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Heat treatment effects on impact toughness of 9Cr–1MoVNb and 12Cr–1MoVW steels irradiated to 100 dpa [1]

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Abstract

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 ≈20 dpa at 365°C and to ≈100 dpa at 420°C in the Fast Flux Test Facility (FFTF). In previous work, the same steels were irradiated in FFTF to 4–5 dpa at 365°C and 35–36 dpa at 420°C. The tests indicated that prior austenite grain size, which was varied by the different normalizing treatments, affected the impact behavior of the 9Cr–1MoVNb but not the 12Cr–1MoVW. Tempering had relatively little effect on the impact behavior of both steels. Conclusions are presented on how heat treatment can be used to optimize impact properties. © 1998 Elsevier Science B.V. All rights reserved.

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1. Introduction

This paper examines how heat treatment and irradiation to high doses affect the Charpy impact behavior of the 9Cr–1MoVNb and 12Cr–1MoVW steels that have been considered in the past as candidate alloys for fusion reactor applications. Charpy results for these steels with similar heat treatments were previously presented after 4–5 dpa at 365°C and 35–36 dpa at 420°C [1]. In this paper the results are extended to ≈20 dpa at 365°C and ≈100 dpa at 420°C.

2. Experimental procedure

Two plates ($88.9 \times 152 \times 9.5$ mm^3) each of 9Cr–1MoVNb (modified 9Cr–1Mo) steel and 12Cr–1MoVW (Sandvik HT9) steel were austenitized 1 h at 1040°C in air and air cooled; two similar plates of each steel were also austenitized 1 h at 1100°C and air cooled. One of the two plates with each normalization treatment was tempered 1 h at 760°C and one at 2.5 h at 780°C. Details on chemical composition and the processing of the two steels have been published [1].

Subsize Charpy specimens measuring $3.3 \times 3.3 \times 25.4$ mm^3 with a 0.51-mm-deep 30° V-notch and a 0.05–0.08-mm root radius were taken from the center of the normalized-and-tempered plates in the rolling direction with the notch running transverse to the rolling direction (L–T orientation). Specimens were irradiated at 365°C and 420°C in the Fast Flux Test Facility (FFTF). At 365°C, specimens with all four heat treated conditions (austenitized at 1040°C and 1100°C and tempered at 760°C and 780°C) were irradiated to a nominal fluence of 5.12×10^{22} n/m^2, ≈19.5 dpa (referred to as 20 dpa). For the 420°C irradiations, only specimens austenitized at 1040°C and tempered at 760°C and 780°C were irradiated. Nominal fluences at 420°C were 2.28×10^{27} n/m^2, ≈100.4 dpa, for the 9Cr–1MoVNb steel and 2.26×10^{27} n/m^2, ≈99.5 dpa, for the 12Cr–1MoVW steel (the doses will be referred to as 100 dpa).

Six Charpy specimens from each normalized-and-tempered condition were irradiated. Details on the test equipment and the procedure for testing the subsize Charpy specimens have been published [2]. Transition

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temperatures were determined at half the upper shelf energy.

3. Results

Irradiation caused an increase in the ductile–brittle transition temperature (DBTT) and a decrease in the upper-shelf energy (USE) for all heat-treated conditions (Table 1) at both irradiation temperatures (Figs. 1–3).

The largest variation in Charpy properties due to heat treatment was for the 9Cr–1MoVNb steel irradiated at 365°C (Fig. 1(a)), although the 12Cr–1MoVW steel developed the largest shift in DBTT (ΔDBTT) (Fig. 3(a)). Both before and after irradiation to 5 and 20 dpa at 365°C, the 9Cr–1MoVNb steel specimens austenitized at 1040°C had a significantly lower DBTT than those austenitized at 1100°C (for the same tempering condition) (Fig. 1(a)). Of the two 9Cr–1MoVNb steel plates austenitized at 1100°C, specimens from the one tempered at 780°C had the lowest transition temperature before and after irradiation at 365°C. For the specimens austenitized at 1040°C and irradiated at 365°C, those tempered at 780°C had a slight advantage (lower DBTT) over those tempered at 760°C after the 5 dpa irradiation, but there was no difference after 20 dpa (Fig. 1(a)). The ΔDBTTs for the 9Cr–1MoVNb steel irradiated at 365°C were different for the different austenitization treatments, with the steel austenitized at 1040°C having the smallest ΔDBTT (Fig. 3(a)). There was little effect of the tempering treatment on ΔDBTT.

Much less difference in the transition temperatures was observed for the 12Cr–1MoVW steel than the 9Cr–1MoVNb steel. This was true for both the normalized-and-tempered condition and after irradiation at 365°C (Fig. 1(a)). After irradiation to 20 dpa at 365°C, the 12Cr–1MoVW steel 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 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)).

Only plates austenitized at 1040°C were irradiated at 420°C, where irradiation caused an increase in the transition temperature for both the 9Cr–1MoVNb and 12Cr–1MoVW steels (Fig. 2(a)), with the ΔDBTT 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. Although the differences were not great, the ΔDBTT for the 9Cr–1MoVNb tempered at 780°C was slightly greater than after tempering at 760°C (Fig. 3(b)). More scatter occurred for the ΔDBTT of the 12Cr–1MoVW steel for the different tempering conditions when the results for the two irradiation fluences are examined (Fig. 3(b)).

Both before and after irradiation at 365°C, the change in USE for the 9Cr–1MoVNb steel was much

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

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

[a] Steels were air cooled after the 1040°C and 1100°C austenitization; temperatures are in °C.

[b] DBTT was determined at $\frac{1}{2}$ the upper shelf energy.

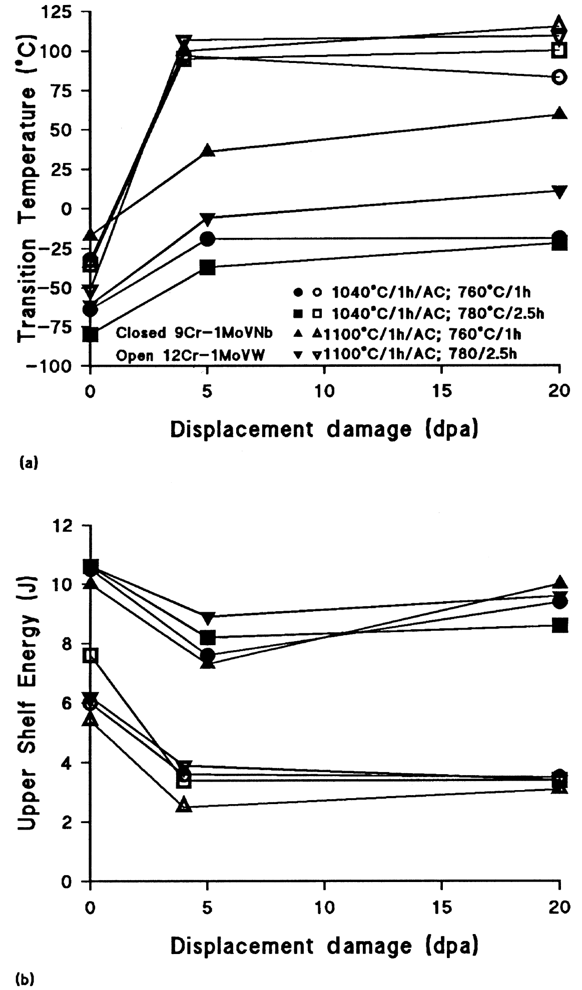

Fig. 1. (a) Ductile–brittle transition temperature and (b) upper-shelf energy as a function of displacement damage for 9Cr–1MoVNb and 12Cr–1MoVW steels with four different heat treatments after irradiation at 365°C in FFTF.

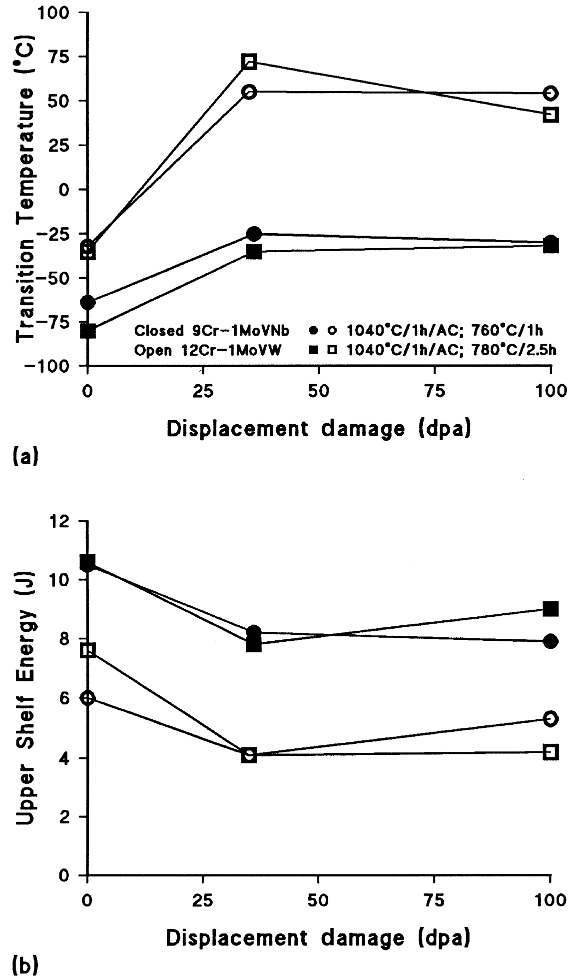

Fig. 2. (a) Ductile–brittle transition temperature and (b) upper-shelf energy as a function of displacement damage for 9Cr–1MoVNb and 12Cr–1MoVW steels with two different heat treatments after irradiation at 420°C in FFTF.

less dependent on heat treatment than was the DBTT, although there was somewhat more variation after irradiation at 356°C than before (Fig. 1(b)). In the case of the 12Cr–1MoVW steel, there was a relatively large variation in USE before irradiation and after 5 dpa, but very little variation after 20 dpa at 365°C (Fig. 1(b)). After irradiation at 420°C, tempering conditions had only a minor effect on the USE of 9Cr–1MoVNb and 12Cr–1MoVW steels (Fig. 2(b)).

4. Discussion

The new higher fluence results presented in this paper 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 on the lower-fluence irradiations [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. Although it is recognized that heat treatment can affect more than prior austenite grain size and precipitate character, these will be the only effects discussed here. In the future, we hope to examine other effects, such as lath and packet size and precipitate morphology and precipitate distribution.

The objective of using different austenitizing temperatures was to change the prior austenite grain size.

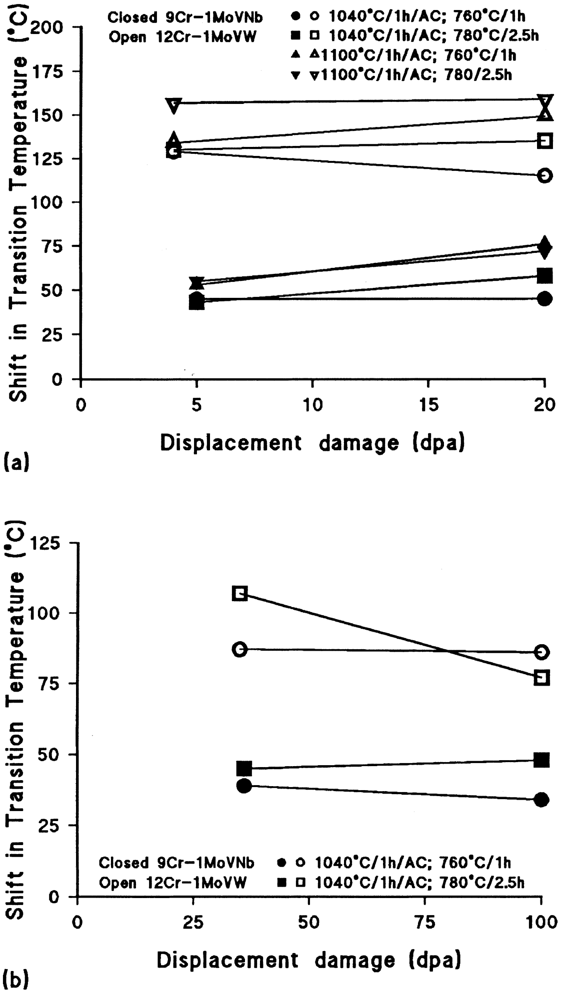

Fig. 3. Shift in ductile–brittle transition temperature as a function of displacement damage for 9Cr–1MoVNb and 12Cr–1MoVW steels (a) with four different heat treatments after irradiation at 365°C in FFTF and (b) with two different heat treatments after irradiation at 420°C in FFTF.

The austenitization temperature affected the prior austenite grain size: the average grain size after austenitizing at 1040°C and 1100°C was estimated at 16 and 22 μm, respectively, for the 9Cr–1MoVNb steel and 22–45 and 90–124 μm, respectively, for the 12Cr–1MoVW steel [1]. The smaller change in prior austenite grain size with heat treatment for the 9Cr–1MoVNb steel was attributed to the strong effect of niobium on restricting grain growth in the austenite during austenitization [5]. Austenite grain size can affect the Charpy transition temperature: transition temperature generally increases with increasing grain size [6].

Despite the relatively small variation in grain size for the 9Cr–1MoVNb steel, it still showed a larger variation in transition temperature than the 12Cr–1MoVW steel (Table 1 and Fig. 1(a)). For a given grain size (austenitization temperature), tempering at the higher temperature gave a lower transition temperature for the 9Cr–1MoVNb steel, which is expected, because the higher tempering temperature lowers the strength. There was much less effect of tempering on the 12Cr–1MoVW steel, which is discussed later.

In the previous paper, impact properties for the steels thermally aged at 400°C for 5000, 10 000, and 20 000 h were presented [1]. Little change in properties of the 9Cr–1MoVNb and 12Cr–1MoVW steels occurred after thermal aging for 20 000 h. Although the exposure in the reactor at 420°C was somewhat greater than 20 000 h, the most significant changes caused by aging at 400°C were the slight decreases in DBTT and slight 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 thermal aging that occurred simultaneously with irradiation.

The relative differences in DBTT of the 9Cr–1MoVNb steel with the different normalized-and-tempered treatments remained after 5 dpa at 365°C (Fig. 1(a)). After 20 dpa, the difference for the plates austenitized at 1100°C remained, but there was a convergence in the DBTT for the two plates austenitized at 1040°C (Fig. 1(a)). 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 ΔDBTT 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 that is used. For the 9Cr–1MoVNb steel, the 1 h 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, as indicated by these results.

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 ΔDBTT 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 in terms of DBTT and ΔDBTT was attributed to the larger volume of $M_{23}C_6$ precipitates in the 12Cr–1MoVW steel [1]. The 12Cr–1MoVW steel contains nominally 0.2% C, compared to 0.1% C for the 9Cr–1MoVNb steel, and thus the 12Cr–1MoVW steel contains over twice as much precipitate [8]. Precipitates were postulated to minimize the role of the grain boundaries for the 12Cr–1MoVW steel [1]. 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 ΔDBTT (Fig. 3(a)) with different heat treatments after irradiation at 365°C. The 12Cr–1MoVW steel plates with the smallest grain size had the lowest DBTT after 20 dpa, but the effect of tempering temperature was different from what was expected: the 12Cr–1MoVW steel tempered at 780°C had the higher 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 occurs for the 9Cr–1MoVNb steel (Fig. 1(a)), which contains the smaller particles [1,8]. The results indicate that the 2.5 h temper 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 DBTT after irradiation at 420°C indicate that for the 9Cr–1MoVNb steel, the saturation with fluence that occurs is essentially independent of the tempering conditions (Fig. 2(a)). A similar conclusion follows for the 12Cr–1MoVW steel, except the specimens tempered at 780°C appears to go through a maximum. That is, after 35 dpa, the steel tempered at 780°C with the larger precipitate particles has the highest DBTT, but after 100 dpa, the two converge. Previous work indicated that the precipitates grow during irradiation [11]. As applied to the present study, this probably means that particles in the plate tempered at 760°C reached a size during the 100 dpa irradiation where the further irradiation-enhanced growth does not affect fracture properties, thus giving the plates tempered at 760°C and 780°C a similar DBTT. This may also explain the apparent convergence of the DBTT for the 9Cr–1MoVNb steel after 100 dpa at 420°C (Fig. 2(a)) and 20 dpa at 365°C (Fig. 1(a)).

The change in the USE with heat treatment and irradiation appears more random than for the DBTT. In most cases, USE values after 20 dpa at 365°C or 100 dpa at 420°C 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 as high or higher than the USE of the 12Cr–1MoVW steel in the unirradiated condition.

The superiority of the 9Cr–1MoVNb steel is probably partly a reflection of the larger carbon concentration of the 12Cr–1MoVW steel. The precipitation of α' in this steel [11] could also play a role by causing more hardening. More carbon is required in 12Cr–1MoVW steel to avoid δ-ferrite during normalization. This was alluded to previously [1] when the 12Cr–1MoVW steel was compared to a 12Cr–0.9Mo–0.3V–0.14C steel [3], which showed a significant effect of austenitizing temperature on impact properties. The main difference between the latter steel and the 12Cr–1MoVW steel involves the carbon. Based on microstructural studies of the 12Cr–0.9Mo–0.3V–0.14C steel [3], it was concluded [1] that this steel showed a larger prior austenite grain size effect than the 12Cr–1MoVW steel because the lower carbon content in the 12Cr–0.9Mo–0.3V–0.14C steel caused a finer precipitate distribution to form. This implies that the DBTT of 12Cr–1MoVW could be affected by lowering carbon content, but to accomplish this, other alloying modifications would be required to avoid δ-ferrite during normalization.

The results indicate that the change in Charpy properties with irradiation dose saturates, and saturation occurs by the lower dose used in these experiments. The ΔDBTT 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 steels irradiated in the Experimental Breeder Reactor (EBR-II), little change in strength occurred between specimens irradiated to 9–13 and 23–25 dpa [12]. Likewise, a saturation in DBTT occurred for these two steels irradiated to 13 and 26 dpa in EBR-II [13]. 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 [14]. A maximum was also observed for 8Cr–2WVTa steel (F82H) irradiated at 400°C to 12, 21, and 34 dpa in the High Flux Isotope Reactor (HFIR) [15]. Khabarov et al. [16] found a peak in strength for 13Cr2MoNbVB steel irradiated to 4–85 dpa in the BN-350 reactor at 350–365°C.

One explanation for the maximum in strength with fluence is that irradiation-enhanced softening offsets part of the irradiation hardening [14–16]. This is not completely unexpected, since thermal aging will cause a reduction of strength due to carbide coarsening and dislocation recovery. However, this would only occur after extremely long aging times at a temperature as low as 400°C [17], although it could be accelerated by irradiation.

The ΔDBTT is related to hardening, and until recently [18], no results have been reported that show a maximum in the DBTT or ΔDBTT with fluence. In the experiment where Khabarov et al. [16] found a maximum in yield stress with dose for the 13Cr2MoNbVB, no maximum was observed for the DBTT. Recently, however, Kohno et al. [18] found a maximum in DBTT for a 9Cr–2W–0.2V–0.1Ta–0.1C steel irradiated in FFTF up to 60 dpa at 410°C. Evidence of a possible maximum in DBTT in the present experiment was seen for the 12Cr–1MoVW steel normalized at 1040°C and tempered 2.5 h at 780°C (Fig. 2(a)), although this could be due to scatter in the data.

5. Summary and conclusions

Different normalizing-and-tempering treatments were used on the 9Cr–1MoVNb and 12Cr–1MoVW steels to study the effect of heat treatment on Charpy impact toughness before and after irradiation in FFTF at 365°C to ≈20 dpa and at 420°C to ≈100 dpa. Previously these same materials were 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 and on the tempering conditions. The shift in DBTT caused by irradiation of this steel was relatively independent of heat treatment, and 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 steel 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. Although it is recognized that in addition to prior austenite grain size, heat treatment can also affect precipitate characteristics, martensite lath and packet size, and precipitate character. In this work, however, only prior austenite grain size and precipitate size were addressed.

Austenitization temperature, and thus prior austenite grain size, had less effect on the DBTT 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 ΔDBTTs 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 steel. 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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