structural materials for fusion first-wall/blanket applications because of their intrinsic low-activation, favorable thermal-physical properties, and good compatibility with lithium [1,2]. A primary candidate based on a balanced consideration has been the V–(4–5)Cr–(4–5)Ti class of alloys. Until a few years ago, with essentially all irradiation testing done in fast reactors at test temperatures >400°C, these alloys displayed significant resistance to radiation damage [3]. However, recent studies at lower temperatures have shown substantially reduced radiation resistance, manifested in lower ductility and enhanced hardening [4,5]. To investigate these temperature-sensitivity issues, which may limit the service temperatures of the materials in fusion devices, additional irradiation experiments were conducted in the ATR, BOR-60, and HFIR test reactors in the temperature range ≈200–500°C. The mechanical properties being investigated include tensile, impact, and irradiation creep. The results of this investigation are the subject of this paper.

2. Experimental procedures

2.1. Test materials and specimens

The majority of test specimens for this study were prepared from four V–(4–5)Ti–(4–5)Cr alloys and one V–4Fe–4Ti alloy. Table 1 shows the nominal compositions of these alloys. The 832665 alloy [6] in this group is the first large industrial heat produced in the US for fusion materials research; the others are smaller laboratory heats produced in the US, Russia and Japan.

The tensile specimens for the study were of the SS-3 design and had nominal gauge dimensions of 0.76 mm (t) × 1.52 mm (w) × 7.6 mm (l). They were machined from cold-rolled sheets with the longitudinal direction parallel to the final rolling direction of the sheets. Before the irradiation, the specimens were annealed in vacuum at 950°C or 1000°C for 1 or 2 h. The pre-irradiation annealing is a secondary test variable for this study.

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Table 1
Chemical composition of alloys investigated

Heat number	Ingot size (kg)	Nominal composition (wt%)	Impurity content (wppm)			
			O	N	C	Si
832665	500	V–3.8Cr–3.9Ti	310	85	80	780
BL-47	30	V–4.1Cr–4.3Ti	350	220	200	870
T87	30	V–5.0Cr–5.0Ti	380	90	110	550
VX8[a]	100	V–3.7Cr–3.9Ti	350	70	300	500
VM9407[b]	1	V–3.6Fe–4.3Ti	370	7	N/A	210

[a] From Russia. Contains (in wppm) 1120 Al, 280 Fe, 500 Co, 270 Mo, 1280 Nb and 19 Zr.
[b] From Tohoku University, Japan.

The Charpy impact specimens were 1/3-size and had dimensions of 3.3 mm (t) $\times$ 3.3 mm (w) $\times$ 25.4 mm (l). They were machined from cold-rolled plates and had a 30°, 0.61 mm-deep notch with a root radius of 0.08 mm, except for the BL-47 specimens, which had a notch angle of 45° from an earlier fabrication campaign. The notch orientation was perpendicular to the final rolling direction. The impact specimens were annealed in vacuum at 1000°C for 1 h before the irradiation.

The creep specimens were pressurized thin-wall tubes of 25.4 mm length, 4.57 mm OD, and 0.254 mm wall thickness. The tubing was produced by either drawing (832665 heat) or machining (VM9407 heat). The specimens were annealed at 1000°C for 1 h in vacuum before the pressurization.

2.2. Irradiation histories

The specimens were irradiated in three experiments: ATR-A1 in the Advanced Test Reactor (ATR) in the US, Fusion-1 in the BOR-60 reactor in Russia, and RB-12J in the High-Flux Isotope Reactor (HFIR) in the US.

The ATR-A1 test vehicle was a capsule consisting of 15 vertically stacked stainless steel subcapsules. As ATR is a water-cooled mixed-spectrum reactor, thermal neutron filters made of gadolinium were incorporated to protect the specimens from excessive V-to-Cr transmutation. For impurity control and temperature uniformity, high-purity lithium was used to bond the specimens inside the subcapsules. The ATR-A1 irradiation was conducted in the A10 channel of the reactor from November 1995 to May 1996 and achieved a peak neutron damage of 4.7 displacements-per-atom (dpa). Nominal test temperatures were 200°C and 300°C.

The Fusion-1 experiment [7] was a 508 mm long, lithium-bonded stainless steel capsule in BOR-60. The test specimens were arranged in 10 equal-height tiers inside the capsule over the middle 361 mm region of the core. Irradiation was conducted in the G23 Cell from July 1995 to June 1996. The calculated specimen temperatures during the irradiation were 320 ± 20°C and the calculated specimen displacement damage was 17–19 dpa. As BOR-60 is a sodium-cooled fast reactor with negligible thermal flux, neutron filtering was not necessary.

The HFIR RB-12J experiment utilized a temperature-controlled, gas-bonded capsule. Irradiation was completed in 10 cycles from February 1997 to July 1998 in core location RB-5B. To curtail the atypical V-to-Cr transmutation in HFIR, which is water-cooled, europium liners were used as the thermal neutron filter. The nominal test temperature of the specimens was 500°C, and the attained peak damage dose was 6.0 dpa.

2.3. Post-irradiation testing procedures

Post-irradiation tensile tests were conducted at room temperature in air or at irradiation temperature in high-purity argon. The tests were performed with an Instron machine without an attached specimen extensometer. The measured crosshead displacement was used to obtain the gauge section extension after subtracting the slack in the grip and the deformation of the load frame. For consistency, the strain rate for all tests was 0.5 mm/min, or $1.09 \times 10^{-3} s^{-1}$.

Charpy impact tests were conducted in air with a Dynatup drop-weight tester. A thermocouple spot-welded to the end of the specimen monitored the specimen temperature at impact. None of the specimens were degassed for hydrogen before the impact tests, because that procedure has been shown to have little effect on measured impact properties. Furthermore, it would have undesirably subjected the specimens to temperatures greater than that experienced during irradiation.

The diameters of the irradiation creep specimens were measured before and after the irradiation with a precision laser profilometer at five axial locations (x/l of 0.1, 0.3, 0.5, 0.7 and 0.9) at 9° azimuthal intervals. The precision of the measurements was $\approx 5 \times 10^{-4}$ mm, or $\approx$0.01% in strain. The 19 azimuthal readings were averaged to yield the mean diameter for each axial location. To exclude end effects from the welded end plugs, only the middle three diameters were used to determine the average diameter and effective creep strain.

3. Results and discussion

3.1. Tensile properties

Tensile specimens were irradiated in the ATR-A1 and BOR-60 Fusion-1 experiments. The test results show that yield strength (YS) and ultimate tensile strength (UTS) of all irradiated materials increased significantly, by a factor of ≈3–4, over those of the non-irradiated controls. At the same time, there is a significant loss of work-hardening ability, manifested by the small measured uniform elongation (typically <1%) and the closeness of the YS and UTS values. Following yielding, specimens failed rapidly due to plastic instability. A set of representative data from the tensile tests is shown in Table 2. The Heat VX8 specimen showed marginally greater uniform elongation in the room-temperature test; however, when tested at the irradiation temperature, its elongation was essentially as low as those of the other specimens. Within the data scatter, therefore, it appears that all the heats of vanadium-base alloys included in this test behaved similarly. The minor variations in pre-irradiation heat treatment, ranging from 1 to 2 h at 950°C or 1000°C for the 832665 material, produced little difference.

Post-test fractographic examinations revealed the fracture to be mostly ductile shear with no discernible cleavage failure in the lower-dpa ATR-A1 specimens. In the higher-dpa BOR-60 specimens, however, both ductile shear and transgranular cleavage fractures were noted. This observation is consistent with the generally greater elongation and reduction-in-area in the lower-dpa ATR-A1 specimens, thus suggesting a dose effect on radiation damage. Apparently, saturation was not reached at the ATR-A1 dose of ≈5 dpa.

Side surfaces of deformed tensile gauge section showed slip bands, characteristic of dislocation channeling formed during testing, in essentially all samples. A typical example is shown in Fig. 1. Short transverse cracks initiated at the surface were also found; they were perpendicular to the direction of the applied tensile load but did not propagate far into the specimens.

3.2. Charpy impact properties

Charpy specimens were irradiated in both the ATR-A1 and BOR-60 Fusion-1 experiments, but only the ATR-A1 specimens have been evaluated thus far. The available test data show that the low-temperature irradiation caused a significant drop in the upper-shelf energy and a marked increase in the ductile-to-brittle transition temperature (DBTT). Results for the 832665 and BL-47 heats are shown in Fig. 2. For the 832665 material irradiated at either ≈230 or 300°C, upper-shelf energy is 2–3 J, substantially below the ≈12–15 J for the non-irradiated material. While the DBTT could not be accurately determined due to the limited number of specimens, within the resolution of the data, it appears that the DBTT for the irradiated 832665 material is ≈150–200°C, which is significantly higher than the approximate −190°C for the non-irradiated control specimens. For the BL-47 material, the findings are approximately the same. As expected, below the DBTT the fracture was mostly cleavage cracks and above the DBTT the fracture was mostly ductile tear.

Table 2
Summary results of tensile tests. All tests were conducted at a strain rate of $1.09 \times 10^{-3} s^{-1}$. Values in parentheses are those of non-irradiated materials at comparable temperatures [a]

Test	Specimen	Heat	Pre-irradiation heat treatment (°C/h)	Irradiation temperature (°C)	Test temperature (°C)	Dpa	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	RA (%)
ATR-A1	71-F	832655	1000/1	280	290	4.3	945 (208)	983 (343)	0.7 (19)	2.1 (27)	16 (93)
	47-E	BL-47	1000/1	302	290	4.6	844 (229)	866 (381)	0.5 (18)	4.9 (24)	12
	72-D	T87	1000/1	300	290	4.1	880 (262)	941 (405)	1.1 (14)	4.1 (21)	32 (91)
BOR-60 Fusion-1	71-1	832655	1000/1	318	318	17	913	932	0.7	1.3	≈0
	71-2	832655	1000/1	318	23	17	1115	1120	0.3	0.4	≈0
	71-2H1	832655	1000/2	318	318	17	892	926	0.4	2.2	≈0
	71-2H2	832655	1000/2	318	23	17	1100	1115	0.3	0.5	≈0
	71-A	832655	950/2	318	318	17	953	962	0.4	1.3	≈0
	71-B	832655	950/2	318	23	17	1120	1125	0.5	0.8	≈0
	69-1	VX8	1000/1	323	323	19	909	936	0.5	2.3	17
	69-2	VX8	1000/1	323	23	19	1135	1170	1.4	2.8	4
	72-1	T87	1000/1	323	323	19	953	955	0.1	1.8	10
	72-2	T87	1000/1	323	23	18	1145	1150	0.4	0.4	≈0

[a] YS: 0.2% offset yield strength; UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation; RA: reduction of gauge cross-sectional area.

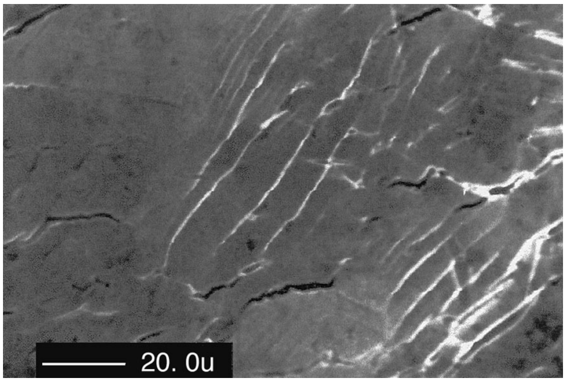

Fig. 1. Typical example of surface steps found on side surfaces of deformed tensile gauge section, indicating occurrence of dislocation channeling.

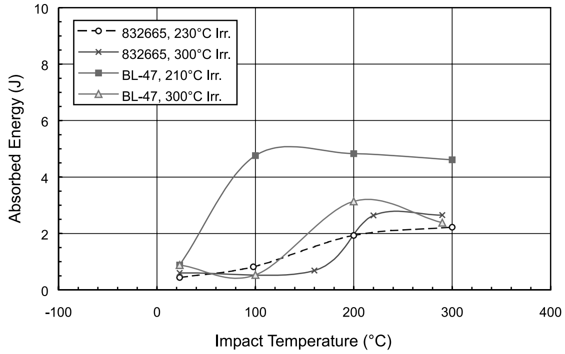

Fig. 2. Impact properties of 832665 and BL-47 Charpy specimens after ATR-A1 irradiation. Upper-shelf energies for irradiated specimens, ≈2–5 J, are substantially lower than those for non-irradiated controls, ≈11–15 J.

3.3. Irradiation creep

Sixteen pressurized-tube creep specimens were irradiated in this study: ten in the ATR-A1 experiment and six in the HFIR-12J experiment. In each test specimen group, one sample was not pressurized to serve as the control for stress-free swelling. None of the specimens ruptured during irradiation. Due to the relatively low-irradiation temperature (<500°C), the thermal creep component is deemed insignificant in these specimens.

The specimen's internal pressure loading and the measured diametral strain were converted to the wall-averaged effective von Mises stress and effective creep strain. The results are shown in Fig. 3 along with those from a prior torsional creep experiment performed by Troyanov et al. [8] in BR-10.

The data from this study show a consistent trend of increasing strain rate with stress, even though there is notable data scatter due to the low attained neutron damage, low measured strains, and limited number of test specimens. Within the resolution, however, the data from this study appear not to show the bilinear behavior – sharply increased strain rate at stress >≈120 Mpa – that Troyanov reported. Reducing the data uncertainty, including determining whether a bilinear behavior exists for the vanadium-base alloy, would be an important objective for the future irradiation creep experiments.

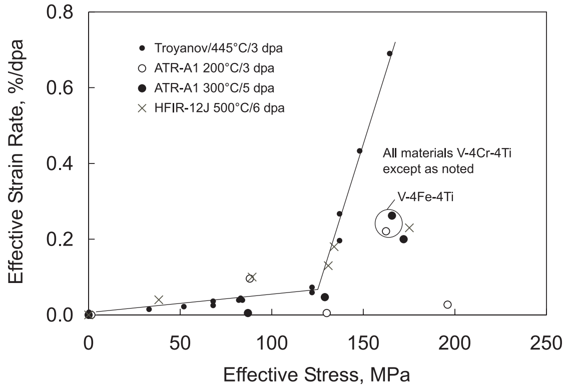

Fig. 3. Data set of effective creep strain rates from this study and from torsional creep tests by Troyanov et al. [8]. Correlation given by Troyanov (solid line) shows bilinear stress dependence.

4. Conclusions

Results from tensile and Charpy impact testing of samples after low-temperature irradiation showed significant hardening and embrittlement. Hardening increased the strength of the materials by about three-fold and reduced the ductility to <1% elongation. Heat-to-heat compositional variation appears to have little effect on fracture behavior because all heats of V–(4–5)Cr–(4–5)Ti alloys in this study behaved similarly. Likewise, variation in pre-irradiation annealing treatment also seems to have no effect. Slip localization and dislocation channeling provided the only plastic deformation noted in these specimens. Radiation damage appears to be dose-dependent, and there appears to be an increase in radiation damage from 5 to 17 dpa.

The impact properties of these materials also degraded significantly because of the low-temperature irradiation. Upper-shelf energy decreased from ≈11–15 J to ≈2–5 J and the DBTT increased from −190°C to ≈50–200°C. Below the DBTT, fracture was mostly cleavage cracks; above the DBTT the fracture was mostly ductile tear.

Due to the limited data, uncertainties in irradiation creep are still a major issue. Reducing the data uncertainty, including the existence of a bilinear behavior, is one of the most important objectives for the future irradiation creep experiments.

Acknowledgements

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