



## Experience in irradiation testing of low-activation structural materials in fast reactor BOR-60

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### Abstract

A Russia/US collaborative irradiation experiment on low-activation structural materials, mainly vanadium alloys, was recently completed in the BOR-60 reactor at the Research Institute of Atomic Reactors (RIAR) in Russia. The experiment, designated Fusion-1, was designed to investigate the effects of neutron-induced displacement damage on the microstructure and mechanical properties of these materials at low temperatures, 300–360°C. For purpose of heat transfer and impurity control, the specimens were lithium-bonded to the capsule. After reaching the goal damage dose of 20 displacements per atom (dpa) in approximately 1 year of irradiation, the capsule was discharged and disassembled in a hot cell at RIAR. All specimens were retrieved from the capsule. This paper summarizes the experiment, covering capsule construction, lithium purification and filling, irradiation, disassembly and specimen retrieval, and calculated results of specimen temperatures and damage dose. © 1998 Elsevier Science B.V. All rights reserved.

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### 1. Introduction

Vanadium-base alloys containing Ti and Cr are strong candidates for fusion structural applications because of their good high-temperature properties and low neutron-induced activation. A preferred alloy composition has been V-(4–5)Cr-(4–5)Ti, based on balanced consideration of strength, ductility, weldability and radiation resistance [1–9]. Two large heats, both with a nominal composition of V-4Cr-4Ti, were recently produced in the US: a 0.5-ton heat for basic research and a 1.2-ton heat to be used in making radiative divertor components for the DIII-D tokamak.

Most of the past neutron damage studies on V-Ti-Cr alloys in the US were performed in the fast reactors

FFTF and EBR-II and in the temperature regime of 400–600°C [10–12]. Temperatures substantially below 400°C were unattainable in these two reactors because of the high sodium inlet temperatures, ~360–370°C. In these >400°C irradiations, many of the alloys exhibited good radiation resistance even after exposure to high fluence.

Extending the irradiation data base to lower temperatures, however, is important because such temperatures are anticipated in at least a portion of the fusion reactor operation. Moreover, at lower temperatures, radiation hardening and embrittlement in vanadium alloys are expected to be more significant than at higher temperatures.

While it is feasible to conduct low-temperature irradiation tests in water-cooled mixed-spectrum reactors, and some are being done, there are notable drawbacks. The most significant are the atypical transmutation reactions, such as V(n, $\gamma$ )Cr, which may significantly alter the alloy composition during irradiation. These trans-

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mutation reactions are predominantly caused by thermal neutrons, which are largely absent in a fusion reactor environment [13,14]. BOR-60 is a fast reactor with a low sodium inlet temperature, 300–330°C. It was therefore well suited for the Fusion-1 experiment, whose objective was to study the effects of radiation damage in materials at low temperatures.

**2. Test specimens and capsule construction**

The Fusion-1 capsule contained 614 miniature test specimens consisting of tensile, Charpy impact, compact tension, and Auger analysis specimens and transmission electron microscopy (TEM) disks.

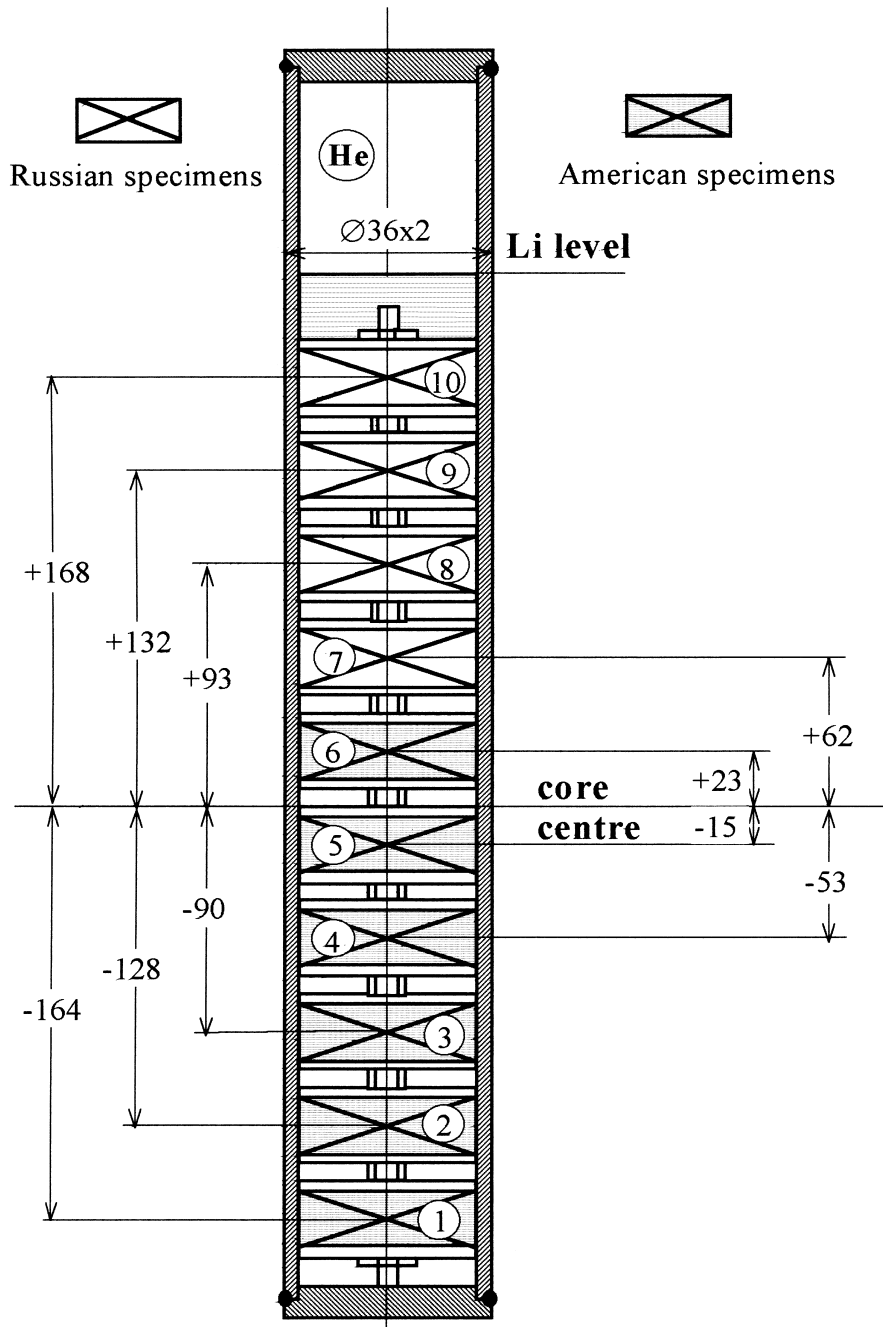


Fig. 1. Scheme of specimens disposition in the “FUSION-1” capsule.

The key variable for the US test matrix was alloy composition. A large number of the specimens were made from the 0.5-ton heat of V–4Cr–4Ti alloy; others were made from smaller laboratory heats with compositions ranging from V–3Cr–3Ti to V–6Cr–6Ti. Some of the laboratory heats had enhanced B or Si contents to study the effects of helium generation from the B(n, $\alpha$ )Li reactions or silicon addition. Because welding is an important issue with vanadium alloys, specimens made from gas-tungsten arc (GTA), electron-beam (EB), and laser weldments were included in the experiment. All specimens, except weld samples, were annealed before irradiation. The annealing parameters were 950°C, 1000°C, or 1050°C for 1 or 2 h. Some of the weldment specimens received postweld heat treatment, others did not.

The Russian specimens included pure V, binary V–(3–6)Cr, V–(3–6)Ti and V–(3–6)Fe alloys and ternary V–(4–6)Cr–(4–10)Ti alloys. For the V–4Cr–4Ti and V–5Cr–10Ti alloys, the specimens included both base metal and weldments from GTA and EB welds. Sheets from these alloys were produced by SSC RF RIIM.

The Fusion-1 capsule was a sealed stainless steel tube of 36 mm OD, 32 mm ID, and 508 mm length. The specimens were arranged in 10 tiers in the capsule: the lower six for the US and the upper four for Russia, as illustrated in Fig. 1. Each tier was about 30 mm high and separated from the others by stainless steel disks.

After the specimens were loaded, the capsule preassembly was shipped to the Institute of Physics and Power Engineering in Russia for lithium filling and final closure welding. To minimize tritium generation during irradiation, depleted lithium was used. Before the filling, the lithium was purified with the apparatus shown in Fig. 2. The purification process consisted of filtration with a 5–10  $\mu$ m stainless steel filter followed by gettering with hot zirconium foils at  $\sim$ 600°C for  $\sim$ 6 h. While this process did not further reduce the already-low carbon impurity content ( $\sim$ 30 ppm), it markedly decreased the nitrogen content from 200 to  $<$ 3 ppm. The purified lithium was loaded into the capsule in a stand at  $\sim$ 250°C under a vacuum of  $\sim$ 10<sup>–4</sup> Torr (Fig. 3). To reduce the likelihood of cavitation, a mechanical vibrator was used to shake the capsule during the lithium filling. Afterwards, the capsule was cooled to room temperature and back-filled with argon. The assembled capsule was radiographed and all specimens were found to be at their proper positions.

To achieve the desired low temperatures for the specimens, a modified hex can was used for the Fusion-1 experiment. This can contained an intermediate tube separating the centrally located capsule and the outer hex can: the inner annulus was the flow channel for the sodium coolant; the outer annulus, with a closed top, was made into a dead space to trap argon, the reactor cover gas. The stagnant argon gas created an insulating

gap between the cooler Fusion-1 capsule and the hotter neighboring fuel subassemblies. Because of hardware limitations, there were no thermocouples in the vehicle to monitor the specimen temperatures.

### 3. Irradiation conditions

The Fusion-1 capsule was irradiated in the G-23 cell, located in the fifth row, 196 mm from the BOR-60 core center. The irradiation started on 13 July 1995 and encompassed two operating campaigns: the 61st summer (13 July–12 November 1995) and 62nd winter (26 December 1995–7 June 1996). The histories of reactor power and sodium inlet temperature during these two campaigns are summarized in Fig. 4.

In both operating campaigns, reactor power and sodium inlet temperature were allowed to vary from time to time to comply with power output demands. The highest specimen temperatures occurred in the winter campaign when both the reactor power and sodium inlet temperature were high,  $\sim$ 50 MW and 330°C, respectively. The lowest specimen temperatures, conversely, occurred in the summer campaign when both the reactor power and inlet temperature were low,  $\sim$ 40 MW and 300°C, respectively.

The time-average irradiation temperature of specimens was calculated as middle-weighted value in a dependence of temperature and time on each of temperature-power level exceeding 100 h. In Fig. 5 it is lower trace. The upper one is height dependence of the specimen temperatures for the most strenuous reactor operation regime, typical for winter time: power level is 50 MW, sodium flowrate is 900 m<sup>3</sup>/h, sodium inlet temperature is 330°C, radiation specific heating is 5 W/g. At any axial position in the capsule, the variation of specimen temperature from the mean due to the power and coolant temperature changes was approximately  $\pm$ 16°C.

Sodium inlet temperature of the BOR-60 reactor may be hardly made below than 330°C at 50 MW power, but at 40 and 30 MW it may be regulated in wide limits from 270°C to 350°C. So, if the special efforts will be made then the specimens average temperature in such a capsule may be lowered up to 295–300°C.

Neutron spectrum and flux data required for the calculation of displacement damages and transmutations were obtained from two sources: a prior in-depth spectrometry study of a sibling cell D23 and an analysis of flux monitors incorporated in the Fusion-1 experiment. From these two analyses, a 100-group flux spectrum was generated for the Fusion-1 experiment. At the peak location near the core axial midplane, the predicted fast ( $E > 0.1$  MeV) and total neutron flux were 1.8 and  $2.3 \times 10^{15}$  n/cm<sup>2</sup> s, respectively, at a reactor power of 60 MW [15]. The error for the fast flux was estimated to be

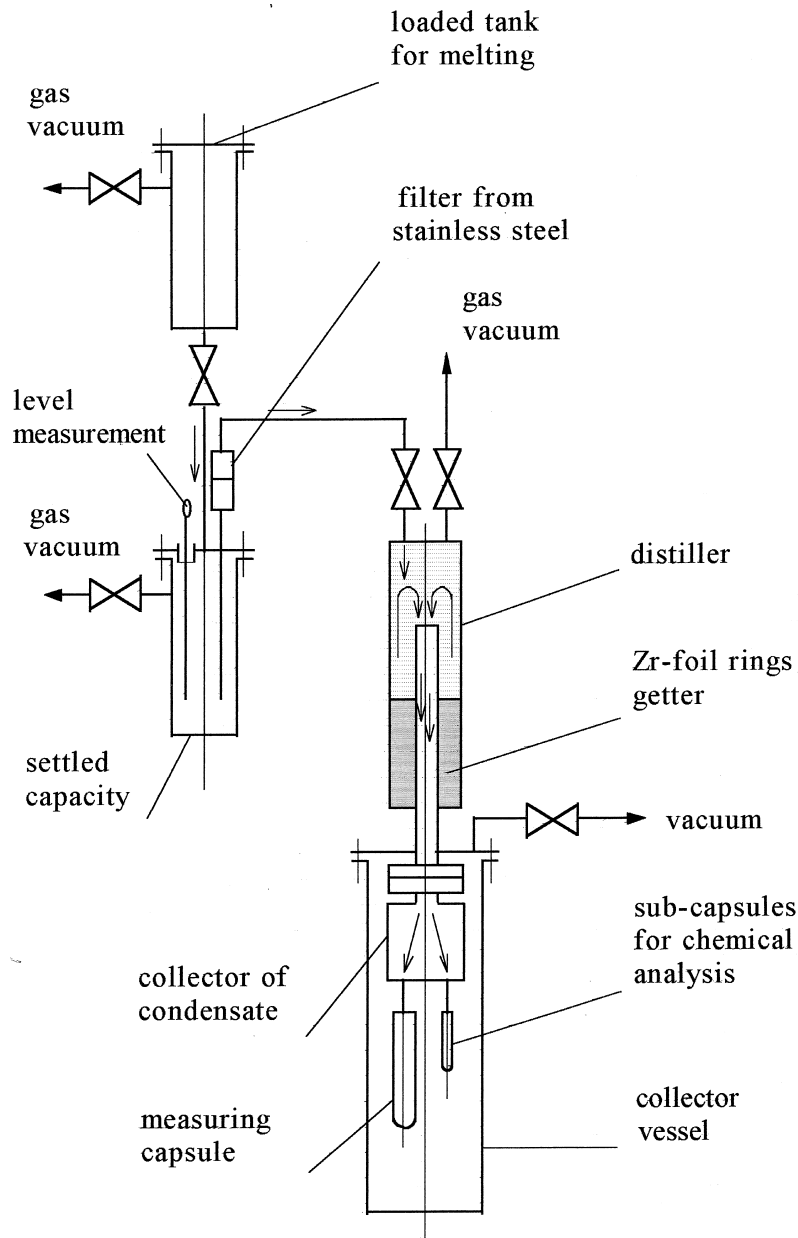


Fig. 2. Principal scheme of stand for Li distillation.

$\pm 15\%$ . Fusion-1's G23 cell is a valid sibling to the D23 cell because of the identical distance to the core center and the similarity of the surrounding core loading.

The flux monitors included in the Fusion-1 experiment were small disks (1.0 mm diam  $\times$  0.1 mm thick) of natural Nb, natural Fe, and enriched  $^{63}\text{Cu}$ . These flux monitors were measured after the irradiation with X-ray and gamma spectrometers at the RIAR. The resulting spectrum data confirmed good agreement with that of the D23 cell in neutron energies up to 1 MeV. Above

that, there were some noticeable deviations, attributed to the presence of lithium in the Fusion-1 capsule.

The axial flux profile in Fusion-1 was determined with the TRIGEX code in conjunction with results of activation measurements previously made with stainless steel specimens in an assembly in the sibling D-23 cell. The resulting profile is shown in Fig. 6. Based on this profile and using the SPECTERD computer code [16], the radiation damage dose for specimens at different tiers was calculated; results are shown in Table 1.

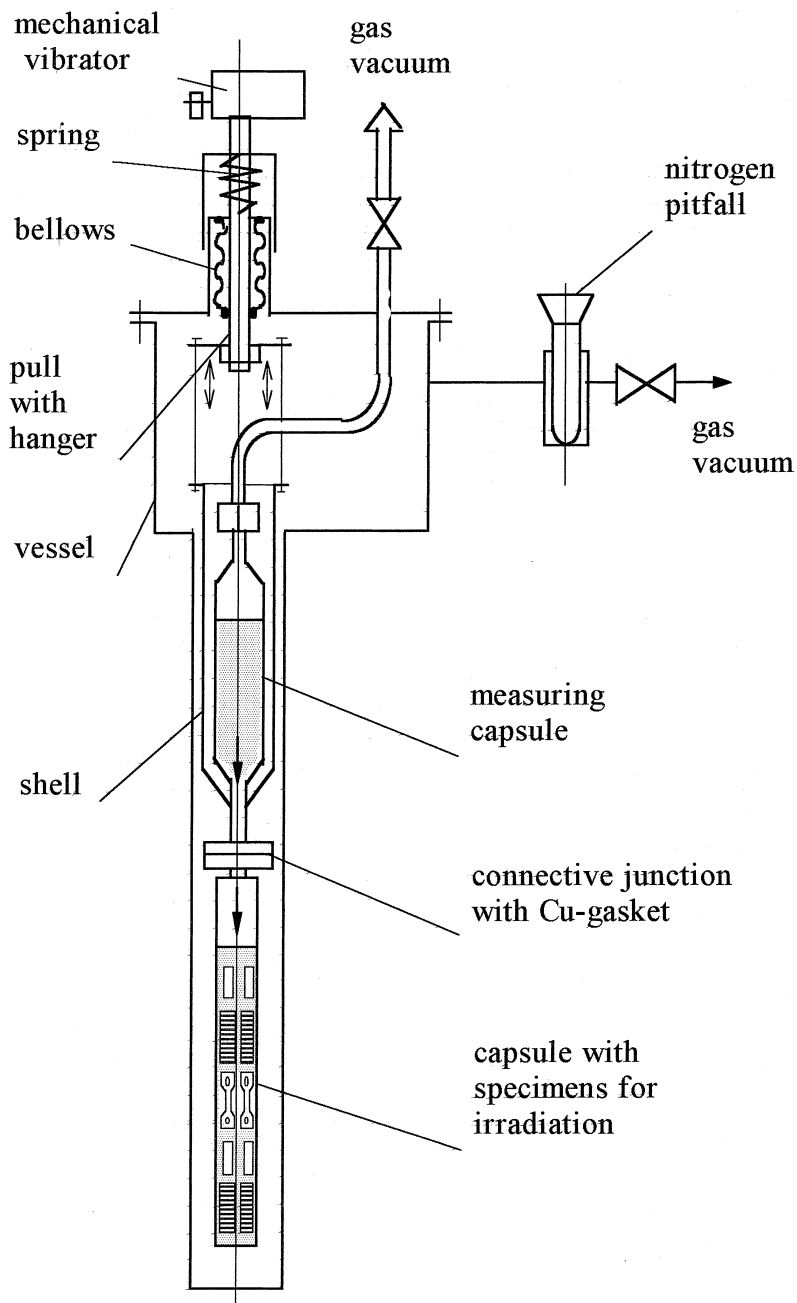


Fig. 3. Principal scheme of stand for capsule filling by Li.

To confirm that the irradiation would not cause excessive alteration of the compositions of the specimens, calculations on transmutation were performed. These calculations were conducted with the TRANS\_MU code [17] using input from ENDF/B-V file and ADL-3 Library [18] for natural isotope compositions, decay constants, and nuclear reaction cross-sections. The results, shown in Table 2, indicate that except for specimens

with the boron addition, the extent of transmutation was minimal.

#### 4. Capsule disassembly

Because the Fusion-1 capsule was lithium-bonded, a critical step in the capsule disassembly and specimen

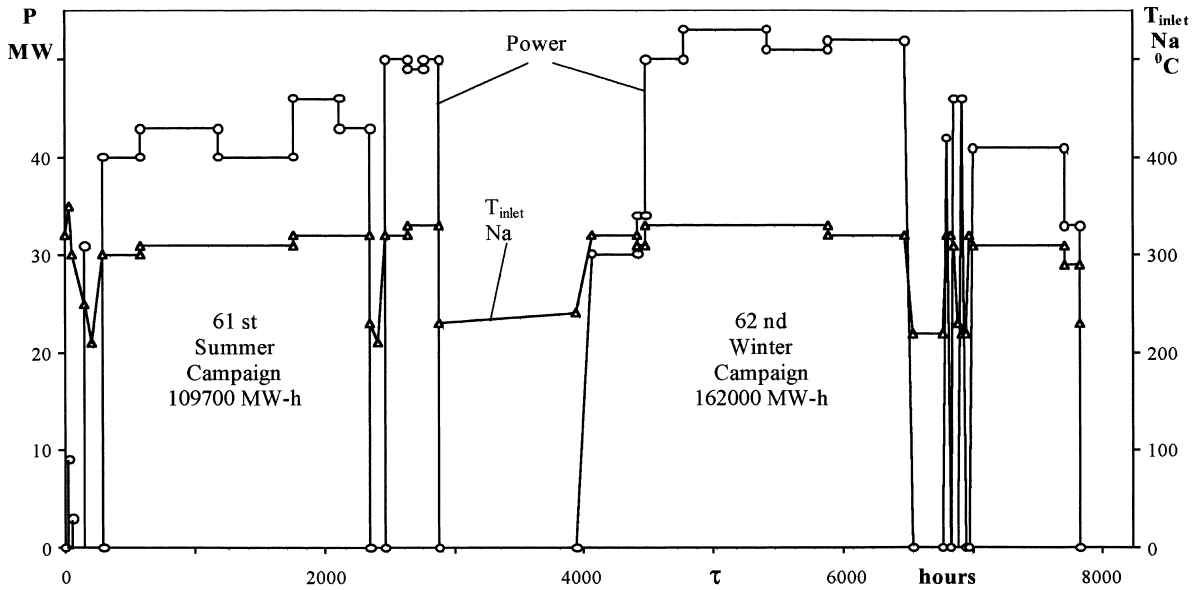


Fig. 4. Power and sodium inlet temperature during 61st summer and 62nd winter campaigns 1995–1996 years of the BOR-60 reactor.

retrieval was the removal of the lithium bond. Several possible methods were considered, and the selected method called for using heated mineral oil to melt and remove the lithium bond. To ascertain that exposure to oil at elevated temperature would not affect the properties of the specimens, extensive ex-cell confirmatory tests were conducted before disassembly. The results

show benign reaction to the oil exposure, specifically, no measurable up-take of interstitial impurities even after prolonged exposure.

The capsule was disassembled and the specimens were retrieved in an air cell at the RIAR. After the top and bottom end plugs of the capsule was removed with a low-speed saw, the capsule was immediately immersed in a bath of mineral oil. The oil was then heated to 250 $^{\circ}C$  to melt the lithium bond (melting point 181 $^{\circ}C$ ) to

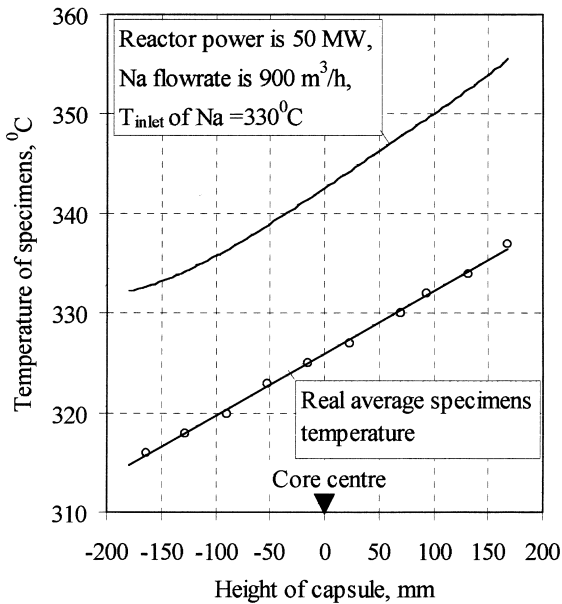


Fig. 5. Specimens temperature distribution on the “FUSION-1” capsule height.

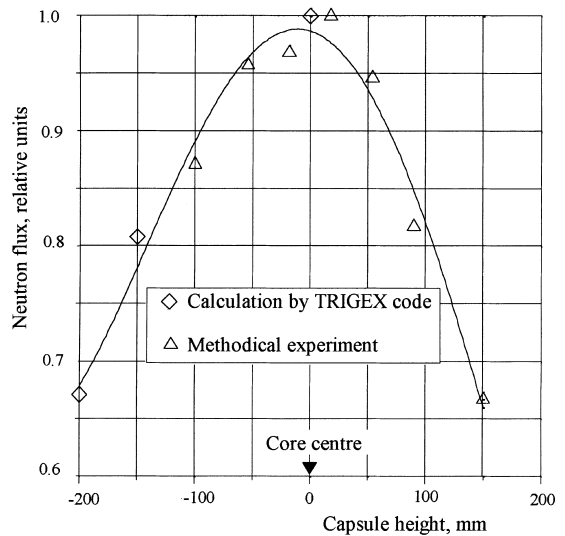


Fig. 6. Height distribution of neutron flux in the G-23 cell of the BOR-60 reactor.

Table 1

Neutron flux at the reactor power of 60 MW, neutron fluence and radiation damage dose for each of tiers with specimens in the "FUSION-1"

Tier no.	Z (mm)	Neutron flux ( $\times 10^{15}$ n/cm <sup>2</sup> · s)		Neutron fluence ( $\times 10^{22}$ n/cm <sup>2</sup> )		Radiation damage dose (for V-4Cr-4Ti) (dpa)
		$E > 0$	$E > 0.1$ MeV	$E > 0$	$E > 0.1$ MeV	
1	-164	1.725	1.354	2.8	2.2	15
2	-128	1.909	1.498	3.1	2.4	17
3	-90	2.093	1.643	3.4	2.7	18
4	-53	2.220	1.742	3.6	2.8	19
5	-15	2.272	1.783	3.7	2.9	20
6	+23	2.236	1.754	3.6	2.9	19
7	+62	2.105	1.652	3.4	2.7	18
8	+93	1.932	1.516	3.2	2.5	17
9	+132	1.656	1.300	2.7	2.1	14
10	+168	1.366	1.072	2.2	1.7	12

facilitate the se-paration of the specimens from the lithium. The retrieved specimens from the oil bath were rinsed in alcohol at room temperature to remove the residual oil and lithium and other possible surface contaminants. The completing operation consisted of the specimens cleaning from  $\alpha$ -contamination in the ultrasonic bath with alcohol. All test specimens in the Fusion-1 experiment were successfully retrieved in this manner.

Table 2

Most substantial transmutants accumulation rate in G-23 cell of fifth row of the BOR-60 reactor

Matrix element or alloy	Daughter element	Accumulation rate (appm/dpa)
V-4Cr-4Ti	Chromium	20
Vanadium	Chromium	22
Chromium	Vanadium	1
	Hydrogen	1.4
Titanium	Hydrogen	1.4
99,84 <sup>7</sup> Li+	Tritium	11.1
+0,16 <sup>6</sup> Li	Helium-4	11.4
Carbon	Beryllium	0.4
	Helium	0.6
	Boron	40
Nitrogen	Carbon	39
	Hydrogen	39
	Helium	40
Oxygen	Carbon	3.7
	Helium	3.7
Zirconium	Molybdenum	1.8
Boron of natur. isotop. composition	Lithium	536
	Beryllium	0.8
	Helium	538
	Magnesium	0.8
Silicon	Phosphorous	0.4
	Hydrogen	2.5
	Helium	0.8

## 5. Conclusions

The Fusion-1 experiment was a successful and comprehensive collaboration between Russia and the US on irradiation testing of low-activation structural materials. More than 600 test specimens of mostly vanadium-base alloys were irradiated. Both the damage dose (20 dpa) and irradiation temperature (300–360°C) targets were met.

With the dwindling number of fast test reactors worldwide, the successful completion of the Fusion-1 experiment attested to the value and potential of BOR-60 for the fusion community. The main advantages of BOR-60 over mixed-spectrum reactors are its higher dose rate and absence of thermal neutrons that could cause atypical transmutation in the test materials. For future experiments in BOR-60, more tightly controlled reactor power and coolant temperature could be implemented to mitigate the specimen temperature fluctuation experienced in the Fusion-1 experiment. Higher-temperature experiments could be conducted by incorporating gas gaps in the capsule to regulate heat transfer.

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## References

- [1] D.L. Smith, H.M. Chung, B.A. Loomis, H.-C. Tsai, J. Nucl. Mater. 233–237 (1996) 356.
- [2] H.M. Chung, L. Nowicki, D. Busch, D.L. Smith, Fusion Reactor Materials Semiannual Progress Report, DOE/ER-0313/19, 1996, p. 17.

- [3] A.N. Gubbi, A.F. Rowcliffe, D.J. Alexander, M.L. Grossbeck, W.S. Eatherly, L.T. Gibson, *ibid* p. 37.
- [4] A.N. Gubbi, A.F. Rowcliffe, *J. Nucl. Mater.* 233–237 (1996) 497.
- [5] H.M. Chung, R.V. Strain, H.-C. Tsai, J.-H. Park, D.L. Smith, *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/20, 1996, p. 55.
- [6] A.N. Gubbi, A.F. Rowcliffe, W.S. Eatherly, L.T. Gibson, *ibid* p. 67.
- [7] J.P. Smith, W.R. Johnson, R.D. Stambaugh, P.W. Trester, D.L. Smith, E.E. Bloom, *J. Nucl. Mater.* 233–237 (1996) 421.
- [8] B.G. Gieseke, C.O. Stevens, M.L. Grossbeck, *J. Nucl. Mater.* 233–237 (1996) 488.
- [9] G.R. Odette, G.E. Lucas, E. Donahue, J.W. Shekherd, *ibid* p. 502.
- [10] B.A. Loomis, L.J. Nowicki, D.L. Smith, *J. Nucl. Mater.* 212–215 (1994) 790.
- [11] B.A. Loomis, H.M. Chung, L.J. Nowicki, D.L. Smith, *ibid* p. 799.
- [12] H.M. Chung, B.A. Loomis, D.L. Smith, *J. Nucl. Mater.* 233–237 (1996) 466.
- [13] H. Tsai, R.V. Strain, I. Gomes, A.G. Hins, D.L. Smith, *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/18, 1995, p. 81.
- [14] H.M. Chung, L. Nowicki, D.L. Smith, *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/20, 1996, p. 84.
- [15] A.I. Tellin et al. *Experimental Investigation of Space-Energetical Neutron Distribution in the BOR-60 Reactor*, Reprint of RIAR-1 (853), Dimitrovgrad, 1996.
- [16] L.R. Greenwood, R.K. Smither, *SPECTERD: Neutron Damage Calculations for Materials Irradiations*, PNL/FPP/TM-197, Richland, 1985.
- [17] G.A. Shimansky, *Algorithm of the Transmutations Calculation with the Complex Errors Checking*, VANT, Nuclear Constants series, issue 2, Dimitrovgrad, 1995, p. 137.
- [18] O. Grudzevich, A.V. Zelensky, A.V. Ignatiuk, A.B. Pashchenko, *Atomic Energy* 76 (2) (1994) 124.