ROOM-TEMPERATURE FRACTURE IN V-(4-5)CR-(4-5)TI TENSILE SPECIMENS IRRADIATED IN FUSION-1 BOR-60 EXPERIMENT*

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SUMMARY

Specimens of V-(4-5)Cr-(4-5)Ti alloys were irradiated to ≈18 dpa at 320°C in the Fusion-1 capsule inserted into the BOR-60 reactor. Tensile tests at 23°C indicated dramatic yield strength increase (>300%), lack of work hardening, and minimal (<1%) total elongations. SEM analysis of fracture and side surfaces were conducted to determine reduction in area and the mode of fracture. The reduction of area was negligible. All but one specimen failed by a combination of ductile shear deformation and cleavage crack growth. Transgranular cleavage cracks were initiated by stress concentrations at the tips of the shear bands. In side-view observations, evidence was found of slip bands typically associated with dislocation channeling. No differences due to pre-irradiation heat treatment and heat-to-heat composition variations were detected. The only deviation from this behavior was found in V-4Cr-4Ti-B alloy, which failed in the grip portion by complete cleavage cracking.

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1 tive of this study was to determine the room temperature tensile properties and fracture r ensile specimens of V-(4-5)Cr-(4-5)Ti alloys irradiated at ≈320°C in the BOR-60 reactor.

I UCTION

1 emperature irradiation performance data of V-4C-4Ti alloys are needed to establish g on the low-temperature limit of fusion reactors operation. These temperatures are also r transient operation of the reactors in start-up, shut-down, and idle modes. Recently, a s irradiation experiments was performed in various irradiation facilities to evaluate the ii performance of V-4Cr-4Ti alloys at low doses (0.1-6 dpa) [1-5]. The present irradiation e it was designed to complement the low-dose data with information on the irradiation performance of V-(4-6)Cr-(4-6)Ti alloys in the temperature range of 310-350°C and at neutron fluences of 15 to 20 dpa. The irradiation was a part of the Russian Federation (RF)-U.S. collaborative Fusion-1 experiment in BOR-60 reactor. Specimens were delivered to Argonne National Laboratory and testing was initiated in this reporting period. The six fractured tensile specimens tested at 23°C were provided for our examination by the ANL Vanadium Alloy Development Program.

In this study, three specimens of the 500kg heat (#832665) of V-4Cr-4Ti alloy after various annealing treatments (1 h at 1000°C, 2 h at 1000°C, and 2 h at 950°C) were evaluated. The three additional specimens were V-4Cr-4Ti-250wppm^{10}B (heat QN74) and V-5Cr-5Ti (heat T87) U.S. alloys, and the V-4Cr-4Ti alloy manufactured in the RF (heat VX8). The goals of the present work were to evaluate the mode of fracture and establish reasons for the dramatic yield strength increase, loss of macroscopic work hardening, and lack of elongation. The results of the tensile tests on these specimens are reported in a separate submission [6]. Here, we report on the findings of scanning electron microscopy (SEM) analysis of the fracture and side surfaces of the tensile specimens tested at 23°C.

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*Work supported by U.S. Department of Energy Office of Fusion Energy Research and Argonne National Laboratory Division of Educational Programs, under Contract W-31-109-Eng-38.

EXPERIMENTAL PROCEDURES

The ANL tensile specimens included in the Fusion-1 experiment were machined from sheet stock to SS-3 geometry specifications (gauge dimensions: 7.62 mm length, 1.52 mm width, 0.76 mm thickness). The long direction of the specimens was parallel to the final rolling direction of the sheets. Two variables were investigated in the Fusion-1 experiment. First was the alloy's composition effects (listed in Table 1) and the second was the preirradiation heat treatment (listed in Table 2). The purpose of boron addition to the heat QN74 was to evaluate the effects of He generated by B(n,α)Li reactions. The ANL specimens were irradiated at ≈320°C to ≈18 dpa. The temperature varied by ±16°C during the irradiation period from July 1995 through June 1996 due to changes in sodium coolant inlet temperature. The coolant inlet temperature was dictated by the reactor's seasonal output demands. BOR-60, a fast breeder reactor, is used to generate electric power for the city of Dimitrovgrad, and in summer the power demands are lower. Following irradiation, the capsule was disassembled at the Research Institute of Atomic Reactors (RIAR). During disassembly specimens were exposed to mineral oil at 250°C followed by alcohol rinses at room temperature to dissolve the residual Li. Cleaned specimens were shipped to the USA.

Table 1. Nominal compositions of the four investigated alloys

Heat ID	Nominal Composition (wt.%)	Major Impurities (wppm)			
		O	N	C	Si
832665	V-3.8Cr-3.9Ti	310	85	80	780
T87	V-5Cr-5Ti	380	90	110	550
QN74 [a]	V-4Cr-4.1Ti-B	350	220	200	870
VX8 [b]	V-3.7Cr-3.9Ti	350	70	300	500

[a] Contains 250 appm of ^{10}B.

[b] *Manufactured in Russia; contains (in wppm) 1120 Al, 280 Fe, 500 Co, 270 Mo, 1280 Nb, and 19 Zr.*

Table 2. Preirradiation heat treatments of investigated specimens

Specimen ID	Heat ID	Pre-Irradiation Annealing
BL71-2	832665	1 h at 1000°C
BL71-2H-2	832665	2 h at 1000°C
BL71-B	832665	2 h at 950°C
BL70-2	QN74	1 h at 1000°C
BL72-2	T87	1 h at 1000°C
BL69-2	VX8	1 h at 1000°C

Details of specimen handling and the testing procedures are given in the accompanying report [6]. The tensile tests were conducted at 23°C and a 0.0011 s^{-1} strain rate. In all tests, yield strengths were dramatically higher (≈300%) than in an unirradiated control specimens (≈350-400 MPa at 23°C). Slip bands formed and led to minimal work hardening and limited total elongations in specimens from heats 832665, T87 and VX8. The only specimen with appreciable macroscopic plastic deformation was the VX8 alloy. The specimen of QN74 failed in the grip area with no plastic deformation. The results of the tensile tests are given in Table 3.

After tensile testing, the specimens were examined by SEM in the ANL Alpha-Gamma Hot Cell Facility. For the reduction-in-area determinations, specimens were mounted in a vertical clip holder and oriented with cross-section parallel to the image plane. For the side-view observations, specimens were tilted ≈45°. Photomicrographs were acquired on Type 55 Polaroid film producing both a positive print and a negative. The negatives were digitized by a Leafscan45 prepress negative scanner. The areas of deformed cross-sections were measured on digitized images. Figures for this report were prepared with Adobe PhotoShop software, and photographic quality output was generated by a Codonics NP-1600 die sublimation printer.

Table 3. Tensile data for ANL specimens irradiated in the Fusion-1 experiment in BOR-60

Specimen ID	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
BL71-2	1115	1120	0.3	0.4
BL71-2H-2	1100	1115	0.3	0.5
BL71-B	1120	1125	0.5	0.8
BL70-2 [a]	-	-	-	-
BL72-2	1145	1150	0.4	0.4
BL69-2	1135	1170	1.4	2.8

[a] Specimen failed within the grip portion at 727 MPa with no plastic deformation.

RESULTS

SEM Fractography

Low magnification (≈40X) head-on orientation fractographs of the five tensile specimens that fractured within the gauge-length portion are presented in Fig. 1(a-e). A perspective view of the QN74 specimen is shown in Fig. 1(f). Reduction-of-area values obtained from these photomicrographs are listed in Table 4; all of these values are negligible. The slight variations from the pretesting values in the cross-sectional areas measured from photomicrographs of the fractured specimens are within the experimental error arising from misalignment of the specimens (≈3%). In all cases, however, fracture occurred by a combination of ductile shear and transgranular brittle cracking. Percentages of the cross-sectional area showing cleavage fracture are also listed in Table 4. Ductile shear fracture predominated in the specimens of V-4Cr-4Ti alloys (832665 and VX8). The percentage of brittle area in the VX8 specimen was higher than in the BL71-2. The difference in fracture behavior of these two specimens becomes apparent in observations with a perspective view (Fig. 2). Shear fracture occurred in BL71-2, while the VX8 alloy fractured transversely after limited necking. In both specimens, cleavage cracks propagated transversely to the loading direction.

Higher-magnification fractographs were obtained to evaluate the direction of cleavage crack propagation. The transitional regions, where cracks transformed from ductile to brittle mode were also examined. Typical examples are shown in Fig. 3. In all five cases, the flow direction of the "river pattern" indicates that the cleavage cracks initiated at a tip of shear band and propagated toward the surface of the specimen. This crack propagation direction suggests that the cleavage cracks were initiated by stress concentrations at the tip of the dislocation pileups impinging on grain boundaries.

The specimen of QN74 alloy fractured within the grip portion, as shown in Fig. 1(f). Close examination of the fracture surface revealed that the cleavage crack initiated at an outer edge of the specimen and propagated toward the pinhole. The "river pattern" visible in Fig. 4 flows to the pinhole. The average plane of the fracture surface was perpendicular to the loading direction. However, the crack changed the local orientation from grain to grain, following randomly oriented cleavage planes. Intergranular cracks were not observed.

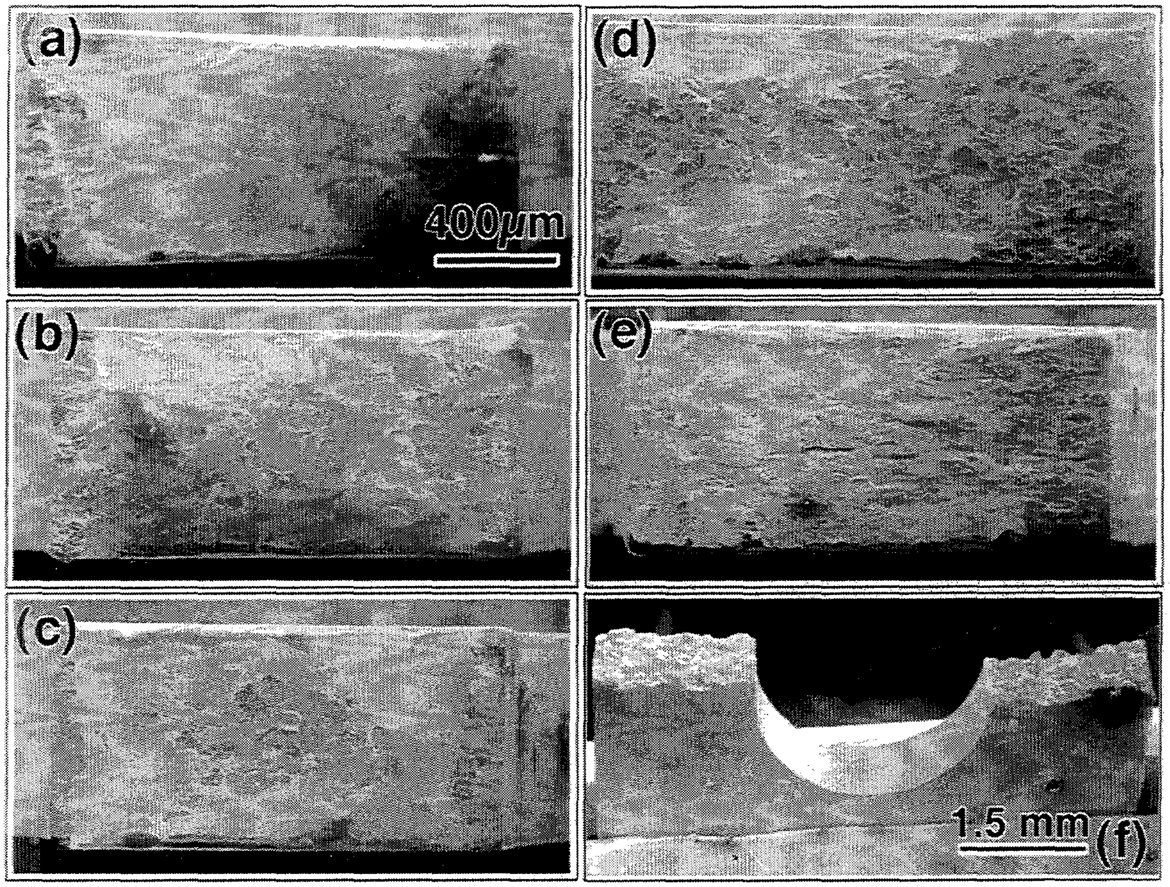

Fig. 1. SEM fractographs showing both ductile and brittle fracture areas of ANL tensile specimens irradiated in Fusion-1 experiment and tested at 23°C. Head-on views used to determine reduction in area: (a) BL71-2, (b) BL71-2H-2, (c) BL71-B, (d) BL72-2, (e) BL69-2, and (f) side view of grip portion of BL70-2 specimen.

Table 4. Reduction in cross-sectional area and fraction of brittle fracture surface in specimens of V-(4-5)-(4-5)Ti alloys irradiated in Fusion-1 capsule in BOR-60 reactor and tested at 23°C

Specimen ID	Cross-Sectional Area (mm^2)		Reduction	Percentage of
	Unirradiated	Post-irradiation	in Area (%)	Cleavage Fracture (%)
BL71-2	1.162	1.150	<< 3	15.83
BL71-2H-2	1.116	1.102	<< 3	65.78
BL71-B	1.185	1.178	<< 3	31.37
BL69-2	1.040	0.997	≈ 4	23.97
BL72-2	1.071	1.065	<< 3	72.05

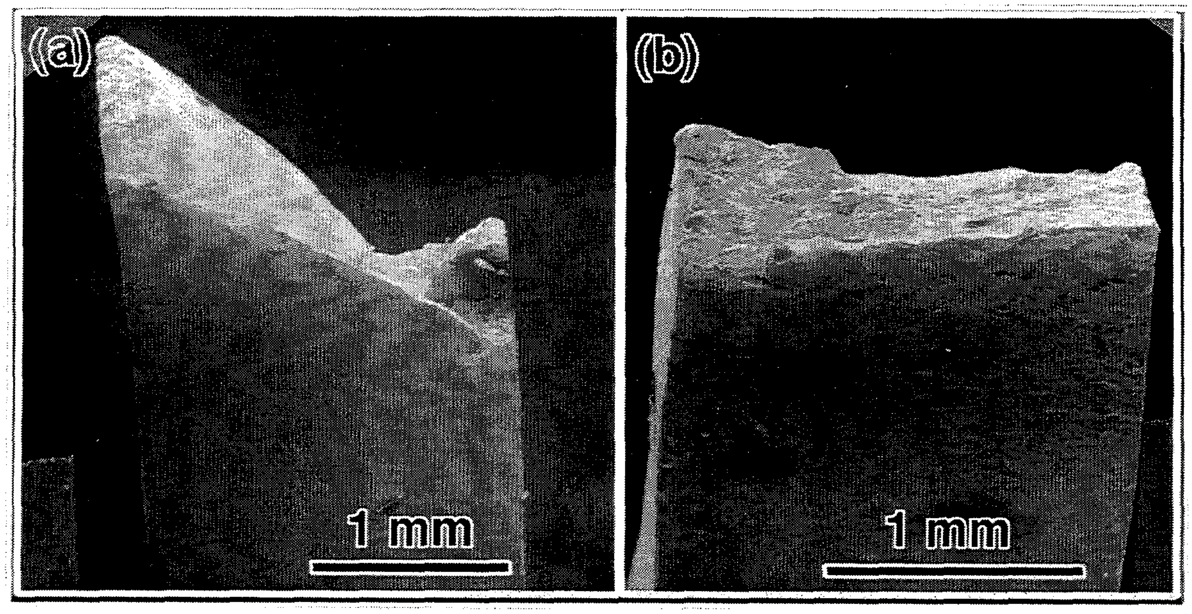

Fig. 2. Perspective views of specimens of V-4Cr-4Ti alloys with same preirradiation annealing schedule (1 h at 1000°C): (a) shear fracture in Heat 832665 (BL71-2) and (b) mixed-mode fracture in VX8 (BL69-2).

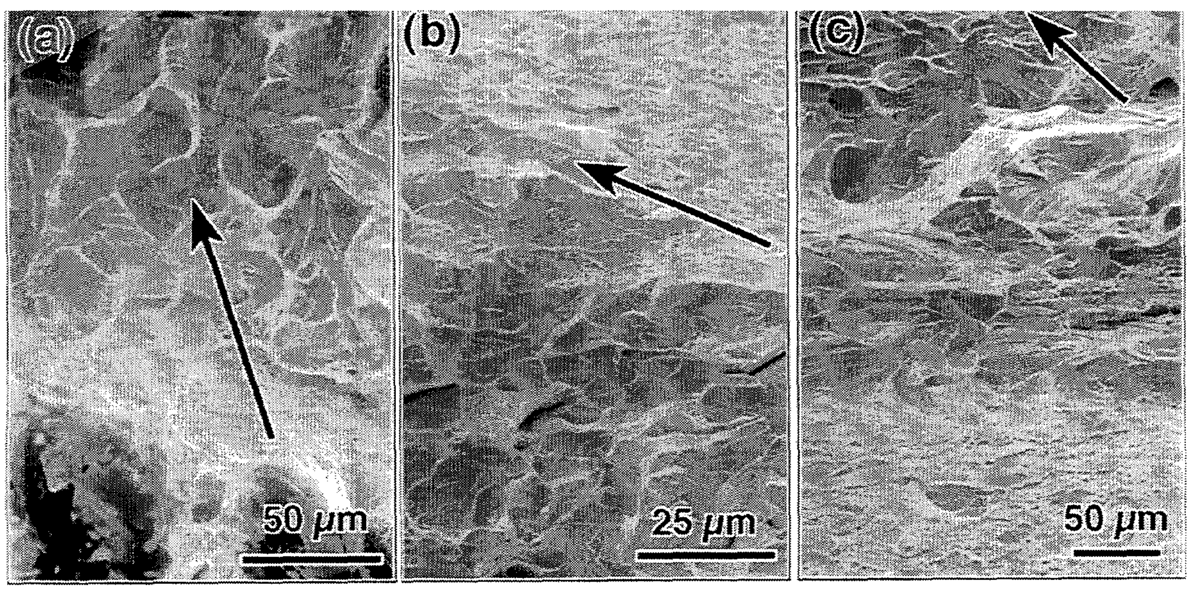

Fig. 3. SEM fractographs illustrating transformation from shear ductile to cleavage cracks: (a) BL71-2, (b)BL72-2, and (c) BL69-2. Arrows indicate directions of cleavage crack propagation.

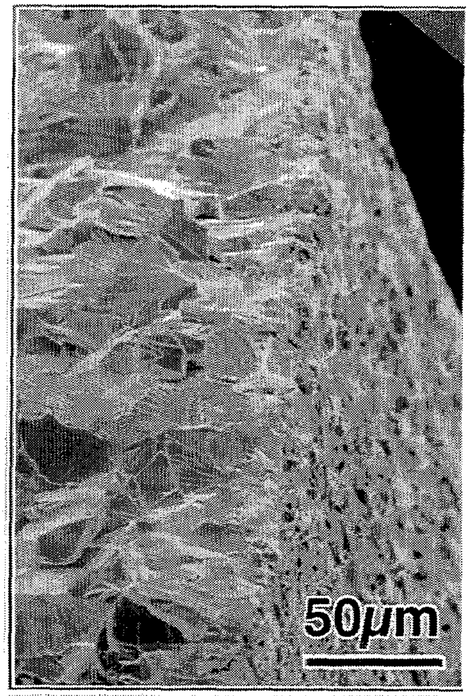

Fig. 4. View of inner surface of pinhole and fracture surface of QN74 specimen. Crack grew from outer specimen surface towards pinhole.

Side Surface Examinations

Side surfaces were examined to determine if slip bands characteristic of dislocation channeling formed during testing. They were observed in all five specimens that fractured in the gauge portion. Typical examples from the BL71-2H-2 and from the VX8 specimens are shown in Fig. 5. Short transverse cracks initiated at the surface were also found; they grew perpendicular to the direction of the applied tensile load, but did not extend far into the specimens. These cracks and slip bands were observed only in the necked regions.

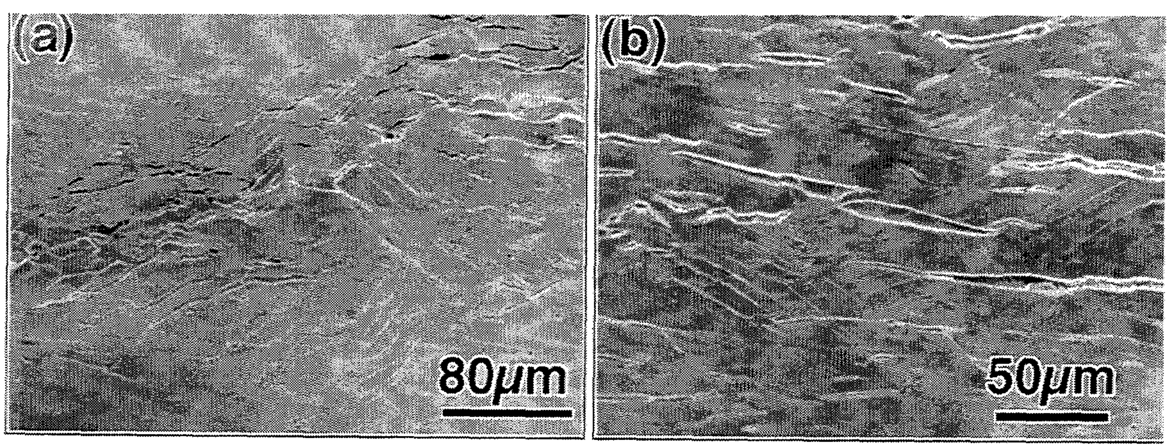

Fig. 5. Typical examples of surface steps indicating occurrence of dislocation channeling found on the side surfaces of the (a) heat 832665 - 2h anneal at 950°C, and (b) in VX8 specimen. The surface cracks also visible in these photomicrographs are perpendicular to tensile direction.

DISCUSSION

The occurrence of slip bands, shear bands, and shear cracks indicate that dislocation channels play a major role in the fracture of low-temperature-irradiated V-4Cr-4Ti alloys. Yield strengths observed in the present test are only slightly higher than those found in the earlier investigations of V-4Cr-4Ti alloy specimens irradiated to ≈6 dpa at 325°C in the HFR reactor [7], to ≈4 dpa at 390°C in the X530 experiment in the EBR-II reactor [8], and to ≈4 dpa at 280°C in the ATR-A1 experiment [9]. The same specimen geometry as in the present study was used in the EBR-II and ATR work. The HFR irradiation study used bar specimens with 4-mm-diameter gauge portions. All of these irradiations led to increased yield strength, lack of work hardening, and minimal elongations. In fractography evaluations of specimens from the X530 and ATR-A1 experiments, surface steps were found [10]. Dislocation channels were observed by TEM in the specimens irradiated at 390°C [11]. In the HFR study, a combination of cleavage and shear cracking was found [7]. Those data, combined with the results of the current study, indicate that irradiation of V-4Cr-4Ti below 400°C leads to detrimental hardening. They also indicate that dislocation channeling is not an effect of specimen geometry but rather an intrinsic response of the radiation-hardened alloy to the applied stress. From an engineering point of view, even the limited plastic deformation provided by the shear bands is preferred to complete brittle fracture. In additional papers, we identified the platelet precipitates formed in V-4Cr-4Ti during heavy ion and fast neutron irradiations at low temperatures (≤420°C). We also described their role in irradiation hardening [12, 13]. Further alloy design work to prevent irradiation-induced precipitate formation or to change the particle geometry will be necessary to improve ductility of V-4Cr-4Ti after low-temperature irradiation.

Heat-to-heat compositional variations did not play a major role in the response of the specimens to loading. Although the V-5Cr-5Ti alloy (T87) showed a considerably higher percentage of brittle fracture than did the V-4Cr-4Ti alloys, both ductile shear and brittle cracking contributed to its failure. Variations of annealing treatment of V-4Cr-4Ti (heat 832665) did not suppress the brittle cracking.

FUTURE WORK

Fractography study will be continued to evaluate the tensile specimens tested at irradiation temperatures. TEM investigations of cross-sections of irradiated and deformed specimens will also be conducted to determine effects of irradiation on the microstructure and the interaction mode of glide dislocations with radiation defects. The 3-mm discs irradiated in BOR-60 have not yet been delivered to ANL. Evaluations of these specimens will commence as soon as they arrive.

CONCLUSIONS

1. Both ductile-shear and transgranular cleavage fracture were observed in V-(4-5)Cr-(4-5)Ti alloys irradiated in the Fusion-1 capsule in the BOR-60 reactor after tensile testing at 23°C. The reductions of area were negligible except for that in the VX8 alloy which showed ≈4% reduction in area. A V-4Cr-4Ti-250appm^{10}B specimen fractured by cleavage within the grip portion with no significant plastic deformation in the gauge length. Intergranular fracture was not observed in any of the specimens.
2. Heat-to-heat compositional variations and preirradiation annealing treatments had no effects on the fracture behavior. In all specimens, slip localization by shear bands and dislocation channeling was observed. Cleavage cracks initiate at the shear bands and propagate toward the surfaces of the specimens.
3. Slip localization and dislocation channeling provide the only plastic deformation found in these specimens. Occurrence of any plastic flow in radiation-hardened V-(4-5)Cr-(4-5)Ti alloys with yield strengths ≥1000 MPa is noteworthy.

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