Tensile and Impact Properties of Vanadium-Base Alloys Irradiated at Low Temperatures in the ATR-A1 Experiment* H. Tsai, L. J. Nowicki, M. C. Billone, H. M. Chung, and D. L. Smith (Argonne National Laboratory)

Summary

Subsize tensile and Charpy specimens made from several V-(4-5)Cr-(4-5)Ti alloys were irradiated in the ATR-A1 experiment to study the effects of low-temperature irradiation on mechanical properties. These specimens were contained in lithium-bonded subcapsules and irradiated at temperatures between ≈200 and 300°C. Peak neutron damage was ≈4.7 dpa. Postirradiation testing of these specimens has begun. Preliminary results from a limited number of specimens indicate a significant loss of work-hardening capability and dynamic toughness due to the irradiation. These results are consistent with data from previous low-temperature neutron irradiation experiments on these alloys.

Objective

The objective of this task is to study the effects of low-temperature neutron irradiation on the mechanical properties of vanadium-base alloys. The irradiation was conducted in the ATR-A1 experiment, a collaborative effort between the U.S. Department of Energy and the Japanese Monbusho.

Background

Vanadium-base alloys [1,2] are attractive candidate structural materials for fusion reactors because of their intrinsic low activation, favorable thermal-physical properties, and good compatibility with lithium. The primary candidates are alloys in the V-(4-5)Cr-(4-5)Ti class [3]. Until a few years ago, with essentially all irradiation testing performed in fast reactors at test temperatures >400°C, these alloys also displayed significant resistance to radiation damage. However, recent irradiation studies [4,5] at lower temperatures (≈80-400°C), have shown significantly reduced resistance to radiation. From several irradiation experiments conducted at ≈80-400°C, it has been reported that both the V-4Cr-4Ti alloys (from the 500-kg Heat 832665 and Laboratory Heat BL-47) and the V-5Cr-5Ti alloy (from Laboratory Heat BL-63) exhibited low uniform elongation, i.e., loss of work-hardening ability, during tensile testing. After the low-temperature irradiations, these alloys also exhibited significant embrittlement, manifested by low impact energy and high ductile-brittle-transition temperature (DBTT). The purpose of the ATR-A1 experiment was to further explore how this temperature sensitivity affects irradiation behavior.

Experimental Procedure

ATR-A1 Irradiation History

The ATR-A1 irradiation experiment, [6,7] consisting of 15 vertically stacked, stainless steel subcapsules, was carried out in the Advanced Test Reactor (ATR). The test specimens were contained inside gadolinium thermal neutron filters (to minimize V-to-Cr transmutation) in the subcapsules and thermally bonded with lithium. The irradiation was conducted in the A10 channel of the reactor in Cycles 108A, 108B, and 109A. The total exposure was 133 effective full power days and the achieved peak neutron damage was 4.7 displacements per atom (dpa) in the test materials. The calculated temperatures and dpa values for specimens in each subcapsule are given in Table 1. Following the irradiation, the subcapsules were disassembled and the lithium bond was removed with liquid ammonia and alcohol. The U.S. specimens have been

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disseminated to Oak Ridge National Laboratory (ORNL), Pacific Northwest National Laboratory (PNNL), and Argonne National Laboratory (ANL).

Table 1. Calculated irradiation conditions of ATR-A1 experiment

Axial Position	Subcapsule Number	Test Materials	Specimen Temp. (°C)[a]	Dpa
15 (top)	AS1	V-alloys	139/144	0.7
14	AS3	V-alloys	186/194	1.5
13	AS4	Fe-allloys	263/277	2.2
12	AS5	V-alloys	198/212	3.0
11	AS6	V-alloys	223/234	3.5
10	AS8	V-alloys	246/259	3.9
9	AS9	V-alloys	273/286	4.3
8	AS10	V-alloys	288/302	4.6
7	AS11	V-alloys	285/300	4.7
6	AS12	V-alloys	282/295	4.5
5	AS14	V-alloys	284/300	4.1
4	AD16	Fe-alloys	337/355	3.8
3	AS7	V-alloys	287/303	3.0
2	AS13	V-alloys	245/258	2.3
1	AS17	V-alloys	204/213	1.5

[a] First value is for Cycles 108A and 109A, with a lobe power of 25 MW; second value is for Cycle 108B, with a lobe power of 27 MW. Rounded averages of the two are used in this report.

Test Materials and Specimens

The test specimens for this study were prepared from three V-(4-5)Ti-(4-5)Cr alloy heats: 832665, BL-47, and T-87. The nominal compositions of these three alloys are shown in Table 2.

Table 2. Composition of the three 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

The tensile specimens for the study at ANL consisted of 15 base-metal specimens and two weldment specimens. The nominal gauge dimensions of the base-metal specimens, size SS-3, were 0.76 mm thick x 1.52 mm wide x 7.6 mm long. The specimens were machined from cold-rolled sheets with the longitudinal direction parallel to the final rolling direction of the sheets. After the machining, the base-metal tensile specimens were annealed in a vacuum that was better than 10^{-7} torr at 1000°C for 1 h before the irradiation.

The two weldment specimens were prepared by bead-on-plate welding onto a piece of annealed plate with a laser beam. The beam traveled in a direction that was perpendicular to the rolling direction of the plate. After the welding, the specimens, also of SS-3 size, were machined from the plate with the weldment at the center and across the width of the gauge section. The weldment specimens were given only a hydrogen-outgassing at 400°C for 1 h in vacuum (i.e., without a postweld heat treatment or the nominal 1000°C anneal) prior to the irradiation.

The Charpy specimens for the study at ANL consisted of 19 base-metal specimens and eight weldment specimens. The base-metal specimens were 1/3-size, i.e., 3.3 mm thick x 3.3 mm wide x 25.4 mm long, machined from cold-rolled plates; they contained a 30°, 0.61-mm-deep notch with a root radius of 0.08 mm, except for some of the BL-47 specimens, which had a notch angle of 45° from an earlier fabrication campaign. The notch orientation (i.e., crack propagation direction) was perpendicular to the final rolling direction: L-S (crack into the thickness direction of the plate) for the 832665 and T87 specimens, and L-T (crack into the width of the plate) for the BL-47 specimens. Three of the 832665 specimens were fatigue precracked. After the machining/precracking, the base-metal specimens were annealed in vacuum at 1000°C for 1 h before the irradiation.

The weldment Charpy specimens were prepared by bead-on-plate welding with an electron-beam (EB) or tungsten-inert-gas (TIG) welder on annealed plates. The direction of weld travel was perpendicular to the rolling direction of the plate. After welding, the specimens were prepared with the V-notch in the weldment in the L-S direction. Although the EB weld specimens were 1/3-size, the TIG weld specimens were smaller, 1.5 (1.5 x 1.5 x 20.0 mm) because of the limit on the irradiation space. Similar to the tensile weldment specimens, the Charpy weldment specimens were given only a hydrogen-outgassing treatment at 400°C for 1 h in vacuum but no postweld heat treatment or annealing before the irradiation.

The list of tensile and Charpy specimens from the ATR-A1 experiment for investigation at ANL are shown in Tables 3 and 4, respectively.

Table 3. Inventory of ATR-A1 tensile specimens for the investigation at ANL

Specimen ID No.	Specimen Type[a]	Heat	Subcapsule	Irrad. Temp.(C)	dpa
71-A	SS-3	832665	AS1	139/144	0.7
71-B	SS-3	832665	AS8	246/259	3.9
71-C	SS-3	832665	AS8	246/259	3.9
71-D	SS-3	832665	AS9	273/286	4.3
71-E	SS-3	832665	AS9	273/286	4.3
71-F	SS-3	832665	AS9	273/286	4.3
47-A	SS-3	BL-47	AS1	139/144	0.7
47-B	SS-3	BL-47	AS17	204/213	1.5
47-C	SS-3	BL-47	AS17	204/213	1.5
47-D	SS-3	BL-47	AS10	288/302	4.6
47-E	SS-3	BL-47	AS10	288/302	4.6
72-A	SS-3	T87	AS1	139/144	0.7
72-B	SS-3	T87	AS17	204/213	1.5
72-C	SS-3	T87	AS14	284/300	4.1
72-D	SS-3	T87	AS14	284/300	4.1
71-LZ-A	SS-3	832665-weld	AS8	246/259	3.9
71-LZ-B	SS-3	832665-weld	AS10	288/302	4.6

[a] SS-3: 25.4 mm overall specimen length, gauge 0.76 mm thick x 1.52 mm wide x 7.6 mm long mm long.

Table 4. Inventory of ATR-A1 Charpy Specimens for the Investigation at ANL

Specimen ID No.	Heat	Configuration[a]	Weld[b]	Subcap.	Irrad. Temp(°C)	dpa
71-A	832665	1/3, M, 30, L-S	–	AS1	139/144	0.7
71-B	832665	1/3, M, 30, L-S	–	AS5	198/212	3.0
71-C	832665	1/3, M, 30, L-S	–	AS6	223/234	3.5
BL71W-39	832665	1/3, M, 30, L-S	–	AS6	223/234	3.5
BL71W-54	832665	1/3, M, 30, L-S	–	AS6	223/234	3.5
BL71W-20	832665	1/3, M, 30, L-S	–	AS9	273/286	4.3
BL71W-21	832665	1/3, M, 30, L-S	–	AS10	288/302	4.6
BL71W-22	832665	1/3, M, 30, L-S	–	AS10	288/302	4.6
BL71W-30	832665	1/3, M, 30, L-S	–	AS10	288/302	4.6
47-09	BL-47	1/3, M, 30, L-S	–	AS9	273/286	4.3
47-A	BL-47	1/3, M, 45, L-T	–	AS1	139/144	0.7
47-B	BL-47	1/3, M, 45, L-T	–	AS11	285/300	4.7
47-C	BL-47	1/3, M, 45, L-T	–	AS11	285/300	4.7
47-D	BL-47	1/3, M, 45, L-T	–	AS11	285/300	4.7
47-E	BL-47	1/3, M, 45, L-T	–	AS17	204/213	1.5
47-F	BL-47	1/3, M, 45, L-T	–	AS17	204/213	1.5
47-G	BL-47	1/3, M, 45, L-T	–	AS17	204/213	1.5
71E-A	832665	1/3, M, 30, L-S	EB	AS5	198/212	3.0
71E-B	832665	1/3, M, 30, L-S	EB	AS5	198/212	3.0
71E-C	832665	1/3, M, 30, L-S	EB	AS7	287/303	3.0
71E-D	832665	1/3, M, 30, L-S	EB	AS7	287/303	3.0
47-H	BL-47	1.5, M, 30, L-S	TIG	AS9	273/286	4.3
47-I	BL-47	1.5, M. 30, L-S	TIG	AS9	273/286	4.3
47-J	BL-47	1.5, M, 30, L-S	TIG	AS9	273/286	4.3
47-K	BL-47	1.5, M, 30, L-S	TIG	AS9	273/286	4.3
BL71W-27	832665	1/3, P, 30, L-S	–	AS14	284/300	4.1
BL71W-40	832665	1/3, P, 30, L-S	–	AS14	284/300	4.1
BL71W-45	832665	1/3, P, 30, L-S	–	AS14	284/300	4.1

[a] 1/3-size: 3.3 x 3.3 x 25.4 mm; 1.5-size: 1.5 x 1.5 x 20.0 mm;
M: machined blunt notch; P: precracked notch;
30: notch angle of 30°; 45: notch angle of 45°;
L-S or L-T: notch (crack propagation) direction.

[b] EB: electron beam welding; TIG: tungsten-inert-gas welding.

Results and Discussion

Tensile Tests

Four tensile tests, one each for the 832665, T87, and BL-47 base metal and one for the laser weldment, have been completed. All four tests were conducted in high-purity argon at 290°C, near the specimens' irradiation temperature. The tests were performed with an Instron machine without an extensometer attached to the specimen. Extensions due to the slack in the grips and the deformation of the load frame were subtracted from the crosshead displacement to obtain the correct gauge section extension. The strain rate for all of the tests was 1.09×10^{-3}/s.

In all four cases, the yield strength of the material increased significantly over the nonirradiated material because of the irradiation. At the same time, a significant loss of work-hardening ability was manifested by the small measured uniform elongation. After yielding, the base-metal specimens failed rapidly because of plastic instability. The recorded load-crosshead displacement curve for the T87 heat specimen is shown in Fig. 1. The peak load of 108 kg corresponds to an ultimate tensile strength of 941 MPa. The load-displacement profiles for other base-metal specimens are similar. The weld specimen was extremely brittle and displayed no measurable plastic deformation before it abruptly failed at a calculated engineering stress of 607 MPa. The tensile test results are summarized in Table 5.

Table 5. Summary of tensile test results for a limited number of ATR-A1 specimens.[a]

Specimen ID No.	Material	0.2% offset Yield Strength (Mpa)	Ultimate Tensile Strength (MPa)	Uniform Elongation (%)	Total Elongation (%)
71-F	832665	945 (208)	983 (343)	0.7 (19)	2.1 (27)
47-E	BL-47	844 (229)	866 (381)	0.5 (18)	4.9 (24)
72-D	T87	880 (262)	941 (405)	1.1 (14)	4.1 (21)
71-LZ-B	832665-weld	N/A[b]	N/A	≈0	≈0

[a] All tests were conducted at 290°C at a strain rate of 1.09 x 10^{-3}/s. Values in parentheses are those of nonirradiated control materials at comparable temperatures (300-337°C) [8].

[b] Specimen failed at 607 MPa with no measurable plastic deformation before failure.

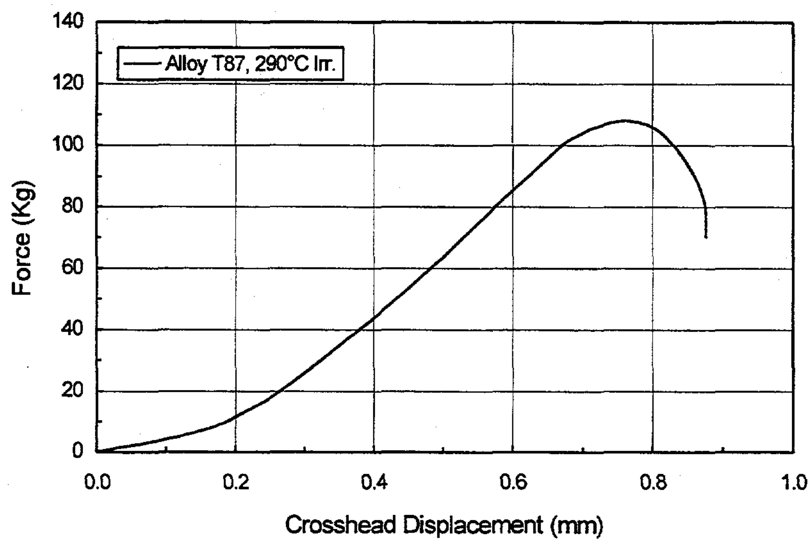

Fig. 1. Load-displacement curve for specimen 72-D (Alloy T87) after irradiation in the ATR-1.

Impact Tests

Seventeen Charpy impact tests have been completed. The tests were conducted in air with a Dynatup drop-weight tester. The specimen temperature during the impact test was measured with a thermocouple that was spot-welded to the end of the specimen. For tests in which temperatures were above ambient, a hot-air blower was used to provide the heating. None of the specimens were degassed for hydrogen before the impact tests, because that procedure has been shown to have little effect on the measured impact properties [4,9]. Furthermore, if the established degassing procedure were to be used, it would require that the specimens be heated to 400°C, which would be substantially above the irradiation temperatures.

The test data show that the ATR-A1 irradiation caused a significant drop of the upper-shelf energy of the material and a marked increase in the DBTT. The results for the blunt-notch base-metal specimens of the 832665 and BL-47 heats are shown in Fig. 2. For the 832665 heat irradiated at either ≈230 or 300°C, the upper-shelf energy is between 2 and 3 J, which is substantially below the ≈12-15 J for the nonirradiated material [9,10]. Although the DBTT could not be accurately determined because the number of specimens was limited, 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 nonirradiated control specimens. Results for the irradiated BL-47 material are approximately the same. (The upper-shelf energy and DBTT of the nonirradiated BL-47 material are ≈11-14 J and -190°C, respectively [11].) The BL-47 specimens irradiated at 300°C appear to have a slightly lower upper-shelf energy (≈2-3 J) than those irradiated at ≈210°C (≈5 J). This temperature dependence was noted before by D. J. Alexander et al. [4], who, based on resistivity measurements, attributed this effect to the increased effectiveness of defect clusters as barriers to dislocation movement at temperatures >200°C due to increased oxygen and carbon migration to the clusters.

As expected, and consistent with previous findings, the precracked 832665 specimens showed even lower absorbed energy than did their blunt-notch siblings. The measured data were 0.5 J at 23°C and 0.7 J at 290°C.

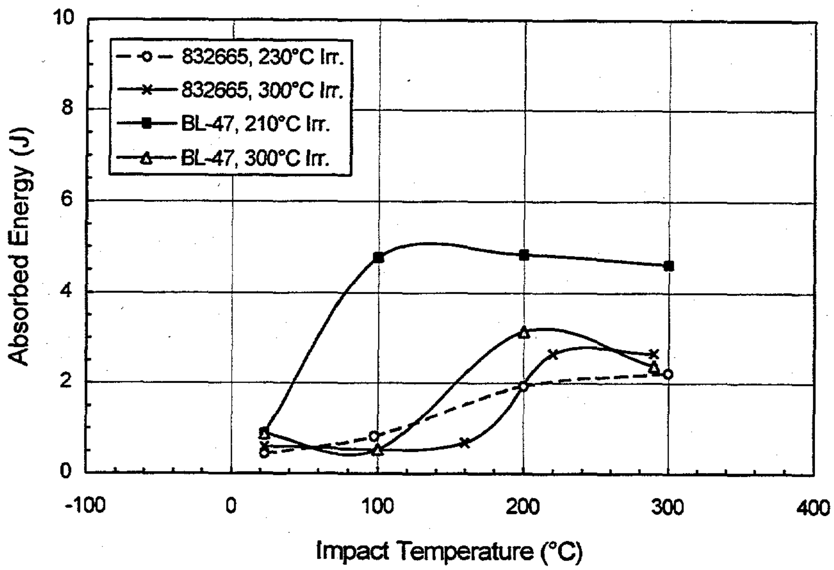

Fig. 2. Impact properties of 832665 and BL-47 specimens (1/3 size, blunt notch) after the ATR-A1 irradiation. Upper-shelf energies for the irradiated specimens, ≈2-5 J, are substantially lower than those for nonirradiated controls, ≈11-15 J.

Future Activities

The remainder of the tensile and impact tests will be completed. The fracture surfaces of selected specimens will be examined by scanning electron microscopy to delineate the fracture mode. Areal reduction of tensile specimens will be measured. Attempts will be made to prepare transmission electron microscopy specimens from the gauge section of tensile specimens near the rupture to study the interplay between dislocation channeling and hardening.

Conclusions

Results from testing three V-(4-5)Ti-(4-5)Cr alloys irradiated in the ATR-A1 experiment to ≈4-5 dpa confirmed their susceptibility to hardening and embrittlement during low-temperature neutron irradiation. Yield strength increased approximately three-fold to ≈800-900 MPa, whereas uniform elongation decreased to ≈1% or less. The impact properties of these materials were also degraded significantly.

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