

## HEAT TREATMENT EFFECTS ON IMPACT TOUGHNESS OF 9Cr-1MoVNb AND 12Cr-1MoVW STEELS IRRADIATED TO 100 dpa—R. L. Klueh and D. J. Alexander (Oak Ridge National Laboratory)

### OBJECTIVE

The goal of this study is to evaluate the impact behavior of irradiated ferritic steels and relate the changes in properties to the heat treatment of the steel.

### SUMMARY

Plates of 9Cr-1MoVNb and 12Cr-1MoVW steels were given four different heat treatments: two normalizing treatments were used and for each normalizing treatment two tempers were used. Miniature Charpy specimens from each heat treatment were irradiated to  $\approx 19.5$  dpa at  $365^\circ\text{C}$  and to  $\approx 100$  dpa at  $420^\circ\text{C}$  in the Fast Flux Test Facility (FFTF). In previous work, the same materials were irradiated to 4-5 dpa at  $365^\circ\text{C}$  and 35-36 dpa at  $420^\circ\text{C}$  in FFTF. The tests indicated that prior austenite grain size, which was varied by the different normalizing treatments, had a significant effect on impact behavior of the 9Cr-1MoVNb but not on the 12Cr-1MoVW. Tempering treatment had relatively little effect on the shift in DBTT for both steels. Conclusions are presented on how heat treatment can be used to optimize impact properties.

### PROGRESS AND STATUS

#### Introduction

Neutron irradiation effects on the toughness of ferritic/martensitic steels is a prime concern for steels considered for fusion reactor applications. Irradiation at temperatures up to  $\approx 450^\circ\text{C}$  can cause increases in the ductile-brittle transition temperature (DBTT) and decreases in the upper shelf energy (USE), as determined by a Charpy impact test. Heat treatment variations affect the kind of microstructure (e.g., prior austenite grain size, dislocation structure, and the character of the precipitates) developed in the steels, and microstructure affects the mechanical properties, such as the impact behavior.

This paper examines how heat treatment and irradiation to high doses affect the Charpy impact behavior of the 9Cr-1MoVNb (modified 9Cr-1Mo) and 12Cr-1MoVW (Sandvik HT9) steels that have been considered in the past as candidate alloys for fusion reactor applications. Although these two steels are no longer prime candidates for fusion applications, the results are important, because similar behavior should occur in the ferritic/martensitic reduced-activation steels that are now being considered for fusion. Results for these steels with similar heat treatments were previously presented after 4-5 dpa at  $365^\circ\text{C}$  and 35-36 dpa at  $420^\circ\text{C}$  [1]. In this paper the results are extended to irradiation of  $\approx 20$  dpa at  $365^\circ\text{C}$  and  $\approx 100$  dpa at  $420^\circ\text{C}$ .

#### Experimental Procedure

The 9Cr-1MoVNb steel was from an argon-oxygen decarburized (AOD) and electroslag-remelted (ESR) heat (heat 30176) processed by Carpenter Technology into 25.4-mm-thick plate. The 12Cr-1MoVW steel (Sandvik HT9 composition) was from an AOD/ESR melt that was processed into hot-rolled plate (heat 9607-R2) by Universal Cyclops. Compositions for the test materials fall within the specifications for the steels and have been published [1]. Sections of the 25.4-mm plate were rolled to 9.5-mm thickness, and pieces of these plates measuring 88.9 by 152 by 9.5 mm were normalized and tempered. Two plates of 9Cr-1MoVNb and two plates of 12Cr-1MoVW were austenitized 1 h at  $1040^\circ\text{C}$  in air and air cooled; two plates of each steel were also austenitized 1 h at  $1100^\circ\text{C}$  and air cooled. Then one plate with each normalization treatment was tempered 1 h at  $760^\circ\text{C}$  or 2.5 h at  $780^\circ\text{C}$ .

Subsize Charpy specimens measuring 3.3 by 3.3 by 25.4 mm with a 0.51-mm-deep 30° V-notch and a 0.05-to 0.08-mm-root radius were taken from the center of the normalized-and-tempered plates along the rolling direction with the notch running transverse to the rolling direction (L-T orientation). Specimens were irradiated in the Fast Flux Test Facility (FFTF) in the Materials Open Test Assembly (MOTA).

For the 420°C irradiations, specimens austenitized at 1040°C and tempered at 760 and 780°C were used. Six Charpy specimens from each heat-treated condition were irradiated to nominal fluences of  $2.26 \times 10^{27}$  n/m<sup>2</sup> for the 12Cr-1MoVW steel to produce  $\approx 99.5$  dpa and  $2.28 \times 10^{27}$  n/m<sup>2</sup> for the 9Cr-1MoVNb steel to produce  $\approx 100.4$  dpa (the doses of both will be referred to as 100 dpa). Irradiations at 365°C were on specimens austenitized at 1040 and 1100°C and tempered at 760 and 780°C; they were irradiated to a nominal fluence of  $5.12 \times 10^{22}$  n/m<sup>2</sup>,  $\approx 19.5$  dpa (referred to as 20 dpa).

Details on the test equipment and the procedure for testing the subsize Charpy specimens have been published [2]. Individual Charpy data sets were fitted with a hyperbolic tangent function to obtain transition temperatures and upper shelf energies. Transition temperatures were determined at half the upper shelf energy.

## Results

Specimens with all four heat treatments were irradiated at 365°C, but only specimens austenitized at 1040°C were irradiated at 420°C. Irradiation caused an increase in DBTT and a decrease in USE for all heat-treated conditions (Table 1). Only relatively minor differences were observed between the properties of the steels irradiated at 365°C to 4-5 dpa [1] and 20 dpa (Fig. 1) and between those irradiated at 420°C to 35-36 dpa [1] and 100 dpa (Fig. 2). For all heat treatments, the shift in DBTT ( $\Delta$ DBTT) for the 12Cr-1MoVW steel was over twice that for the 9Cr-1MoVNb steel (Fig. 3).

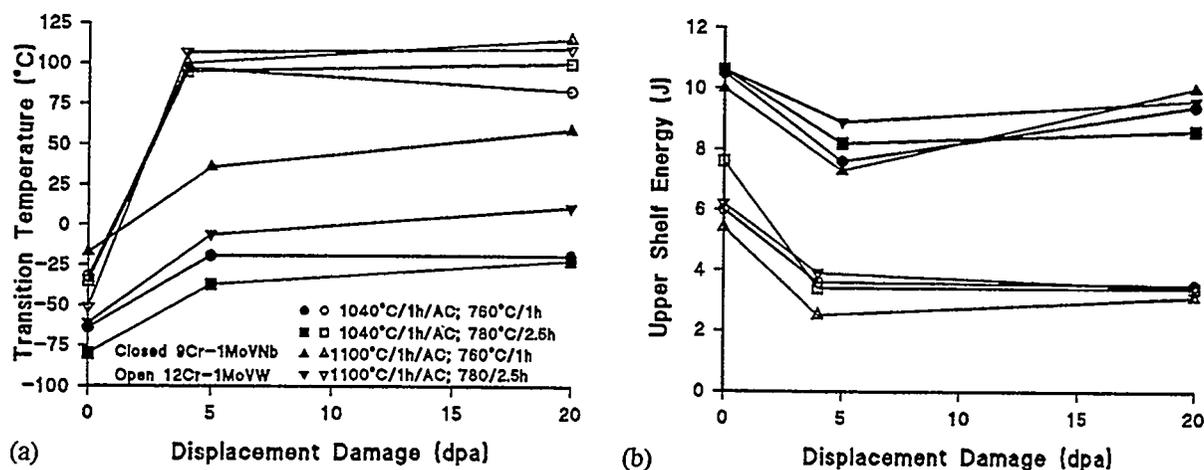


Fig. 1 (a) DBTT and (b) USE as a function of displacement damage for 9Cr-1MoVNb and 12Cr-1MoVW steels with four different heat treatments after irradiation at 365°C in FFTF.

Table 1. Impact properties of 9Cr-1MoVNb and 12Cr-1MoVW irradiated at 365 and 420°C

Heat Treatment <sup>a</sup>	DBTT <sup>b</sup> , °C			USE, J		
	Unirr	4/5 dpa	20 dpa	Unirr	4/5 dpa	20 dpa
<u>9Cr-1MoVNb Steel—365°C</u>						
1040/1h;760/1h	-64	-19	-19	10.5	7.6	9.4
1040/1h;780/2.5h	-80	-37	-22	10.6	8.2	8.6
1100/1h;760/1h	-17	36	59	10.0	7.3	10.0
1100/1h;780/2.5h	-61	-6	11	10.6	8.9	9.6
<u>12Cr-1MoVW Steel—365°C</u>						
1040/1h;760/1h	-32	97	83	6.0	3.6	3.5
1040/1h;780/2.5h	-35	95	100	7.6	3.4	3.4
1100/1h;760/1h	-34	100	115	5.4	2.5	3.1
1100/1h;780/2.5h	-51	107	109	6.2	3.9	3.4
	Unirr	35/36 dpa	100 dpa	Unirr	35/36 dpa	100 dpa
<u>9Cr-1MoVNb Steel—420°C</u>						
1040/1h;760/1h	-64	-25	-30	10.5	8.2	7.9
1040/1h;780/2.5h	-80	-35	-32	10.6	7.8	9.0
<u>12Cr-1MoVW—420°C</u>						
1040/1h;760/1h	-32	55	54	6.0	4.1	5.3
1040/1h;780/2.5h	-35	72	42	7.6	4.1	4.2

<sup>a</sup> Steels were air cooled after the 1040 and 1100°C austenitization; temperatures are in °C.

<sup>b</sup> DBTT was determined at ½ the upper shelf energy.

The largest variation caused by the heat treatment occurred for the 9Cr-1MoVNb steel irradiated at 365°C. The plates of 9Cr-1MoVNb austenitized at 1100°C had the highest DBTT before irradiation [Fig 1(a)] and developed the largest  $\Delta$ DBTT [Fig. 3(a)] after irradiation to 5 and 20 dpa at 365°C. Of the two 9Cr-1MoVNb plates austenitized at 1100°C, the one tempered at 780°C had the lowest transition temperature before and after irradiation. The two plates austenitized at 1040°C had lower transition temperatures than after the 1100°C heat treatment. Tempering at 780°C gave a slight advantage in the unirradiated condition and after 5 dpa, but there was no difference after 20 dpa. Fig. 3(a) shows that the  $\Delta$ DBTT for the 9Cr-1MoVNb steel irradiated at 365°C was different for the two different austenitization treatments, but there was little effect of the tempering treatment.

Much less difference in the transition temperatures was observed for the 12Cr-1MoVW steel irradiated at 365°C [Fig 1(a)]. The plate given the 1100°C austenitization and the 780°C temper

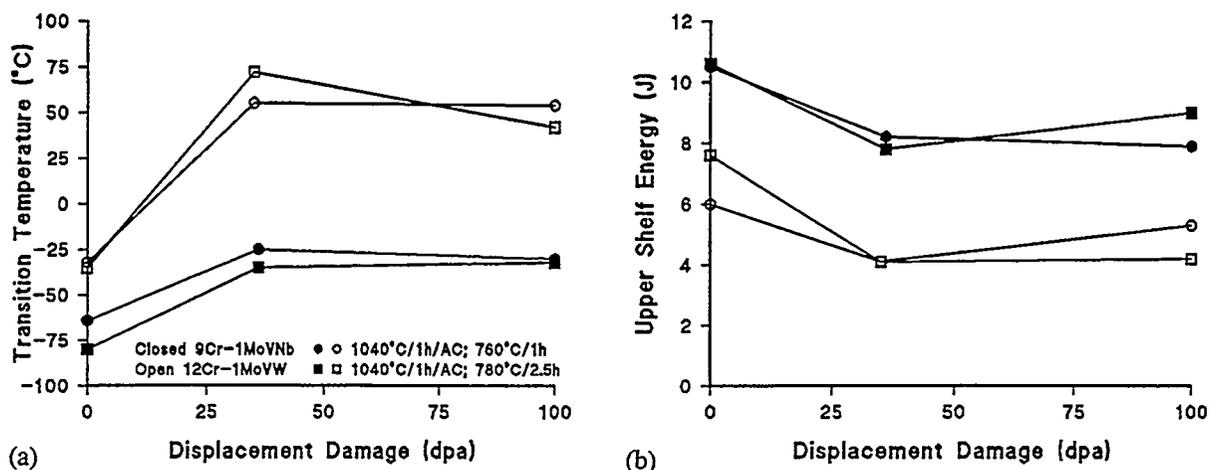


Fig. 2 (a) DBTT and (b) USE as a function of displacement damage for 9Cr-1MoVNb and 12Cr-1MoVW steels with two different heat treatments after irradiation at 420°C in FFTF.

had the lowest transition temperature before irradiation, with little difference being observed for the transition temperatures for the other three heat treatments. After irradiation to 20 dpa at 365°C, specimens with the 1100°C austenitization treatment had the highest transition temperatures, with the plate austenitized at 1040°C and tempered at 760°C having the lowest transition temperature. The variation in transition temperatures was greatest for the 12Cr-1MoVW steel plates after irradiating to 20 dpa at 365°C. This greater variation in properties with heat treatment is also evident for the shift in transition temperature [Fig. 3(a)].

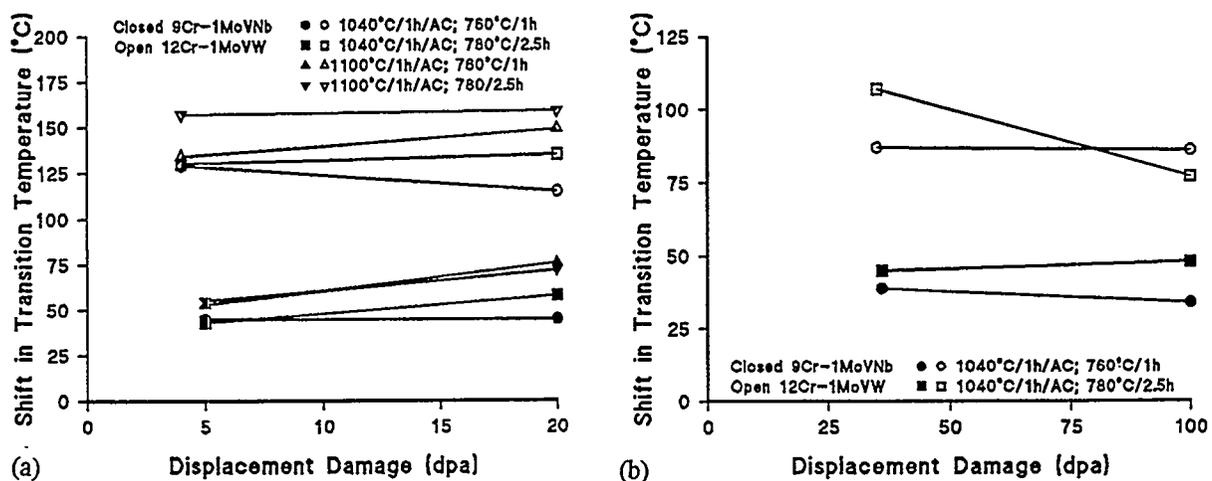


Fig. 3 Shift in DBTT as a function of displacement damage for 9Cr-1MoVNb and 12Cr-1MoVW steels (a) with four different heat treatments after irradiation at 365°C in FFTF and (b) with two different heat treatments after irradiation at 420°C in FFTF.

Both before and after irradiation at 365°C, the USE for the 9Cr-1MoVNb steel was much less dependent on heat treatment than was the DBTT, although there was somewhat more variation after irradiation than before [Fig. 1(b)]. After 20 dpa, the 9Cr-1MoVNb plates austenitized at 1040°C showed the smallest  $\Delta$ DBTT. In the case of the 12Cr-1MoVW steel, there was a relatively large variation in USE before irradiation and after 5 dpa, but much less variation after 20 dpa at 365°C [Fig. 1(b)].

Only plates austenitized at 1040°C were irradiated at 420°C (Fig. 2). Irradiation at 420°C caused an increase in the transition temperature for both the 9Cr-1MoVNb and 12Cr-1MoVW steels, with the  $\Delta$ DBTT for the 12Cr-1MoVW steel being about twice that for the 9Cr-1MoVNb steel [Fig. 3(b)]. For neither steel was there a large effect of tempering temperature on the transition temperature, either in the unirradiated condition or after 35-36 dpa and 100 dpa [Fig. 2(a)]. Likewise, tempering temperature caused little difference on the USE for the steels in the unirradiated and irradiated conditions [Fig. 2(b)]. Although the differences were not great, the  $\Delta$ DBTT for the 9Cr-1MoVNb tempered at 780°C was slightly greater than after tempering at 760°C [Fig. 3(b)]. Slightly more scatter occurred for the  $\Delta$ DBTT of the 12Cr-1MoVW steel with the different tempering conditions [Fig. 3(b)].

### Discussion

The new higher fluence results presented in this report generally confirm the conclusions reached previously [1] that heat treatment can affect properties after irradiation—especially the transition temperature and the shift in transition temperature. In the previous paper [1], the results were compared to results from other investigators who investigated the effect of heat treatment in the normalized-and-tempered [3] and irradiated [4] conditions. Those comparisons will not be repeated, since the conclusions reached previously are not changed by the results presented here.

The objective of using different austenitizing temperatures was to change the prior-austenite grain size. Heat treatment had a relatively small effect on the prior-austenite grain size of the 9Cr-1MoVNb steel, but it had a larger effect on the 12Cr-1MoVW steel: the average grain size after austenitizing at 1040 and 1100°C was estimated at 16 and 22  $\mu$ m, respectively, for the 9Cr-1MoVNb and 22-45 and 90-124  $\mu$ m, respectively, for the 12Cr-1MoVW [1]. The smaller prior austenite grain size variation with heat treatment for the 9Cr-1MoVNb steel was attributed to the strong effect of niobium on restricting grain growth of the austenite during the austenitization treatment [5]. Despite the relatively small variation in grain size for the 9Cr-1MoVNb, it still showed a larger variation in transition temperature than the 12Cr-1MoVW [Table 1 and Fig. 1(a)].

Austenite grain size can affect the transition temperature: transition temperature generally increases with increasing grain size [6], which was observed for the 9Cr-1MoVNb. For a given grain size (austenitization temperature), tempering at the higher temperature gave a lower transition temperature, which is also expected, because the higher tempering temperature lowers the strength.

In the previous paper, impact properties for the steels thermally aged at 400°C for 5000, 10000, and 20000 h were presented [1]. Charpy properties were little changed after thermal aging for 20000 h [1]. Although the exposure in the reactor at 420°C was somewhat greater than 20000 h, the most significant changes caused by aging were slight decreases in DBTT and increases in USE for the 9Cr-1MoVNb steel austenitized at 1100°C and tempered at 760°C [1]. These changes are opposite to the effects observed during irradiation. Thus, any properties degradation observed following irradiation cannot be attributed to the thermal aging that occurred simultaneously with irradiation.

The relative differences in DBTT of the 9Cr-1MoVNb steel that were present in the normalized-and-tempered condition remained after 5 dpa [Fig. 1(a)]. After 20 dpa, the difference for the plates

austenitized at 1100°C remained, but there was little difference in the DBTT for the two plates austenitized at 1040°C [Fig. 1(a)]. Thus, grain size eventually determined the DBTT for the steel austenitized at 1040°C. In the case of the 9Cr-1MoVNb plate austenitized at 1100°C, the properties did not converge for the two different tempering treatments. However, the  $\Delta$ DBTT for the different tempering conditions did show convergence [Fig. 3(a)]. These results indicate that, at least for the 9Cr-1MoVNb and steels like it, it may be possible to improve the irradiation resistance of the impact properties by the thermo-mechanical treatment. For the 9Cr-1MoVNb steel, the 1h at 760°C temper was the optimum temper determined when the steel was developed [7]. As this work indicates, raising that temperature, which would lower the strength, would not improve the impact properties after irradiation. It would be interesting to determine whether a further reduction in the prior-austenite grain size would improve the post-irradiation impact properties.

Although the 12Cr-1MoVW steel showed a somewhat larger variation in prior-austenite grain size than the 9Cr-1MoVNb steel, it showed a smaller variation in transition temperature for the four heat treated conditions in both the normalized-and-tempered condition and after irradiation at 365°C. It showed the most variation after 20 dpa, where the specimens with the smallest prior-austenite grain size again had the lowest transition temperature [Fig. 1(a)]. The  $\Delta$ DBTT had a similar, though larger, variation [Fig. 3(a)].

Previously the difference in fracture behavior of the 12Cr-1MoVW steel relative to the 9Cr-1MoVNb steel was attributed to the larger volume of carbide precipitates, mainly  $M_{23}C_6$  [1]. The 12Cr-1MoVW contains 0.2% C, compared to 0.1% C for the 9Cr-1MoVNb, and thus, the 12Cr-1MoVW contains over twice as much precipitate [8]. Precipitates were postulated to minimize the role of the grain boundaries for the 12Cr-1MoVW [1], and these precipitates could affect the fracture process because the larger, brittle precipitate particles in the 12Cr-1MoVW steel could cause a larger initial crack size for crack initiation during fracture. Carbide particles are believed to be a source of cracks in steels [9,10]. Possible confirmation of this is the relative behavior of the DBTT [Fig. 1(a)] and  $\Delta$ DBTT [Fig. 3(a)] with different heat treatments. Although the 12Cr-1MoVW plates with the smallest grain size had the lowest DBTT after 20 dpa, the effect of tempering temperature was different from what was expected: the steel tempered at 780°C had the higher DBTT and  $\Delta$ DBTT. The opposite is expected, because under most conditions, a higher tempering temperature reduces the strength, which should improve fracture properties [5,6]. However, the higher tempering temperature will also produce larger precipitate particles, thus enhancing fracture. Note that the opposite effect occurs for the 9Cr-1MoVNb steel [Fig. 1(a)], which contains the smaller particles [1,8]. The results indicate that the tempering treatment of 2 h at 780°C that is often used for the 12Cr-1MoVW steel could be replaced by shorter times at a lower temperature, thus providing an improved strength without a reduction in toughness.

The observations on the transition temperature after irradiation at 420°C indicate that for the 9Cr-1MoVNb steel, the saturation with fluence that occurs is independent of the tempering conditions [Fig. 2(a)]. A somewhat similar conclusion follows for the 12Cr-1MoVW steel. Here, it appears that after 35 dpa, and the steel tempered at 780°C with the larger precipitate particles has the highest DBTT. However, after 100 dpa, the two converge. This probably means that the particles in the plate tempered at 760°C reached a size where the further irradiation-enhanced growth does not affect fracture properties, thus giving the plates tempered at 760 and 780°C a similar DBTT.

The change in the USE with respect to heat treatment and irradiation appears more random than the transition temperature. In most cases, the USE values after 20 dpa at 365°C or 100 dpa at 420°C irradiations were equal to or greater than those after the previous irradiations. The relatively small change in USE for the 9Cr-1MoVNb steel up to 100 dpa at 420°C and 20 dpa at 365°C shows the superior behavior of this steel. The 9Cr-1MoVNb steel has a higher USE than that for the 12Cr-1MoVW steel in the normalized-and-tempered condition, thus making the relative change considerably less. After all irradiations, the USE of the 9Cr-1MoVNb steel remained higher than the USE of the 12Cr-1MoVW steel in the unirradiated condition.

The superiority of the 9Cr-1MoVNb steel is probably a reflection of the larger carbon concentration of the 12Cr-1MoVW steel. A high carbon concentration is required in the 12Cr-1MoVW steel to avoid  $\delta$ -ferrite formation during normalization. This was alluded to in the previous paper [1] when the 12Cr-1MoVW was compared to a 12Cr-0.9Mo-0.3V-0.14C steel of Little et al. [3], which showed a significant effect of austenitizing temperature on impact properties. The main difference between the steel of Little et al. and the 12Cr-1MoVW steel of the present study involves the carbon. Based on the microstructural studies described by Little et al. [3], it was concluded [1] that their steel showed a much larger prior austenite grain size effect than the 12Cr-1MoVW steel because of the lower carbon content in the steel of Little et al. [3], which caused a much finer precipitate distribution to form. This implies that the DBTT of the 12Cr-1MoVW could be affected significantly by lowering the carbon content, but to accomplish this, other alloying modifications would be required to avoid  $\delta$ -ferrite formation during normalization.

The results indicate that the change in Charpy properties with irradiation dose saturates, and saturation is achieved by the lower dose used in these experiments. The shift in DBTT is related to hardening, which is measured as an increase in yield stress. Hardening is also generally thought to saturate with fluence. For 9Cr-1MoVNb and 12Cr-1MoVW steel irradiated in the Experimental Breeder Reactor (EBR-II), little or no change in strength occurred between specimens irradiated to 9-13 and 23-25 dpa [11]. Likewise, a saturation in DBTT occurred for these two steels irradiated to 13 and 26 dpa in EBR-II [12]. However, for a series of Cr-W-V-Ta steels with 2.25, 7, 9, and 12 % Cr irradiated to 25, 35 and 60 dpa at 400°C in FFTF, hardening went through a maximum [13]. A similar observation was made on an 8Cr-2WVTa steel (F82H) irradiated at 400°C to 12, 21, and 34 dpa in the High Flux Isotope Reactor (HFIR) [14]. Khabarov et al. [15] observed a peak in strength for the Russian steel 13Cr2MoNbVB after irradiation in the BN-350 reactor over the range 4 to 85 dpa at 350-365°C.

One explanation for the maximum in strength with fluence is that irradiation-enhanced softening offsets part of the irradiation hardening [13-15]. This would not be completely unexpected, since thermal aging will cause a reduction of strength due to carbide coarsening and dislocation recovery. However, this should only occur after extremely long aging times at a temperature as low as 400°C [16], although it could be accelerated by irradiation.

The shift in DBTT is related to the hardening, but there do not appear to be any results that show a maximum in the DBTT or shift in DBTT with fluence. In the same experiment where Khabarov et al. [15] found a maximum in yield stress with dose for the 13Cr1MoNbVB irradiated in BN-350, no maximum was observed for the DBTT. Likewise, there was no indication of a maximum in the shift in DBTT for the 9Cr-1MoVNb and 12Cr-1MoVW steels irradiated in the present experiment, even after 100 dpa.

## SUMMARY AND CONCLUSIONS

Different normalizing-and-tempering treatments were used on 9Cr-1MoVNb and 12Cr-1MoVW steels to study the effect of heat treatment on Charpy impact toughness before and after irradiation. Plates of the steels were austenitized at two temperatures (1040 and 1100°C) to vary the prior austenite grain size, and two tempering treatments (1 h at 760 and 2 h at 780°C) were used for each austenitizing temperature. Subsize Charpy specimens were tested in each heat treated condition before irradiation and after irradiation in FFTF at 365°C to  $\approx$ 20 dpa; two heat-treated conditions were also tested after irradiation in FFTF at 420°C to  $\approx$ 100 dpa. Previously these same materials had been irradiated to 4-5 dpa at 365°C and 35-36 dpa at 420°C.

As normalized and tempered, the DBTT of the 9Cr-1MoVNb steel depended on the austenitizing temperature (prior austenite grain size) and on the tempering conditions. The shift in DBTT caused by irradiation for this steel was relatively independent of heat treatment, which meant that after irradiation the relative difference in DBTT for the steel given the different heat treatments was similar to what it was before irradiation. These observations suggest that to insure a low DBTT for 9Cr-1MoVNb after irradiation, it

should be heat treated to produce a low DBTT before irradiation. The best method to do this is by reducing the prior austenite grain size.

Austenitization temperature, and thus prior austenite grain size, had less effect on the transition temperature of the normalized-and-tempered and the irradiated 12Cr-1MoVW steel than the 9Cr-1MoVNb steel. Tempering treatment also had a small effect. The shift in DBTT was relatively independent of heat treatment, but the shifts for the 12Cr-1MoVW steel were over twice those for 9Cr-1MoVNb steel. Therefore, it does not appear possible to use heat treatment to reduce the effect of irradiation on the DBTT of 12Cr-1MoVW. Because of the lack of a heat treatment effect on DBTT, however, it may be possible to use this steel without tempering to the low strength levels at which the steel is usually used.

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