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Effect of Heat Treatment and Tantalum on Microstructure and Mechanical Properties of Fe-9Cr-2W-0.25V Steel

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Abstract: An Fe-9Cr-2W-0.25V-0.07Ta-0.1C (9Cr-2WVTa) steel has a smaller prior-austenite grain size than this same composition without tantalum (9Cr-2WV) when the steels are given a similar heat treatment. Except for prior-austenite grain size, the microstructures of the steels are similar before irradiation, and they develop similar changes in microstructure during irradiation. Nevertheless, the 9Cr-2WVTa shows less effect of irradiation on the Charpy behavior. To determine the effect of grain size on the Charpy properties of the 9Cr-2WV and 9Cr-2WVTa, specimens of the two steels were given various normalization heat treatments to produce different prior austenite grain sizes, and the tensile and impact properties were determined. For the smaller prior-austenite grain sizes, the 9Cr-2WV steel had impact properties similar to or better than those of the 9Cr-2WVTa steel. Differences in the microstructures of the steels were used to explain the observations and what they mean for developing steels with improved properties.

Keywords: ferritic/martensitic steels, tensile properties, impact properties, prior-austenite grain size, heat treatment

Introduction

A reduced-activation steel with nominal composition (in wt %) Fe-9.0Cr-2.0W 0.25V-0.07Ta-0.1C (9Cr-2WVTa) developed for fusion reactor applications had excellent mechanical properties [*1*] and superior resistance to irradiation embrittlement as demonstrated by a relatively small increase (32ºC) in the ductile-brittle transition temperature ( ) DBTT) when irradiated to 28 dpa at 250-400ºC [*2-9*]. The impact properties of a similar composition but without the tantalum (9Cr-2WV) developed a shift almost twice as great (61ºC) when the steels were given the same normalizing-and-tempering heat treatment (austenitized at 1050ºC and tempered at 750ºC) (Fig. 1) [*2-5,9*].

The 9Cr-2WVTa steel had a smaller prior-austenite grain size after the 1050ºC austenitization treatment, but otherwise the microstructures were similar before

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irradiation and showed similar changes during irradiation. In addition to the difference in embrittlement behavior, irradiation behavior of the 9Cr-2WVTa steel differed from the 9Cr-2WV steel and most similar 8-12% Cr steels in two ways. First, the shift in DBTT of the 9Cr-2WVTa steel irradiated at . 400ºC did not saturate with fluence by 28 dpa, whereas for the 9Cr-2WV steel and most similar steels, saturation occurs at <10 dpa. This is shown in Fig. 1, and although the increase for 9Cr-2WVTa was gradual and small, it did appear to continue to increase with dose. The second difference was that the shift in DBTT for the irradiated 9Cr-2WVTa steel increased with increasing irradiation temperature (Fig. 1), whereas it decreased for the 9Cr-2WV steel, as it does for most similar steels [*4-6*]. In this case, because the DBTT of the 9Cr-2WV steel before irradiation was considerably higher than that of the 9Cr-2WVTa, after irradiation it was still higher than that of the 9Cr-2WVTa. However, they developed similar increases in DBTT during irradiation [*8*].

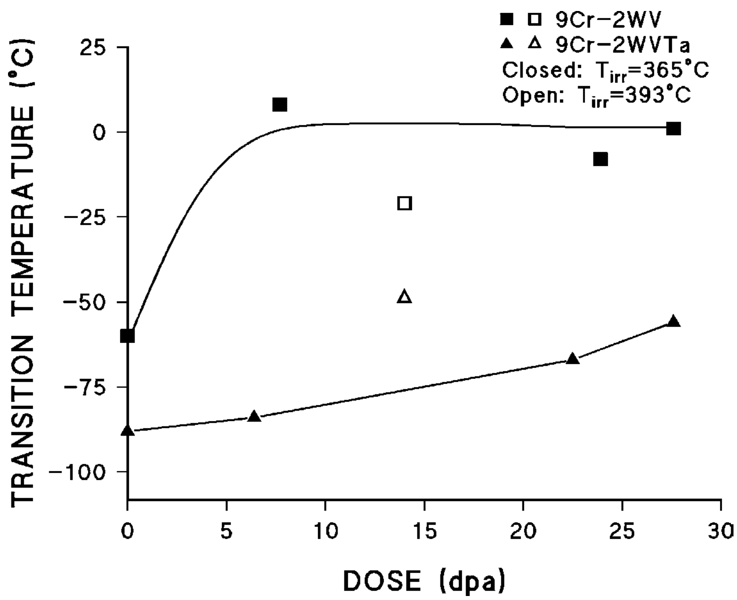

Figure 1—*Transition Temperature of 9Cr-2WV and 9Cr-2WVTa as a Function of Dose*

The improved properties of the 9Cr-2WVTa steel and the differences from other steels during irradiation were attributed to tantalum in solution [*5,8,9-11*]. Tantalum was determined to be in solution from transmission electron microscopy (TEM) studies that analyzed the amount of tantalum in precipitates [*10*] and by atom probe analyses [*11*]. Tantalum in solution caused a grain refinement and affected the impact properties, partly due to the smaller prior austenite grain size and partly due to an effect on the fracture stress of the steel [*5,8,9*].

It was hypothesized that the different behavior of 9Cr-2WV and 9Cr-2WVTa during irradiation was caused by a loss of tantalum from solution by precipitation [*5,8*]. Since the loss of tantalum will be diffusion controlled, it should proceed throughout the irradiation, leading to a continued increase in the DBTT, as observed, if tantalum has an effect other than just the effect on grain size. Likewise, an increase in the irradiation temperature should increase the rate of precipitation, as equilibrium will be achieved at a higher rate. Precipitation of tantalum during irradiation was confirmed by Kimura et al.

for a 9Cr-2WVTa steel irradiated in FFTF at 460ºC, when they found that tantalum-rich M_6C precipitated [*12*]. They did not observe any tantalum-rich precipitates when irradiated at the lower temperature of 390ºC for the fluence used, which is in agreement with the observations on the unusual temperature effect discussed above.

From these observations, it was postulated that if the effect of tantalum in solution was lost by precipitation, then once equilibrium for tantalum is reached, the difference in behavior of the 9Cr-2WV and 9Cr-2WVTa steels would be caused by the difference in prior-austenite grain size and any tantalum remaining in solution, which could be quite small. This appears to be the case, since there is little difference in the ΔDBTT of the steels after irradiation at the higher temperature (Fig. 1). Therefore, it might be possible to get similar irradiation resistance without tantalum [*5,6*] if the prior-austenite grain size of 9Cr-2WV could be reduced. In this paper, different heat treatments were used to vary the prior-austenite grain size of 9Cr-2WV and 9Cr-2WVTa steels. The effect of the heat treatments on the microstructure and the tensile and Charpy properties were determined.

Experimental Procedure

The 9Cr-2WV and 9Cr-2WVTa steels are nominally (in wt. %) Fe-9.0Cr-2.0W-0.25V-0.1C without and with 0.06% Ta, respectively. They were prepared as 18 kg electro-slag remelted heats by Combustion Engineering, Inc., Chattanooga, Tennessee. In addition to the Cr, W, V, C, and Ta, elements normally found in such steels (e.g., Mn, Si, etc.) were adjusted to levels typical of commercial steel processing practice. Compositions for the two steels are shown in Table 1.

Table 1—*Chemical compositions of the steels (wt. %)*[a]

Steel	C	Mn	Si	Cr	W	V	Cu	N	Ta
9Cr-2WV	0.12	0.51	0.23	8.95	2.01	0.24	0.03	0.029	
9Cr-2WVTa	0.11	0.44	0.21	8.90	2.01	0.23	0.03	0.022	0.06

[a] P=0.015, S=0.008, Balance Fe.

Specimens were taken from a 6.35-mm plate after pieces of the plate were given several normalizing and tempering heat treatments. Normalization involved an austenitization treatment followed by cooling in flowing helium gas. Specimens were austenitized as follows: 0.033, 0.25, and 0.5 h (5, 15, and 30 min) at 950ºC, 0.5 h at 1000ºC, and 0.5 h at 1050ºC. All specimens were tempered 1 h at 750ºC.

Tensile specimens 25.4-mm long with a reduced gage section 7.62-mm long by 1.52-mm wide by 0.76-mm thick were machined from the 6.35-mm plate. Tensile tests were at room temperature on a 120-kN Instron universal test machine at a nominal strain rate of $1 \times 10^{-3}\ s^{-1}$. Two tests were conducted for each test condition.

Charpy specimens were one-third size V-notch specimens measuring 3.3 x 3.3 x 25.4 mm with a 0.51-mm-deep 30º V-notch and a 0.05- to 0.08-mm-root radius that were machined from normalized-and-tempered 6.35-mm plates. Specimens were machined from normalized-and-tempered plate along the rolling direction with the notch transverse to the rolling direction (L-T orientation). The DBTT was determined at an energy level midway between the upper and lower shelf energies. Details on the test procedure for the subsize impact specimens have been published [*13-15*].

Results

Mechanical Properties

The tensile and Charpy data are given in Table 2 for the five normalizing conditions: 950ºC/5 min, 950ºC/15 min, 950ºC/30 min, 1000ºC/15 min, 1050ºC/30 min; all were tempered 1 h at 750ºC. Figure 2 shows the room-temperature yield stress for the five normalizing conditions. Despite the differences in normalizing conditions, there was

Table 2—*Tensile and Charpy Data*

Steel	Austenitization Conditions	Yield Stress[a]	Ultimate Strength	Uniform Elongation	Total Elongation	Charpy DBTT[b]	Charpy USE
9Cr-2WV	950C/5m	560	698	8.1	18.8	-109	11.8
9Cr-2WV	950C/15m	548	716	8.2	19.1	-105	11.9
9Cr-2WV	950C/30m	551	698	8.2	18.5	-85	12.6
9Cr-2WV	1000C/15m	548	691	7.8	18.8	-95	11.8
9Cr-2WV	1050C/1h	549	659	4.7	12.3	-60	8.4
9Cr-2WVTa	950C/5m	549	691	7.9	17.4	-99	11.6
9Cr-2WVTa	950C/15m	559	691	8.1	18.4	-99	11.0
9Cr-2WVTa	950C/30m	563	693	7.9	18.7	-94	12.1
9Cr-2WVTa	1000C/15m	572	700	7.2	17.6	-89	11.4
9Cr-2WVTa	1050C/1h	544	652	4.3	12.3	-88	11.2

[a] The tensile data represent the average of two tests.
[b] One-third size Charpy specimens were tested; the DBTT was calculated as one-half the difference between the upper shelf and lower shelf energies.

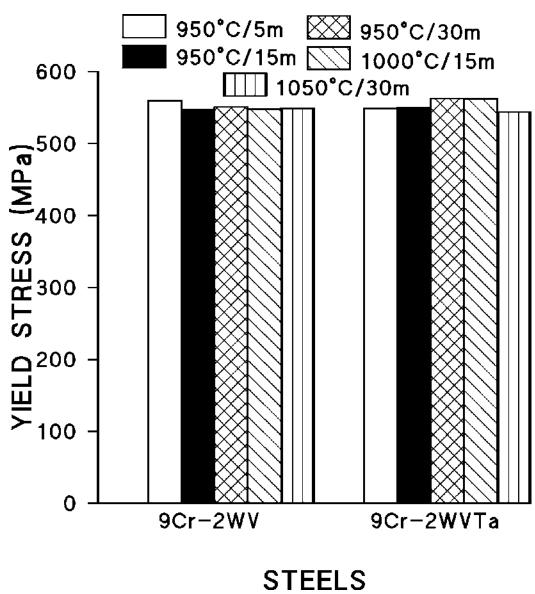

Figure 2—*Room Temperature Yield Stress of 9Cr-2WV and 9Cr-2WVTa Steels Given Different Heat Treatments*

little difference in yield stress among the specimens of the 9Cr-2WV and 9Cr-2WVTa and little difference between the two steels. A similar observation was made on ultimate tensile strength (Table 2). Total elongations (Fig. 3) also showed no change when the austenitizing temperature was 950 or 1000ºC, but for 1050ºC, there was about a 30% decrease in elongation. A similar effect was observed for uniform elongation (Table 2).

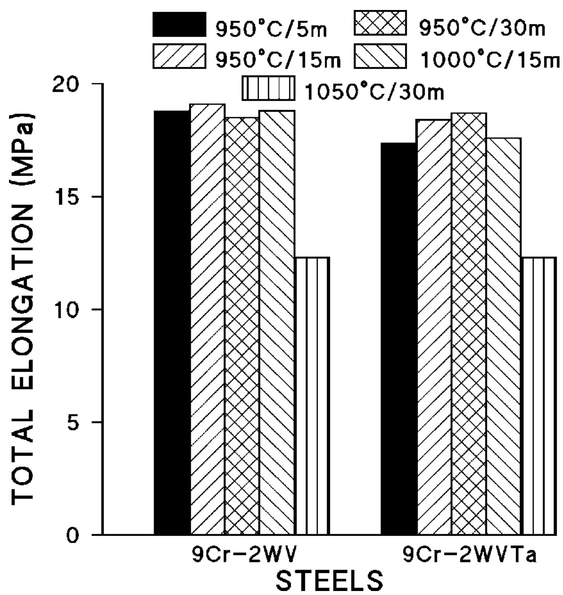

Figure 3—*Room Temperature Total Elongation of 9Cr-2WV and 9Cr-2WVTa Steels Given Different Heat Treatments*

The effect of the different heat treatments on the DBTT values measured for the 9Cr-2WVTa steel with 1/3-size Charpy specimens was rather small for all heat treatments (Fig. 4). For the 9Cr-2WV, on the other hand, there was more change, especially for the 1050ºC austenitization treatment. The upper-shelf energy (USE) (Fig. 5) showed essentially no change with austenitization temperature for the 9Cr-2WVTa. Likewise, there was no change for the 9Cr-2WV for the 950 and 1000ºC austenitization treatments. However, just as was the case for the total elongation, there was a relatively large change (decrease) when austenitized at 1050ºC.

Microstructure

The effect of tantalum on the prior-austenite grain size for the 9Cr-2WV and 9Cr-2WVTa steels is obvious in the 100% tempered martensite microstructures of the two steels shown in Fig. 6 following austenitization at 1050ºC for 0.5 h. Grain sizes were estimated by optical microscopy after the different anneals (Fig. 7). There was a large increase in the grain size of the 9Cr-2WV steel with an increase in time at 950ºC and with an increase in temperature from 950 to 1000 to 1050ºC. Less change occurred for the 9Cr-2WVTa than the 9Cr-2WV for all of the heat treatment conditions. The largest difference in grain size for the two steels occurred for those austenitized at 1050ºC. The results indicate that tantalum is very effective in reducing austenite grain growth.

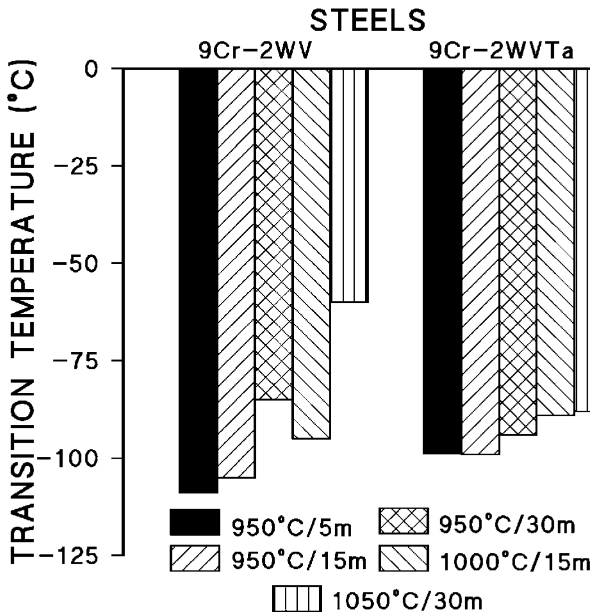

Figure 4—*Charpy Transition Temperature of 9Cr-2WV and 9Cr-2WVTa Steels Given Different Heat Treatments*

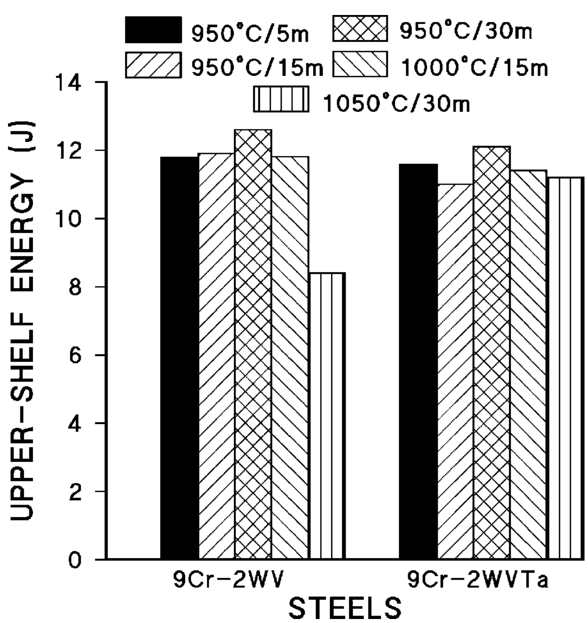

Figure 5—*Upper-Shelf Energy of 9Cr-2WV and 9Cr-2WVTa Steels Given Different Heat Treatments*

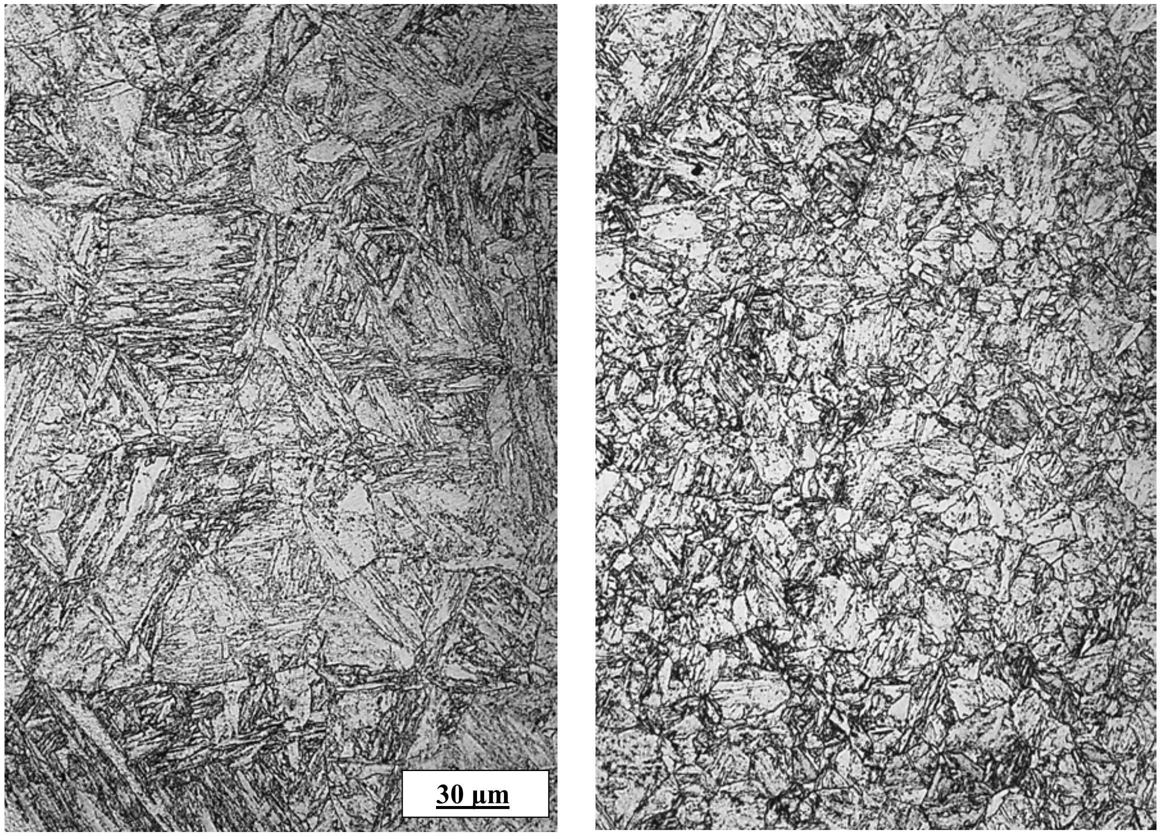

Figure 6— *Photomicrographs of 9Cr-2WV (left) and 9Cr-2WVTa (right) Steels Austenitized for 0.5 h at 1050ºC*

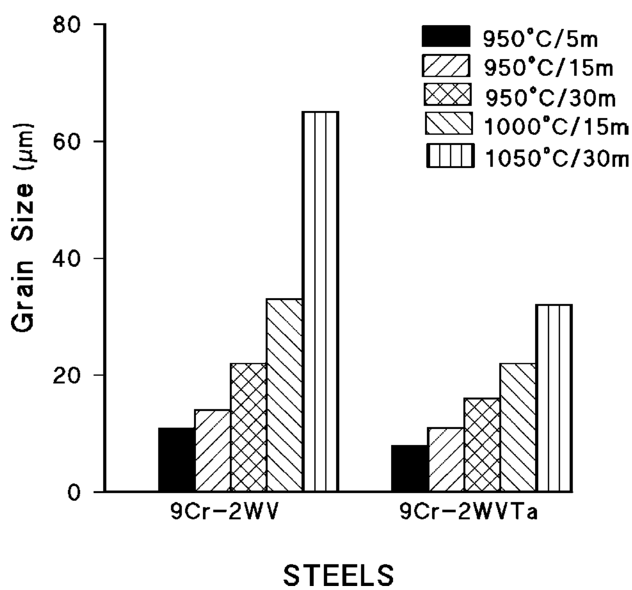

Figure 7— *Prior-Austenite Grain Size of 9Cr-2WV and 9Cr-2WVTa Steels Given Different Heat Treatments*

Grain-size measurements at low magnification (100X) indicated that microstructures for all heat treatments were 100% martensite for both steels. This was verified for 9Cr-2WV by transmission electron microscopy (TEM), where a typical tempered martensite structure was observed for all heat-treated conditions. With the exception of the prior-austenite grain size, there was little difference in the structure of the steels heat treated for different times at 950ºC and at the other two temperatures (Fig. 8). The lath sizes also did not vary substantially for the different heat treatments. The average lath width was estimated at 0.3-0.6 μm.

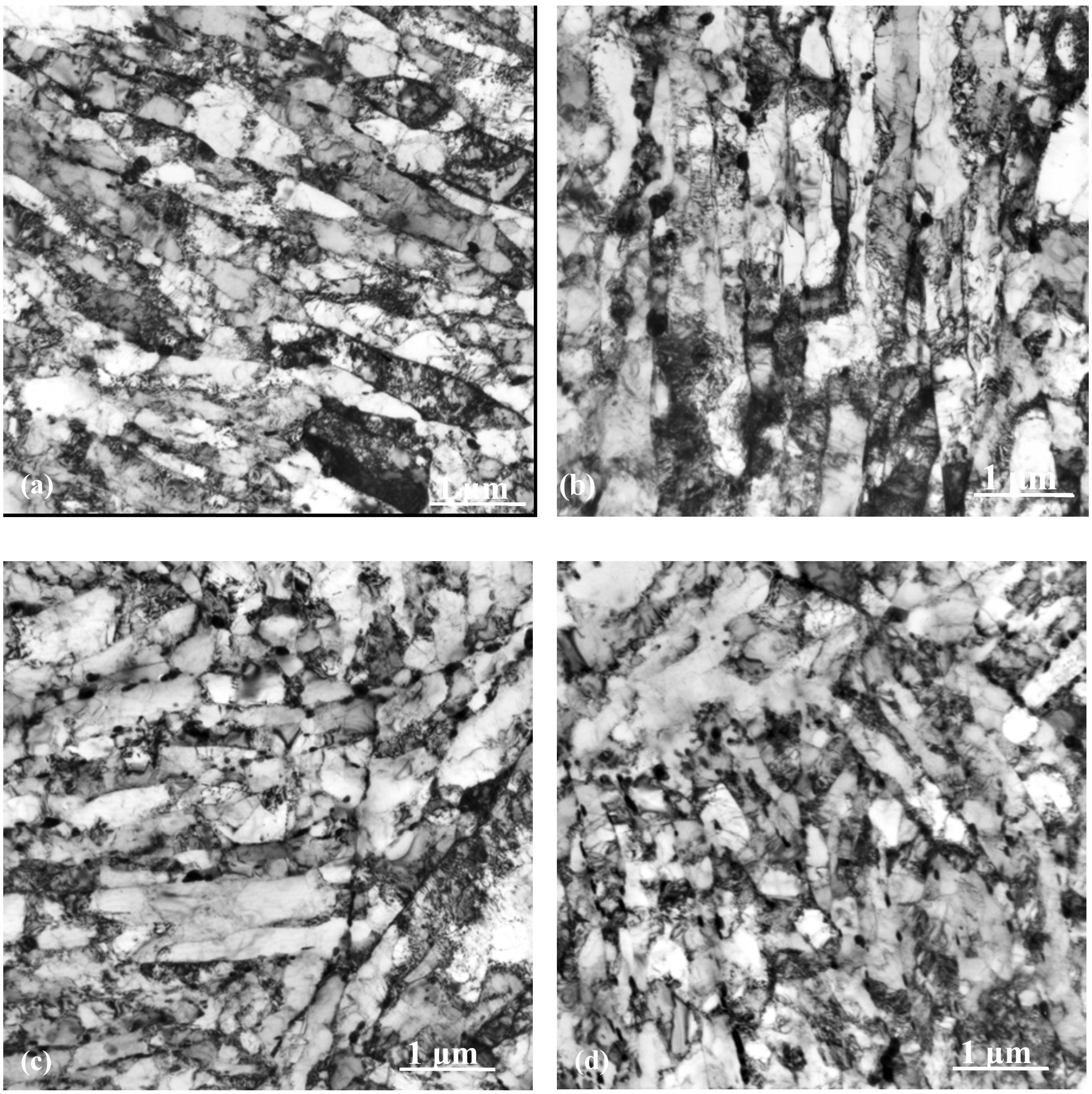

Figure 8— *TEM Micrographs of the 9Cr-2WV Steel Austenitized at (a) 5 min at 950ºC (b) 15 min at 950ºC, (c) 30 min at 950ºC, and (d) 15 min at 1000ºC*

The 9Cr-2WVTa steel had a somewhat different microstructure after it was austenitized at 950ºC for 5 min: it contained regions of ferrite [Fig. 9(a)] that evidently had not transformed to austenite during the exposure at 950ºC. A smaller amount of ferrite was found in the specimen heat treated at 950ºC for 15 min [Fig. 9(b)]. A small amount was also observed after 30 min at 950ºC, but none was present after the 1000 and 1050ºC heat treatments.

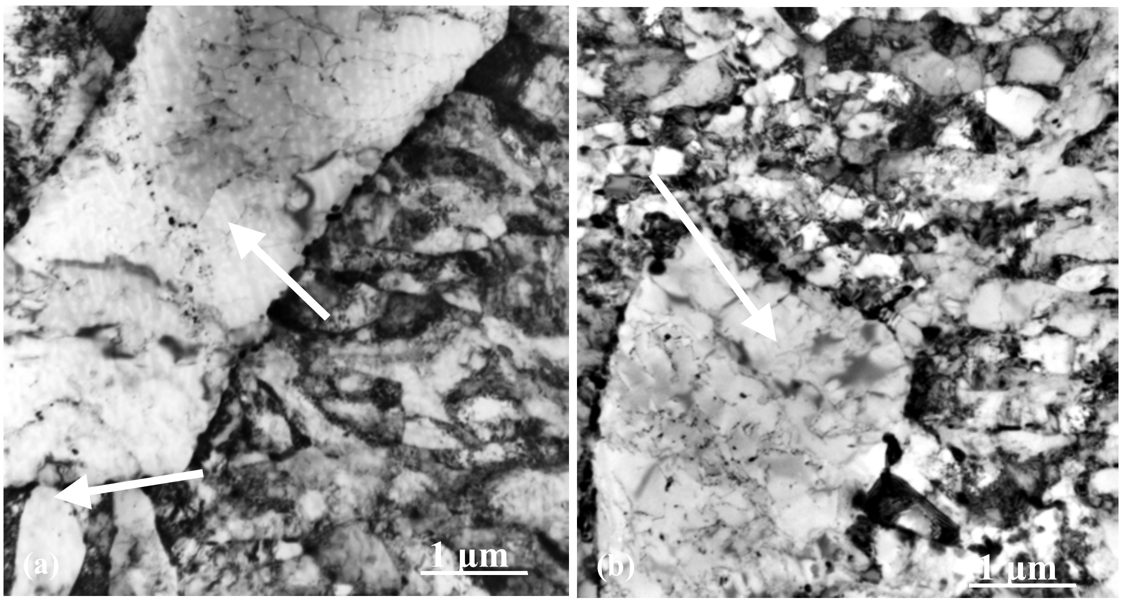

Figure 9—*TEM Micrographs of 9Cr-2WVTa Steel Austenitized (a) 5 min at 950ºC and (b) 15 min at 950ºC Showing Ferrite (Arrows)*

Since the presence of ferrite had not been detected by optical microscopy when the specimens were examined for prior-austenite grain size at low magnification, the steels were re-examined and the ferrite identified. At high magnification (500X), the ferrite could be easily observed (Fig. 10). The amount of ferrite was estimated at around 10-15% and 3-6% in the steels austenitized 5 and 15 min at 950ºC, respectively. Although none was observed by TEM after the 30 min anneal at 950ºC, a small amount (<1%) was observed by optical microscopy. None was detected for the steel austenitized for 15 min at 1000ºC by either optical microscopy or TEM;

The lath structure of the martensite in the 9Cr-2WVTa steel was similar to that of the 9Cr-2WV steel with a similar lath size (Fig. 11).

Although no attempt was made to identify the precipitates in this work, the precipitate morphology and distribution were typical of those in most 9-12Cr steels (Fig. 12). Precipitates have been studied and identified in the 9Cr-2WV and 9Cr-2WVTa steels [*13,14*], and the majority are $M_{23}C_6$ (estimated at 100-200 nm diameter at a number density of 10^{19}-10^{20} m^{-3} in both steels). These are the large precipitates on the prior-austenite grain boundaries and lath boundaries in Fig. 12. The other precipitate that has been identified is small MX particles in the matrix. These are present at a lower number density (estimated at 20-50 nm diameter and 10^{17}-10^{18} m^{-3}) than the $M_{23}C_6$ [*13,14*].

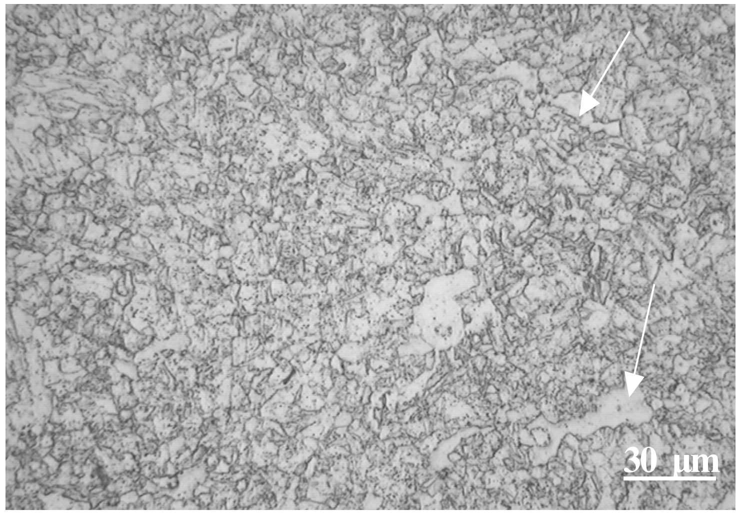

Figure 10─*Optical Photomicrograph of 9Cr-2WVTa Steel Austenitized 5 min at 950ºC; Arrows Indicate Ferrite*

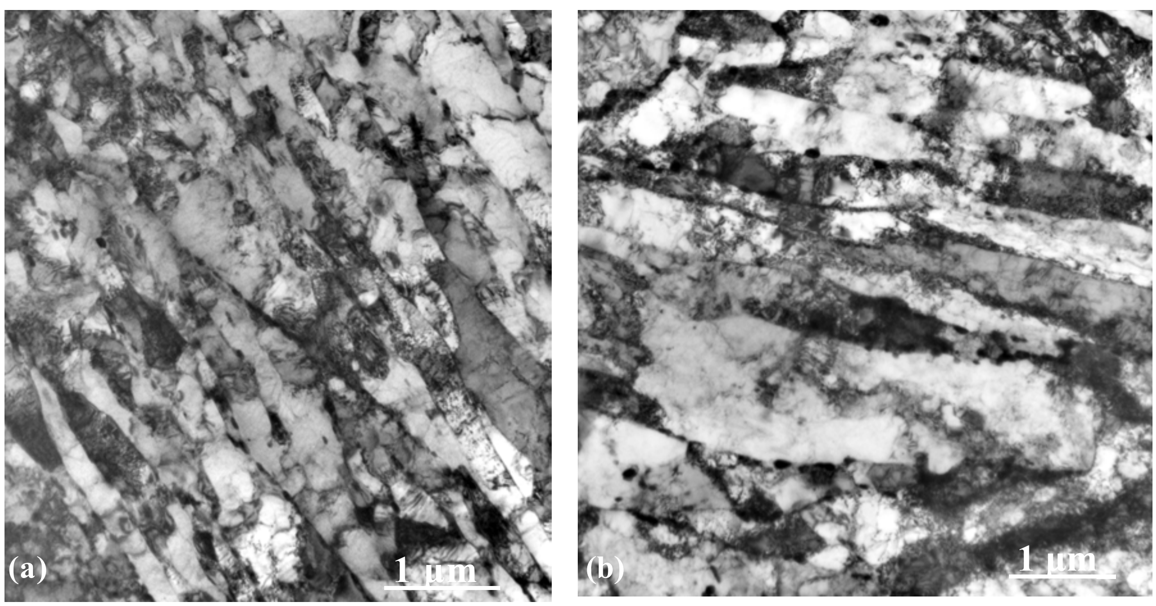

Figure 11─*TEM Photomicrographs of (a) 9Cr-2WV and (b) 9Cr-2WVTa Showing the Similar Martensite Lath Sizes*

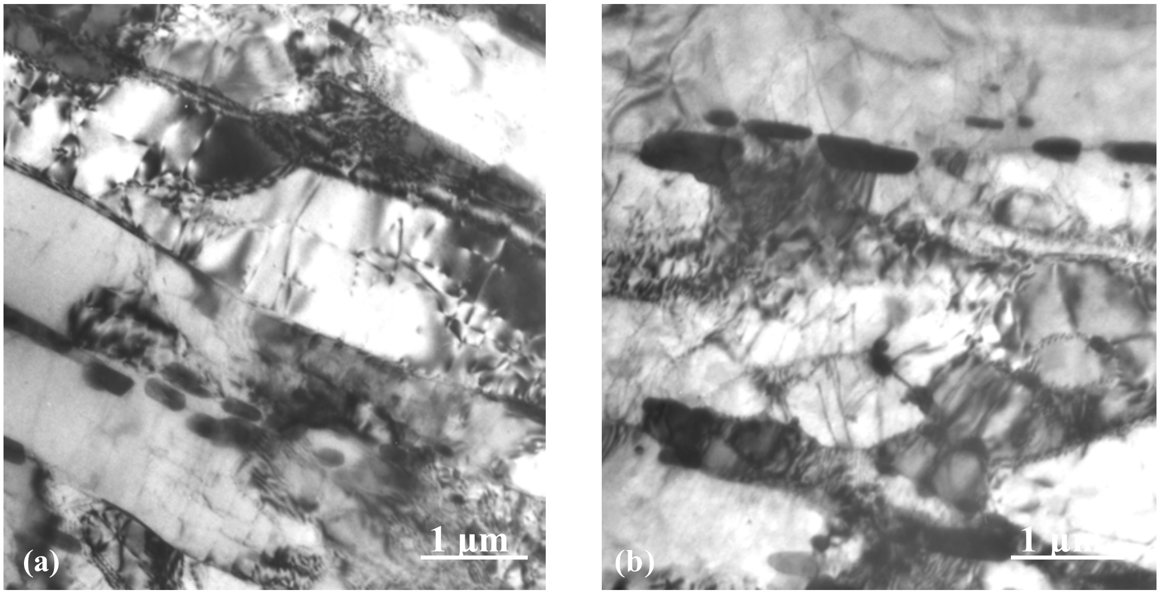

Figure 12─*TEM Photomicrographs of (a) 9Cr-2WV and (b) 9Cr-2WVTa Showing the Typical Precipitate Microstructures of the Steels (Austenitized at 1050ºC)*

Discussion

The results indicate that tantalum plays a complicated role in the mechanical properties and microstructural behavior of the 9Cr-2WVTa steel. Despite differences in the prior-austenite grain sizes of the 9Cr-2WV and 9Cr-2WVTa steels and the change in grain size with different heat treatments, room temperature yield stress and ultimate tensile strength were essentially unaffected, indicating that room-temperature strength is determined by the martensite microstructure and is unaffected by the prior austenite grain size. Ductility was only affected at the highest austenitization temperature (1050ºC), and again, there was little difference between the 9Cr-2WV and 9Cr-2WVTa. Thus, it is difficult to see how this could be affected by prior-austenite grain size, since there was a large difference in grain size of the two steels after the 1050ºC heat treatment (Fig. 7) but essentially no difference in uniform or total elongation. This similarity in strength and ductility was observed previously in both the unirradiated and irradiated conditions [*5,8*].

The DBTT change of the 9Cr-2WV steel for the different heat treatments appears reflective of a prior-austenite grain size effect, since there did not appear to be any other obvious microstructural changes to account for the change of the DBTT. Precipitation appeared to be similar for the different heat treatments. This might be expected, since most of the precipitates form during the tempering treatment. The USE appeared much less affected than the DBTT; it changed (decreased) only after the 1050ºC anneal, similar to the change in ductility.

Charpy properties of the 9Cr-2WVTa steel showed relatively little change: there was a slight increase in DBTT with an increase in temperature and time at temperature of the austenitization treatment. This change probably reflects the much smaller change in prior-austenite grain size, and it may also reflect the previous conclusion that there is a component of the fracture resistance of this steel that can be attributed to tantalum in solution [*5,8*].

Despite the smaller grain size of the tantalum-containing steel after the 950ºC heat treatment for 5 and 15 min, the 9Cr-2WV steel had a DBTT comparable to that of the 9Cr-2WVTa steel for these heat treatments (Table 2 and Fig. 4). This is probably caused by the ferrite in these two samples of 9Cr-2WVTa. The presence of δ-ferrite has been shown to affect the transition temperature of other 9 and 12% Cr steels [*16,17*], and the ferrite in the present steels could act in the same way.

The interesting observation that after the two short-time (5 and 15 min) heat treatments at 950ºC the 9Cr-2WVTa contained ferrite and the 9Cr-2WV did not is another indication of the effect of tantalum. It is assumed that the transformation of ferrite to austenite on heating proceeds by nucleation and growth, which means this is another observation that tantalum retards the growth of austenite (it could also retard nucleation), the same conclusion reached by the observations on grain size differences between 9Cr-2WV and 9Cr-2WVTa.

In light of the observation that tantalum is lost from solution during irradiation, the objective for this work was to determine if the steel without tantalum could be made to perform as well as the steel with tantalum. Since irradiation has an effect on fracture properties by increasing the transition temperature, this comparison was made on the Charpy properties. If the comparison is made between the low-temperature anneals (950 and 1000ºC) of the 9Cr-2WV and the 1050ºC anneal used for the 9Cr-2WVTa previously, then it is possible to improve the unirradiated Charpy properties of the 9Cr-2WV to be as good or better than those of the 9Cr-2WVTa. This is demonstrated in Fig. 13, where the DBTT are plotted against the prior-austenite grain size and the similarity for the two steels for the smaller grain sizes are evident. It is seen that a 1000ºC austenitization temperature with a DBTT of ≈-90ºC (Table 2) could be used to get a comparable DBTT to that of the 9Cr-2WVTa annealed at 1050ºC. It is assumed that with a finer prior-austenite grain size, the ΔDBTT for 9Cr-2WV steel for similar irradiations at 365ºC to those of Fig. 1 should be less than observed previously, and it could approach that of the 9Cr-2WVTa. Likewise, at 393ºC, where the ΔDBTT of the 9Cr-2WV was similar to that of the 9Cr-2WVTa (Fig. 1), the steels would be expected to have similar DBTTs after irradiation to a fluence similar to that used previously [*8*]. Therefore, by decreasing the prior-austenite grain size of the 9Cr-2WV steel, it is expected that the Charpy properties of this steel could be made as good or better than those of the 9Cr-2WVTa steel.

Although the grain size of the 9Cr-2WV—and the 9Cr-2WVTa—could be reduced by using a lower austenitizing temperature or another thermomechanical processing technique, it is not clear that it would be advisable. No effect of the small grain size on creep properties has been determined, but the fine grain size should reduce the creep strength at high temperatures. Therefore, it appears that the use of tantalum to reduce the grain size and contribute in other ways to the fracture resistance may be the best way to achieve the properties required. However, if it were necessary to remove tantalum—say for nuclear considerations [*18*]—then it should still be possible to produce a comparable steel without tantalum, especially since the additional effect of tantalum on fracture is lost during irradiation.

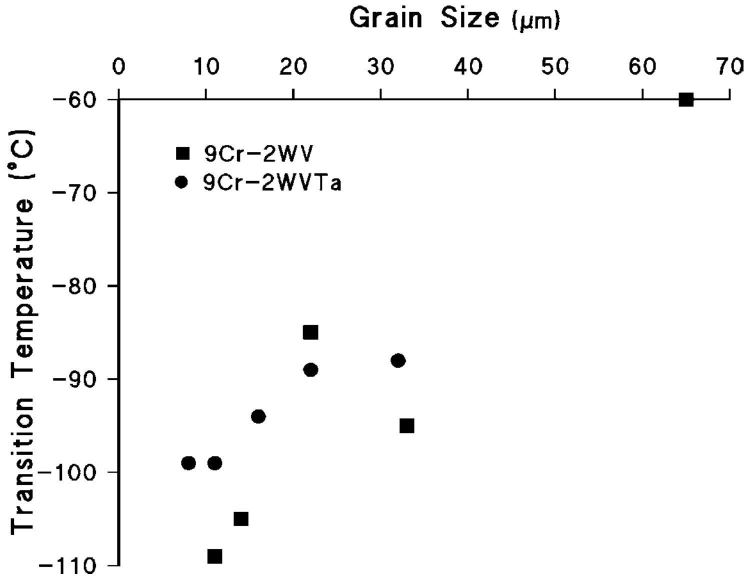

Figure 13—*Transition Temperature as a Function of Prior-Austenite Grain Size For 9Cr-2WV and 9Cr-2WVTa Steels After Different Austenitization Treatments*

Summary and Conclusions

The effect of prior-austenite grain size and tantalum on a nominal Fe-9Cr-2W-0.25V-0.10C (9Cr-2WV) steel was determined by examining the effect of austenitization time and temperature on the room-temperature tensile properties and Charpy properties of the 9Cr-2WV steel and this steel with 0.06% Ta (9Cr-2WVTa). To vary the prior-austenite grain size of the normalized steel, specimens were austenitized for 5, 15, and 30 min at 950ºC, 15 min at 1000ºC, and 30 min at 1050ºC, followed by a rapid cool in flowing helium gas. All specimens were tempered 1 h at 750ºC. The results can be summarized as follows:

- Tantalum causes grain refinement in the 9Cr-2WVTa, but it does not cause other significant differences in microstructure (martensite lath size and precipitates) compared with 9Cr-2WV.
- Neither prior-austenite grain size nor tantalum affected the room-temperature strength.
- Room-temperature ductility was unaffected except after the 1050ºC heat treatment, where total and uniform elongation decreased; similar decreases occurred for the 9Cr-2WV and 9Cr-2WVTa steels.
- The DBTT of the 9Cr-2WV steel showed significant increases with increasing prior-austenite grain size, whereas, the 9Cr-2WVTa was much less sensitive to prior-austenite grain size.
- The USE of the 9Cr-2WVTa was unaffected by the different heat treatments, and the USE of the 9Cr-2WV was unaffected except after the 1050ºC austenitization, where it was reduced significantly.

It should be possible to produce a 9Cr-2WV steel with tensile and impact properties as good as those of the 9Cr-2WVTa by reducing the prior-austenite grain size. The effect of such a small grain size on the creep properties has not been determined.

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