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## TENSILE AND CHARPY IMPACT PROPERTIES OF IRRADIATED REDUCED-ACTIVATION FERRITIC STEELS

**REFERENCE:** Klueh, R. L. and Alexander, D. J., "Tensile and Charpy Impact Properties of Irradiated Reduced-Activation Ferritic Steels," Effects of Radiation on Materials: 18th International Symposium, ASTM STP 1325, R. K. Nanstad, M. L. Hamilton, F.A. Garner, and A.S. Kumar, Eds., American Society for Testing and Materials, 1997.

**ABSTRACT:** Tensile tests were conducted on eight reduced-activation Cr-W steels after irradiation to 15-17 and 26-29 dpa, and Charpy impact tests were conducted on the steels irradiated to 26-29 dpa. Irradiation was in the Fast Flux Test Facility at 365°C on steels containing 2.25-12% Cr, varying amounts of W, V, and Ta, and 0.1%C. Previously, tensile specimens were irradiated to 6-8 dpa and Charpy specimens to 6-8, 15-17, and 20-24 dpa. Tensile and Charpy specimens were also thermally aged to 20000 h at 365°C. Thermal aging had little effect on the tensile behavior or the ductile-brittle transition temperature (DBTT), but several steels showed a slight increase in the upper-shelf energy (USE). After  $\approx$ 7 dpa, the strength of the steels increased (hardened) and then remained relatively unchanged through 26-29 dpa (i.e., the strength saturated with fluence). Post-irradiation Charpy impact tests after 26-29 dpa showed that the loss of impact toughness, as measured by an increase in DBTT and a decrease in the USE, remained relatively unchanged from the values after 20-24 dpa, which had been relatively unchanged from the earlier irradiations. As before, the two 9Cr steels had the most irradiation resistance.

**KEYWORDS:** ferritic steels, reduced-activation, tensile properties, impact properties

Alloy development programs are in progress in Japan, the European Union, the United States, and Russia to develop reduced-activation or fast induced-radioactivity decay (FIRD) ferritic steels for fusion power plant applications [1]. Components of a plant constructed from such steels could be more readily disposed of or recycled after service than if they were made from conventional alloys [2]. A FIRD or reduced-

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activation alloy cannot contain niobium and molybdenum [2], important constituents in conventional Cr-Mo steels of interest for fusion, which in the U.S. fusion program included 2 1/4Cr-1Mo, 9Cr-1MoVNb, and 12Cr-1MoVW.

The eight experimental steels discussed in this paper were based on compositions of conventional Cr-Mo steels with molybdenum replaced by tungsten and niobium replaced by tantalum [1]. Nominal compositions are given in Table 1, along with the designation for each steel. As a generic designation, the new class of steels will be referred to as Cr-W steels, chromium and tungsten being the primary constituents after iron. (An exception to this is the 2 1/4CrV steel, which contains no tungsten.) This designation follows the procedure used for the Cr-Mo steels, after which these steels are patterned, even though both types of steel may contain other alloying elements (i.e., V, Ta, etc.).

TABLE 1--Nominal compositions for reduced-activation steels

Alloy	Nominal Chemical Composition <sup>a</sup> (wt %)				
	Cr	W	V	Ta	C
2.25CrV	2.25		0.25		0.1
2.25Cr-1WV	2.25	1.0	0.25		0.1
2.25Cr-2W	2.25	2.0			0.1
2.25Cr-2WV	2.25	2.0	0.25		0.1
5Cr-2WV	5.0	2.0	0.25		0.1
9Cr-2WV	9.0	2.0	0.25		0.1
9Cr-2WVTa	9.0	2.0	0.25	0.12	0.1
12Cr-2WV	12.0	2.0	0.25		0.1

<sup>a</sup> Balance iron.

Information on microstructure [3], tempering and tensile behavior [4], and Charpy impact behavior [5] of the eight FIRD steels in the unirradiated condition has been reported. Results were also published on the tensile properties after irradiation to 6-8 dpa [6] and the Charpy properties after irradiation to 6-8 [6], 15-17 [7], and 23-24 dpa [8] at 365°C in the Fast Flux Test Facility (FFTF). Charpy specimens have now been irradiated to 26-29 dpa and tensile specimens have been irradiated to 15-17 and 26-29 dpa at 365°C in the FFTF. In this paper, these data will be presented and combined with the previous data to analyze the effect of irradiation on the properties. Observations on impact behavior are extremely useful because neutron irradiation causes an increase in the ductile-brittle transition temperature (DBTT) and a decrease in upper-shelf energy (USE) of ferritic steels; those effects generally reflect a degradation in fracture toughness. Developing steels with minimal changes in these parameters is crucial if ferritic steels are to be useful structural materials for fusion.

## EXPERIMENTAL PROCEDURE

Eight electroslag-remelted heats of FIRD steels with the nominal compositions and alloy designations given in Table 1 were prepared by Combustion Engineering, Inc. These steels were used in previous studies, and melt compositions have been published [3]. In addition to nominal compositions of Cr, W, V, C, and Ta, elements normally found in steels, such as Mn, Si, etc., were adjusted to levels typical of commercial practice.

The steels were normalized and tempered prior to aging and irradiation. The 2 1/4Cr-2W steel was austenitized 1 h at 900°C and air cooled. The other seven heats contained vanadium and were austenitized 1 h at 1050°C and air cooled; the higher normalizing temperature assured that any vanadium carbide dissolved during austenitization. The 2 1/4CrV, 2 1/4Cr-1WV, and 2 1/4Cr-2W steels were tempered 1 h at 700°C; the other five heats were tempered 1 h at 750°C.

Tensile specimens were machined from 0.76-mm sheet. In this case, heat treatments were made on the machined specimens and were carried out in a helium atmosphere. Rapid cooling was achieved by pulling the specimens from the tube furnace and cooling in flowing helium. The tensile specimens were 25.4 mm long and had a reduced gage section of 7.62 mm long by 1.52 mm wide by 0.76 mm thick. Tensile tests were made at 365°C in vacuum on a 120-kN Instron universal machine at a nominal strain rate of  $\approx 1 \times 10^{-3}/s$ .

One-third size Charpy specimens measuring 3.3 x 3.3 x 25.4 mm with a 0.51-mm-deep 30° V-notch and a 0.05- to 0.08-mm-root radius were machined from normalized-and-tempered 15.9-mm plates. Specimens were machined from along the rolling direction with the notch transverse to the rolling direction (L-T orientation). The absorbed energy values were fit with a hyperbolic tangent function to permit the USE and DBTT to be evaluated. The DBTT was determined at an energy level midway between the upper and lower shelf energies. Details on the test procedure for the subsize Charpy specimens have been published [9].

For each irradiation condition, six Charpy specimens and two tensile specimens from each heat were irradiated in the Materials Open Test Assembly (MOTA) of FFTF in the below-core specimen canister, a sodium "weeper" operating at  $\approx 365^\circ\text{C}$ . Fluence was determined from flux monitors in the irradiation canisters; there was some variation for different specimens, depending on their position in the canister. Specimens were irradiated to  $1.7-2.1 \times 10^{26}$ ,  $4.2-4.5 \times 10^{26}$ ,  $5.9-6.3 \times 10^{26}$ , and  $5.5-6.3 \times 10^{26} \text{ n/m}^2 (\text{E} > 0.1 \text{ MeV})$ , which produced 6-8, 15-17, 23-24, and 26-29 dpa, respectively. Helium concentrations were calculated to be less than 1 appm.

Tensile and Charpy specimens were aged for 5000, 10000, and 20000 h at 365°C, and these specimens along with the unaged specimens (data previously reported) were tested at 365°C as controls.

## RESULTS

Tensile and Charpy results will be presented separately. The new results will be presented within the context of the results previously obtained [6-8], so that the effects of irradiation fluence on mechanical property behavior can be analyzed.

## Properties Before and After Thermal Aging

Tensile Behavior--Aging for 5000, 10000, and 20000 h at 365°C had relatively little effect on yield stress [Fig. 1(a)], ultimate tensile strength [Fig. 1 (b)], uniform elongation [Fig. 1(c)], and total elongation [Fig. 1(d)] (Table 2). Several of the steels showed a slight increase in strength (2 1/4CrV, 9Cr-2WV, 9Cr-2WVTa, and 12Cr-2WV), with most of the increase occurring in the first 5000 h. Despite this hardening in the first 5000h, there was often an increase in ductility after the 5000 h age, although the elongations displayed considerable scatter. The 2 1/4CrV and 2 1/4Cr-1WV were the strongest steels, and the 2 1/4Cr-2W was the weakest (these three steels were tempered at 700°C). Of the five steels tempered at 750°C, the 2 1/4Cr-2WV was the strongest, and the 5Cr-2WV the weakest. The strengths of the 9Cr-2WV and 9Cr-2WVTa were essentially the same before and after aging.

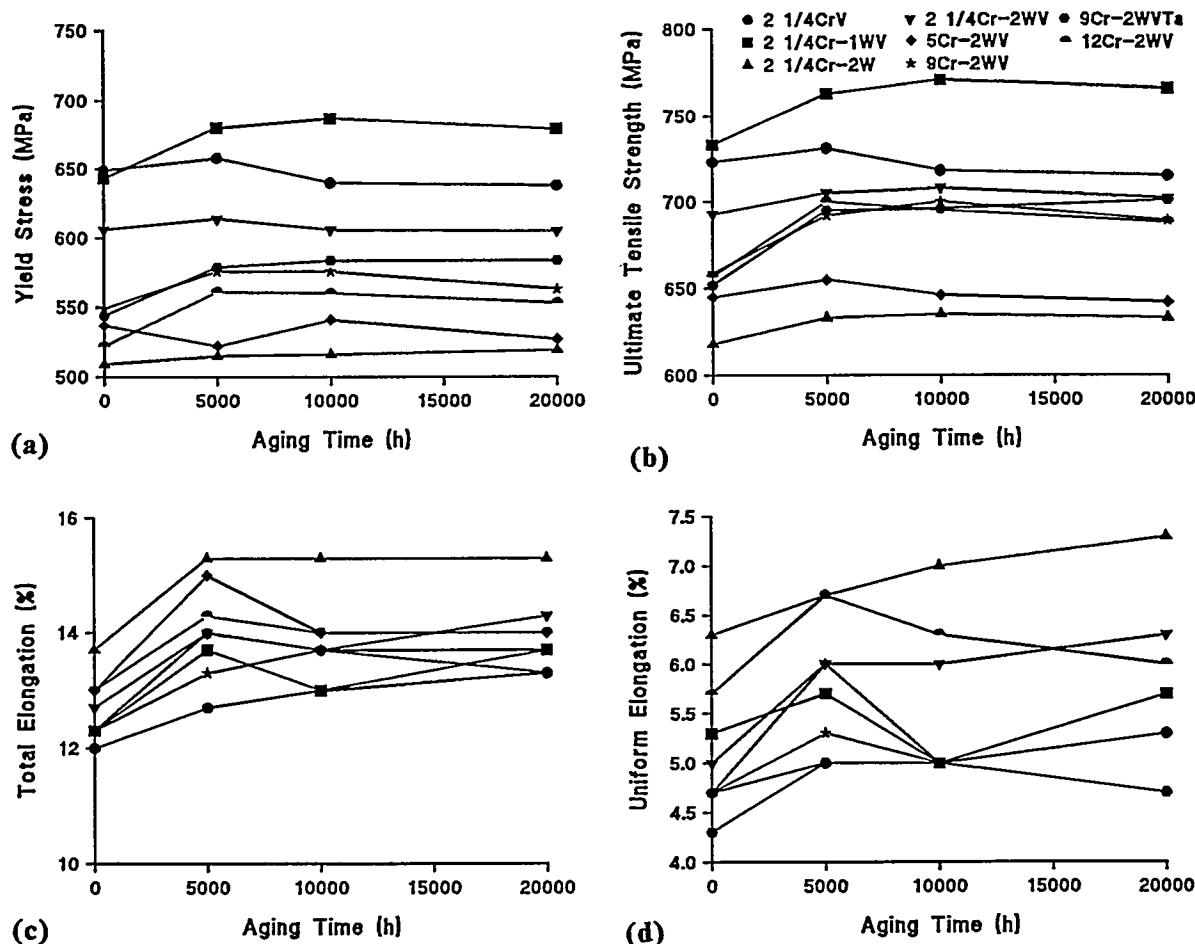


Fig. 1--The (a) yield stress, (b) ultimate tensile strength, (c) uniform elongation, and total elongation as a function of aging time at 365°C for the eight reduced-activation steels.

TABLE 2--Tensile properties of thermally aged reduced-activation steels<sup>a</sup>

Steel	Aging Time (h)	Strength (Mpa)		Elongation (%)	
		Yield	Ultimate	Uniform	Total
2 1/4CrV	0	649	723	4.7	12.0
	5000	658	731	5.0	12.7
	10000	640	718	5.0	13.0
	20000	638	715	5.3	13.3
2 1/4Cr-1WV	0	643	733	5.3	12.3
	5000	680	763	5.7	13.7
	10000	687	771	5.0	13.0
	20000	679	766	5.7	13.7
2 1/4Cr-2W	0	509	618	6.3	13.7
	5000	515	633	6.7	15.3
	10000	516	635	7.0	15.3
	20000	519	633	7.3	15.3
2 1/4Cr-2WV	0	606	693	5.0	12.7
	5000	614	705	6.0	14.0
	10000	606	708	6.0	13.7
	20000	605	702	6.3	14.3
5Cr-2WV	0	537	645	4.7	13.0
	5000	522	655	6.0	15.0
	10000	541	646	5.0	14.0
	20000	527	642	5.3	14.0
9Cr-2WV	0	549	659	4.7	12.3
	5000	576	692	5.3	13.3
	10000	576	700	5.0	13.7
	20000	563	689	5.3	13.7
9Cr-2WVTa	0	544	652	4.3	12.3
	5000	579	695	5.0	14.0
	10000	584	696	5.0	13.7
	20000	584	701	4.7	13.3
12Cr-2WV	0	522	657	5.7	13.0
	5000	561	700	6.7	14.3
	10000	560	695	6.3	14.0
	20000	553	688	6.0	14.0

<sup>a</sup> Values are the average of two tests

Charpy Behavior--Charpy impact properties showed relatively little change after aging for 0, 5000, 10000, and 20000 h at 365°C (Table 3). The DBTT values [Fig. 2(a)] for several of the the steels showed a slight change, usually in the initial 5000 h. The 9Cr-2WVTa had the lowest DBTT, and the four 2 1/4 Cr steels and the 12Cr-2WV steel had the highest values. The USE [Fig. 2(b)] of most of the steels showed an increase with aging time with the largest change usually occurring within the first 5000 h. The 9Cr-2WVTa steel had the highest USE after 10000 h, but then decreased at 20000 h to a value similar to values for the other steels.

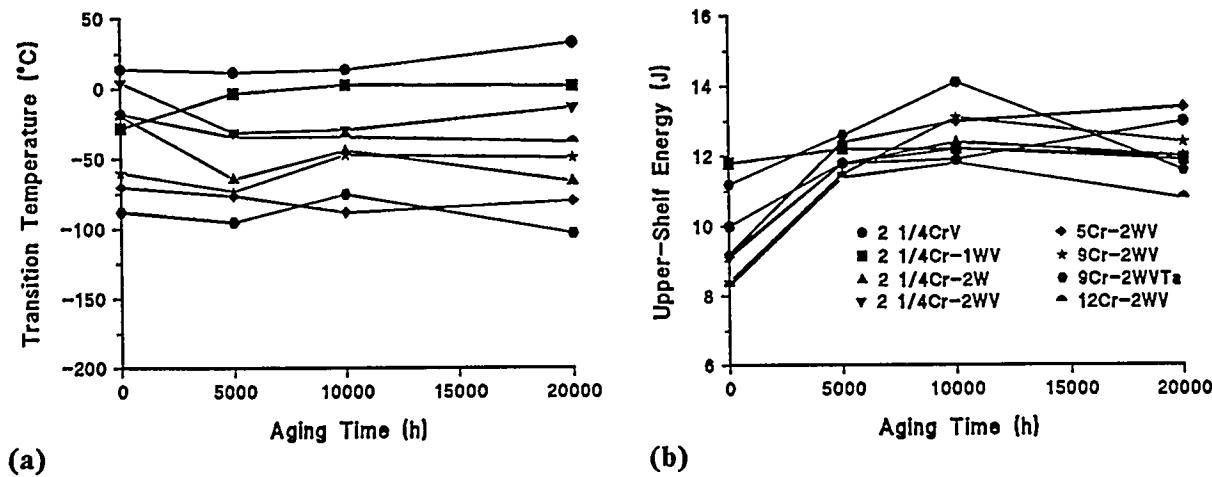


Fig. 2--The (a) ductile-brittle transition temperature and (b) upper-shelf energy as a function of aging time at 365°C for the eight reduced-activation steels.

#### Properties After Irradiation

Tensile behavior--Irradiation hardened the steels (Table 4), as measured by the yield stress [Fig. 3(a)] and ultimate tensile strength [Fig. 3(b)]. Hardening appeared to saturate with fluence, although the curve for the 2 1/4CrV appeared to go through a maximum. For this discussion, the results are concluded to indicate saturation; that is, there was a strength increase during the first increment of irradiation, after which it remained essentially constant with further irradiation. The curves for strength after irradiation fell into two groups: the 2 1/4Cr-2W, 5Cr-2WV, 9Cr-2WV, and 9Cr-2WVTa fell into a group showing the lowest strength and the 2 1/4CrV, 2 1/4Cr-1WV, 2 1/4Cr-2WV, and 12Cr-2WV fell in a group showing a considerably higher strength. The two 9Cr steels had the lowest strength after irradiation.

Uniform [Fig. 3(c)] and total elongation [Fig. 3(d)] decreased with fluence. As opposed to the separation into two groups observed for the strength, the ductility appeared to fall into three groups. The 9Cr-2WV and 9Cr-2WVTa steels had the highest ductility after irradiation, followed by the 2 1/4Cr-2W and 5Cr-2WV, and then the third group containing the 2 1/4CrV, 2 1/4Cr-1WV, 2 1/4Cr-2WV, and 12Cr-2WV. Although

TABLE 3--Charpy impact properties of thermally aged reduced-activation steels

Steel	Aging Time (h)	DBTT <sup>a</sup> (°C)	Upper-Shelf Energy (J)
2 1/4CrV	0	14	10.0
	5000	12	11.8
	10000	14	11.9
	20000	33	13.0
2 1/4Cr-1WV	0	-28	11.8
	5000	-3	12.2
	10000	3	12.2
	20000	2	11.9
2 1/4Cr-2W	0	-19	9.2
	5000	-64	11.8
	10000	-44	12.4
	20000	-66	12.0
2 1/4Cr-2WV	0	4	9.1
	5000	-31	11.8
	10000	-29	12.2
	20000	-13	12.0
5Cr-2WV	0	-70	9.2
	5000	-76	12.4
	10000	-88	13.0
	20000	-80	13.4
9Cr-2WV	0	-60	8.4
	5000	-73	11.5
	10000	-47	13.1
	20000	-49	12.4
9Cr-2WVTa	0	-88	11.2
	5000	-95	12.6
	10000	-75	14.1
	20000	-103	11.6
12Cr-2WV	0	-18	8.3
	5000	-34	11.4
	10000	-34	11.8
	20000	-38	10.8

<sup>a</sup>Evaluated at an energy level halfway between the upper and lower shelves

the ductility data showed considerable scatter, the decrease in uniform elongation appeared to saturate with fluence.

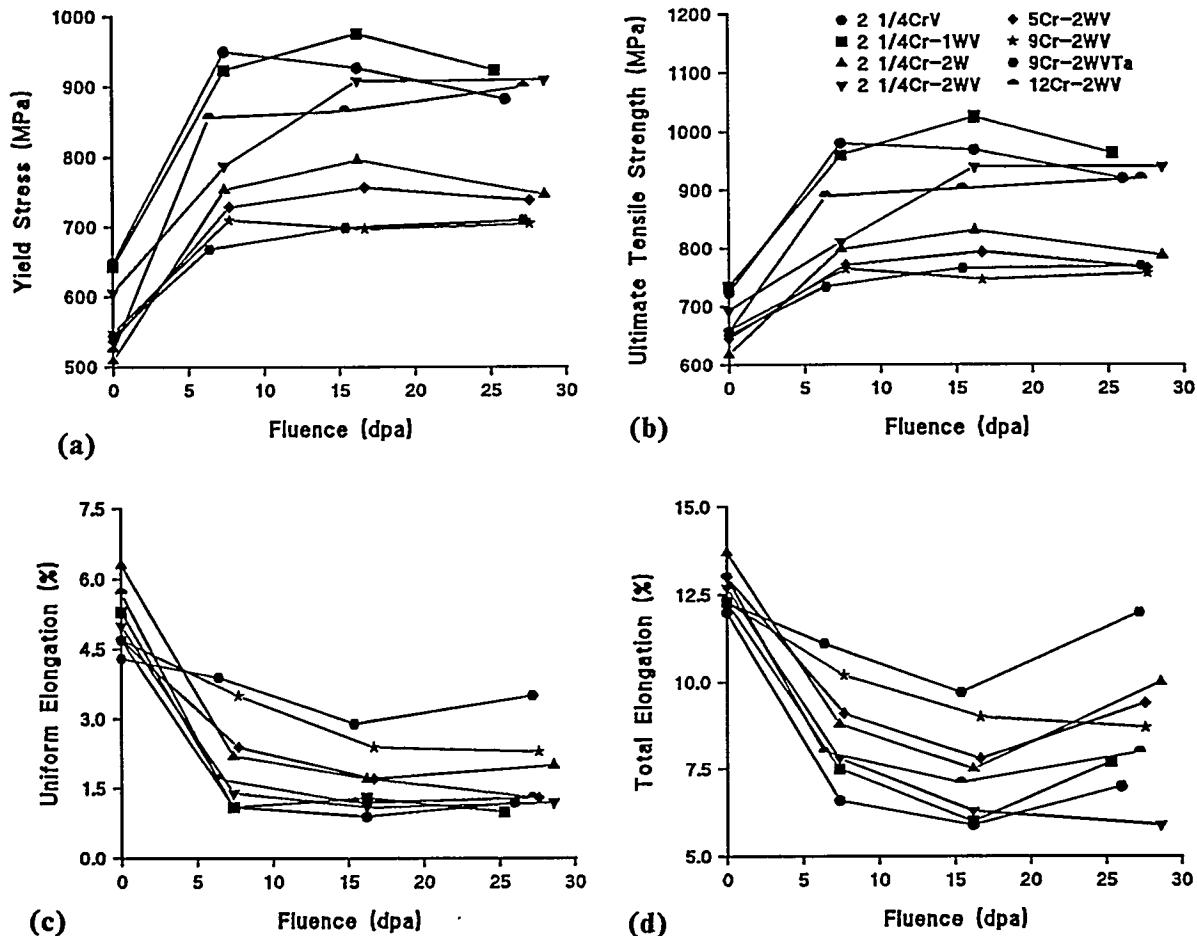


Fig. 3--The (a) yield stress, (b) ultimate tensile strength, (c) uniform elongation, and (d) total elongation as a function of fluence for the eight reduced-activation steels irradiated in FFTF at 365°C.

Charpy Behavior--The Charpy data for irradiations up to 26-29 dpa continued the trends noted for the previous irradiations (Table 5) [6-8]. After an initial increase in DBTT with fluence [Fig. 4(a)], the change in DBTT of most of the steels appeared to level off with increasing fluence, indicating an approach to a saturation in the shift in DBTT ( $\Delta$ DBTT). The only exception was the 9Cr-2WVTa, which showed a continuous increase in DBTT over the entire fluence range, although the increase was slight. Despite the slight increase, it was obvious that the 9Cr-2WVTa showed superior behavior at all fluences. The 9Cr-2WV and the 5Cr-2WV steels showed the next best behavior.

TABLE 4--Tensile properties of Cr-W steels irradiated in FFTF at 365°C<sup>a</sup>

Steel	Fluence (dpa)	Strength (Mpa)		Elongation (%)	
		Yield	Ultimate	Uniform	Total
2 1/4CrV	0	649	723	4.7	12.0
	7.4	950	980	1.1	6.4
	16.2	937	968	0.9	7.0
	26.0	883	920	1.2	7.0
2 1/4Cr-1WV	0	643	733	5.3	12.3
	7.4	924	959	1.3	7.5
	16.2	976	1026	1.0	7.5
	25.4	924	963	1.4	7.7
2 1/4Cr-2W	0	509	618	6.3	13.7
	7.4	754	799	2.2	8.8
	16.2	796	830	1.7	7.5
	28.6	747	787	2.0	10.0
2 1/4Cr-2WV	0	606	693	5.0	12.7
	7.4	788	810	1.4	7.8
	16.2	909	939	1.1	5.3
	28.6	910	940	1.2	5.9
5Cr-2WV	0	537	645	4.7	13.0
	7.7	729	771	2.4	9.1
	16.7	757	793	1.7	7.8
	27.6	739	766	1.4	11.2
9Cr-2WV	0	549	659	4.7	12.3
	7.7	710	764	3.5	10.2
	16.7	697	745	2.3	9.0
	27.6	705	756	2.3	8.7
9Cr-2WVTa	0	544	652	4.3	12.3
	6.4	669	734	3.9	11.1
	15.4	699	765	2.9	9.7
	27.2	710	769	3.5	12.0
12Cr-2WV	0	522	657	5.7	13.0
	6.4	857	890	1.7	8.0
	15.4	866	902	1.2	7.1
	27.2	900	932	1.3	8.0

<sup>a</sup> Values are the average of two tests

The USE [Fig. 4(b)] decreased with fluence and leveled off (saturated), although for several steels it decreased slightly between the third and fourth irradiations. By far the best steel was the 9Cr-2WVTa, with the 9Cr-2WV and 5Cr-2WV second best. Comparison of Charpy curves for the two most irradiation-resistant steels indicates the superiority of the 9Cr-2WVTa steel by showing that the curves for the 9Cr-2WVTa [Fig. 5(a)] after irradiation approach the curve for the unirradiated 9Cr-2WV steel [Fig. 5(b)].

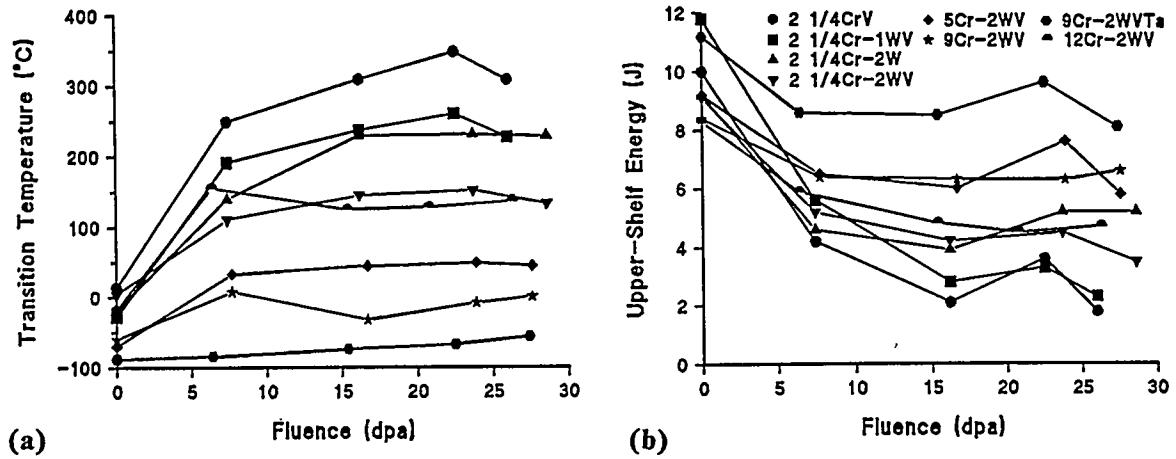


Fig. 4--The (a) ductile-brittle transition temperature and (b) upper-shelf energy as a function of fluence for the eight reduced-activation steels irradiated in FFTF at 365°C.

Large increases in DBTT and large decreases in USE ( $\Delta$ USE) were observed for the four low-chromium steels and the 12Cr-2WV. The behavior of the 2 1/4Cr-2WV and 12Cr-2WV were comparable and displayed the smallest change in DBTT of these five steels. The 2 1/4CrV had the largest  $\Delta$ DBTT and  $\Delta$ USE, followed by the 2 1/4Cr-1WV and the 2 1/4Cr-2W, although the USE of the latter steel at the highest fluences was comparable to that of the 2 1/4Cr-2WV and 12Cr-2WV. The difference between the best and worst of the steels can be seen by comparing the curves for 2 1/4CrV [Fig. 5(c)] with those for 9Cr-2WV [Fig. 5(a)] and 9Cr-2WVTa in [Fig. 5(b)].

## DISCUSSION

### Properties Before and After Thermal Aging

The microstructures of the normalized-and-tempered 0.76-mm sheets and 15.9-mm plates from which tensile and Charpy specimens were taken, respectively, have been examined [3]. There was a difference in microstructure of the 2 1/4 Cr steels in the two geometries. All of these low-chromium steels were  $\approx$ 100% bainite when heat treated as 0.76-mm sheet. When heat treated as 15.9-mm plate, all but the 2 1/4Cr-2W steel contained a duplex microstructure of tempered bainite and polygonal ferrite: 2 1/4CrV [Fig. 6(a)] contained  $\approx$ 30% tempered bainite, 70% ferrite; 2 1/4Cr-1WV [Fig. 6(b)] contained  $\approx$ 55% tempered bainite, 45% ferrite; and 2 1/4Cr-2WV [Fig. 6(d)] was  $\approx$ 80% tempered bainite, 20% ferrite. The 2 1/4Cr-2W steel [Fig. 6(c)] was 100% tempered bainite. Microstructures were the same for the high-chromium steels in both geometries: the 5Cr-2WV [Fig. 7(a)], 9Cr-2WV [Fig. 7(b)], and 9Cr-2WVTa [Fig. 7(c)] steels were

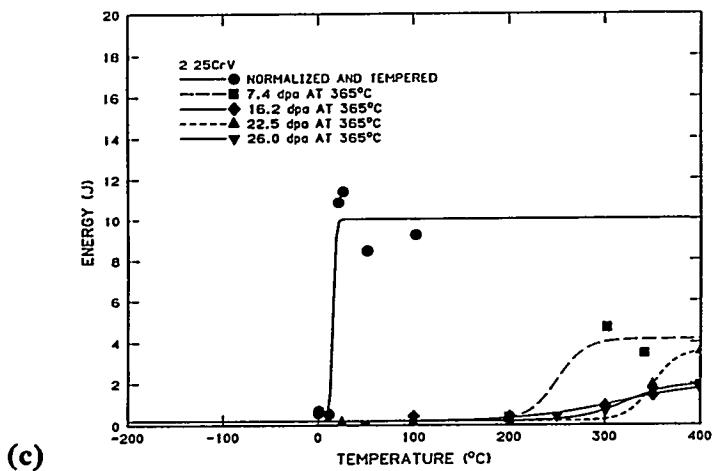
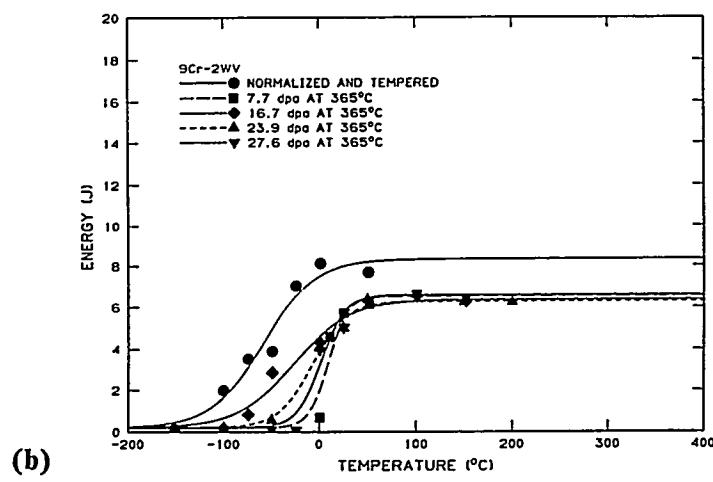
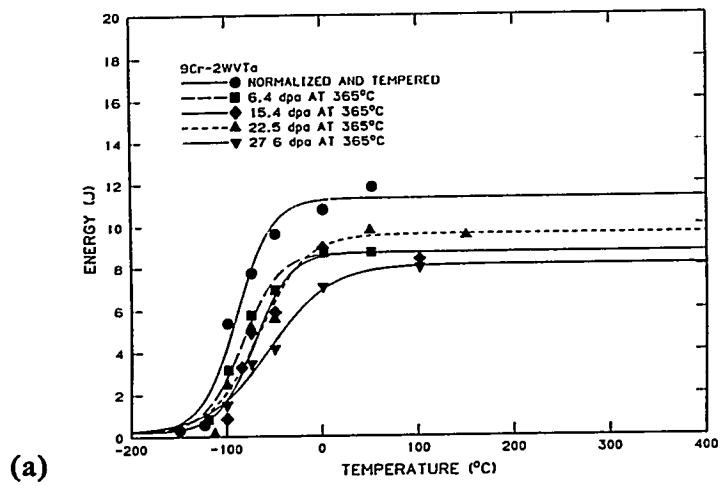


Fig. 5--Charpy curves for the normalized-and-tempered and irradiated (a) 9Cr-2WVTa, (b) 9Cr-2WV steels, and (c) 2 1/4CrV.

TABLE 5--Charpy impact properties of Cr-W steels irradiated in FFTF at 365°C

Steel	Fluence (dpa)	DBTT (°C)	ΔDBTT (°C)	Upper-Shelf Energy (J)	ΔUSE (%)	Δσ <sub>y</sub> (%)
2 1/4CrV	0	14		10.0		
	7.4	250	236	4.2	-58	46
	16.2	310	296	2.1	-79	44
	22.5	349	335	3.6	-64	
	26.0	309	295	1.8	-82	36
2 1/4Cr-1WV	0	-28		11.8		
	7.4	192	220	5.6	-53	44
	16.2	238	266	2.8	-76	52
	22.5	261	289	3.3	-72	
	26.0	228	256	2.3	-81	44
2 1/4Cr-2W	0	-19		9.2		
	7.4	140	159	4.6	-50	48
	16.2	230	249	3.9	-58	56
	23.7	232	251	5.2	-43	
	28.6	229	248	5.2	-43	47
2 1/4Cr-2WV	0	4		9.1		
	7.4	111	107	5.2	-43	30
	16.2	145	141	4.2	-54	50
	23.7	152	148	4.5	-51	
	28.6	133	129	3.5	-62	50
5Cr-2WV	0	-70		9.2		
	7.7	33	103	6.5	-29	36
	16.7	45	115	6.0	-35	41
	23.9	49	119	7.6	-17	
	27.6	45	115	5.8	-37	38
9Cr-2WV	0	-60		8.4		
	7.7	8	68	6.4	-24	30
	16.7	-32	28	6.3	-25	27
	23.9	-8	52	6.3	-25	
	27.6	1	61	6.6	-21	28
9Cr-2WVTa	0	-88		11.2		
	6.4	-84	4	8.6	-23	23
	15.4	-74	14	8.5	-24	28
	22.5	-67	21	9.6	-14	
	27.6	-56	32	8.1	-28	31
12Cr-2WV	0	-18		8.3		
	6.4	156	174	5.9	-29	64
	15.4	125	143	4.8	-42	66
	20.8	128	146	4.5	-46	
	26.3	137	155	4.7	-43	72

<sup>a</sup>Evaluated at an energy level halfway between the upper and lower shelves

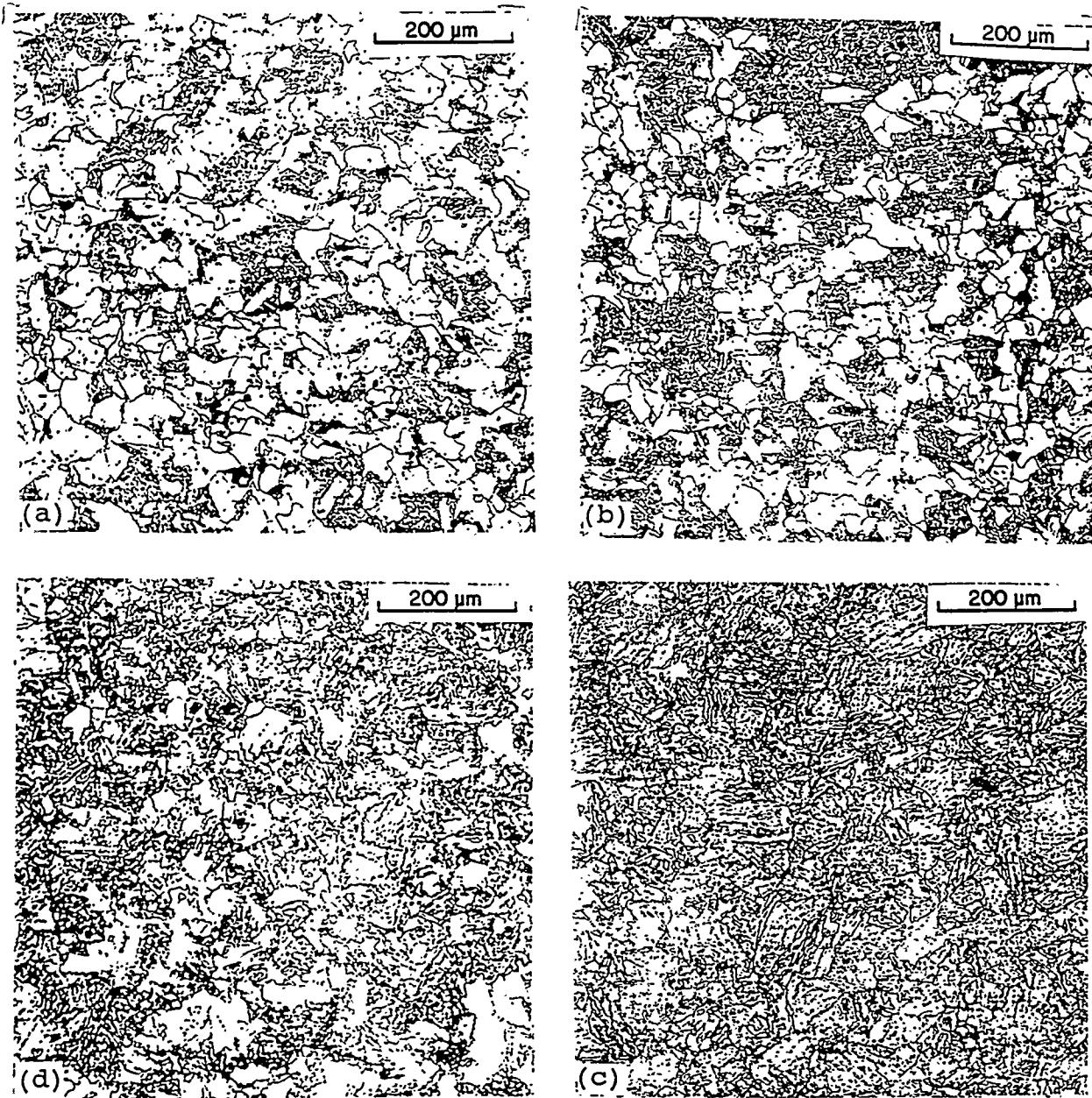


Fig. 6--Normalized-and-tempered microstructures of (a) 2 1/4CrV, (b) 2 1/4Cr-1WV, (c) 2 1/4Cr-2W, and (d) 2 1/4Cr-2WV steels.

100% tempered martensite, and the 12Cr-2WV [Fig. 7(d)] steel was tempered martensite with  $\approx 25\%$   $\delta$ -ferrite [3].

Before irradiation, the three 2 1/4Cr steels with vanadium contained  $M_7C_3$ ,  $M_3C$ , and MC precipitates [3]. The 2 1/4Cr-2W contained  $M_7C_3$ ,  $M_3C$ ,  $M_{23}C_6$ , and  $M_2X$ . The 9Cr-2WV, 9Cr-2WVTa, and 12Cr-2WV contained primarily  $M_{23}C_6$  and small amounts of MC; some of the MC in the 9Cr-2WVTa contained tantalum, although most was vanadium

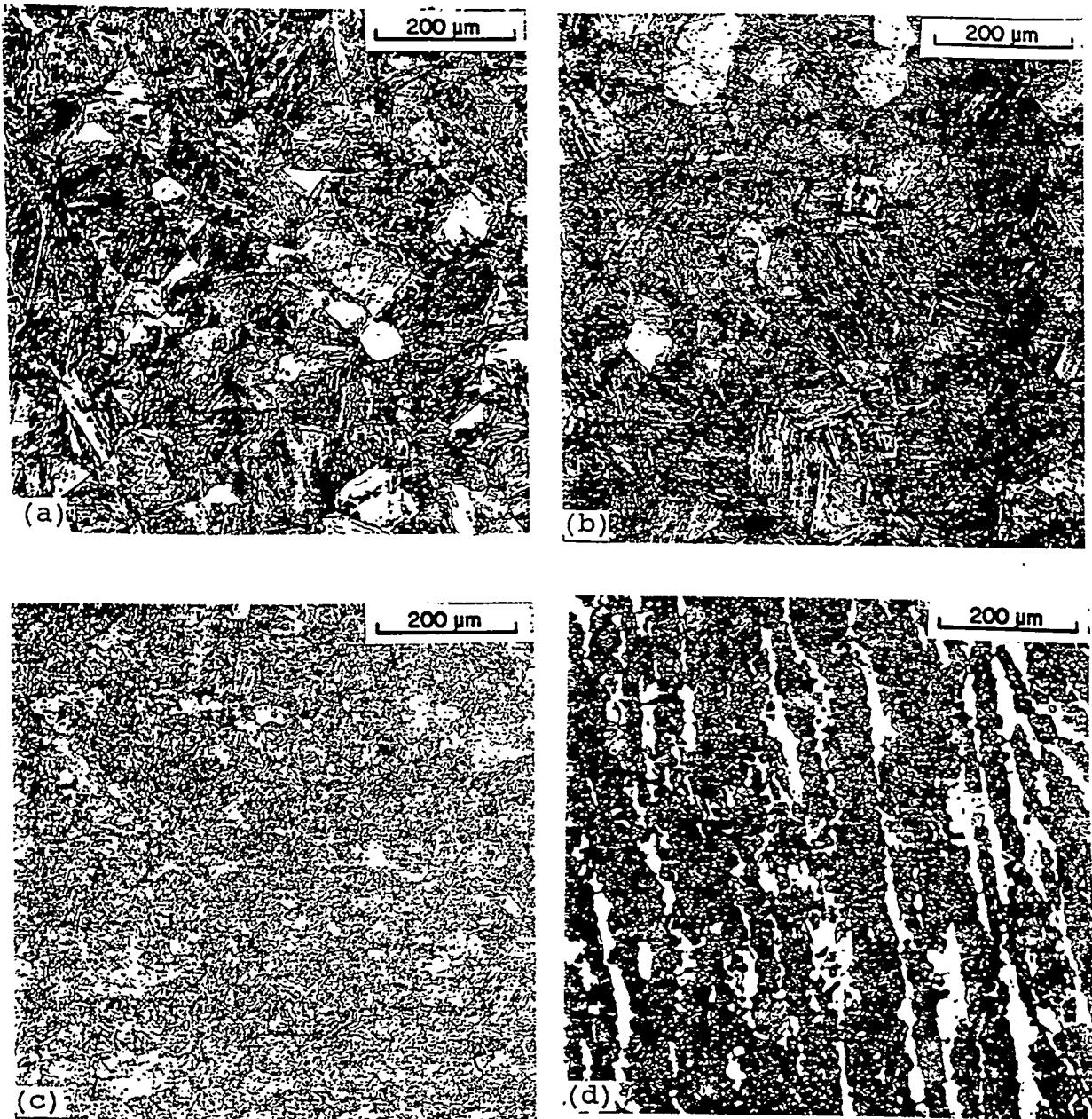


Fig. 7--Normalized-and-tempered microstructures of (a) 5Cr-2WV, (b) 9Cr-2WV, (c) 9Cr-2WVTa, and (d) 12Cr-2WV steels.

rich, as it was in the 9Cr-2WV. The 5Cr-2WV contained mainly M<sub>7</sub>C<sub>3</sub>, with small amounts of M<sub>23</sub>C<sub>6</sub> and MC, thus bridging the gap between the M<sub>7</sub>C<sub>3</sub> found in the steels with 2 1/4% Cr and the M<sub>23</sub>C<sub>6</sub> found in the steels with 9% Cr [3].

The relative strengths of the steels before aging or irradiation [Figs. 1(a) and 1(b)] were the result of the heat treatment given the steels. The 2 1/4CrV and 2 1/4Cr-1WV were stronger than the other steels because these steels were tempered at 700°C, while all

but the 2 1/4Cr-2W were tempered at 750°C. The 2 1/4Cr-2W was the weakest, despite being tempered at 700°C, because it did not contain the strong carbide-forming element vanadium, and thus, it was not strengthened by MC. Of the steels that contained vanadium and were tempered at 750°C, the 2 1/4Cr-2WV was the strongest, followed by 9Cr-2WVTA, 9Cr-2WV, 12Cr-2WV, and the 5Cr-2WV steels. There was essentially no difference in the strength of the 9Cr-2WV and the 9Cr-2WVTA in the unirradiated condition. All of the steels had adequate ductility [Figs. 1(b) and 1(c)]. Aging at 365°C to 20000 h had little effect on strength and ductility of any of the steels; properties remained similar or slightly improved compared to the unaged steel.

Thermal aging had little effect on the DBTT of the steels [Fig. 2(a)]. However, the DBTT behavior of the unirradiated steels after thermal aging reflected the different microstructures. Steels with a 100% tempered martensite and a 100% tempered bainite microstructure--the 5Cr-2WV, 9Cr-2WV, 9Cr-2WVTA and 2 1/4Cr-2W--had the lowest DBTT values. The other four steels had duplex microstructures, each containing some polygonal or delta ferrite. The DBTT values increased with increasing amount of ferrite, from the 12Cr-2WV and 2 1/4Cr-2WV, with 20-25%, to the 2 1/4Cr-1WV, with  $\approx$ 45%, and the 2 1/4CrV, with  $\approx$ 70% ferrite.

The USE of most of the steels increased slightly during aging, especially during the initial 5000 h period. The reason for this is not known, although a slight supersaturation of carbon may have been present from the tempering treatment, which is relieved by the low-temperature aging treatment. Little diffusion of any substitutional elements would be expected at 365°C.

### Properties After Irradiation

Irradiation hardening saturated with fluence with the amount of hardening depending on microstructure [Fig. 3(a) and (b)]. By comparing the relative increase in yield stress,  $\Delta\sigma_y$  (Table 5) for the different steels, it is seen that the steels with 100% tempered martensite (5Cr-2WV, 9Cr-2WV, and 9Cr-2WVTA) hardened the least. The  $\Delta\sigma_y$  of the two 9Cr steels was the lowest ( $\approx$ 30% increase), with that for the 5Cr steel slightly higher (35-40%). The four 2 1/4Cr steels, which had 100% bainite microstructures in the tensile specimens, hardened about the same amount (40-50%), even though they had different strengths before irradiation. Therefore, it appears that tempered bainite hardens more than tempered martensite. The 12Cr-2WV steel contained  $\approx$ 75% tempered martensite, and it hardened the most (60-70%), suggesting that the difference between this steel and the 100% tempered martensitic steels must have been the 25%  $\delta$ -ferrite present in the microstructure. The effect of fluence on ductility was inverse to the hardening in that an increase in hardening was accompanied by a decrease in ductility [Fig. 3(c) and 3(d)].

Despite the difference in microstructure of the 2 1/4 Cr steels used for the tensile and Charpy specimens, the effect of fluence on Charpy impact behavior was generally similar to the effect on strength. With the exception of the 9Cr-2WVTA, which appeared to show a slight increase with fluence (this will be discussed below), the effect of irradiation on the DBTT and USE saturated with fluence. Irradiation had the least effect on the DBTT and USE of the three 100% martensitic steels. Of these, the 9Cr-2WVTA was superior to the other steels, followed by 9Cr-2WV and 5Cr-2WV, in agreement with the previous results

[6-8]. The steels with the next best irradiation resistance were the 2 1/4Cr-2WV and the 12Cr-2WV steels, which contained 20-25% polygonal and delta ferrite, respectively. These steels saturated at a similar DBTT and USE, although the 2 1/4Cr-2WV had the lowest  $\Delta$ DBTT (Table 5).

For the 2 1/4Cr steels containing vanadium and a duplex bainite-polygonal ferrite microstructure, it appeared that the the  $\Delta$ DBTT increased with the amount of ferrite in the microstructure. The 2 1/4CrV steel, which contained the most ferrite ( $\approx 70\%$ ), showed the largest  $\Delta$ DBTT, followed by the the 2 1/4Cr-1WV and the 2 1/4Cr-2WV, the steels with  $\approx 45$  and  $25\%$ , respectively. Because of the difference in microstructures in the 2 1/4Cr steel specimens used in the tensile and Charpy experiments, the relationship between the increase in strength and  $\Delta$ DBTT must be interpolated. By comparing tensile results for the 12Cr-2WV and the other martensitic steels, it appears that hardening was influenced substantially by the amount of ferrite in the microstructure. The similarity of the DBTT for the 12Cr-2WV and 2 1/4Cr-2WV steels, which both contain similar amounts of ferrite, appears to support that conclusion [Fig. 4(a)]. Likewise, it is concluded that the relative behavior of the  $\Delta$ DBTT of the 2 1/4CrV, 2 1/4Cr-1WV, and 2 1/4Cr-2WV is determined by the amount of the polygonal ferrite present in the microstructure.

Anderko et al. [13] showed that for 12Cr steels containing  $\delta$ -ferrite, it is  $M_{23}C_6$  on martensite/ $\delta$ -ferrite boundaries rather than the  $\delta$ -ferrite itself that causes a deterioration of the Charpy properties. Considerable  $M_{23}C_6$  forms on the interfaces in the 12Cr-2WV in the unirradiated condition [3], which means that  $M_{23}C_6$  could control fracture in the 12Cr-2WV. Precipitates (mainly  $M_7C_3$ ) may also control the behavior of the 2 1/4Cr-2WV [3]. This implies that reducing the size of the precipitates at ferrite/martensite or ferrite/bainite boundaries would be the most likely way to minimize  $\Delta$ DBTT. In the 2 1/4Cr-2WV, the polygonal ferrite can be eliminated by heat treating. It has been demonstrated that heat treating to produce 100% bainite significantly lowered the DBTT of unirradiated 2 1/4Cr-2WV [10].

The results on the 5Cr-2WV, 9Cr-2WV, and 9Cr-2WVta indicate that tempered martensite is more irradiation resistant than tempered bainite. More information on the irradiation resistance of tempered bainite will become known after Charpy specimens of 2 1/4Cr-2WVstel in the fully bainitic condition have been irradiated.

The properties of the 2 1/4Cr-2W appeared anomalous with the other 2 1/4Cr steels because this steel was 100% tempered bainite but had a much higher DBTT and  $\Delta$ DBTT than the 2 1/4Cr-2WV, which was 25% ferrite. The DBTT of the irradiated 2 1/4Cr-2W were similar to those of the 2 1/4Cr-1WV, which contained 45% ferrite. A major difference between the 2 1/4Cr-2WV and 2 1/4Cr-2W is that the 2 1/4Cr-2W does not contain vanadium. During irradiation the vanadium-containing carbides are more stable than the  $M_7C_3$  and  $M_3C$  that dominate in the 2 1/4Cr-2W [3]. The larger precipitates in the 2 1/4Cr-2W after irradiation could enhance its susceptibility to fracture relative to the steels with a more stable precipitate structure (e.g., 2 1/4Cr-2WV).

Similar explanations for the low-chromium steels have previously been used to conclude that for maximum irradiation resistance, these steels containing the combination of tungsten and vanadium must be irradiated in the entirely bainitic condition [10]. It has also been pointed out that the type of bainite formed could play a role in the irradiation resistance.

The two 9Cr steels show the least hardening and have the best impact properties after irradiation. The properties of the 9Cr-2WVTa were exceptional after the previous three irradiations, showing only a small  $\Delta$ DBTT (21°C after 22.5 dpa) [6-8]; similar behavior was observed in the present experiment. Not only does it show a very small  $\Delta$ DBTT (32°C) after over 27 dpa, but because it has such a low DBTT in the unirradiated condition, the DBTT after irradiation remains substantially below that for the other steels. It was pointed out previously that the  $\Delta$ DBTT data for the previous three irradiations for 9Cr-2WVTa indicated that there was a gradual increase in the post-irradiation DBTT with increasing dpa [8]. That trend continued in the present experiment [Fig. 4(a)]. However, even after the 27.4 dpa, the DBTT of -56°C for the 9Cr-2WVTa is still comparable to the DBTT of the 9Cr-2WV before irradiation (the 9Cr-2WV had the second lowest DBTT before irradiation). A similar conclusion applies to 9Cr-1MoVNb (modified 9Cr-1Mo, Grade 91), which has one of the lowest  $\Delta$ DBTT values ( $\approx$ 50°C) of the conventional steels considered for fusion applications [7].

One of the interesting aspects in comparing the 9Cr-2WV and 9Cr-1MoVNb steels with the 9Cr-2WVTa steel is that the difference in Charpy properties of these steels before and after irradiation occurs despite there being little difference in the strength of the 9Cr-2WVTa and the other two steels before and after irradiation. This can be seen in the similar irradiation hardening that occurred for the 9Cr-2WV and 9Cr-2WVTa steels as measured by  $\Delta\sigma_y$  (Table 5).

Transmission electron microscopy examination of the normalized-and-tempered 9Cr-2WV and 9Cr-2WVTa revealed only minor differences prior to irradiation [3,11]. Likewise, there was no marked difference in microstructure after irradiation, with similar numbers of dislocation loops formed in both steels during irradiation [11]. Thus, the similarity of strength of the two steels before and after irradiation is not unexpected. However, without any gross differences in the microstructure of the two steels, the only other major difference to account for the difference in Charpy properties is the tantalum in solid solution. Based on the amount of tantalum that appeared to be present in the MC carbides of the 9Cr-2WVTa, it was estimated that most of the tantalum remained in solid solution (or was incorporated in the  $M_{23}C_6$  precipitate) [11]. An atom probe analysis of the unirradiated 9Cr-2WVTa steel indicated that  $\approx$ 90% of the tantalum remained in solution in the normalized-and-tempered condition [12].

Tantalum in solution in the 9Cr-2WVTa can probably account for the smaller prior-austenite grain size in that steel than in the 9Cr-2WV; a smaller lath (subgrain) size might also be expected but was not observed. This smaller grain size was originally used to explain the difference between the 9Cr-2WV and 9Cr-2WVTa steels [6]. A smaller grain size can lead to a lower DBTT in the normalized-and-tempered condition. However, this explanation was subsequently questioned because in the normalized-and-tempered condition, the two steels had similar yield stresses, and they also had a similar yield stress as the 9Cr-1MoVNb, which had the smallest grain size of the three steels [7]. After  $\approx$ 20 dpa, the  $\Delta$ DBTT of the 9Cr-2WV and 9Cr-1MoVNb were similar, but above the value for the 9Cr-2WVTa [8]. This occurred even though there were differences in the microstructural changes that occurred in the 9Cr-2WV and 9Cr-1MoVNb during irradiation, while the microstructural changes in the 9Cr-2WV and 9Cr-2WVTa were similar [11].

These observations lead to the conclusion that microstructure (grain size, precipitate type, etc.) does not provide the sole explanation for the observations on mechanical property changes. It appears that tantalum in solution must cause a higher fracture stress for 9Cr-2WVTa than 9Cr-2WV, and the combination of tungsten and tantalum in the 9Cr-2WVTa leads to a higher fracture stress than produced by molybdenum and niobium in 9Cr-1MoVNb.

The observation that the  $\Delta$ DBTT of the 9Cr-2WVTa appeared to increase slightly with fluence appears to in agreement with such an explanation. This increase would follow if tantalum is being removed from solution during irradiation and being incorporated in the existing or new precipitates. If this were the case, the  $\Delta$ DBTT of the 9Cr-2WVTa would be expected to increase with fluence as tantalum is removed from solution. Eventually, it might be expected to approach the  $\Delta$ DBTT for the 9Cr-2WV. Even if that were to happen, however, the 9Cr-2WVTa should still have the lowest DBTT after irradiation because of the lower DBTT before irradiation.

## SUMMARY AND CONCLUSIONS

Tensile and Charpy impact properties of eight reduced-activation Cr-W ferritic steels have been determined after irradiation in FFTF at 365°C. Tensile specimens were irradiated to 6-8, 15-17, and 26-29 dpa and the Charpy specimens to  $\approx$  6-8, 15-17, 20-24, and 26-29 dpa (results for all but the tensile irradiations to 15-17 and 26-29 dpa and the Charpy irradiations to 26-29 dpa were presented previously). Chromium concentrations in the eight steels ranged from 2.25 to 12wt% (all steels contained 0.1%C). The 2.25Cr steels contained variations of tungsten and vanadium (2 1/4CrV, 2 1/4Cr-1WV, 2 1/4Cr-2W) and steels with 2.25, 5, 9, and 12% Cr contained a combination of 2% W and 0.25% V (2 1/4Cr-2WV, 5Cr-2WV, 9Cr-2WV, and 12Cr-2WV). A 9Cr steel with 2% W, 0.25% V, and 0.07% Ta (9Cr-2WVTa) was also irradiated. The microstructures of the 2 1/4Cr steels were bainite with various amounts of polygonal ferrite, while the two 9Cr steels and the 5Cr steel were 100% martensite. The 12Cr-2WV steel was martensite with  $\approx$ 25%  $\delta$ -ferrite. The properties of the steels with 100% martensite were superior to those with the duplex structures of bainite and ferrite or martensite and ferrite.

Irradiation caused an increase in strength during the first irradiation period (6-8 dpa), but there was little further hardening for the subsequent irradiations, indicating that the hardening saturated with fluence. The DBTT increased with irradiation, and USE decreased, but indications were that saturation occurred for most of the steels after the initial 6-8 dpa irradiation. The 2 1/4Cr-2WV steel had the most irradiation resistance of the four 2 1/4 Cr steels, but it was concluded that this resistance would be improved if it were 100% bainite (it contained  $\approx$ 25% polygonal ferrite). The 9Cr steels were least affected by irradiation, with the 9Cr-2WVTa showing only a 32°C increase in DBTT after  $\approx$ 28 dpa. This was the only steel that showed a slight increase in the shift with increasing fluence, with the 32°C shift being an increase from shifts of 4, 14, and 21°C in the previous irradiations to  $\approx$ 6.4, 15.4, and 22.5 dpa, respectively. Despite the slight increase, 32°C is one of the lowest shifts in DBTT for this type of steel irradiated to these conditions, and it compares with a 61°C shift for the 9Cr-2WV, which had the second

lowest shift. The advantage for the 9Cr-2WVTa over the 9Cr-2WV is further enhanced by the much lower DBTT of the 9Cr-2WVTa before irradiation. The advantage of the 9Cr-2WVTa was attributed to the tantalum in solution, and the increase in DBTT with irradiation was thought to be caused by a loss of the tantalum from solution.

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