

**TENSILE PROPERTIES OF VANADIUM-BASE ALLOYS IRRADIATED IN THE FUSION-1 LOW-TEMPERATURE EXPERIMENT IN THE BOR-60 REACTOR** - H. Tsai, J. Gazda, L. J. Nowicki, M. C. Billone, and D. L. Smith (Argonne National Laboratory)

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60 is a sodium-cooled fast reactor in Russia with the low coolant inlet temperature of 300°C. The objective of the Fusion-1 experiment in BOR-60 was to study the effects of neutron dose in vanadium-base alloys in the 300-350°C temperature regime. Prior to this experiment, neutron testing of vanadium-base alloys in mixed-spectrum reactors and the fast reactor EBR-II at temperatures below ≈400°C had shown significant radiation hardening and embrittlement of materials even at a relatively low neutron dose. The Fusion-1 experiment, a joint U.S.-Russia effort to develop fusion materials, was designed to evaluate this low-temperature sensitivity at the high neutron fluence of ≈20 displacements per atom (dpa).

## SUMMARY

The irradiation has been completed and the test specimens have been retrieved from the lithium-bonded capsule at the Research Institute of Atomic Reactors (RIAR) in Russia [1]. During this reporting period, the Argonne National Laboratory (ANL) tensile specimens were received from RIAR and initial testing and examination of these specimens at ANL has been completed. The results, corroborating previous findings [2-5], showed a significant loss of work hardening capability in the materials. There appears to be no significant difference in behavior among the various heats of vanadium-base alloys in the V-(4-5)Cr-(4-5)Ti composition range. The variations in the preirradiation annealing conditions also produced no notable differences.

## EXPERIMENTAL PROCEDURE

### Test Specimens

The tensile specimens submitted by ANL for the Fusion-1 experiment were 1/3-sized sheet specimens, with an overall length of 25.4 mm and a gauge section that was 7.62 mm long x 1.52 mm wide x 0.76 mm thick. The long direction of the gauge section was parallel to the final rolling direction of the sheets.

The primary test variable of the ANL specimens was alloy composition: V-4Cr-4Ti (Heat 832665), V-5Cr-5Ti (Heat T87), V-4Cr-4Ti with addition of 250 appm of boron (QN74), and a Russian heat of V-4Cr-4Ti (VX8). Table 1 shows the nominal compositions of these alloys. Also included in the test were weldment specimens from bead-on-plate laser welds of the Heat 832665 material. The purpose of adding boron to the QN74 alloy was to study the effects of helium generation from the B(n,α)Li reactions.

The secondary test variable was preirradiation thermal-mechanical treatment: all specimens except the weldment were annealed in vacuum at 950 or 1000°C for 1 or 2 h to remove the prior cold work (≈50%). The laser weldment specimens received only a hydrogen outgassing at 400°C for 1 h in vacuum after the welding, i.e., without a postweld heat treatment or the nominal 1000°C annealing.

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Table 1. Nominal composition of the four alloys investigated

Heat Number	Ingot Size (kg)	Nom. Composition (wt.%)	Impurity Content (wppm)			
			O	N	C	Si
832665	500	V-3.8Cr-3.9Ti	310	85	80	780
T87	30	V-5.0Cr-5.0Ti	380	90	110	550
QN74 <sup>a</sup>	15	V-4.0Cr-4.1Ti-B	350	220	200	870
VX8 <sup>b</sup>	100	V-3.7Cr-3.9Ti	350	70	300	500

<sup>a</sup>Contains  $\approx$ 250 appm of <sup>10</sup>B.

<sup>b</sup>From Russia. Contains (in wppm) 1120 Al, 280 Fe, 500 Co, 270 Mo, 1280 Nb, and 19 Zr.

### Irradiation Summary

The Fusion-1 test vehicle was a single, 508-mm-long, lithium-bonded stainless steel capsule with a 36-mm OD and 32-mm ID. [1]. Inside the capsule, the test specimens were arranged in 10 equal-height tiers over the middle 361-mm region of the core. The ANL tensile specimens were placed below the core midplane, in Tiers 2, 3, and 4 (Tier 1 being at the bottom of the capsule) to attain the desired low specimen temperatures.

During the irradiation, which started in July 1995 and ended in June 1996, the reactor power and sodium inlet temperature were allowed to vary from time to time to comply with seasonal power output demands. Consequently, the specimen temperatures fluctuated during these operations. Based on the results of thermal analyses, the deviation from the time-averaged means was approximately  $\pm 16^\circ\text{C}$ . The calculated time-averaged temperatures for specimens in tiers 2, 3, and 4 were 318, 320, and 323°C, respectively.

Displacement damage and transmutations in the specimens were calculated from the neutron flux and spectrum data determined from prior in-depth spectrometry measurements and the flux monitors incorporated in the Fusion-1 capsule. For Tiers 2, 3, and 4, the calculated displacement damage in the specimens was 17, 18, and 19 dpa, respectively. Because of the lack of thermal neutrons, atypical transmutations were insignificant. For specimens with added boron, the calculated helium generation rate in the material was  $\approx$ 500 appm per dpa.

### Capsule Disassembly

Because of the relatively large size of the fusion-1 capsule, the conventional method of using liquid ammonia to dissolve the lithium bond was impractical. An alternative technique of using heated mineral oil to melt and remove the lithium bond was adopted. (This method had been used before at RIAR to disassemble similar lithium-bonded capsules.) To ascertain that the exposure to oil at a temperature of  $\approx 250^\circ\text{C}$  for several hours would not adversely affect the properties of the specimens, extensive confirmatory tests were conducted at ANL. The results of these confirmatory tests showed only benign effects from the oil exposure and no measurable uptake of interstitial impurities.

The capsule disassembly was completed in an air cell at the RIAR. After the top and bottom end plugs of the capsule were removed with a low-speed saw, the capsule was immediately immersed in a bath of mineral oil at room temperature. The oil was then heated to 250°C to melt the lithium, which floated to the surface of the oil bath. The oil was then cooled to room temperature and the specimens were retrieved from the bath. The specimens were then repeatedly rinsed in alcohol at room temperature to remove residual oil and lithium and other possible surface contaminants.

## RESULTS OF TENSILE TESTS

Thirteen tensile tests have been completed. The tests were conducted either at room temperature in air or at the irradiation temperature in high-purity flowing argon. Because the tests were performed without a specimen extensometer, the gauge elongation was determined from the crosshead displacement with the extension due to the slack in the grip and the deformation of the load frame subtracted. The strain rate for all tests was  $1.1 \times 10^{-3}$ /s. The results of these tests are summarized in Table 2.

Table 2. Tensile data for ANL specimens irradiated in Fusion-1 experiment<sup>a,b</sup>

Specimen ID No.	Material/Heat	Preirrad.	Test Temp. (°C)	YS <sup>c</sup> (MPa)	UTS <sup>c</sup> (MPa)	UE <sup>c</sup> (%)	TE <sup>c</sup> (%)	RA <sup>c,g</sup> (%)
		Heat Treatment (°C/h)						
71-2	V-4Cr-4Ti/832655	1000/1	23	1115	1120	0.3	0.4	<≈3
71-1	V-4Cr-4Ti/832655	1000/1	318	913	932	0.7	1.3	tbd
71-2H2	V-4Cr-4Ti/832655	1000/2	23	1100	1115	0.3	0.5	<≈3
71-2H1	V-4Cr-4Ti/832655	1000/2	318	892	926	0.4	2.2	tbd
71-B	V-4Cr-4Ti/832655	950/2	23	1120	1125	0.5	0.8	<≈3
71-A	V-4Cr-4Ti/832655	950/2	318	953	962	0.4	1.3	tbd
72-2	V-5Cr-5Ti/T87	1000/1	23	1145	1150	0.4	0.4	<≈3
72-1	V-5Cr-5Ti/T87	1000/1	323	953	955	0.1	1.8	tbd
70-2	V-4Cr-4Ti-B/QN74	1000/1	23	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>
70-1	V-4Cr-4Ti-B/QN74	1000/1	323	<sup>e</sup>	842	<sup>e</sup>	<sup>e</sup>	tbd
69-2	V-4Cr-4Ti/RF VX8	1000/1	23	1135	1170	1.4	2.8	<≈4
69-1	V-4Cr-4Ti/RF VX8	1000/1	323	909	936	0.5	2.3	tbd
71-LZ-1	832665/Laser	400/1	320	<sup>f</sup>	782	0.0	0.0	tbd

<sup>a</sup>All specimens were irradiated in the narrow temperature range of 318-323°C (time-averaged) and the dpa range of 17-19.

<sup>b</sup>All tests were conducted at a strain rate of  $1.1 \times 10^{-3}$ /s.

<sup>c</sup>YS: 0.2% offset yield strength; UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation; RA: reduction in area, tbd: to be determined.

<sup>d</sup>Specimen failed at grip at 727 MPa with no measurable plastic deformation before failure.

<sup>e</sup>Chart-drive malfunction prevented determination of these quantities.

<sup>f</sup>No measurable plastic deformation; offset yield strength could not be determined.

<sup>g</sup>See Ref. 6.

The yield strength of all of the materials was found to have increased significantly (by a factor of 3 to 4) over those of the nonirradiated materials. At the same time, there was a notable loss of work hardening ability, manifested by the small measured uniform elongation. All specimens failed rapidly due to plastic instability after yielding. Figure 1 shows the recorded tensile loading curves for Specimens 71-2 (Heat 832665) and 69-2 (Heat VX8), both of which were tested at room temperature. The load-displacement profiles of other specimens are similar. When tested at room temperature, the specimen with added boron broke at the grip before reaching the yield point, attesting to the material's brittleness after irradiation. The laser weldment specimens, although fractured at the gauge section, was also brittle and showed no measurable elongation. Uniform elongations for all other specimens were small (typically <1%). The Heat VX8 specimen

showed a marginally greater uniform elongation at room temperature; however, when tested at the irradiation temperature, its uniform elongation was essentially the same as those of the other specimens. Within the data scatter, therefore, it appears that all of the heats of vanadium-base alloys included in this test behaved similarly. The minor variations of the preirradiation heat treatment of the Heat 832665 specimens produced little effects.

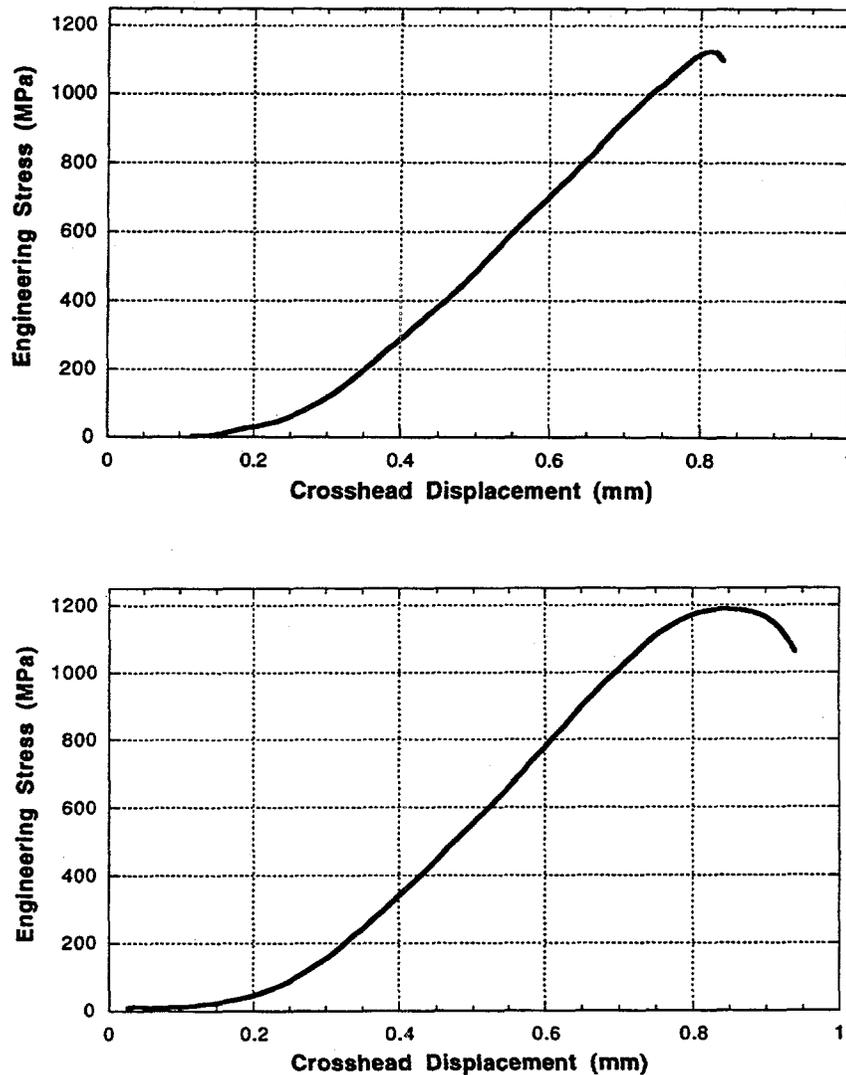


Fig. 1. Tensile loading curves for Specimens 71-2 (top, Heat 832665) and 69-2 (bottom, Heat VX8), both tested at room temperature. The gauge length of both specimens was 7.62 mm.

Fractographic examination with a scanning electron microscope is in progress. The objective of the examination is to determine the mode of fracture and the reduction of gauge cross-sectional area. The fractography of the room-temperature specimens has been completed. All fractures appear to be a mixture of ductile shear and brittle cleavage. Consistent with the elongation data, the reduction-in-area values were all small, as shown in Table 2. A more complete report of the fracture examination can be found in Ref. 6.

## FUTURE ACTIVITIES

Fractographic examination will be completed. Additional tests will be conducted at lower strain rates to study dislocation channeling effects. The results from the present and future tests will be compared with X530, ATR-A1 and HFBR test data to better define the low-temperature irradiation effects.

## REFERENCES

1. V. Kazakov et al., "Experience in Irradiation Testing of Low-Activation Structural Materials in Fast Reactor BOR-60," 8th Int. Conf. on Fusion Reactor Mater., Sendai, Japan, Oct. 26-31, 1998 (Proc. to be published).
2. H. Chung, H. Tsai, L. J. Nowicki, and D. L. Smith, "Tensile Properties of Vanadium Alloys Irradiated at 390°C in EBR-II," Fusion Materials Semiannual Progress Report for Period Ending June 30, 1997, DOE/ER-0313/22, pp.18-21.
3. S. J. Zinkle et al., "Effects of Fast Neutron Irradiation to 4 dpa at 400°C on the Properties of V-(4-5)Cr-(4-5)Ti Alloys," Fusion Materials Semiannual Progress Report for Period Ending December 31, 1996, DOE/ER-0313/21, pp.73-78.
4. D. J. Alexander et al., "Effects of Irradiation at Low Temperature on V-4Cr-4Ti," Fusion Materials Semiannual Progress Report for Period Ending June 30, 1996, DOE/ER-0313/20, pp.87-95.
5. H. Tsai et al., "Tensile and Impact Properties of Vanadium-Base Alloys Irradiated at Low Temperatures in the ATR-A1 Experiment," Fusion Materials Semiannual Progress Report for Period Ending December 31, 1997, DOE/ER-0313/23, pp.70-76.
6. J. Gazda, M. Meshii and H. Tsai, "Room Temperature Fracture in V-(4-5)Cr-(4-5)Ti Tensile Specimens Irradiated in Fusion-1 BOR-60 Experiment," *ibid.*