MECHANICAL PROPERTIES OF UNIRRADIATED AND IRRADIATED REDUCED-ACTIVATION MARTENSITIC STEELS WITH AND WITHOUT NICKEL COMPARED TO PROPERTIES OF COMMERCIAL STEELS—R. L. Klueh, M. A. Sokolov, and N. Hashimoto (Oak Ridge National Laboratory)

### **OBJECTIVE**

The objective of this work is to develop an understanding of the effect of irradiation on mechanical properties of reduced-activation ferritic/martensitic steels that are of interest for fusion applications and to use that knowledge to develop steels with improved properties.

### **ABSTRACT**

Tensile and Charpy specimens of four normalized-and-tempered martensitic steels were irradiated to 23-33 dpa at 376–405°C in the Experimental Breeder Reactor (EBR-II). The steels were the ORNL reduced-activation steel 9Cr-2WVTa and that containing 2% Ni (9Cr-2WVTa-2Ni), modified 9Cr-1Mo (9Cr-2WVTa), and Sandvik HT9 (12Cr-1MoVW). Two tempering conditions were used for 9Cr-2WVTa and 9Cr-2WVTa-2Ni: 1 hr at 700°C and 1 h at 750°C. The 9Cr-1MoVNb and 12Cr-1MoVW were tempered 1 h at 760°C. These heat treatments produced tempered-martensite microstructures for all steels except 9Cr-2WVTa-2Ni tempered at 750°C, where, a duplex structure of tempered and untempered martensite formed. Based on changes in tensile and Charpy impact properties, the results demonstrated the superiority in strength and ductility of the 9Cr-2WVTa reduced-activation steel over the commercial steels. Comparison of the mechanical properties after irradiation of 9Cr-2WVTa-2Ni and 9Cr-2WVTa steels indicated a favorable effect of nickel that could lead to development of a heat treatment for improved irradiation resistance.

### Introduction

The 9Cr reduced-activation ferritic/martensitic steels are candidates for applications as first wall and blanket structural materials for future fusion reactors. Displacement damage by neutron irradiation of this type of steel below 425–450°C hardens the steel lattice, causing an increase in strength and a decrease in toughness. The effect on impact toughness is measured in a Charpy test as an increase in the ductile-brittle transition temperature (DBTT) and a decrease in the upper-shelf energy (USE).

The possible effect of helium on hardening and embrittlement is important because large amounts of transmutation helium will form in a ferritic/martensitic steel first wall of a fusion reactor. Nickel-doped 9 and 12 Cr steels have been irradiated in the High Flux Isotope Reactor (HFIR) to study the effect of helium on fracture [1]. Helium is formed in a mixed-spectrum reactor such as HFIR by a two-step transmutation reaction between <sup>58</sup>Ni and the thermal neutrons; natural nickel contains 68% <sup>58</sup>Ni. This technique allows for the simultaneous production of displacement damage from the fast neutrons in the spectrum and helium from the thermal neutrons, thus simulating what will happen in a fusion reactor first wall.

Results from irradiation experiments on nickel-doped 9Cr-1MoVNb and 12Cr-1MoVW steels in HFIR at 300 and 400°C indicated that helium caused embrittlement in addition to that due to displacement damage alone [2–4]. On the other hand, some low-temperature irradiation experiments of nicked-doped 9Cr reduced-activation steels irradiated under conditions where no helium formed showed that the nickel-doped steels hardened more than steels without the nickel addition [5,6]. These results indicated that the nickel-doping simulation technique should be used with caution, especially below about 300°C.

In this paper, tensile and Charpy properties are reported for the reduced-activation steel ORNL 9Cr-2WVTa and this steel containing 2% Ni (9Cr-2WVTa-2Ni) after irradiation in the Experimental Breeder Reactor (EBR-II)—a fast reactor where little helium forms. The objective was to determine whether there is increased hardening in the nickel-containing steel compared to the steel without nickel. In addition, the commercial non-reduced-activation steels modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-

1MoVW) were irradiated and tested to determine differences between reduced-activation and conventional steels.

## **Experimental Procedure**

Compositions and designations of the steels used in this experiment are given in Table 1. In the original Oak Ridge National Laboratory (ORNL) program to develop reduced-activation steels [7], an 18-kg heat of the electroslag-remelted 9Cr-2WVTa steel was produced by Combustion Engineering Inc, Chattanooga, TN. Material from that heat was used as the master alloy to prepare 450-g vacuum arcmelted button heats of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels. The 9Cr-2WVTa heat was a remelt of the master alloy so that the steels could be compared after similar processing.

Elementa 9Cr-2WVTa 9Cr-2WVTa-2Ni 9Cr-1MoVNbb 12Cr-1MoVW<sup>c</sup> С 0.098 0.098 0.092 0.20 Si 0.19 0.19 0.15 0.17 0.39 0.38 0.48 0.57 Mn Ρ 0.014 0.012 0.014 0.016 S 0.003 0.004 0.003 0.003 Cr 8.71 8.55 8.32 12.1 Мо < 0.01 < 0.01 0.86 1.04 W 2.17 2.15 < 0.01 0.61 Ni 0.02 2.01 0.09 0.51 0.23 0.23 0.20 V 0.29 Nb <0.01 <0.01 0.06 <0.001 0.06 0.06 Та Ν 0.016 0.016 0.054 0.027

Table 1. Chemical composition of the steels tested

The small heats were cast as 25.4 mm x 12.7mm x 152 mm ingots, after which they were rolled to 6.4-mm plate and 0.76 mm sheet. The steels were normalized by austenitizing for 0.5 h at 1050°C in a helium atmosphere, after which they were quickly cooled in flowing helium. Specimens were irradiated in two tempered conditions: 1 h at 700°C and 1 h at 750°C.

The modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) steels were from large commercial-size heats that have been irradiated previously and were included in this experiment as benchmarks for the reduced-activation steels.

Tensile specimens 44.5-mm long with a reduced gage section of 20.3 x 1.52 x 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. Tests were conducted on irradiated and unirradiated specimens at 400°C (near the irradiation temperature) in vacuum on a 44-kN Instron universal testing machine at a nominal strain rate of  $4 \times 10^{-4} \, \text{s}^{-1}$ .

Two tensile specimens of each heat and each heat-treated condition were irradiated in the COBRA experiment in EBR-II at temperatures of 390 to 395°C. Fluence was determined from flux monitors in the irradiation canisters. There were minor variation for different specimens, depending on their position in the canisters, but the individual sets of specimens for a given steel and heat treatment were kept together in the canisters and experienced the same irradiation conditions. Tensile specimens were irradiated to  $6.7 \times 10^{26}$  to  $6.9 \times 10^{26}$  n/m<sup>2</sup> (E>0.1 MeV), which produced between 32 and 33 dpa.

<sup>&</sup>lt;sup>a</sup>Balance iron

<sup>&</sup>lt;sup>b</sup>Modified 9Cr-1Mo steel

<sup>&</sup>lt;sup>c</sup>Sandvik HT9

One-third-size Charpy specimens measuring 3.3 x 3.3 x 25.4 mm with a 0.51-mm-deep 30° V-notch and a 0.05- to 0.08-mm-root radius were machined from normalized-and-tempered 6.4-mm plates. Specimens were machined with the longitudinal axis along the rolling direction and the notch transverse to the rolling direction (L-T orientation). The absorbed energy vs. temperature values were fit with a hyperbolic tangent function to permit the USE and DBTT to be consistently evaluated. The DBTT was determined at an energy level midway between the upper- and lower-shelf energies. Details of the test procedure for the subsize Charpy specimens have been published [10–12].

Six Charpy specimens of each heat and each heat-treated condition were irradiated in the COBRA experiment in EBR-II at temperatures of 376 to 405°C. Individual sets of Charpy specimens for a given steel and heat treatment were kept together, although the conditions were somewhat different from the tensile specimens. Charpy specimens were irradiated to  $5.1 \times 10^{26}$  to  $6.9 \times 10^{26}$  n/m² (E>0.1 MeV), which produced between 23 and 33 dpa. Helium concentrations for both the tensile and Charpy specimens were calculated as 3–6 appm, depending on the dose and composition (the 6 appm was for the steel containing 2% Ni).

#### Results

First, results for 9Cr-2WVTa steel with and without nickel will be presented to demonstrate the effect of tempering and the effect of nickel. After that, results for the reduced-activation 9Cr-2WVTa and 9Cr-2WVTa-2Ni will be compared with the commercial modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) steels.

# 9Cr2WVTa and 9Cr-2WVTa-2Ni Steels

## **Tensile Properties**

In the unirradiated condition, the strength of the 9Cr-2WVTa was substantially higher and ductility lower when tempered at 700°C than at 750°C (Table 2 and Figs. 1 and 2). The strength of the 9Cr-2WVTa-2Ni after the 700°C temper was essentially the same as that for 9Cr-2WVTa tempered at 700°C. The yield stress for 9Cr-2WVTa-2Ni also decreased when tempered at 750°C compared to 700°C, but the relative change was much less than for 9Cr-2WVTa (≈5% vs. 26% decrease). When tempered at 750°C, the 9Cr-2WVTa-2Ni was considerably stronger than the 9Cr-2WVTa. There was no difference in the ultimate tensile strength of the 9Cr-2WVTa-2Ni for the two tempering conditions, and contrary to observations on 9Cr-2WVTa, the total elongation after the 750°C temper was less than after the 700°C temper, and there was no change in uniform elongation.

Table 2	Tancila data	for unirradiated	l and irradiated	ctable tactad	<b>△+ //∩∩</b> 0℃*
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Steel	Tompor	Irradiation	Strength (MPa)		Elongation (%)		
Sieei	Temper	IITaulation	Yield	Yield Ultimate		Uniform Total	
9Cr-2WVTa	700°C	Unirradiated	701	758	2.7	9.7	
9Cr-2WVTa	700°C	390°C/32.6dpa	698	732	1.2	6.5	
9Cr-2WVTa	750°C	Unirradiated	517	595	3.3	11.0	
9Cr-2WVTa	750°C	390°C/32.6dpa	613	645	1.3	7.1	
9Cr-2WVTa -2Ni	700°C	Unirradiated	707	788	2.7	11.3	
9Cr-2WVTa -2Ni	700°C	390°C/32.6dpa	649	690	3.4	10.0	
9Cr-2WVTa -2Ni	750°C	Unirradiated	674	787	2.7	8.3	
9Cr-2WVTa -2Ni	750°C	390°C/32.6dpa	637	699	3.4	10.8	
9Cr-1MoVNb	760°C	Unirradiated	562	636	1.4	4.6	
9Cr-1MoVNb	760°C	395°C/32.1dpa	612	626	0.7	5.3	
12Cr-1MoVW	760°C	Unirradiated	558	697	1.9	4.3	
12Cr-1MoVW	760°C	395°C/32.1dpa	720	776	1.7	4.0	

<sup>\*</sup>Data are for the average of two tests.

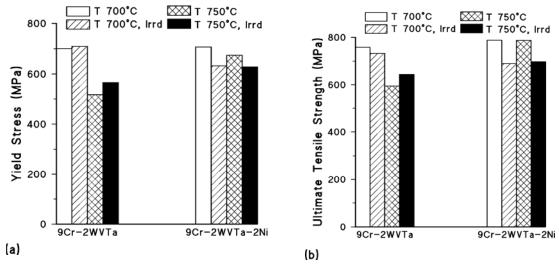


Fig. 1. (a) Yield stress and (b) ultimate tensile strength of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

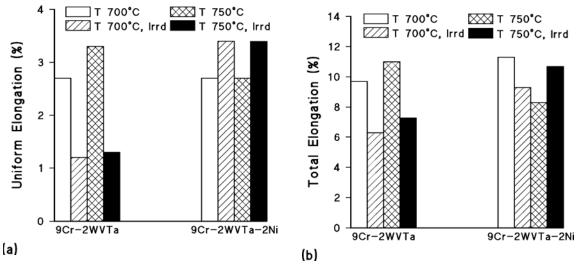


Fig. 2. (a) Uniform elongation and (b) total elongation of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

Irradiated tensile specimens were tested at 400°C (Table 2 and Figs. 1 and 2), which is near the irradiation temperature (390°C). There was little effect of irradiation on strength for the 9Cr-2WVTa tempered at 700°C and on the 9Cr-2WVTa-2Ni for both tempering conditions (Fig. 1). A small amount of irradiation hardening was observed for 9Cr-2WVTa after the 750°C temper. The effect of irradiation on ductility (Fig. 2) appeared to be greater for 9Cr-2WVTa than 9Cr-2WVTa-2Ni. For both tempering conditions, uniform elongation of the 9Cr-2WVTa-2Ni increased after irradiation, while that for 9Cr-2WVTa decreased. Total elongation of the 9Cr-2WVTa also decreased after irradiation for both tempering conditions, but the total elongation for 9Cr-2WVTa-2Ni decreased slightly after the 700°C temper and increased slightly after the 750°C temper.

# **Charpy Properties**

Charpy data are summarized in Table 3. Both the 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels with either the 700 and 750°C tempers had low DBTTs in the unirradiated condition (Fig. 3). Nickel had a beneficial effect on the transition temperature prior to irradiation, for in both heat-treated conditions, the nickel-containing steel had the lowest value.

Steel	Temper	Irrdn	Dose	Uirrd	Irrd	DBTT	Uirrd	Irrd USE
Sieei		Temp		DBTT	DBTT	Shift	USE	
9Cr-2WVTa	700°C	376°C	23 dpa	-77°C	-26°C	51°C	9.6 J	7.1 J
901-2VVV1a	750°C	390°C	33 dpa	-81°C	-98°C	0°C	14.0 J	9.0 J
9Cr-2WVT-2Ni	700°C	404°C	26 dpa	-97°C	-81°C	16°C	9.1 J	7.6 J
9CI-2VVV I-2IVI	750°C	390°C	33 dpa	-125°C	-86°C	39°C	12.8 J	7.1 J
9Cr-1MoVNb	760°C	405°C	24 dpa	-17°C	31°C	48°C	10.0 J	8.0 J
12Cr-1MoVW	760°C	405°C	24 dpa	-34°C	54°C	84°C	5.4 J	3.5 J

Table 3. Charpy data for unirradiated and irradiated steels

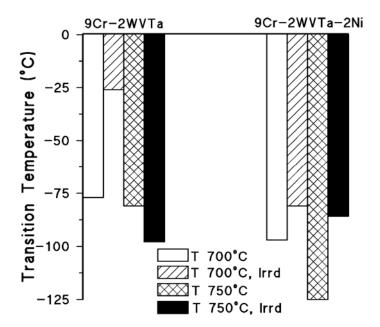


Fig. 3. Charpy impact transition temperature of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels in the unirradiated and irradiated conditions.

After irradiation of the two steels tempered at 700°C, the 9Cr-2WVTa steel showed the larger shift in DBTT (Fig. 3). For the steels tempered at 750°C, the steel without nickel showed a decrease in DBTT, while the DBTT of the nickel-containing steel increased 39°C. Although the 9Cr-2WVTa-2Ni showed the larger increase in DBTT, the final values for the two steels were similar (-98 and -86°C, respectively), because of the lower DBTT of the nickel-containing steel in the unirradiated condition.

Before irradiation, the USE values for the 9Cr-2WVTa were slightly greater than those for 9Cr-2WVTa-2Ni (Fig. 4); the values for both steels after the 750°C temper were higher than after the 700°C temper. Irradiation caused a decrease in the USE, with the largest decrease occurring for the steels with the 750°C temper. As a result, after irradiation there was little difference in the USE of the steels given the different tempering treatments, with the 9Cr-2WVTa having a slight advantage after the 750°C temper.

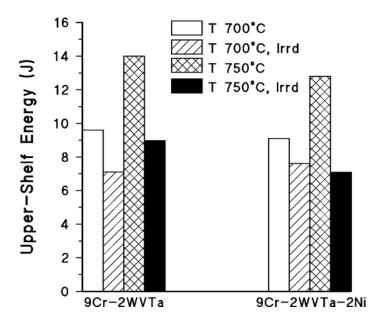


Fig. 4. Charpy upper-shelf energy of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels in the unirradiated and irradiated conditions.

# Comparison of Reduced-activation and Commercial Steels

Comparison of the reduced-activation 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels with the conventional 9Cr-1MoVNb and 12Cr-1MoVW steels was made for the reduced-activation steels tempered 1 h at 750°C. The commercial steels were both tempered 1 h at 760°C (typical tempering conditions for these steels).

## **Tensile Properties**

In the unirradiated condition, there was relatively little difference in strength among the four steels (Table 2 and Fig. 5), with the 9Cr-2WVTa-2Ni somewhat stronger than the others. Despite the higher strength, the ductility was not significantly affected (Fig. 6). The 9Cr-2WVTa and 9Cr-2WVTa-2Ni had significantly higher ductility than the 9Cr-1MoVNb and 12Cr-1MoVW steels.

For all the steels but the 9Cr-2WVTa-2Ni, both the yield stress and ultimate tensile strength indicated that relatively minor irradiation hardening occurred, but in the case of 9Cr-2WVTa-2Ni, slight softening occurred (Fig. 5). This was reflected in the ductility (Fig. 6), where uniform and total elongation of the 9Cr-2WVTa-2Ni steel increased. The total elongation of the 9Cr-1MoVNb also increased slightly after irradiation. The 9Cr-2WVTa-2Ni steel had the highest uniform and total elongation of the four steels after irradiation. The greatest decrease in ductility was observed for the 9Cr-2WVTa steel, although after irradiation it had ductility as good as or better than that for 9Cr-1MoVNb and 12Cr-1MoVW. Although these latter two steels showed relatively minor ductility changes, they had lower ductilities than the reduced-activations steels after irradiation, as they also did before irradiation.

## **Charpy Properties**

In both the unirradiated and irradiated conditions, transition temperatures of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels tempered at 750°C had significant advantages over those for the commercial steels tempered at 760°C (Table 3 and Fig. 7). After irradiation, the commercial 9Cr-1MoVNb and 12Cr-1MoVW steels had DBTT values above room temperature, whereas values for the two reduced-activation steels were well below 0°C. This occurred despite the 9Cr-1MoVNb and 12Cr-1MoVW steels being irradiated at a higher temperature and to a lower dose (Table 3). Indeed, in both the unirradiated and irradiated

conditions, the transition temperature values of the 9Cr-2WVTa and 9Cr-2WVTa-2Ni tempered at 700°C were much better than either of the commercial steels tempered at 760°C (Table 3).

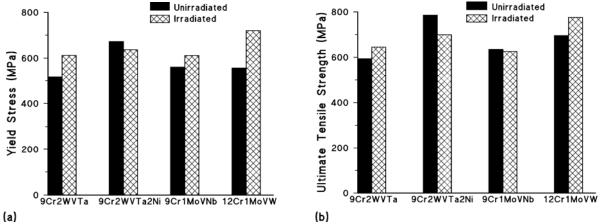


Fig. 5. (a) Yield stress and (b) ultimate tensile strength of unirradiated and irradiated 9Cr-2WVTa, 9Cr-2WVTa-2Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels.

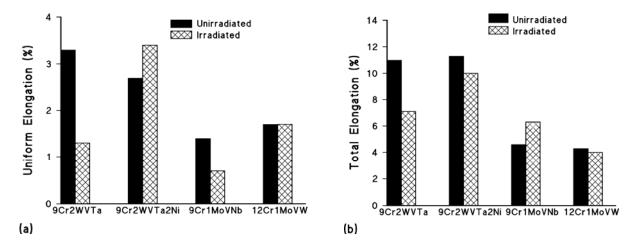


Fig. 6. (a) Uniform and (b) total elongation of unirradiated and irradiated 9Cr-2WVTa, 9Cr-2WVTa-2Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels.

Before irradiation, the USE values of the 9Cr-2WVTa and 9Cr-2WVTa-2Ni tempered at 750°C were also better than those of the 9Cr-1MoVNb and 12Cr-1MoVW steels tempered at 760°C (Fig. 8). After irradiation, USE values of 9Cr-2WVTa, 9Cr-2WVTa-2Ni, and 9Cr-1MoVNb were similar, and all three were considerably higher than that for 12Cr-1MoVW steel. Both before and after irradiation, USE values for the 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels tempered at 700°C (Fig. 8) were comparable to the commercial steels before and after irradiation, even though the latter steels were tempered at a higher temperature.

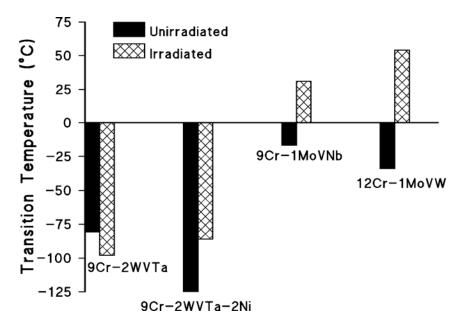


Fig. 7. Charpy impact transition temperature of 9Cr-2WVTa, 9Cr-2WVTa-2Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels in the unirradiated and irradiated conditions.

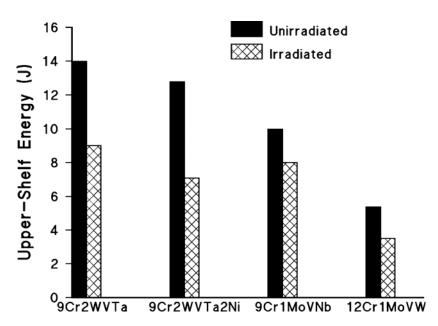


Fig. 8. Charpy impact upper-shelf energy of 9Cr-2WVTa, 9Cr-2WVTa-2Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels in the unirradiated and irradiated conditions.

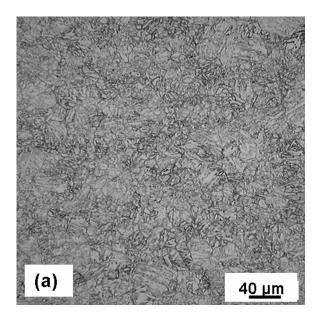
### **Discussion**

As discussed above, the primary reason for irradiating 9Cr-2WVTa-2Ni is that an  $(n,\alpha)$  reaction between <sup>58</sup>Ni and thermal neutrons in a mixed-spectrum reactor produce helium, thus providing a simulation technique for studying helium effects for fusion applications. Irradiation of the steels in a fast reactor, such as EBR-II, where very little helium forms, provides a control for irradiation in HFIR where much larger amounts of helium are generated.

Nickel is an austenite-stabilizing element that causes a reduction in  $A_1$ , the equilibrium temperature above which ferrite begins to transform to austenite. If the tempering temperature is above the  $A_1$ , then any austenite formed during tempering will transform to martensite during air cooling from the tempering temperature, and such a "normalized-and-tempered" steel will contain untempered martensite.

Previous helium-effects studies were made on steels with and without nickel tempered below the A<sub>1</sub> (usually at 700°C for several hours) to similar strengths [2]. In the present experiment, the 750°C temper was used to investigate the effect of untempered martensite, which should provide a strengthening effect.

For the commercial 9Cr-1MoVNb and 12Cr-1MoVW steels, previous work showed that the addition of 2% nickel lowered their  $A_1$  temperatures to  $\approx 710^{\circ}\text{C}$  [11]. In the absence of the 2% Ni, the  $A_1$  of the steels exceeds  $800^{\circ}\text{C}$ . Because of the similarity of the commercial and reduced-activation steels, it was assumed that  $A_1$  for the 9Cr-2WVTa-2Ni steel would also be between 700 and 750°C. This was verified by the comparison of the change in the unirradiated strengths of the 9Cr-2WVTa and 9Cr-2WVTa-2Ni for the two tempering conditions (Table 2 and Fig. 1). After a  $700^{\circ}\text{C}$  temperature, the two steels have similar strengths. Therefore, the presence of untempered martensite is indicated in the observation that the yield stress for 9Cr-2WVTa decreased 184 MPa, compared to a much smaller 33 MPa decrease for 9Cr-2WVTa-2Ni. The reduced strength for the steel without nickel given the  $750^{\circ}\text{C}$  temper is the expected behavior. Thus, the 9Cr-2WVTa-2Ni must contain considerable untempered martensite to account for the much smaller decrease and that there was no change in the ultimate tensile strength. Optical microscopy revealed a duplex structure in the 9Cr-2WVTa-2Ni that was not present in the 9Cr-2WVTa, because the untempered martensite in the 9Cr-2WVTa-2Ni etched darker than the tempered martensite (Fig. 9).



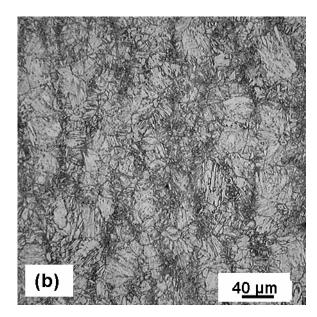


Fig. 9. Microstructures of normalized (a) 9Cr-2WVTa and (b) 9Cr-2WVTa-2Ni steels tempered at 750°C. The dark-etching regions in the 9Cr-2WVTa-2Ni are untempered martensite present in the steel because the tempering temperature was above the  $A_1$ .

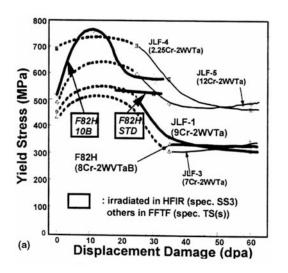
The tensile results for the 9Cr-2WVTa and 9Cr-2WVTa-2Ni steel are most interesting. First, before irradiation of the steels tempered at 750°C, the 9Cr-2WVTa-2Ni was 30% stronger than the 9Cr-2WVTa (Fig. 1). Second, a most unusual observation after irradiation was that the 9Cr-2WVTa-2Ni softened for both tempering conditions. The 9Cr-2WVTa tempered at 700°C also softened, but hardening occurred for this steel tempered at 750°C.

Despite the relatively small irradiation hardening, the uniform and total elongations of the 9Cr-2WVTa steel decreased for both tempering temperatures. However, the uniform elongation of the 9Cr-2WVTa-

2Ni, which was the harder steel containing untempered martensite after the 750°C temper, actually increased for both tempering conditions, as did the total elongation for this steel tempered at 750°C. A slight decrease in total elongation was observed for the 9Cr-2WVTa-2Ni steel tempered at 700°C. The ductility of the nickel-containing steel after irradiation was superior to that of the steel without nickel for all conditions.

This unexpected behavior of the 9Cr-2WVTa-2Ni was also indicated when the steels tempered at 750°C were compared with 9Cr-1MoVNb and 12Cr-1MoVW steels tempered at 760°C. In this case, the 9Cr-2WVTa-2Ni was the only steel not hardened, although none of the other three steels hardened significantly (Fig. 5). Likewise the 9Cr-2WVTa-2Ni steel had the best ductility after irradiation. Comparison of the results for the different steels also demonstrated the excellent behavior of the reduced-activation 9Cr-2WVTa steel relative to the two commercial steels.

The relatively minor hardening—and softening—was unexpected from previous experiments where these steels were irradiated at ≈400°C [12–14]. An explanation can be found in results from several investigators who found a peak in irradiation hardening with increasing fluence for reduced-activation steels (Fig. 10) [15] and commercial-type Cr-Mo steels [16,17]. For the reduced-activation steels, the reduction in irradiation hardening with increasing dose begins to approach the unirradiated value near 30 dpa [Fig. 10(a)]. This is similar to the dose achieved in the specimens in the present experiment that showed this behavior. Figure 10(b) indicates that total elongation decreased to a plateau around 30 dpa. There is an indication of a minimum in total elongation for one of the steels in Fig. 9(b), but the minimum is beyond 30 dpa. An explanation for the peak in strength is that irradiation-enhanced recovery of the microstructure offsets irradiation hardening [17].



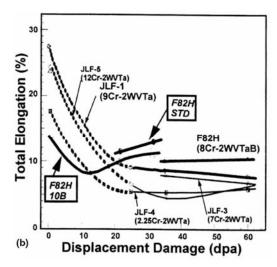


Fig. 10. The (a) yield strength and (b) total elongation as a function of irradiation dose for several reduced-activation steels [14].

The positive effect of nickel and untempered martensite in irradiated 9Cr-2WVTa-2Ni steel tempered at 750°C can also be explained in terms of an effect of irradiation-enhanced recovery and, in particular, irradiation-enhanced recovery of the untempered martensite. In this case, the effect of recovery is dominant from the beginning of the irradiation, and it is hypothesized that the higher rate of recovery of the untempered martensite partially or wholly offsets irradiation hardening. This assumes that the effect is not due to the nickel, which is a reasonable assumption, since there is relatively little difference in the tensile properties between the 9Cr-2WVTa and 9Cr-2WVTa-2Ni tempered at 700°C, either before or after irradiation.

The microstructure of the 9Cr-2WVTa-2Ni is expected to be a duplex structure with hard zones (untempered martensite) and soft zones (tempered martensite), as indicated in Fig. 9. The excellent ductility and strength of the steel implies that ductility is determined by the tempered martensite, and the strength is determined by the untempered martensite. If this explanation is correct, then the irradiation resistance of reduced-activation 9Cr-2WVTa steel could be improved by tempering above the  $A_1$  or adding nickel to the steels. The former is preferred because nickel additions are known to decrease creep strength [18]. Experiments would be required to determine the relative amount of untempered and tempered martensite for optimum properties. This microstructure could be tailored to produce a higher-strength steel with less irradiation hardening. Since irradiation hardening causes embrittlement, as measured in a Charpy impact test, less embrittlement should occur. The Charpy results discussed below support this conclusion.

Irradiation-enhanced recovery can also explain the observations on the lack of hardening for the steels tempered at 700°C (Table 2). These steels were tempered to a significantly lesser extent than was the 9Cr-2WVTa steel tempered at 750°C. Tempering at 700°C instead of 750°C results in smaller and more numerous precipitates plus a higher dislocation density. Equilibrium for the steels involves larger precipitates and a reduced dislocation density, and in the absence of irradiation, it is approached by thermal aging. Enhanced diffusion rates due to the excess vacancies and interstitials produced by irradiation should speed up the recovery/aging process and also enhance vacancy/interstitial annihilation. Irradiation-enhanced thermal aging has been observed for irradiations at 550°C [19].

In the normalized-and-tempered condition, the Charpy results demonstrated the excellent impact properties of the reduced-activation 9Cr-2WVTa steel compared to the commercial steels modified 9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) (Figs. 7 and 8). Even for the 700°C temper (a low tempering temperature for these martensitic steels) the stronger normalized-and-tempered 9Cr-2WVTa had excellent impact properties relative to the commercial steels.

Excellent Charpy impact behavior was also observed for the 9Cr-2WVTa-2Ni steel, with the irradiation resistance almost as good (after the 750°C temper) or better (after the 700°C temper) than the steel without nickel. This occurred with the 750°C temper despite the presence of martensite and a 30% greater strength than the steel without nickel.

Recent irradiations of 9Cr-2WVTa-type steels with and without nickel at 200-270°C in fast reactors found excess hardening and a larger shift in DBTT in the nickel-containing steel compared to the steel without nickel [5,6]. This is contrary to the present experiment where there was no indication that the nickel-containing steel hardened excessively relative to the steel without nickel or had a larger shift in DBTT. The difference in hardening behavior for the present tests and the previous tests that showed no hardening effect caused by nickel when irradiated in a fast reactor [20,21] and the previous tests that showed hardening [5,6] appear to be due the different irradiation temperatures. The low-temperature, nickel-enhanced irradiation hardening was attributed to finer defect clusters in the nickel-containing steels [5]. Such clusters apparently become unstable at the higher temperatures of the present experiments [22,23].

Previous work to determine helium effects was on the 9Cr-1MoVNb and 12Cr-1MoVW steels with and without nickel additions irradiated in EBR-II and FFTF, fast reactors where little helium forms, and in HFIR, a mixed-spectrum reactor where much higher helium concentrations form [2-4]. Observations on the 9Cr-1MoVNb and 12Cr-1MoVW steels with and without nickel indicated that there was no increased hardening or enhanced shift in DBTT attributable to nickel for the steels irradiated in the fast reactor [20], as observed for the 9Cr-2WVTa-2Ni steel in this experiment. However, when irradiated in HFIR to ≈40 dpa and over 200 appm He, a larger shift with little difference in hardening was observed for the nickel-containing steel. This additional shift was attributed to helium causing a decrease in the fracture stress that causes a transition to intergranular fracture on the lower shelf [2].

## **Summary and Conclusions**

Tensile and Charpy specimens of normalized-and-tempered reduced-activation steel 9Cr-2WVTa, this steel with 2% Ni (9Cr-2WVTa-2Ni), and two commercial steels, 9Cr-1MoVNb (modified 9Cr-1Mo) and 12Cr-1MoVW (Sandvik HT9), were irradiated in EBR-II to 20-30 dpa at 376-405°C. The reduced-activation steels were irradiated in two tempered conditions: 1 h at 700°C and 1 h at 750°C; the commercial steels were tempered 1 h at 760°C.

The results demonstrated the superior irradiation resistance to embrittlement of the reduced-activation steels compared to the commercial steels. Results for the 9Cr-2WVTa-2Ni steel indicated that nickel did not adversely affect the hardening or shift in DBTT of this steel relative to the steel without nickel. This occurred even though the microstructure of the 9Cr-2WVTa-2Ni containing untempered martensite after the 750°C temper. In fact, a steel with untempered martensite may have greater irradiation resistance than a steel that is entirely tempered martensite, even though the steel with untempered martensite is considerably stronger. Further work is required to verify the advisability of using a steel such as the 9Cr-2WVTa in a heat-treated condition that produced a duplex microstructure of tempered and untempered martensite.

## Acknowledgements

We wish to thank E. T. Manneschmidt and J. L. Bailey for carrying out the experimental tests and procedures and Drs. T. S. Byun and J. T. Busby for reviewing the manuscript. This research was sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

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