

## IMPACT PROPERTIES OF VANADIUM-BASE ALLOYS IRRADIATED AT <430°C\*

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### SUMMARY

Recent attention to vanadium-base alloys has focused on the effect of low-temperature (<430°C) neutron irradiation on the mechanical properties, especially the phenomena of loss of work-hardening capability under tensile loading and loss of dynamic toughness manifested by low impact energy and high ductile-brittle-transition temperature (DBTT). This paper summarizes results of an investigation of the low-temperature impact properties of V-5Ti, V-4Cr-4Ti, and V-3Ti-Si that were irradiated in several fission reactor experiments, i.e., FFTF-MOTA, EBR-II X-530, and ATR-A1. Irradiation performance of one production-scale and one laboratory heat of V-4Cr-4Ti and one laboratory heat of V-3Ti-Si was the focus of the investigation. Even among the same class of alloy, strong heat-to-heat variation was observed in low-temperature impact properties. A laboratory heat of V-4Cr-4Ti and V-3Ti-1Si exhibited good impact properties whereas a 500-kg heat of V-4Cr-4Ti exhibited unacceptably high DBTT. The strong heat-to-heat variation in impact properties of V-4Cr-4Ti indicates that fabrication procedures and minor impurities play important roles in the low-temperature irradiation performance of the alloys.

### OBJECTIVE

The objective of this research is to evaluate the effects of irradiation on the impact properties of candidate vanadium-base alloys.

### INTRODUCTION

Recent attention to vanadium alloys has focused on low-temperature (<430°C) irradiation performance of V-(4-5)Cr-(4-5)Ti, especially tensile and impact properties after irradiation at <430°C. From several irradiation experiments at 80-430°C, it has been reported that a large-scale (Heat ID #832665) and a laboratory (BL-47) heat of V-4Cr-4Ti<sup>1-5</sup> and a large-scale (BL-63) heat of V-5Cr-5Ti<sup>6</sup> exhibited low uniform elongation as a result of virtual loss of work-hardening capability, although significant susceptibility to loss of work-hardening capability has not been observed for irradiation temperatures  $\geq 500^\circ\text{C}$ . The large-scale heat of V-4Cr-4Ti (#832665) also exhibited severe embrittlement manifested by very low impact energy and high DBTT after irradiation either at 100-275°C in helium environment in the High Flux Beam Reactor (HFBR)<sup>1</sup> or at  $\approx 390^\circ\text{C}$  in lithium environment in EBR-II<sup>6</sup>. In contrast to this, laboratory heats of V-4Cr-4Ti (BL-47) and V-3Ti-1Si (BL-45) have been reported to exhibit excellent impact properties after a conventional irradiation (i.e., a non DHCE) at  $>430^\circ\text{C}$  in Li environment in FFTF.<sup>7</sup> In this work, impact properties were evaluated on V-5Ti, V-4Cr-4Ti, and V-3Ti-1Si alloys that were irradiated at <430°C in several conventional fission reactor experiments, i.e., FFTF-MOTA, EBR-II X-530, and ATR-A1. In some of the experiments, to investigate the impact properties of the latter laboratory heats under more severe conditions, coldworked Charpy impact specimens were irradiated at  $\leq 400^\circ\text{C}$  in lithium environment.

### MATERIALS AND TESTING PROCEDURES

The elemental composition of the alloys investigated in this study and other comparable alloys, determined prior to irradiation, is given in Table 1. Charpy impact specimens were machined from coldworked  $\approx 3.8$ -mm-thick plates and were inserted in the irradiation capsules either in annealed or coldworked state. Annealed specimens from the laboratory heat of V-4Cr-4Ti (BL-47) were annealed at  $\approx 1000^\circ\text{C}$  or  $\approx 1125^\circ\text{C}$  for 1 h in an ion-pumped vacuum system, whereas specimens from the laboratory heats of V-5Ti and V-3Ti-1Si were annealed at  $\approx 1050^\circ\text{C}$  for 1 h. Following irradiation, the Charpy impact specimens were retrieved from the capsules and cleaned ultrasonically in alcohol. Some of the brittle Charpy impact specimens of V-4Cr-4Ti Heat #832665, irradiated at  $\approx 390^\circ\text{C}$  in EBR-II X-530 experiment, were deliberately annealed at  $>430^\circ\text{C}$  to investigate the effect of high-temperature annealing on toughness recovery.

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Table 1. Chemical composition of vanadium alloys

Heat ID	Nominal Comp. (wt.%)	Impurity Concentration (wt. ppm)			
		O	N	C	Si
BL-50	1.0Ti	230	130	235	1050
BL-62	3.1Ti	320	86	109	660
BL-52	3.1Ti	210	310	300	500
BL-46	4.6Ti	305	53	85	160
BL-12	9.8Ti	1670	390	450	245
BL-15	17.7Ti	830	160	380	480
BL-10	7.2Cr-14.5Ti	1110	250	400	400
BL-24	13.5Cr-5.2Ti	1190	360	500	390
BL-40	10.9Cr-5.0Ti	470	80	90	270
BL-41	14.5Cr-5.0Ti	450	120	93	390
BL-43	9.2Cr-4.9Ti	230	31	100	340
BL-49	7.9Cr-5.7Ti	400	150	127	360
BL-63 <sup>a</sup>	4.6Cr-5.1Ti	440	28	73	310
BL-27	3.1Ti-0.25Si	210	310	310	2500
BL-45	2.5Ti-1Si	345	125	90	9900
QN74 <sup>b</sup>	4.0Cr-4.1Ti	480	79	54	350
BL-47	4.1Cr-4.3Ti	350	220	200	870
VX-8 <sup>c</sup>	3.73Cr-3.93Ti	350	70	300	500
832665 <sup>d</sup>	3.8Cr-3.9Ti	310	85	80	783

<sup>a</sup>80-kg heat fabricated with sponge Ti

<sup>b</sup>Contains ≈250 appm B<sup>10</sup>.

<sup>c</sup>100-kg heat, contains (in wppm) 1120 Al, 280 Fe, 500 Co, 270 Mo, 1280 Nb, and 19 Zr.

<sup>d</sup>500-kg heat produced in Teledyne Wha Chang Albany.

<sup>e</sup>All others 15- to 30-kg laboratory heats.

## IRRADIATION CONDITIONS

Details of the recent conventional irradiation experiments at <430°C (i.e., the FFTF-MOTA,<sup>5</sup> HFIR 200J and 400J,<sup>8</sup> EBR-II COBRA-1A2,<sup>9</sup> EBR-II X-530,<sup>10</sup> and ATR A1<sup>11, 12</sup> experiments) are summarized in Table 2. The conventional irradiation in the FFTF is referred to as non-DHCE experiment.

Table 2. Summary of irradiation experiments

Experiment ID	Subcapsule	Environment	Temperature (°C)	dpa	He/dpa Ratio
FFTF-nonDHCE	many	Li	427-600	14-46	-
FFTF-DHCE	many	Li	430-600	14-27	0.4-4.2
HFIR	200J	He	200	10	-
	400J	He	400	10	-
COBRA-1A2	V499	Li	395	36	-
	V495	Li	379	31	-
EBR-II X530	S8	Li	394	4	-
	S9	Li	390	4	-
ATR-A1	many	Li	138-285	4.7	-

## IMPACT PROPERTIES

Charpy impact specimens of the 500-kg heat (#832665) and the 30-kg heat (BL-47) of V-4Cr-4Ti and the 15-kg heats of V-3Ti-1Si (BL-45) and V-5Ti (BL-46) were irradiated at  $<400^{\circ}\text{C}$  in the EBR-II X-530 and ATR-A1 experiments. Orientation of the blunt-notched specimen is illustrated in Fig. 1, annealing history and irradiation parameters are summarized in Table 3.

Table 3. Summary of Charpy impact specimen orientation, annealing history, and irradiation parameters

Heat ID	Composition (wt.%)	Irradiation Experiment	Specimen Orientation	Blunt Notch Angle	Annealing	Irradiation Temperature ( $^{\circ}\text{C}$ )	Irradiation Damage (dpa)	Irradiation Environment
BL-46	4.6Ti	EBR-II-X530	L-S	30	coldworked	$\approx 390$	4	Li
BL-45	2.5Ti-1Si	EBR-II-X530	L-T	30	coldworked	$\approx 390$	4	Li
BL-47	4.1Cr-4.3Ti	EBR-II-X530	L-S or L-T	30 or 45	coldworked	$\approx 390$	4	Li
		ATR-A1	-	30 or 45	1 h at $1000^{\circ}\text{C}^{\text{a}}$	$\approx 202$ or $\approx 270$	4.7	Li
832665	3.8Cr-3.9Ti	EBR-II-X530	-	30 or 45	two-stage anneal <sup>b</sup>	$\approx 390$	4	Li
		ATR-A1	-	30	1 h at $1000^{\circ}\text{C}^{\text{a}}$	$\approx 200$ or $\approx 220$	4.7	Li

<sup>a</sup>Annealed in laboratory in ion-pumped high vacuum.

<sup>b</sup>Annealed in factory in diffusion-pumped vacuum for 2 h at a nominal temperature between  $1050$ - $1070^{\circ}\text{C}$ .

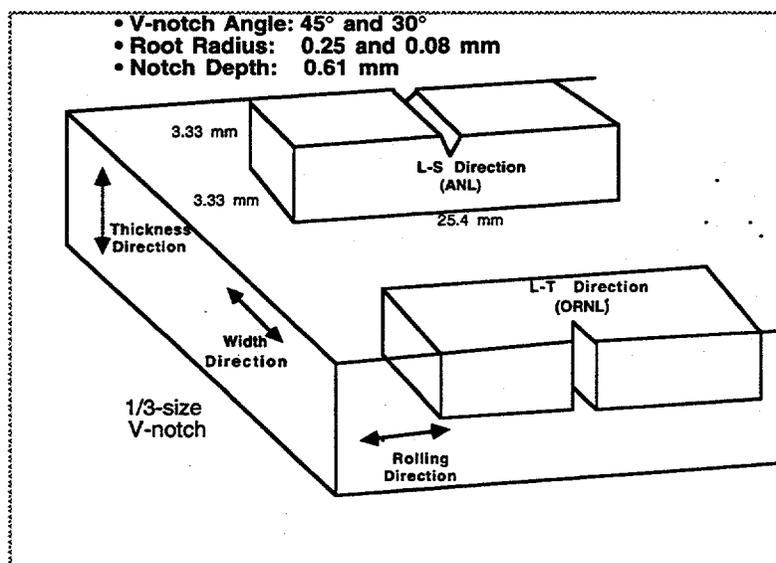


Fig. 1.

Schematic illustration of the geometry and orientation of the Charpy impact specimen

Results of impact testing of the coldworked laboratory heats of V-4Cr-4Ti (BL-47), V-3Ti-1Si (BL-45), and V-5Ti (BL-46) that were irradiated to  $\approx 4$  dpa in Li in the EBR-II X-530 experiment are shown in Figs. 2, 3, and 4, respectively. Similar results obtained from the annealed specimens of the same heats that were irradiated to  $\approx 34$  dpa at  $\approx 430^{\circ}\text{C}$  in non-DHCE in FFTF<sup>7</sup> are also shown in the figures for comparison. The effect of specimen orientation of the coldworked material of V-4Cr-4Ti Heat BL-47 was very significant. Coldworked specimens of the heat machined in L-S orientation exhibited good impact properties even after irradiation at  $\approx 390^{\circ}\text{C}$ ; in fact, the impact properties appear to be comparable to those of the annealed specimens irradiated at  $\approx 427^{\circ}\text{C}$  to 34 dpa in FFTF. Coldworked specimens of V-4Cr-4Ti Heat BL-47 that were machined in L-T orientation exhibited inherently inferior impact properties even before irradiation.

In contrast to the laboratory heat BL-47, the 500-kg heat (#832665) of V-4Cr-4Ti exhibited completely brittle characteristics after irradiation to  $\approx 4$  dpa at  $\approx 390^\circ\text{C}$  in the EBR-II X-530 experiment. This is shown in Fig. 5. Ductile behavior at room temperature could be restored only after post-irradiation reannealing at  $>650^\circ\text{C}$  for  $\approx 20$  min. in high vacuum (see Fig. 6). The 3.8-mm-thick plate of Heat #832665 was inadvertently annealed in factory (nominally at  $\approx 1050^\circ\text{C}$  for 2 h in relatively poor vacuum in a diffusion-oil-pumped system), and the Charpy specimens machined out of the plate were annealed further in high vacuum in a clean ion-pumped system in laboratory.

Charpy impact specimens of the 500-kg heat (#832665) and the 30-kg heat (BL-47) of V-4Cr-4Ti were irradiated at  $200$ - $270^\circ\text{C}$  in Li in the ATR-A1 experiment. Charpy specimens of both heats were machined out of coldworked plate, following annealing at  $1000^\circ\text{C}$  for 1 h in ion-pumped high vacuum. Results of impact testing of these specimens are shown in Fig. 7. Specimens from the laboratory heat BL-47 exhibited better impact properties than the 500-kg heat #832665, although the upper shelf energy was only  $\approx 5$  J and DBTT was as high as  $\approx 60^\circ\text{C}$ .

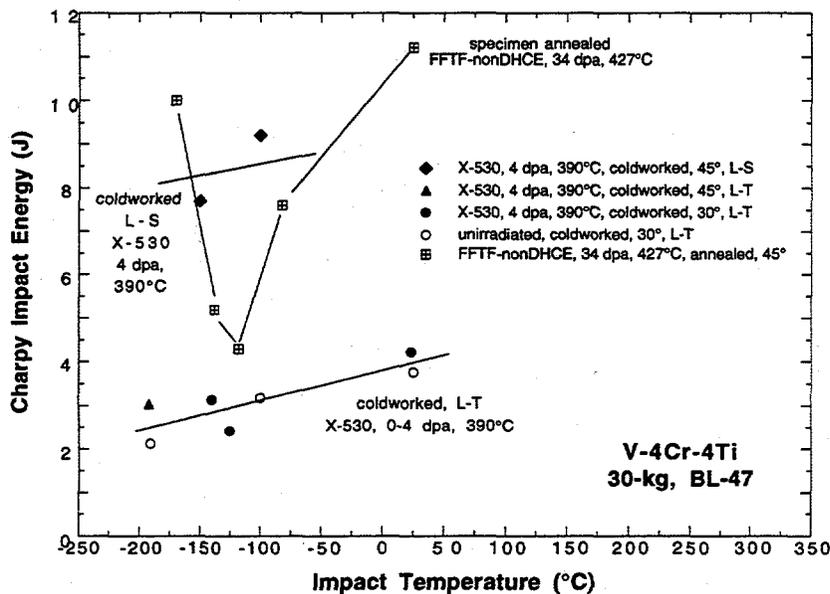


Fig. 2.

Impact properties of coldworked 30-kg heat of V-4Cr-4Ti (BL-47) irradiated to  $\approx 4$  dpa at  $\approx 390^\circ\text{C}$  in EBR-II X-530 experiment. Similar results from specimens annealed and irradiated to  $\approx 34$  dpa at  $\approx 427^\circ\text{C}$  in FFTF non-DHCE are also shown for comparison.

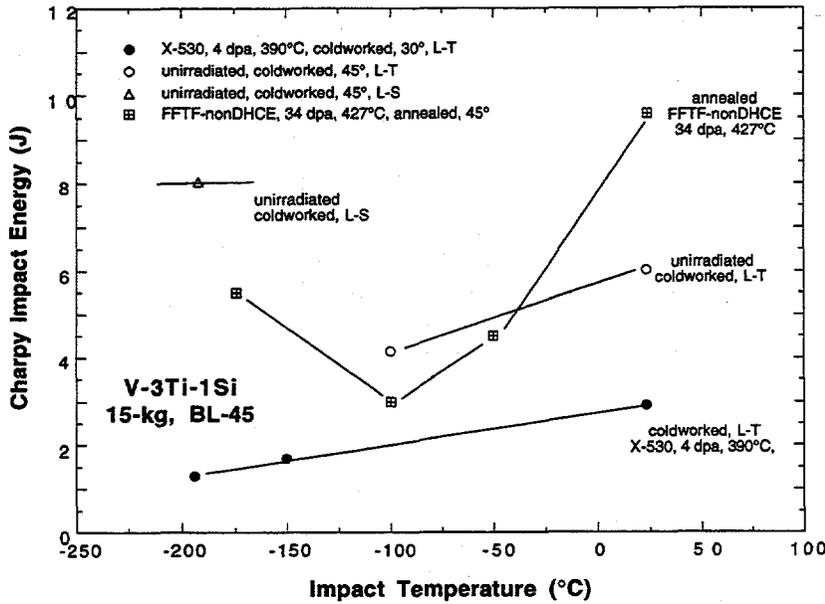


Fig. 3.

Impact properties of coldworked 15-kg heat of V-3Ti-1Si (BL-45) irradiated to  $\approx 4$  dpa at  $\approx 390^\circ\text{C}$  in EBR-II X-530 experiment. Similar results from specimens annealed and irradiated to  $\approx 28$  dpa at  $\approx 430^\circ\text{C}$  in FFTF non-DHCE are also shown for comparison.

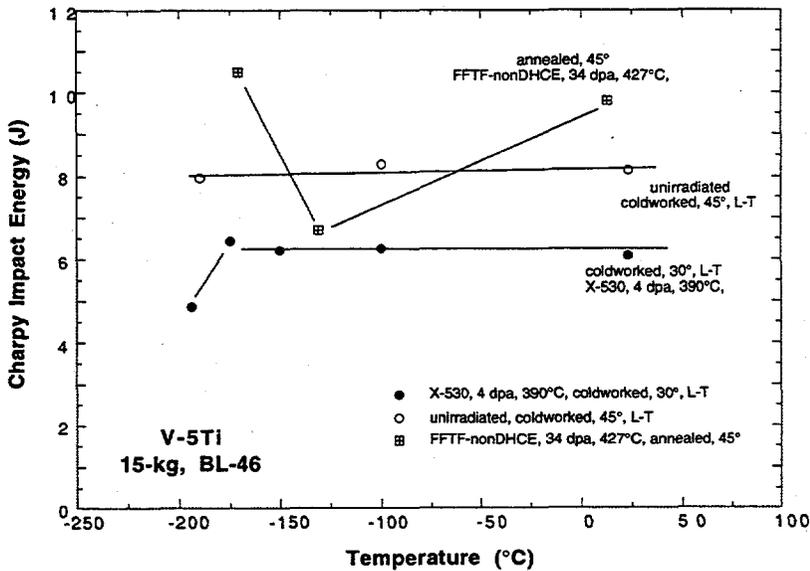


Fig. 4.

Impact properties of coldworked 15-kg heat of V-5Ti (BL-46) irradiated to  $\approx 4$  dpa at  $\approx 390^\circ\text{C}$  in EBR-II X-530 experiment. Similar results from specimens annealed and irradiated to  $\approx 34$  dpa at  $\approx 427^\circ\text{C}$  in FFTF non-DHCE are also shown for comparison.

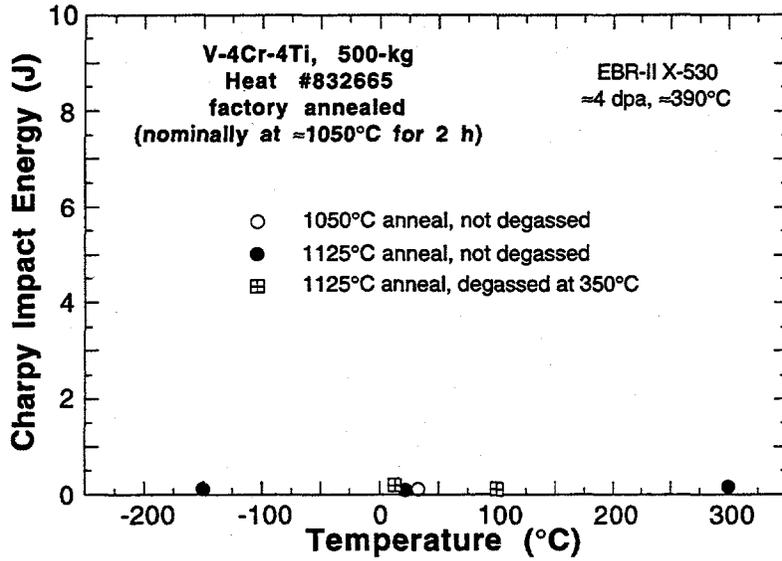


Fig. 5.

Impact properties of doubly annealed 500-kg heat of V-4Cr-4Ti (Heat #832665) irradiated to  $\approx 4$  dpa at  $\approx 390^{\circ}\text{C}$  in the EBR-II X-530 experiment, showing severe embrittlement.

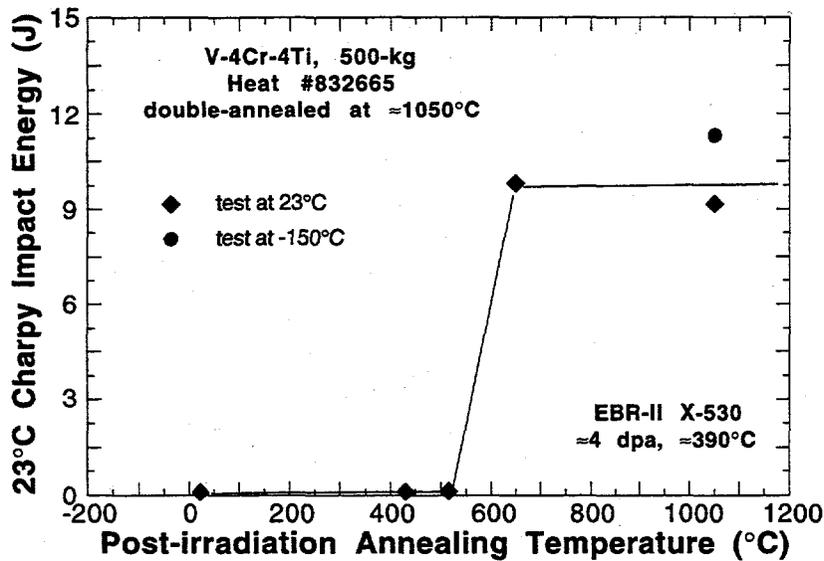


Fig. 6.

Room-temperature impact properties of 500-kg heat of V-4Cr-4Ti (Heat 832665) that were irradiated to  $\approx 4$  dpa at  $\approx 390^{\circ}\text{C}$  in the EBR-II X-530 experiment and reannealed for 20 min. in high vacuum.

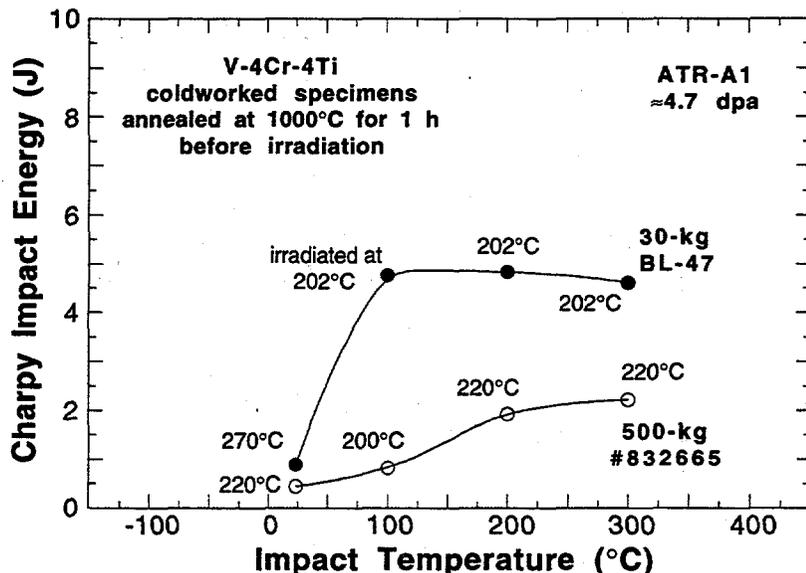


Fig. 7.

Impact properties of 500-kg and 30-kg heats of V-4Cr-4Ti irradiated to  $\approx 4.7$  dpa at  $\approx 200$ - $270^\circ\text{C}$  in Li in the ATR-A1 experiment. All specimens were annealed at  $1000^\circ\text{C}$  for 1 h in an ion pump before irradiation.

## CONCLUSIONS

1. The effect of specimen orientation on the impact properties of coldworked material of a 30-kg laboratory heat of V-4Cr-4Ti (BL-47) was significant. Coldworked specimens of the heat machined in L-S orientation exhibited superior impact properties after irradiation at  $390^\circ\text{C}$  to  $\approx 4$  dpa. High impact energies observed at temperatures as low as  $-100$  to  $-150^\circ\text{C}$  are comparable to those observed for annealed specimens after irradiation at  $\approx 427^\circ\text{C}$  to  $\approx 34$  dpa. Coldworked specimens machined in L-T orientation exhibited, however, inferior impact properties even before irradiation.
2. The 500-kg heat of V-4Cr-4Ti (#832665) exhibited brittle characteristics after irradiation to  $\approx 4$  dpa at  $\approx 390^\circ\text{C}$  in the EBR-II X-530 experiment. Ductile behavior of this heat at room temperature could be restored after post-irradiation reannealing at  $>650^\circ\text{C}$  for  $\approx 20$  min. in high vacuum.
3. Significant heat-to-heat variation in impact properties were observed for V-4Cr-4Ti after irradiation at  $200$ - $430^\circ\text{C}$ . One laboratory heat exhibited consistently better impact properties than the 500-kg heat. The exact mechanism of the strong heat-to-heat variation is, however, not understood.

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