



Effect of chromium, tungsten, tantalum, and boron on mechanical properties of 5–9Cr–WVTaB steels [☆]

R.L. Klueh ^{a,*}, D.J. Alexander ^b, M.A. Sokolov ^a

^a Oak Ridge National Laboratory, Metals and Ceramic Division, P.O. Box 2008, MS 6376, Oak Ridge, TN 37831-6138, USA

^b Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Received 2 January 2002; accepted 15 May 2002

Abstract

The Cr–W–V–Ta reduced-activation ferritic/martensitic steels use tungsten and tantalum as substitutes for molybdenum and niobium in the Cr–Mo–V–Nb steels that the reduced-activation steels replaced as candidate materials for fusion applications. Studies were made to determine the effect of W, Ta, and Cr composition on the tensile and Charpy properties of the Cr–W–V–Ta; steels with 5%, 7%, and 9% Cr with 2% or 3% W and 0%, 0.05%, or 0.10% Ta were examined. Boron has a long history of use in steels to improve properties, and the effect of boron was also examined. Regardless of the chromium concentration, the steels with 2% W and 0.05–0.1% Ta generally had a better combination of tensile and Charpy properties than steels with 3% W. Boron had a negative effect on properties for the 5% and 7% Cr steels, but had a positive effect on the 9% Cr steel. When the 5, 7, and 9Cr steels containing 2% W and 0.05% Ta were compared, the tensile and Charpy properties of the 5 and 9Cr steels were better than those of the 7Cr steel, and overall, the properties of the 5Cr steel were better than those of the 9Cr steel. Such information will be useful if the properties of the reduced-activation steels are to be optimized.

© 2002 Published by Elsevier Science B.V.

1. Introduction

Reduced-activation Fe–Cr–W–V–Ta ferritic/martensitic steels with 7–9Cr, 1.5–2W, 0.2–0.3V, 0.03–0.1Ta (all concentrations in weight percent) are the primary candidates for first wall and blanket structural applications for future magnetic fusion reactors. An Fe–9Cr–2W–0.25V–0.07Ta–0.1C (9Cr–2WVTa) steel was shown to have excellent tensile and impact properties before and after irradiation in a fission reactor [1]. Similar results were obtained for F82H, a 7.5–8Cr–2W–0.2V–0.03Ta–

0.1C steel [2,3], and JLF-1, a 9Cr–2W–0.2V–0.08Ta–0.025N steel [3,4]. Heats of F82H and JLF-1 are being studied in the US, Europe, and Japan in a collaborative program under the auspices of the International Energy Agency [5].

Several studies have been conducted on Cr–2WV steels with chromium concentrations between 2.25% and 12% Cr [1,6,7]. The Cr–2WVTa steels with ≈2–4% Cr can be transformed to bainite, those with 5–10% Cr can be transformed to 100% martensite, and such steels with ≈12% Cr will contain ≈25% δ-ferrite unless extra austenite-forming alloying elements (e.g., C or Mn for reduced-activation steels) are added to the composition. Effects of vanadium [1,7,8] and tungsten [1,7,9] have also been determined; these studies have generally indicated that steels containing 7–9% Cr, 2% W, 0.2–0.3V, and 0.03–0.1% Ta with 0.1% C offer excellent physical and mechanical properties for the proposed application.

The highest operating temperature for these steels is 550–600 °C, similar to the upper operating temperature

[☆] Research sponsored by the Office of Fusion Energy Sciences, US Department of Energy, under contract DE-AC05-00OR22725 with U.T.-Battelle, LLC.

* Corresponding author. Tel.: +1-865 574 5111; fax: +1-865 241 3650.

E-mail address: ku2@ornl.gov (R.L. Klueh).

of the conventional modified 9Cr–1Mo (9Cr–1MoVNb) and Sandvik HT9 (12Cr–1MoVW) steels that the reduced-activation steels replaced for fusion applications. It is of interest to increase the operating temperature for fusion power plants, and one way proposed to do this is by increasing tungsten [9].

Although studies to date indicate the general superiority of 7–10Cr–2WVTa martensitic steels over 2–3% Cr bainitic steels and 12% Cr duplex steels, little information is available concerning the effect of chromium over the 5–10% range, where the steels transform to martensite when normalized or quenched. In this work, the effect of chromium, tungsten, and tantalum was investigated for the 5–9% Cr martensitic steels. The effect of boron was also investigated, because it has often been used in these types of steels to improve properties.

2. Experimental procedure

Compositions of the steels investigated and the designations used in this paper to describe the steels are given in Table 1. Nominally the steels are Fe–5Cr–2W–0.25V–0.1C (designated 5Cr–2WV), the 5Cr–2WV with 0.05% Ta (5Cr–2WVTa1), the 5Cr–2WV with 0.1% Ta (5Cr–2WVTa2), an Fe–5Cr–3W–0.25V–0.1C (5Cr–3WV), the 5Cr–3WV with 0.05% Ta (5Cr–3WVTa1), the 5Cr–3WVTa1 with 0.005% B (5Cr–3WVTa1B1), the 5Cr–3WVTa1 with 0.013% B (5Cr–3WVTa1B2), an Fe–7Cr–2W–0.25V–0.05Ta–0.1C (7Cr–2WVTa), an Fe–7Cr–3W–0.25V–0.05Ta–0.1C (7Cr–3WVTa), the 7Cr–2WVTa with 0.005% B (7Cr–2WVTaB), Fe–9Cr–2W–0.25V–0.05Ta–0.1C (9Cr–2WVTa), and the 9Cr–2WVTa with 0.005% B (9Cr–2WVTaB).

In the original Oak Ridge National Laboratory (ORNL) development program for reduced-acti-

vation steels [10], eight 18-kg electroslag-remelted heats of 2.25%, 5%, 9%, and 12% Cr heats containing various amounts of W, V, and Ta were prepared by Combustion Engineering Inc., Chattanooga, TN. In addition to nominal compositions of Cr, W, V, C and Ta, elements normally found in such steels (e.g., Mn, Si, etc.) were adjusted to levels typical of commercial practice [10]. Material from the 18-kg heats of the 5Cr–2WV steel and 9Cr–2WVTa steels was used as a master alloy to prepare 450-g vacuum arc-melted button heats for all but one of the heats in Table 1. The exception was the 5Cr–3WV, for which material from an 18-kg heat of Fe–2.25Cr–2W–0.25V–0.1C steel was used as the master alloy.

The 450-g heats were cast at ORNL as $12.7 \times 25.4 \times 127$ mm ingots; half of each ingot was hot rolled to a thickness of 6.4 mm and half to a thickness of 0.76 mm. Mechanical properties tests were made on normalized-and-tempered steel. The steels were normalized by first austenitizing 0.5 h at 1050 °C in a helium atmosphere, after which they were quickly cooled in flowing helium. Specimens were tested in two tempered conditions: 1 h at 700 °C and 1 h at 750 °C.

Normalized-and-tempered tensile specimens 44.5-mm long with a reduced gage section of $20.3 \times 1.52 \times 0.76$ mm were machined from the 0.76-mm sheet with gage lengths parallel to the rolling direction. Specimens were heat treated after machining. Tensile tests were conducted at room temperature, 200, 300, 400, 500, and 600 °C in vacuum on a 44-kN Instron universal testing machine at a nominal strain rate of 4×10^{-4} s⁻¹.

One-third-size Charpy specimens $3.3 \times 3.3 \times 25.4$ mm with a 0.51-mm-deep 30° V-notch and a 0.05–0.08-mm-root radius were machined from the normalized 6.4-mm plate along the rolling direction with the notch transverse to the rolling direction (LT orientation). Specimens were tempered after machining. The ab-

Table 1
Chemical composition of steels investigated (wt%)^a

Steel	C	Mn	Si	Cr	V	W	Ta	N	B
5Cr–2WV	0.12	0.49	0.23	5.04	0.24	2.01		0.011	
5Cr–2WVTa1	0.11	0.46	0.20	4.67	0.25	2.11	0.05	0.008	
5Cr–2WVTa2	0.12	0.45	0.19	4.65	0.25	2.14	0.10	0.009	
5Cr–3WV	0.096	0.40	0.12	4.97	0.23	3.00		0.009	
5Cr–3WVTa1	0.11	0.44	0.22	4.63	0.25	3.01	0.05	0.010	
5Cr–3WVTa1B1	0.11	0.43	0.21	4.61	0.25	2.87	0.05	0.010	0.005
5Cr–3WVTa1B2	0.11	0.42	0.21	4.61	0.24	2.99	0.05	0.011	0.013
7Cr–2WVTa	0.12	0.42	0.19	7.01	0.24	2.01	0.05	0.014	
7Cr–3WVTa	0.12	0.42	0.19	6.98	0.24	2.97	0.05	0.014	
7Cr–2WVTaB	0.12	0.42	0.19	7.02	0.24	1.98	0.05	0.013	0.004
9Cr–2WV	0.12	0.51	0.23	8.95	0.24	2.01		0.029	
9Cr–2WVTa	0.11	0.44	0.21	8.90	0.23	2.01	0.06	0.017	
9Cr–2WVTaB	0.094	0.40	0.21	8.38	0.23	2.02	0.06	0.014	0.005

^a Other elements analyzed (highest value of the eleven heats): P ≤ 0.16, S ≤ 0.009, Mo ≤ 0.02, Nb < 0.01, Cu ≤ 0.04, Al ≤ 0.021, As ≤ 0.004, Sn ≤ 0.004.

sorbed energy values were fit with a hyperbolic tangent function to permit the upper-shelf energy (USE) and ductile-brittle transition temperature (DBTT) to be evaluated. The DBTT was determined at an energy level midway between the USE and lower-shelf energy. Details on the test procedure have been published [11–13].

3. Results

3.1. Microstructure

Optical microscopy indicated that all the steels were 100% martensite. Prior-austenite grain sizes were determined (Table 2) using the comparison procedure. Data for the 5Cr steels (Table 2) indicated that tantalum affects prior-austenite grain size, since all steels with tantalum have a smaller grain size than those without tantalum. This tantalum effect was observed previously for the 9Cr–2WVTa steel when compared with a 9Cr–2WV steel [1]. For a 5Cr steel, it appears that increasing the tungsten caused a decrease in the grain size; increasing the boron also caused a reduction in grain size (Table 2). In the 7Cr steel, the effect of tungsten and boron on the grain size was not as obvious.

3.2. Tensile behavior

3.2.1. 5Cr–W–V–Ta–B steels

Fig. 1 shows the room-temperature yield stress (YS) for the seven 5Cr steels in the two normalized-and-tempered conditions. When no tantalum was present, there was no effect of increasing the tungsten from 2% (5Cr–2WV) to 3% (5Cr–3WV). Tantalum additions

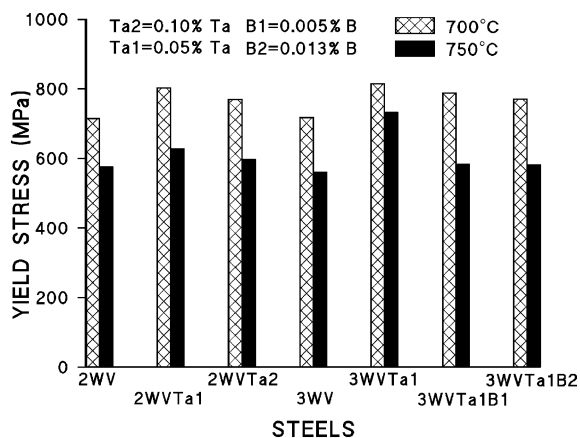


Fig. 1. YS at room temperature of 5Cr steels of different composition.

caused a slight increase in strength for the 5Cr–2WV steel, especially for the 700 °C temper. However, there was no difference between 0.05% and 0.10% Ta. There was no difference in the strength of the 5Cr–2WVTa1 and 5Cr–3WVTa1 steels after the 700 °C temper, but the 5Cr–3WVTa1 was strongest after the 750 °C temper. The addition of B to the 5Cr–3WVTa1 steel produced little difference in the strength of this steel relative to the 5Cr–3WVTa1 for the 700 °C temper, but there was a reduction in strength for the boron-containing steels when tempered at 750 °C.

In Fig. 2 tensile data from room temperature to 600 °C are shown for the five 5Cr steels without boron tempered at 700 °C, and the relative behavior over this temperature range is similar to that observed at room temperature. The steels without tantalum were the weakest (Fig. 2(a)), and there was relatively little difference in the three steels with tantalum (5Cr–2WVTa1, 5Cr–2WVTa2, and 5Cr–3WVTa1), although at 600 °C – highest test temperature – the strength of the 5Cr–3WVTa1 becomes relatively weaker than the other two tantalum-containing steels, even though this steel was the strongest at the lower temperatures. The strength decrease at 600 °C was more pronounced for the ultimate tensile strength (UTS) than the YS (Fig. 2(a)).

Uniform elongation of these five steels after tempering at 700 °C (Fig. 2(b)) did not show large variations. Somewhat more variation occurred for the total elongation, especially at 600 °C, where the 5Cr–2WV and 5Cr–3WVTa1 showed relatively large increases compared to the lower temperatures; the large increase in total elongation at 600 °C for the 5Cr–3WVTa1 accompanies the large decrease in strength at this temperature (Fig. 2(a)).

When the steels were tempered at 750 °C (Fig. 3), the YS of the 5Cr–3WVTa1 steel was considerably higher than that of the other steels up to 500 °C. All of the

Table 2
Grain sizes of normalized-and-tempered steels

Steels	Grain size	
	ASTM no.	Size (μm)
5Cr–2WV	6	45
5Cr–2WVTa1	9	16
5Cr–2WVTa2	10	11
5Cr–3WV	6.5	39
5Cr–3WVTa1	8	22
5Cr–3WVTa1B1	8.5	19
5Cr–3WVTa1B2	10	11
7Cr–2WVTa	10	11
7Cr–3WVTa	9.5	14
7Cr–2WVTaB	9.5	14
9Cr–2WV	5	65
9Cr–2WVTa	7	32
9Cr–2WVTaB	7	32

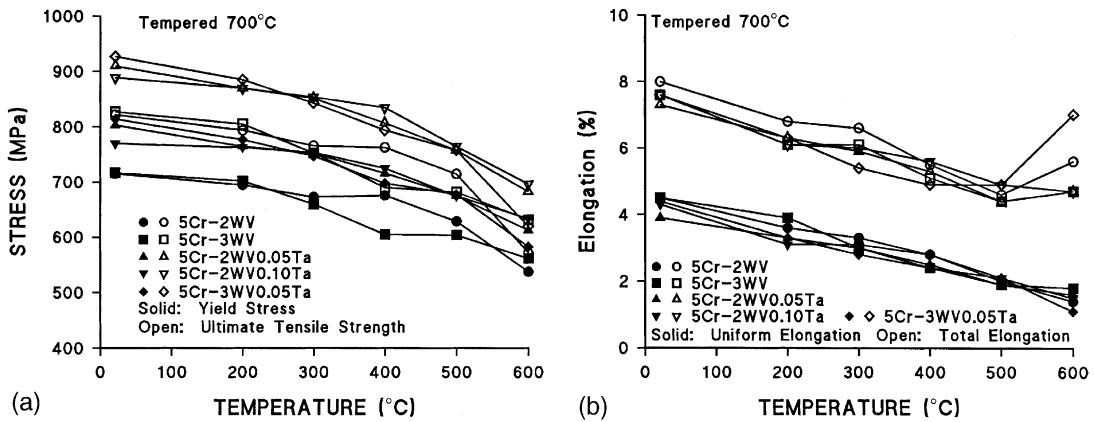


Fig. 2. (a) YS and UTS and (b) uniform and total elongation of 5Cr steels of different composition tempered at 700 °C.

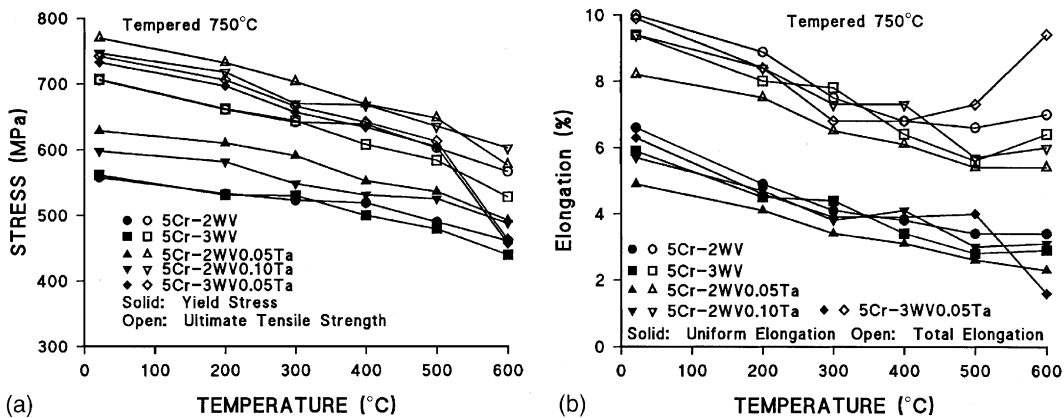


Fig. 3. (a) YS and UTS and (b) uniform and total elongation of 5Cr steels of different composition tempered at 750 °C.

tantalum-containing steels tempered at 750 °C were considerably stronger than those without tantalum, and there was no difference between the 5Cr-2WV and 5Cr-3WV steels (Fig. 3(a)), similar to the observations after tempering at 700 °C. After tempering at 750 °C, there was little difference in the YS and UTS for the 5Cr-3WV1, indicating that the steel had little work-hardening capacity. Because of this small difference in YS and UTS of the 5Cr-3WV1, the UTSs of the 5Cr-2WV1 and 5Cr-2WV2 steels were higher than those of the 5Cr-3WV1, even though the latter steel had a much higher YS from room temperature to 500 °C. As observed for the 5Cr-3WV1 steel after tempering at 700 °C (Fig. 2(a)), there was again a large decrease in YS and UTS in going from 500 to 600 °C relative to the change for the 5Cr-2WV1 and 5Cr-2WV2 steels (Fig. 3(a)). In fact, the YS of the 5Cr-3WV1 at 600 °C fell below that of the 5Cr-2WV1 and 5Cr-2WV2 steels, even though the latter were much weaker at the lower temperatures. The 5Cr-

2WV1 was stronger than the 5Cr-2WV2 at all temperatures but 600 °C, where they had similar strengths.

For the specimens tempered at 750 °C, there was somewhat more variation in the uniform and total elongation (Fig. 3(b)) than for those tempered at 700 °C. The 5Cr-2WV1 had the lowest ductility over the entire range, with considerable variability among the other steels. Again, the 5Cr-3WV1 showed a relatively large increase in total elongation at the higher test temperatures (400–600 °C). Contrary to this increase in total elongation at 600 °C for the 5Cr-3WV1, the uniform elongation showed a relatively large decrease, again emphasizing the lack of strain-hardening capacity for this steel.

When the properties of the tantalum-containing steels with boron were compared to the tantalum-containing steels without boron, the boron-containing steels were the weakest after tempering at both the 700 (Fig. 4(a)) and 750 °C (Fig. 5(a)). The uniform and total

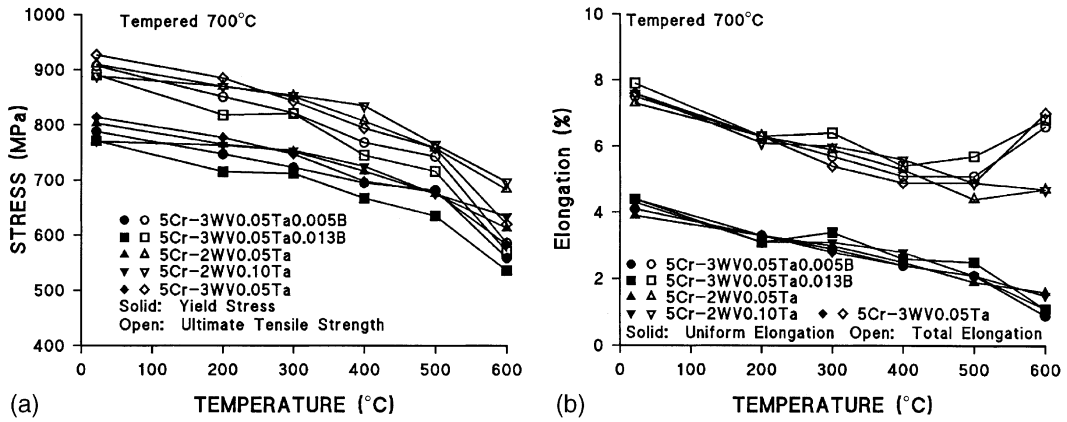


Fig. 4. (a) YS and UTS and (b) uniform and total elongation of 5Cr boron-containing steels of different composition tempered at 700 °C.

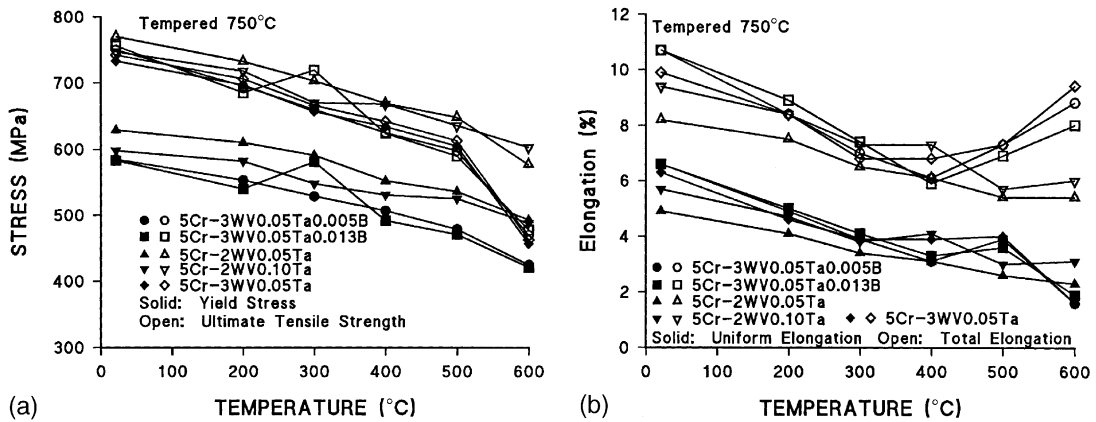


Fig. 5. (a) YS and UTS and (b) uniform and total elongation of 5Cr boron-containing steels compared with other compositions after tempering at 750 °C.

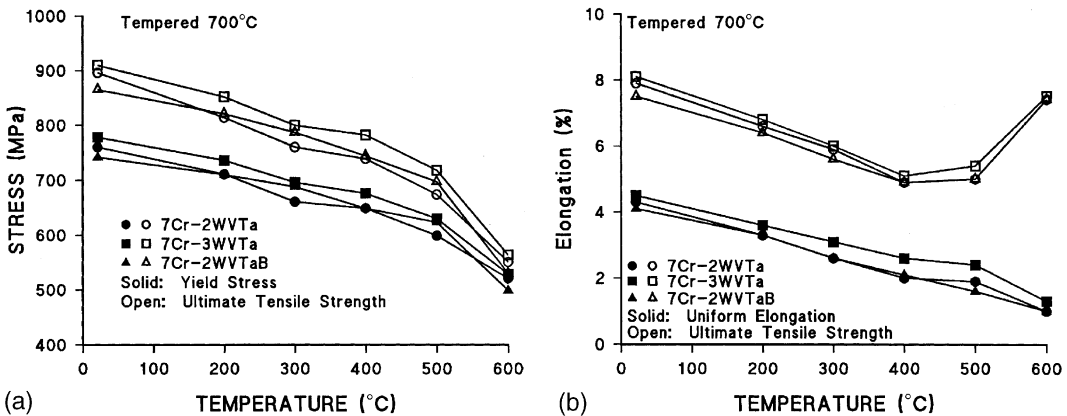


Fig. 6. (a) YS and UTS and (b) uniform and total elongation of 7Cr steels of different composition tempered at 700 °C.

elongations generally reflected the strength behavior in that the ductility was relatively high (Figs. 4(b) and

5(b)). At the highest test temperatures, the total elongation of the boron-containing steels increased, similar

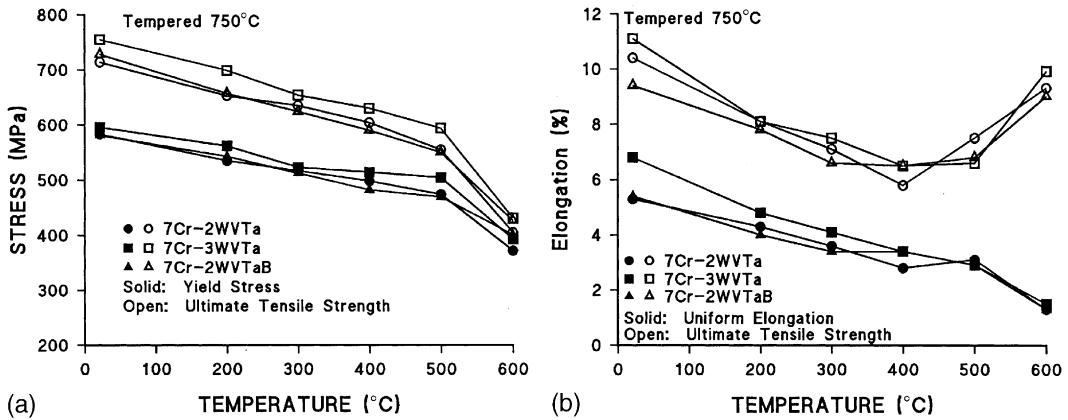


Fig. 7. (a) YS and UTS and (b) uniform and total elongation of 7Cr steels of different composition tempered at 750 °C.

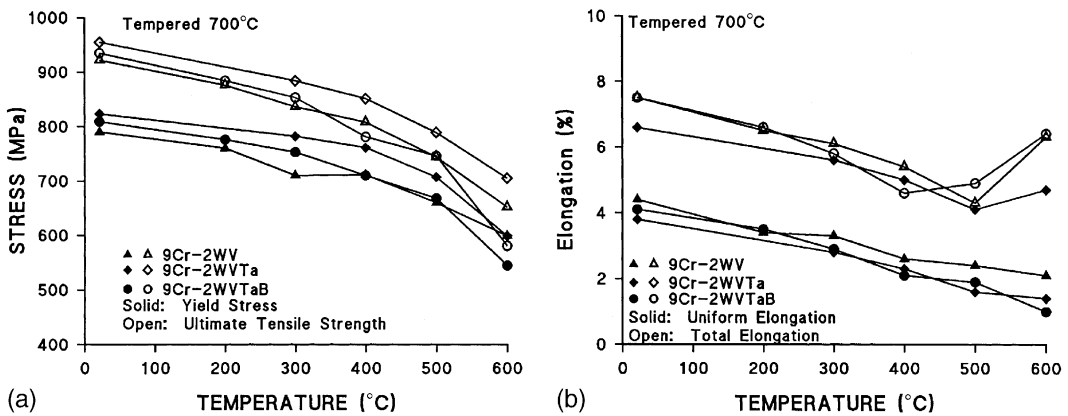


Fig. 8. (a) YS and UTS and (b) uniform and total elongation of 9Cr steels of different composition tempered at 700 °C.

to the increase observed for 5Cr-3WVTa1. At 600 °C, the uniform elongation decreased, which is also similar to the behavior of the 5Cr-3WVTa1.

3.2.2. 7Cr-W-V-Ta-B steels

For the 7Cr composition, the effect of the addition of 1% W or 0.005% B to the 7Cr-2WVTa steel composition was examined.¹ Although the 7Cr-3WVTa steel was generally slightly stronger than the other two steels from room temperature to 600 °C, the difference was relatively small after either the 700 °C (Fig. 6(a)) or 750 °C (Fig. 7(a)) temper. Similarly, there was relatively little

difference in the uniform and total elongation of the three steels (Figs. 6(b) and 7(b)). Despite being the strongest, the 7Cr-3WVTa steel generally had the highest uniform and total elongation. Whereas the increase in total elongation at the highest test temperature for the 5% Cr steels was greatest for the steel containing 3% W, for the 7% Cr steels all three steels showed similar changes.

3.2.3. 9Cr-W-V-Ta-B steels

The addition of tantalum to the 9Cr-2WV composition to produce 9Cr-2WVTa caused an increase in strength after both the 700 °C (Fig. 8(a)) and 750 °C (Fig. 9(a)) temper. After the 700 °C temper, there was little difference between 9Cr-2WV and 9Cr-2WVTaB, except at 600 °C, where the 9Cr-2WV was strongest (Fig. 8(a)), because of a rather abrupt decrease in strength for the 9Cr-2WVTaB. When tempered at 750 °C, the strength of the 9Cr-2WVTaB is similar to that of the 9Cr-2WVTa, except at 600 °C, where there is again

¹ All steels with tantalum and boron except the 5Cr-2WVTa2 and 5Cr-2WVTa1B2 contained about 0.05% Ta and 0.005% B. Therefore, these elements will not be designated by a number for the 7 and 9Cr steels, and when comparisons are made with 5Cr-2WVTa1, the latter will be designated 5Cr-2WVTa.

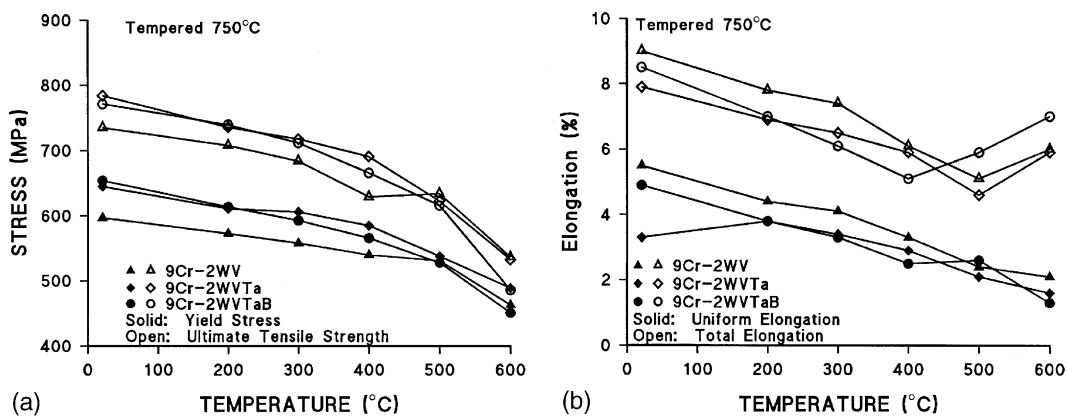


Fig. 9. (a) YS and UTS and (b) uniform and total elongation of 9Cr steels of different composition tempered at 750 °C.

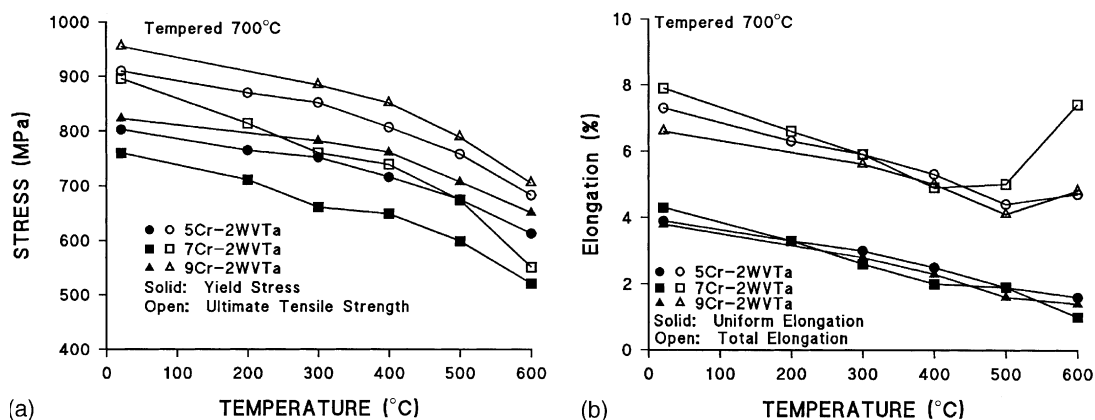


Fig. 10. A comparison of (a) YS and ultimate tensile stress and (b) uniform and total elongation of 5Cr-2WVTa, 7Cr-2WVTa, and 9Cr-2WVTa steels tempered at 700 °C.

a large reduction in the strength of the 9Cr-2WVTaB relative to the other steels, and it becomes the weakest (Fig. 9(a)). The 9Cr-2WV is weakest below 600 °C.

The ductilities do not show large differences between the different steels (Figs. 8(b) and 9(b)). Total elongation of all of the steels increased in going from 500 to 600 °C, with the greatest increase occurring for the 9Cr-2WVTaB steel, which also showed an increase in going from 400 to 500 °C. This steel had the lowest uniform elongation at 600 °C for both tempering conditions.

3.2.4. Comparison of 5Cr, 7Cr, and 9Cr steels

A comparison of the tensile properties of the 5Cr-2WVTa (0.5Ta or Ta1), 7Cr-2WVTa, and 9Cr-2WVTa steels indicated that after the 700 °C temper the 9Cr-2WVTa had a slightly higher YS than the 5Cr-2WVTa, with the YS of the 5Cr-2WVTa considerably above that of the 7Cr-2WVTa steel (Fig. 10(a)). A similar relative difference was observed for the UTS of the 5Cr-2WVTa and 9Cr-2WVTa steels, while that for the 7Cr-2WVTa

was similar to that of the 5Cr-2WVTa at room temperature, and then dropped off precipitously with temperature, until at 600 °C it was below the YS of the other two steels (Fig. 10(a)). Uniform elongations for the steels tempered at 700 °C (Fig. 10(b)) were similar at all test temperatures. Total elongations were also quite similar up to 600 °C, where the 7Cr-2WVTa steel showed a large increase.

After tempering at 750 °C, the 5Cr-2WVTa and 9Cr-2WVTa had similar strengths (YS and UTS), which were considerably above those for the 7Cr-2WVTa (Fig. 11(a)). The ductility behavior (Fig. 11(b)) was similar to that observed after the 700 °C temper.

3.3. Charpy impact behavior

The fracture mode of the steels after a Charpy test was typical for such steels: transgranular cleavage occurred for tests on the lower shelf, ductile transgranular

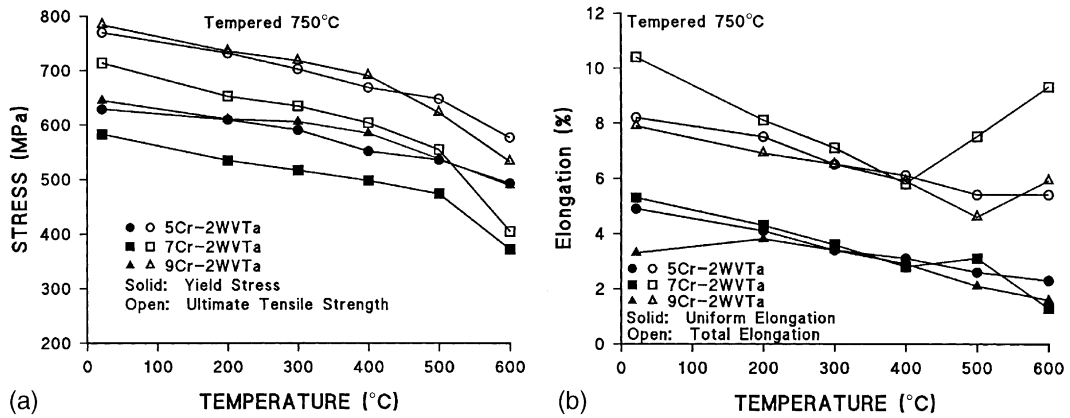


Fig. 11. A comparison of (a) YS and ultimate tensile stress and (b) uniform and total elongation of 5Cr-2WVTa, 7Cr-2WVTa, and 9Cr-2WVTa steels tempered at 750 °C.

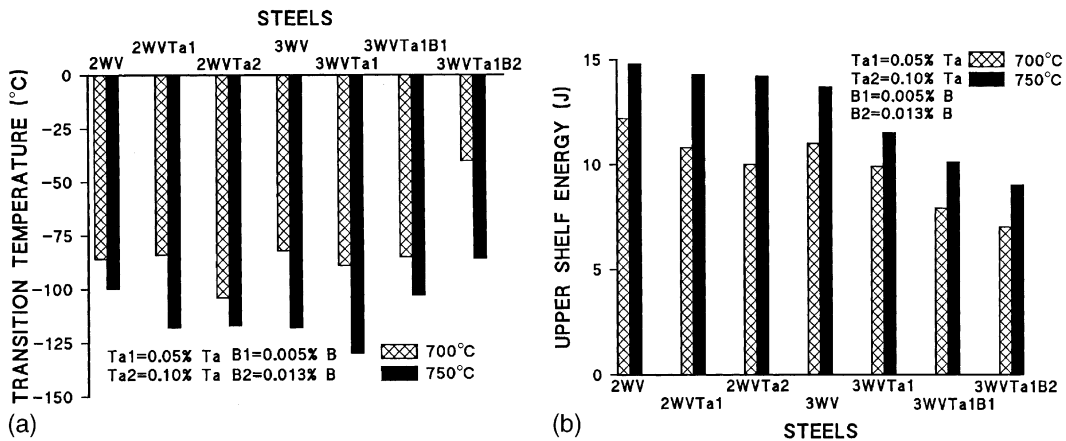


Fig. 12. (a) DBTT and (b) USE of 5Cr steels tempered at 700 and 750 °C.

failure with void coalescence occurred on the upper shelf, and a mixture of the two failure modes occurred in the transition region between the lower shelf and upper shelf.

3.3.1. 5Cr-W-V-Ta-B steels

By comparing the DBTT of the 5Cr-2WV steel with those with 0.05 (5Cr-2WVTa1) and 0.1% Ta (5Cr-2WVTa2) (Fig. 12(a)), it appears that 0.05% Ta has no effect when the steel is tempered at 700 °C, but there is a positive effect after tempering at 750 °C. On the other hand, when 5Cr-2WVTa1, a positive tantalum effect was observed only after tempering at 700 °C, which means that the 5Cr-2WVTa2 has a positive effect over the 5Cr-2WV after tempering at both temperatures. From a comparison of the 5Cr-3WV and 5Cr-3WVTa1, it is seen that the effect of tantalum is similar to that observed between

5Cr-2WV and 5Cr-2WVTa1 (i.e., a positive effect after tempering at 750 °C).

No effect of tungsten was observed when 5Cr-2WV and 5Cr-3WV were compared after tempering at 700 °C, but the 3% W steel had the lowest DBTT after tempering at 750 °C. This same effect of tungsten was observed by comparing the 5Cr-2WVTa1 and 5Cr-3WVTa1. The addition of boron in combination with tantalum caused a deterioration in the DBTT.

Neither tungsten, tantalum, nor boron favorably affected USE (compare the 5Cr-2WV with the other six steels) after either the 700 or 750 °C temper (Fig. 12(b)). After the 750 °C temper, the presence of tantalum in the steels with 2% W (either 0.05% or 0.1%) resulted in the USE values approaching those of the 5Cr-2WV steel. This was also true of the USE for the 5Cr-3WV, but not the 5Cr-3WVTa. As was true for the DBTT, boron caused a marked deterioration in the USE at both tempering conditions.

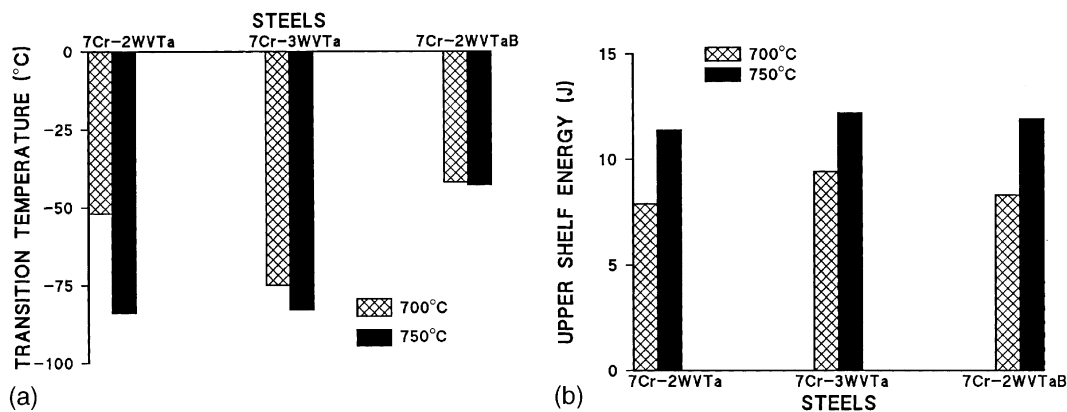


Fig. 13. (a) DBTT and (b) USE of 7Cr steels tempered at 700 and 750 °C.

3.3.2. 7Cr-W-V-Ta-B steels

For the 7% Cr steels, the effect of tungsten and boron was studied. The results confirmed the deterioration of the DBTT by the addition of boron observed in the 5Cr steels (Fig. 13(a)). An increase in tungsten from 2% to 3% favorably affected the DBTT for specimens tempered at 700 °C, but it had no effect on those tempered at 750 °C.

The USE of the 7Cr-3WVTa steel was a little higher than that of the 7Cr-2WVTa steel after both tempers, and the USE of the 7Cr-2WVTaB was similar to that of the 7Cr-2WVTa.

3.3.3. 9Cr-W-V-Ta-B steels

The positive effect of tantalum on the Charpy properties of 9Cr-2WV steel has been documented [6]. Fig. 14(a) shows this effect, and it also shows the effect of 0.005% B on the 9Cr-2WVTa steel. Contrary to the results for the 5Cr and 7Cr steels, boron favorably af-

ected the transition temperature after both the 700 and 750 °C tempers.

The USEs of the 9Cr-2WV, 9Cr-2WVTa, and 9Cr-2WVTaB steels were similar with perhaps a slight advantage for the 9Cr-2WVTaB (Fig. 14(b)).

3.3.4. Comparison of 5Cr, 7Cr, and 9Cr steels

When the 5Cr-2WVTa (0.5Ta-Ta1), 7Cr-2WVTa, and 9Cr-2WVTa steels were compared (Fig. 15(a)), the 5Cr-2WVTa had the lowest DBTT after both tempers. There was very little difference in the values for the 7Cr-2WVTa and 9Cr-2WVTa steels, with the values for the 7Cr-2WVTa being slightly lower. Similarly, the USE of the 5Cr-2WVTa was the largest after each heat treatment. Again, there was relatively little difference between the 7Cr-2WVTa and 9Cr-2WVTa steels, with a slight advantage for the 7Cr-2WVTa steel (Fig. 15(b)).

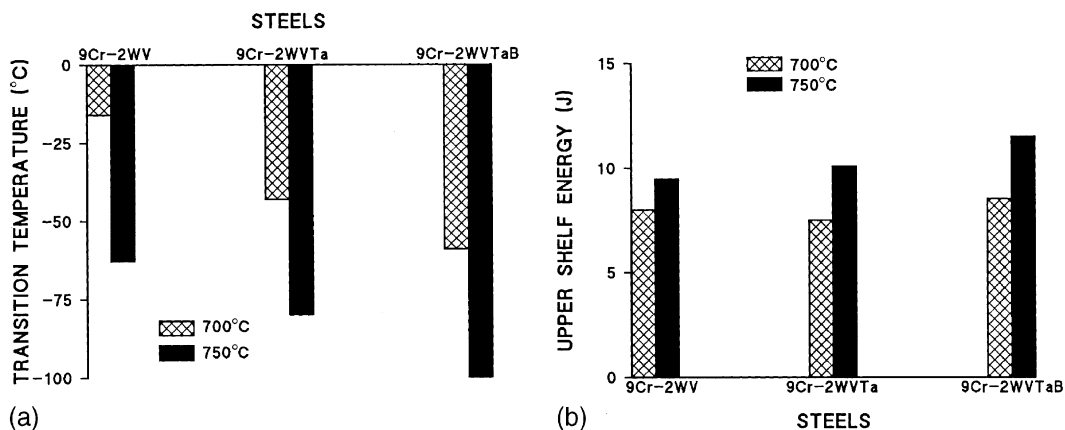


Fig. 14. (a) DBTT and (b) USE of 9Cr steels tempered at 700 and 750 °C.

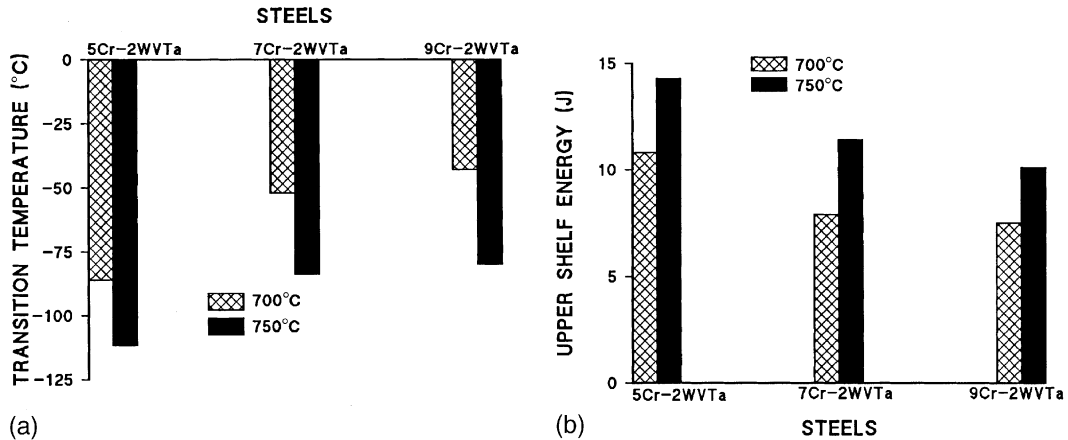


Fig. 15. A comparison of (a) DBTT and (b) USE of 5Cr-2WVTa, 7Cr-2WVTa, and 9Cr-2WVTa steels tempered at 700 and 750 °C.

4. Discussion

The microstructure of 9Cr-2WVTa has been examined previously by transmission electron microscopy (TEM) [10,14,15]. Large particles of $M_{23}C_6$ were observed on prior austenite grain boundaries and on lath boundaries along with small amounts of MC in the matrix [10,14,15]. No TEM was carried out in the present investigation, but the 5Cr-2WVTa, 7Cr-2WVTa, and 9Cr-2WVTa steels were examined previously [15]. Fig. 16 shows TEM microstructures of the steels, and Table 3 summarizes the observations on precipitate sizes and number density along with lath sizes. Lath sizes were similar for the three steels, but there were significant differences in the precipitates [15].

An important observation (Table 3) on the differences in the precipitates in the three steels is that the chromium-rich $M_{23}C_6$ makes up the majority of the precipitate in the 9Cr-2WVTa steel. As the chromium is reduced from 9% to 7%, chromium-rich M_7C_3 appears along with the $M_{23}C_6$. This trend continues when the chromium is reduced to 5%, and the 5Cr-2WVTa steel contains about an order of magnitude less $M_{23}C_6$ and an order of magnitude more M_7C_3 than the 7Cr-2WVTa steel. The 9Cr-2WVTa steel does not contain M_7C_3 . In Fig. 16 it appears that the 5Cr-2WVTa steel contains a much higher density of precipitates than the 7Cr-2WVTa and 9Cr-2WVTa steels. That is because M_7C_3 and $M_{23}C_6$ are the prominent precipitates visible in the figures, and the 5Cr steel contains about an order of magnitude more of these large precipitates.

The other important observation (Table 3) concerns the small carbide (MC) and carbonitride [M(CN)] precipitates in the steels [15]. In the 9Cr-2WVTa steel, spherical vanadium-rich particles and a few tantalum-rich particles were observed and identified as MC [10, 14,15]. The 5Cr-2WVTa and 7Cr-2WVTa steels con-

tained small needle-shaped vanadium-rich particles that atom probe analyses indicated contained both carbon and nitrogen and were concluded to be carbonitrides [15]. The M(CN) particles were smaller than the MC particles in the 9Cr-2WVTa steel and were present at a much higher number density (Table 3), as shown in Fig. 17 for the 7Cr-2WVTa steel. Note that even though the 9Cr-2WVTa steel contained somewhat more nitrogen than the 5Cr and 7Cr steels (Table 1), the fine precipitates that were identified as carbonitrides by atom probe were not observed in the 9Cr steel.

Mechanical properties results obtained in the present investigation on the 5% and 9% Cr steels indicate that tantalum has a positive effect on strength (Figs. 2, 3, 8, 9). For the 5% Cr steels this was observed for both the 2% and 3% W steels with 0.05% Ta, although there was little difference between the strength of the 5Cr-2WV steel with 0.05% and 0.1% Ta.

Tantalum also favorably affected the DBTT of the 5Cr and 9Cr steels. For the 5Cr steels with 0.05% Ta, the effect was significant only after a 750 °C temper (Fig. 12(a)); the 5Cr steel with 0.1% Ta showed an effect after both tempers, as was the case for the 9Cr steels (Fig. 14(a)). The USE did not appear to be affected by tantalum for either the 5Cr (Fig. 12(b)) or 9Cr (Fig. 14(b)) steels. Further TEM is required to explain these observations.

The use of 3% instead of 2% W in the 5Cr steels had no effect on strength in the absence of tantalum. In the presence of 0.05% Ta, there was little effect after the 700 °C temper, but there was a positive effect on YS up to ≈ 500 °C when tempered at 750 °C (Fig. 3(a)). However, at 600 °C, the strength of the 5Cr-3WVTa1 steel deteriorated significantly, thus indicating little or no advantage to the extra tungsten for the 5% Cr steels. Furthermore, although the YS was highest of all the 5Cr steels to 500 °C, there was little difference in the YS and

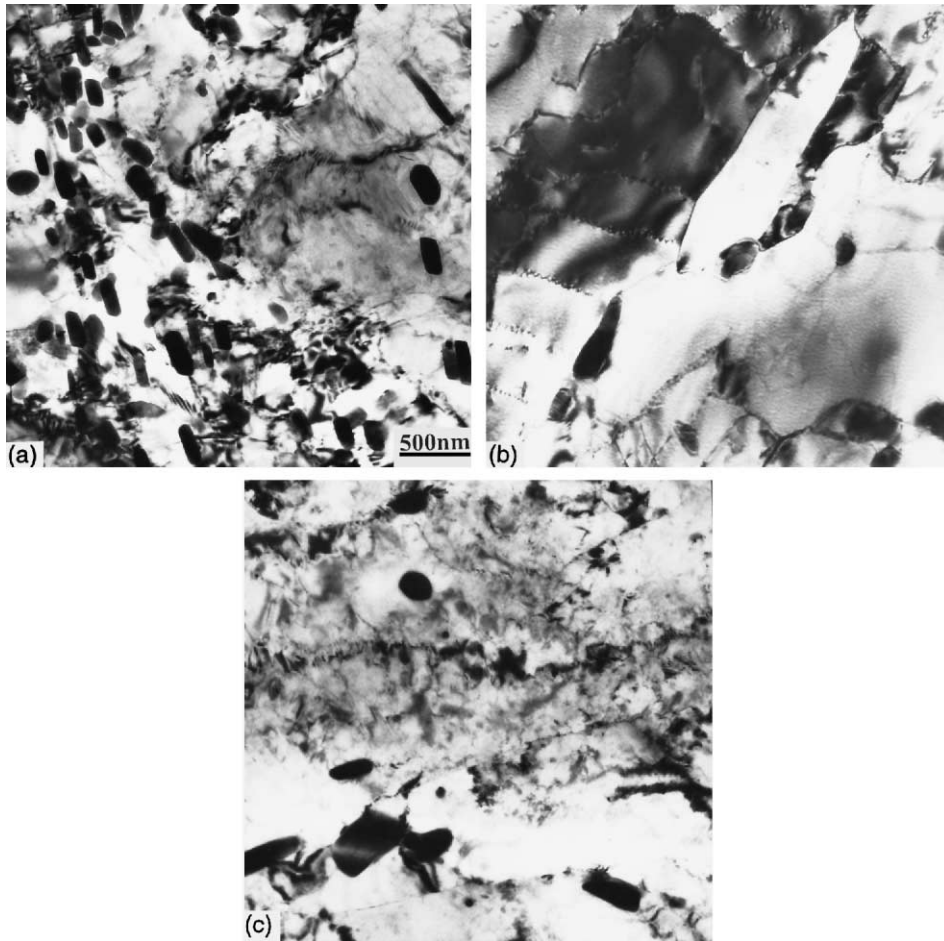


Fig. 16. Transmission electron micrographs of (a) 5Cr-2WVTa, (b) 7Cr-2WVTa, and (c) 9Cr-2WVTa steels [15].

Table 3
Estimate of number and size of microstructural features [15]

Feature ^a	5Cr-2WVTa	7Cr-2WVTa	9Cr-2WVTa
Prior austenite grain size (μm)	16	11	32
Lath size (μm)	0.09–0.2	0.1–0.3	0.1–0.3
M_{23}C_6 N_v (m^{-3})	1.0×10^{18} – 10^{19}	1.0×10^{19} – 10^{20}	1.0×10^{19} – 10^{20}
Diameter (nm)	200–400	100–200	100–200
M_7C_3 N_v (m^{-3})	1.0×10^{20} – 10^{21}	1.0×10^{19} – 10^{20}	ND ^b
Diameter (nm)	100–300	100–300	
MC N_v (m^{-3})	ND	ND	1.0×10^{17} – 10^{18}
Diameter (nm)			20–40
M(CN) N_v (m^{-3})	1.0×10^{21}	1.0×10^{23}	ND
Diameter (nm)	5×30	5×30	

^a N_v is number density.

^b Not detected.

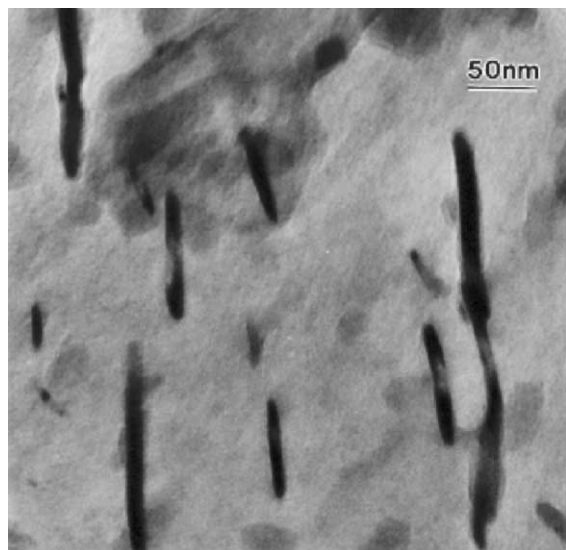


Fig. 17. Transmission electron micrograph of needle-like M(CN) precipitates in the 7Cr-2WVTa steel [15].

UTS, indicating a lack of work-hardening capability. For the 7Cr-2WVTa and 7Cr-3WVTa, the 7Cr-3WVTa steel was generally stronger up to 600 °C, where there was little difference in strength (Figs. 6(a) and 7(a)).

The presence of 3% W seemed to have a positive effect on the DBTT for the 5% Cr steel after the 750 °C temper (Fig. 12(a)). There was essentially no effect of tungsten for the 7Cr steel after the 750 °C temper, with the appearance of a slight effect after the 700 °C temper (Fig. 13(a)). The USE appeared little affected by the extra tungsten for either the 5Cr or 7Cr steels (Figs. 12(b) and 13(b)).

The deterioration of the strength at 600 °C for the high-tungsten steel, especially the 5Cr steel, would appear to be a negative indication for creep-rupture strength, an important consideration for the application of these steels. No such deterioration of strength was observed for the 7Cr steel, although more tungsten did not improve strength. The observed strength deterioration appears to differ from results obtained by Abe et al. [8,9] for 9Cr-XW steels ($X = 0, 1, 2, 3,$ and 4). They found that creep-rupture strength at 600 °C increased with increasing tungsten up to 3%. They did not report the effect of tungsten on tensile properties, but they would be expected to parallel the creep behavior. The DBTT for the 9Cr-XW steels had a minimum at 1% W. The 1% W steel had better impact toughness than the 3% W steel, and the 3% W steel had better creep-rupture properties. Unfortunately, the effect of tungsten on the 9Cr steel was not evaluated in the present work. Based on the Abe et al. observations [8,9] and the more favorable effect of tungsten on the 7Cr steel, it appears

that tungsten has a favorable effect on strength with increasing chromium concentration.

The tensile and Charpy results from the studies on the 5% and 7% Cr steels provide no reason to use boron in a 5% and 7% Cr steel, as has been used in some other such steels. In fact the properties of the boron-containing 5% and 7% Cr steels were inferior to those without boron. However, the boron addition to 9Cr-2WVTa steel did favorably affect the properties. In addition to studying the creep-rupture properties of the 9Cr-XW steels, Abe et al. [8,9] did creep-rupture tests at 600 °C on 9Cr-1WVTaB and 9Cr-3WVTaB steels. They found that the 3% W steel had significantly better properties than the steel with 1% W. This may indicate the combination of W, Ta, and B can have a favorable effect on the tensile, Charpy, and creep properties for 9Cr steels but not on the 5 and 7Cr steels, although the effect of boron on creep properties of these latter steels has not been determined.

One explanation advanced for a favorable effect of boron on creep properties on 9–12% Cr steels is that it is enriched in the $M_{23}C_6$, which stabilizes the precipitate relative to the precipitate without boron. This, in turn, stabilizes the lath martensite structure, thus inhibiting subgrain growth [16]. Boron in 9–12% Cr steels has been found in $M_{23}C_6$ by atom probe field ion microscopy, with the boron located near the surface of the precipitate [17,18]. No enrichment of boron in MX or Laves phase was observed [17,18]. Since a higher number density of smaller particles of $M_{23}C_6$ was observed in the 9Cr steel than the 5Cr steel [15], an effect on the precipitate in the 9Cr steel may be a cause, although more TEM is required to determine the origin of the difference.

The strength comparison of 5Cr-2WVTa, 7Cr-2WVTa, and 9Cr-2WVTa steels indicates that after tempering at 750 °C, there was essentially no difference in the strength of the 5Cr-2WVTa and 9Cr-2WVTa, and they were considerably stronger than the 7Cr-2WVTa (Fig. 11(a)). The ductility of the 5Cr-2WVTa and 9Cr-2WVTa steels were similar. The reason for the comparative strengths is not obvious from the microstructure (Table 3). The 5Cr-2WVTa has a higher number density of a smaller size of the carbonitrides than the fine MX of the 9Cr-2WVTa. However, the 7Cr-2WVTa has a higher density of the carbonitrides than the 5Cr-2WVTa steel. The other difference between the 7 and 5Cr steels is that the 5Cr steel contained more M_7C_3 , but this difference would not be expected to greatly affect the strength, given the large size and low number density of these precipitates.

When the Charpy properties are compared for these three steels, the 5Cr-2WVTa has a clear advantage. In fact, the DBTT (Fig. 15(a)) and USE (Fig. 15(b)) of this steel after tempering at 700 °C are as good or better than those for the 7Cr-2WVTa and 9Cr-2WVTa steels tempered at 750 °C. Since the 9Cr-2WVTa steel needs to be

tempered at 750 °C to get adequate toughness, it would appear from the present tests that the 5Cr–2WVTa steel may have advantages in both strength and impact toughness.

The emphasis of this discussion on the effect of composition has been on the precipitates. The strength and toughness will also depend on the dislocation structure, which can also be affected by the chemical composition through the precipitates and the effect on solid-solution strengthening. For a detailed analysis of the microstructure-mechanical properties correlation, the combination of grain size, lath size, precipitate distribution, dislocation density, etc., would all need to be considered.

A composition of 7.5–10Cr–2WVTa has generally been accepted as best for future fusion applications. If the apparent minimum in strength between 5% and 9% Cr observed here is an accurate representation of the behavior of these steels, then a composition near 9% Cr should be used. The F82H steel presently being used in an International Energy Agency collaborative fusion materials program has a nominal composition of Fe–7.5Cr–2W–0.2V–0.03Ta–0.1C, which may be a low chromium concentration given the results of this work.

5. Summary and conclusions

The effect of Ta, W, B, and Cr composition in Cr–2WV reduced-activation martensitic steels was investigated in the normalized-and-tempered (tempered at 700 and 750 °C) condition. Steels with 5%, 7%, and 9% Cr were examined to determine the effect of increasing tungsten from 2% to 3%, changing tantalum from 0% to 0.1%, and changing boron from 0% to 0.013%. The effect of W, Ta, and B was investigated for the 5Cr steel, W and B for the 7Cr steel, and Ta and B for the 9Cr steel. The results are summarized as follows:

Tantalum effect: Tantalum had a positive effect on strength and DBTT of the 5Cr and 9Cr steels. The USE was not affected by tantalum for either steel.

Tungsten effect: Using 3% W instead of 2% W in the 5Cr steels had no effect on strength in the absence of tantalum. For 0.05% Ta, there was little effect after the 700 °C temper, but there was a positive effect when tempered at 750 °C. The increase to 3% W improved the DBTT for the 5% Cr steel after a 750 °C temper and had a slight effect on the 7% Cr steel after a 700 °C temper. The USE was unaffected by tungsten in the 5Cr and 7Cr steels.

Boron effect: Boron had no favorable effect on the properties of the 5% and 7% Cr steels but did favorably affect the strength and impact properties of 9Cr–2WVTa.

Chromium effect: The 5Cr–2WVTa and 9Cr–2WVTa steels with 0.05% Ta appeared to be near the optimum composition for the compositions examined. After tempering at 750 °C, there was no difference in the strength and ductility of the 5Cr–2WVTa and 9Cr–2WVTa, and they were superior to 7Cr–2WVTa. A composition of 7.5–10Cr–2WVTa has been accepted in the literature as best for future fusion applications. If the apparent minimum in strength between 5% and 9% Cr is an accurate representation, then steels with at least 9% Cr should be used. For the 5Cr–2WVTa, 7Cr–2WVTa, and 9Cr–2WVTa steels, the 5Cr–2WVTa had a clear advantage. The DBTT and USE of this steel after tempering at 700 °C are as good or better than those of the 7Cr–2WVTa and 9Cr–2WVTa tempered at 750 °C. Since 9Cr–2WVTa needs to be tempered at 750 °C for adequate toughness, the 5Cr–2WVTa steel may have advantages in both strength and impact toughness in the unirradiated condition. Further work on thermal creep needs to be performed to assess the potential stability of the 5Cr–2WVTa steel.

Acknowledgements

We wish to thank L.T. Gibson and E.T. Manneschildt for conducting the tensile tests and impact tests, respectively. Drs N. Hashimoto, M.L. Grossbeck, and S.J. Zinkle reviewed the manuscript and provided useful suggestions.

References

- [1] R.L. Klueh, D.J. Alexander, in: R.K. Nanstad, M.L. Hamilton, F.A. Garner, A.S. Kumar (Eds.), *Effects of Radiation on Materials: 18th International Symposium*, ASTM STP 1325, American Society for Testing and Materials, Philadelphia, PA, 1999, p. 911.
- [2] K. Shiba, I. Ioka, J.E. Robertson, M. Suzuki, A. Hishinuma, in: *Proc. Conference on Materials and Nuclear Power*, Institute of Materials, London, 1996, p. 265.
- [3] E.V. van Osch, M.G. Horsten, G.E. Lucas, G.R. Odette, in: M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L. Grossbeck (Eds.), *Effects of Irradiation on Materials: 19th International Symposium*, ASTM STP 1366, American Society for Testing and Materials, West Conshohocken, PA, 2000, p. 612.
- [4] A. Kohyama, Y. Kohno, K. Asakura, H. Kayano, *J. Nucl. Mater.* 212–214 (1994) 684.
- [5] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, *J. Nucl. Mater.* 233–237 (1996) 138.
- [6] H. Kayano, A. Kimura, M. Narui, Y. Sasaki, Y. Suzuki, S. Ohta, *J. Nucl. Mater.* 155–157 (1988) 978.
- [7] D.S. Gelles, in: R.L. Klueh, D.S. Gelles, M. Okada, N.H. Packan (Eds.), *Reduced Activation Materials for Fusion*

- Reactors, ASTM STP 1047, ASTM, Philadelphia, PA, 1990, p. 113.
- [8] F. Abe, T. Noda, H. Araki, S. Nakazawa, *J. Nucl. Mater.* 179–181 (1991) 663.
- [9] F. Abe, H. Araki, T. Noda, *Mater. Sci. Tech.* 6 (1990) 714.
- [10] R.L. Klueh, P.J. Maziasz, *Met. Trans.* 20A (1989) 373.
- [11] D.J. Alexander, R.K. Nanstad, W.R. Corwin, J.T. Hutton, in: A.A. Braun, N.E. Ashbaugh, F.M. Smith (Eds.), *Automation Technology to Fatigue and Fracture Testing*, ASTM STP 1092, American Society for Testing and Materials, Philadelphia, PA, 1990, p. 83.
- [12] D.J. Alexander, R.L. Klueh, in: J.M. Molt (Ed.), *Charpy Impact Test: Factors and Variables*, ASTM STP 1072, American Society for Testing and Materials, Philadelphia, PA, 1990, p. 179.
- [13] M.A. Sokolov, R.K. Nanstad, in: D.S. Gelles, R.K. Nanstad, A.S. Kumar, E.A. Little (Eds.), *Effects of Radiation on Materials: 17th International Symposium*, ASTM STP 1270, American Society for Testing and Materials, Philadelphia, PA, 1996, p. 384.
- [14] J.J. Kai, R.L. Klueh, *J. Nucl. Mater.* 230 (1996) 116.
- [15] R. Jayaram, R.L. Klueh, *Met. Mater. Trans. A* 29A (1998) 1551.
- [16] F. Abe, M. Igarashi, S. Wanikawa, M. Tabuchi, T. Itagaki, K. Kimura, K. Yamaguchi, in: R. Viswanathan, W.T. Bakker, J.D. Parker (Eds.), *Advances in Materials Technology for Fossil Power Plants*, Institute of Materials, London, 2001, p. 79.
- [17] M. Schwind, M. Hättestrand, H.-O. Andrén, in: A. Strang, J. Cawley, G.W. Greenwood (Eds.), *Microstructural Stability of Creep Resistant Alloys for High Temperature Plant Applications*, Institute of Materials, London, 1998, p. 197.
- [18] M. Hättestrand, M. Schwind, H.-O. Andrén, in: R. Viswanathan, J. Nutting (Eds.), *Advanced Heat Resistant Steel for Power Generation*, Institute of Materials, London, 1999, p. 199.