CONF-911111--32 32

DE92 006592

HEAT TREATMENT EFFECTS ON TOUGHNESS OF 9Cr-1MoVNb AND 12Cr-1MoVW STEELS IRRADIATED AT 365°C*

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Abstract

The 9Cr-1MoVNb and 12Cr-1MoVW steels were austenitized at 1040 and 1100°C to produce different prior austenite grain sizes, after which they were given different tempering treatments (1 h at 760 or 2.5 h at 780°C). Subsize Charpy impact specimens from these materials were irradiated at 365°C up to 5 dpa. For 9Cr-1MoVNb steel in the unirradiated condition, the smaller the prior austenite grain size and the higher the tempering temperature, the lower the ductile-brittle transition temperature (DBTT). Regardless of the DBTT in the unirradiated condition, however, the DBTT shift for 9Cr-1MoVNb steel due to irradiation was the same for all heat treatments. This means heat treatment can be used to ensure a lower DBTT before and after irradiation. The 12Cr-1MoVW steel showed little effect of heat treatment on DBTT in the unirradiated condition, and the shift in DBTT was relatively constant. Thus, it appears that heat treatment cannot be used to reduce the effect of irradiation on DBTT for this steel.

*Research sponsored by the Office of Fusion Energy, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

1. Introduction

Ferritic/martensitic steels are relatively immune to void swelling and are being considered as possible structural materials for the first wall and blanket structure of future magnetic fusion reactors. A major concern involves the effect of irradiation on toughness. Toughness changes can be observed by studying the Charpy impact behavior before and after irradiation of steels in a fission reactor. Irradiation can cause a large increase in the ductile-brittle transition temperature (DBTT) and a decrease in the upper-shelf energy (USE). Even if the DBTT is suitably low before irradiation, it can be well above room temperature after irradiation [1-7].

Charpy impact behavior can be affected by heat treatment. Prior austenite grain size can affect the DBTT: generally, the smaller the grain size, the lower the DBTT. The prior austenite grain size depends on austenitization time and temperature. Tempering also affects the behavior: generally, the higher the tempering temperature or the longer the tempering time at a given temperature, the lower the DBTT and the higher the USE, although excessive time at high tempering temperatures can lead to grain growth and a reversal of the trend.

There do not appear to have been any studies on the effect of heat treatment on Charpy impact properties after irradiation for the high-chromium Cr-Mo steels of interest for fusion. In this experiment, Charpy impact specimens of 9Cr-1MoVNb (modified 9Cr-1Mo steel) and 12Cr-1MoVW (the Sandvik HT9 composition) steels were given different heat treatments and then irradiated in the Fast Flux Test Facility (FFTF). The results were compared with results for the same steels in the unirradiated condition.

2. Experimental Procedure

The 9Cr-1MoVNb steel was taken from an argon-oxygen decarburized (AOD) and electroslag-remelted (ESR) heat (Heat 30176) processed by Carpenter Technology into 1-in. plate. The 12Cr-1MoVW steel was from an AOD/ESR melt that was processed into hot-rolled plate (National Fusion Heat 9607-R2). Nominal composition (in weight percent) for 9Cr-1MoVNb is 9Cr-1Mo-0.2V-0.06Nb-0.1C, and for 12Cr-1MoVW is 12Cr-1Mo-0.25V-0.5W-0.5Ni-0.2C. Sections of 15.9-mm plate were rolled to 9.5-mm plate for heat treatment to obtain the specimens. Plates 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 for 1 h at 1040°C and air cooled, and a similar number of plates were austenitized for 1 h at 1100°C and air cooled. Then one plate from each steel with each normalization treatment was tempered 1 h at 760°C or 2.5 h at 780°C.

Subsize Charpy specimens essentially one-third the standard size measuring 3.3 by 3.3 by 25.4 mm with a 0.51-mm-deep 30° V-notch with 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 FFTF in the Materials Open Test Assembly (MOTA) in the MOTA 1E experiment. Six Charpy specimens from each heat and each heat treatment were irradiated in the below-core region of MOTA in a sodium "weeper" at ≈365°C, which is slightly above the coolant ambient temperature. All of the 9Cr-1MoVNb steel specimens were irradiated to a fluence of about 1.3 x 10^{26} n/m^2 (>0.1 MeV), which produces ≈5 dpa. The 12Cr-1MoVW steel specimens were in a slightly different position in the below-core region and received about 1.1 x 10^{26} n/m^2, ≈4 dpa, at 365°C. Less than 1 appm He formed in the specimens during irradiation.

Details on the test procedure for the subsize Charpy specimens have been published [8]. Individual Charpy data sets were fitted with a hyperbolic-tangent function to obtain the transition temperatures and upper-shelf energies.

3. Results and discussion

Prior austenite grain size varied with the austenitization temperature. Table 1 gives grain sizes estimated by optical microscopy. There was only a small difference in average grain diameter for the 9Cr-1MoVNb steel: 20 and 30 mm for the 1040 and 1100°C austenitizing temperatures, respectively. A larger difference was observed for 12Cr-1MoVW steel: 32 and 105 mm for 1040 and 1100°C, respectively. The much smaller amount of grain growth during austenitization of the 9Cr-1MoVNb steel is probably due to the niobium present in this steel.

Results for the Charpy tests for the steels in the unirradiated and irradiated conditions are given in Table 2. In addition to giving the DBTT and USE for both conditions, the shift in DBTT (ΔDBTT) and change in USE (ΔUSE) are also given.

For the 9Cr-1MoVNb steel, prior austenite grain size affected the DBTT of the unirradiated steel (Table 1). The prior austenite grain size effect can be seen by comparing DBTT values for the two austenitizing temperatures with a common tempering temperature. In both cases, the steel austenitized at 1040°C had the lower DBTT. A higher DBTT is seen after the 760°C temper than the 780°C temper (for a given austenitization temper), indicating that the 780°C temper has a greater effect than the 760°C temper. However, for the steel austenitized at 1100°C, tempering at 780°C has a relatively larger effect than it does for the steel austenitized at 1040°C. The USE of the 9Cr-1MoVNb steel in the unirradiated condition appears to be unaffected by heat treatment.

After the 9Cr-1MoVNb steel was irradiated to ~5 dpa, the ΔDBTTs for the four different heat treatments were only slightly different (Table 2). The ΔDBTT for the specimens austenitized at 1100°C (the ones with the largest grain size) were ~10°C higher, which may indicate a slight effect of austenite grain size. However, the tempering conditions had no effect on ΔDBTT. The ΔUSE is also fairly constant, although there appears to be a slight effect of tempering. That is, for a given austenitization temperature, the change in USE was slightly less after the 780°C temper.

The 12Cr-1MoVW steel in the unirradiated condition shows a somewhat different behavior from the 9Cr-1MoVNb steel (Table 2). Although a change in austenitization temperature produced a much larger difference in grain size for this steel than the 9Cr-1MoVNb steel, there was no apparent effect of prior austenite grain size on DBTT. The DBTT values were the same for the steel given the two different austenitizing treatments and then tempered at 760°C. There was a difference when the steels were tempered at 780°C, but the difference was opposite to that expected for a grain-size effect. The specimen austenitized at 1040°C and tempered at 780°C had a DBTT similar to that of the two steels tempered at 760°C, but the steel austenitized at 1100°C and tempered at 780°C had a somewhat lower DBTT (19°C lower). A higher DBTT is expected, however, for a prior austenite grain size effect. Therefore, this difference must be due to the higher tempering temperature. The USE of the 12Cr-1MoVW in the unirradiated condition appears to be affected slightly by tempering (Table 2). In each case, the steel tempered at 780°C had a higher USE than that at 760°C.

Irradiation of the 12Cr-1MoVW steel to ~4 dpa caused ΔDBTTs for all heat treatments that were over twice as large as those observed for the 9Cr-1MoVNb steel (Table 2). After irradiation, there was essentially no difference in DBTT for the four different heat treatments, indicating that heat treatment has relatively little effect on the DBTT of the 12Cr-1MoVW steel in either the unirradiated or irradiated condition. The USEs for all four conditions also appear similar after irradiation. The only noticeable difference is in the USE for the two steels austenitized at 1100°C, where the steel tempered at 780°C had a significantly higher USE than that tempered at 760°C.

The lack of a prior austenite grain size effect in 12Cr-1MoVW steel compared to the 9Cr-1MoVNb steel may be explained by assuming that the precipitate in the microstructure controls the fracture behavior of the 12Cr-1MoVW. This steel contains twice as much carbon as the 9Cr-1MoVNb steel, and in the normalized-and-tempered condition, the 12Cr-1MoVW contains over twice as much precipitate (3.8 wt% precipitate in the 12Cr-1MoVW compared to 1.5 wt% in 9Cr-1MoVNb) [9]. This difference in precipitation can be seen in Fig. 1, where carbide extraction replicas of the 9Cr-1MoVNb and 12Cr-1MoVW steels are shown after a similar normalizing-and-tempering treatment for both steels. (The majority of the precipitate in both steels was shown to be $M_{23}C_6$, with a small amount of MC [9].) These photomicrographs, taken from the work of Vitek and Klueh [9], show the large amount of precipitate in 12Cr-1MoVW steel, which would be expected to affect the fracture process. Furthermore, the precipitate in the 12Cr-1MoVW is distributed fairly uniformly within the prior austenite grains and therefore minimizes the role of the grain boundaries, while for the 9Cr-1MoVNb, precipitate is along prior austenite grain boundaries, which could enhance the role grain boundaries play in the fracture process.

The results indicate that for 9Cr-1MoVNb steel, heat treatment could be used to minimize the effect of irradiation on DBTT. This follows from the observation that the specimens with the smaller grain size had a lower DBTT in the unirradiated condition, and the ΔDBTT was similar regardless of the heat treatment. Thus, the lowest DBTT after irradiation is guaranteed by starting with the lowest possible DBTT before irradiation. Increasing tempering time is an additional route for increasing the initial DBTT. However, reduction of DBTT should be accomplished while at the same time keeping the strength as high as possible. Also, Table 2 shows that additional tempering has a minimal effect on DBTT for the small grain size. Therefore, lowering grain size further would be the best way to optimize strength and toughness, although as seen in Table 1, grain size changed only slightly with the austenitizing temperatures used for this study.

A different conclusion is reached for 12Cr-1MoVW steel. Here it appears that heat treatment has a much smaller effect on DBTT, and this effect is lost after irradiation. The results indicate that it would therefore be appropriate to use the lower tempering temperature, because this would improve the strength of the steel with little effect on toughness. The 780°C tempering temperature has been used extensively in the past, but 760°C (or less) should be acceptable.

Previous studies of 9Cr-1MoVNb and 12Cr-1MoVW steels irradiated in fast reactors indicated that the ΔDBTT saturated with increasing fluence [2,10]. Half-size Charpy specimens of 12Cr-1MoVW irradiated at 365°C to 10 and 17 dpa in FFTF resulted in a ΔDBTT of 161°C at both fluences (the steels were austenitized at 1050°C and tempered 2.5 h at 780°C) [10]. Third-size specimens (the type of specimen used in the present experiment) of this same steel were irradiated simultaneously at 365°C to 10 dpa, and the ΔDBTT was 151°C, essentially the same as for the half-size specimens, an indication that specimen size does not affect ΔDBTT [10]. Hu and Gelles [2] also found saturation when they irradiated half-size specimens of both 9Cr-1MoVNb and 12Cr-1MoVW steels at a higher temperature (390°C) in the Experimental Breeder Reactor (EBR-II). The ΔDBTTs for 9Cr-1MoVNb were 52 and 54°C after irradiation to 13 and 26 dpa, respectively, and for 12Cr-1MoVW shifts of 124 and 144°C were observed for the same fluences.

The magnitude of the ΔDBTT values for the 9Cr-1MoVNb and 12Cr-1MoVW steels observed in the present study is similar to those observed in previous studies [2,10]. Based on the previous results at 365°C, however, it appears that saturation was probably not achieved in the present experiment, where irradiation was to only about 5 and 4 dpa for the 9Cr-1MoVNb and 12Cr-1MoVW steels, respectively. Higher fluences are required to determine if the conclusions on austenite grain size and tempering apply to saturation. More specimens are presently under irradiation in FFTF to determine when saturation occurs and what kind of behavior is exhibited at higher fluences.

4. Summary and conclusions

The 9Cr-1MoVNb and 12Cr-1MoVW steels were austenitized to produce two different prior austenite grain sizes and then given two different tempering treatments. Subsize Charpy specimens were tested before and after irradiation in FFTF at 365°C. The 9Cr-1MoVNb was irradiated to ~5 dpa and the 12Cr-1MoVW to ~4 dpa. The following summarizes the observations and conclusions.

1. As normalized and tempered, the DBTT of the 9Cr-1MoVNb steel depended on the austenitizing (prior austenite grain size) and tempering temperatures. The shift in DBTT caused by irradiation for this steel was relatively independent of heat treatment. This 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.

2. Austenitization temperature, and thus prior austenite grain size, had little effect on the DBTT of the unirradiated 12Cr-1MoVW steel, and tempering temperature had only a slight effect. The shift in DBTT was again relatively independent of heat treatment, but the shifts for the 12Cr-1MoVW steel were over twice those for 9Cr-1MoVNb steel.

3. Observations on 9Cr-1MoVNb steel suggest that the effect of irradiation on DBTT can be ameliorated by reducing the prior austenite grain size.

4. From the results on 12Cr-1MoVW steel, no method is obvious to reduce the effect of irradiation on DBTT. However, because of the lack of a heat treatment effect on DBTT, it may be possible to use this steel without tempering to the low strength levels at which the steel is usually used.

Acknowledgments

Helpful discussions with Drs. J. M. Vitek and R. K. Nanstad are appreciated. The completion of this work was aided by E. T. Manneschmidt, J. J. Henry, and T. N. Jones, who tested the Charpy specimens, N. H. Rouse, who did metallography, and by F. A. Scarboro, who prepared the final manuscript.

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Table 1. Estimated Grain Sizes of 9Cr-1MoVNb and 12Cr-1MoVW Steels

Austenization Temperature °C	ASTM Grain Size Number[a]	Average Grain Diameter, mm
	9Cr-1MoVNb Steel	
1040	8	20
1100	7	32
	12Cr-1MoVW Steel	
1040	7	32
1100	3-4	153

[a]Estimated with grain size eyepiece.

Table 2. Effect of Irradiation at 365°C on Charpy Behavior of 9Cr-1MoVNb and 12Cr-1MoVW Steels

Heat Treatment	DBTT,[a] °C		USE, J		ΔDBTT	ΔUSE
	Unirrad	Irrad	Unirrad	Irrad	°C	J
			9Cr-1MoVNb Steel[b]			
1040/1h/AC + 760/1h	-64	-19	10.5	7.6	45	-2.9
1040/1h/AC + 780/2.5h	-80	-37	10.6	8.2	43	-2.4
1100/1h/AC + 760/1h	-17	36	10.0	7.3	53	-2.7
1100/1h/AC + 780/2.5h	-61	-6	10.6	8.9	55	-1.7
			12Cr-1MoVW Steel[c]			
1040/1h/AC + 760/1h	-32	97	6.0	3.6	129	-2.4
1040/1h/AC + 780/2.5h	-35	95	7.6	3.4	130	-4.2
1100/1h/AC + 760/1h	-34	100	5.4	2.5	134	-2.9
1100/1h/AC + 780/2.5h	-51	107	6.2	3.9	158	-2.3

[a]DBTT was determined at 1/2 the upper shelf.
[b]9Cr-1MoVNb was irradiated to ≈5 dpa.
[c]12Cr-1MoVW was irradiated to ≈4 dpa.

List of Figures

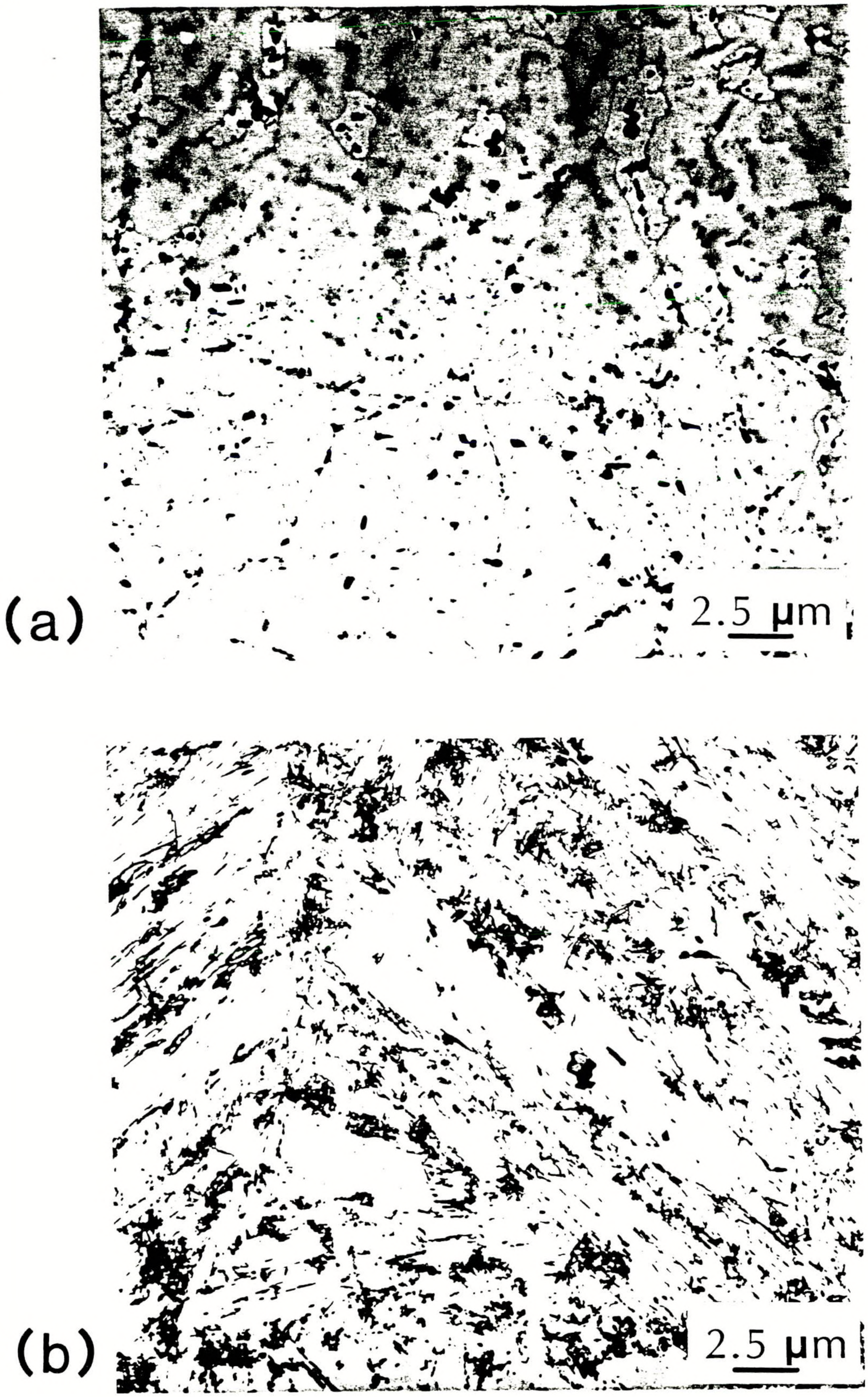

Fig. 1. Extraction replicas of normalized-and-tempered (1040°C/0.5h/AC + 780°C/2.5h/AC) (a) 9Cr-1MoVNb and (b) 12Cr-1MoVW steels.