

## **MECHANICAL PROPERTIES OF IRRADIATED 9Cr-2WVTA STEEL WITH AND WITHOUT NICKEL—R. L. Klueh and M. A. Sokolov (Oak Ridge National Laboratory)**

### **OBJECTIVE**

The objective of this work is to develop an understanding of the effect of transmutation helium on the irradiated mechanical properties of ferritic/martensitic steels for fusion applications and to use that knowledge to develop steels with improved properties.

### **SUMMARY**

Tensile and Charpy specimens of normalized-and-tempered ORNL 9Cr-2WVTa reduced-activation steel and that steel composition containing 2% Ni (9Cr-2WVTa-2Ni) were irradiated at 376–405°C in the Experimental Breeder Reactor (EBR-II) to 23–33 dpa. Steels were irradiated in two tempered conditions: 1 hr at 700°C and 1 h at 750°C. The mechanical properties before and after irradiation of the 9Cr-2WVTa-2Ni steel were quite similar to those of the 9Cr-2WVTa steel, indicating no adverse effect of the nickel. Neither of the steels showed excessive hardening or a large increase in ductile-brittle transition temperature.

### **PROGRESS AND STATUS**

#### **Introduction**

Displacement damage by neutron irradiation of 9Cr reduced-activation martensitic steels below 425–450°C hardens the lattice, causing an increase in strength and a decrease in toughness. The effect on impact toughness (embrittlement) 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).

Possible effects of helium on hardening and embrittlement are important because large amounts of transmutation helium will form in the ferritic/martensitic steel first wall of a fusion reactor. Nickel-doped 9 and 12 Cr steels have been irradiated in the mixed-spectrum High Flux Isotope Reactor (HFIR) to study the effect of helium on fracture [1]. Helium is formed in a mixed-spectrum reactor by a two-step transmutation reaction between  $^{58}\text{Ni}$  and the thermal neutrons in the mixed-neutron spectrum. Irradiation of nickel-doped steels in HFIR causes the simultaneous production of displacement damage and helium in the steel matrix, thus simulating what will happen in a first wall. Results from such irradiation experiments at 400°C have been interpreted to indicate an effect of helium on embrittlement [1].

More recent irradiation experiments of nickel-doped 9Cr reduced-activation steels at 170–270°C indicated that nickel-doped steels hardened more than steels without the nickel [2,3]. These results indicated that nickel-doping should be used at  $\leq 300^\circ\text{C}$  with caution. These hardening effects were well below temperatures where nickel-doped steels were used to indicate helium effects [1].

To more fully understand the effect of nickel, 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) at 376–405°C.

#### **Experimental Procedure**

The nominal composition for the 9Cr-2WVTa steel in wt. % is: Fe-2.0W-0.25V-0.07Ta-0.10C; 9Cr-2WVTa-2Ni had this same composition but with a 2% Ni addition. The steels were produced from a 9Cr-2WVTa steel master alloy. Details on chemical composition, ingot preparation, and plate and sheet fabrication procedures have been published [5].

The steels were irradiated in the normalized-and-tempered condition; normalization involved 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.

Tensile specimens 44.5-mm long with a reduced gage section of 20.3 x 1.52 x 0.76 mm were machined from normalized-and-tempered 0.76-mm sheet with gage lengths parallel to the rolling direction. Tests were conducted on irradiated and unirradiated specimens at 400°C (near the irradiation temperature) in vacuum at a nominal strain rate of  $4 \times 10^{-4} \text{ s}^{-1}$ .

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). Details of the test procedure for the subsize Charpy specimens have been published [6–8].

Two tensile and six Charpy specimens of each heat-treated condition were irradiated at 376 to 405°C in the COBRA experiment in EBR-II. Specimens were irradiated to  $5.1 \times 10^{26}$  to  $6.9 \times 10^{26} \text{ n/m}^2$  ( $E > 0.1 \text{ MeV}$ ), which produced between 23 and 33 dpa. Calculated helium concentrations were between 3 and 6 appm, depending on the dose and composition.

## Results

The yield stress [Fig. 1(a)] for 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels given different tempers before and after irradiation indicated that there was little effect of irradiation on strength for the 9Cr-2WVTa for the 700°C temper and on the 9Cr-2WVTa-2Ni for both tempering conditions. The yield stress of the 9Cr-2WVTa tempered at 750°C increased after irradiation, but for both tempers, ultimate tensile strength showed little change for the 9Cr-2WVTa. For the 9Cr-2WVTa-2Ni, both the yield stress and ultimate tensile strength showed fairly large decreases-irradiation softening (Table 1).

The negative effect of irradiation on total elongation [Fig. 1(b)] was greater for 9Cr-2WVTa than 9Cr-2WVTa-2Ni. Total elongation of the 9Cr-2WVTa decreased after irradiation for both tempering conditions, but total elongation for the 9Cr-2WVTa-2Ni decreased slightly when tempered at 700°C and increased when tempered at 750°C, which reflects the irradiation softening. For both tempering conditions, uniform elongation of 9Cr-2WVTa-2Ni was relatively unchanged after irradiation, while that for 9Cr-2WVTa decreased by  $\approx 60\%$  (Table 1).

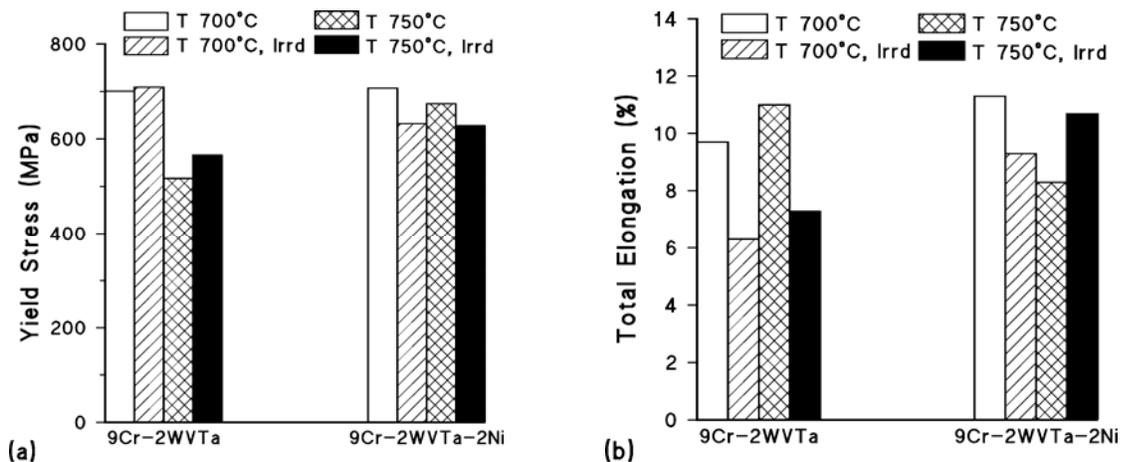


Fig. 1. (a) The yield stress and (b) total elongation of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

Table 1. Tensile data for unirradiated and irradiated steels tested at 400°C

Steel	Temper	Irradiation	Strength (MPa)		Elongation (%)	
			Yield	Ultimate	Uniform	Total
9Cr-2WVTa	700°C	Unirradiated	701	767	2.7	9.7
9Cr-2WVTa	700°C	390°C/32.6dpa	709	745	1.1	6.3
9Cr-2WVTa	750°C	Unirradiated	517	603	3.3	11.0
9Cr-2WVTa	750°C	390°C/32.6dpa	567	598	1.3	7.3
9Cr-2WVTa-2Ni	700°C	Unirradiated	707	798	2.7	11.3
9Cr-2WVTa-2Ni	700°C	390°C/32.6dpa	632	666	2.4	9.3
9Cr-2WVTa-2Ni	750°C	Unirradiated	674	797	2.7	8.3
9Cr-2WVTa-2Ni	750°C	390°C/32.6dpa	629	670	3.1	10.7

Nickel has a beneficial effect on the Charpy transition temperature prior to irradiation, and in both heat-treated conditions, the nickel-containing steel had the lowest value (Table 2 and Fig. 2). For the two steels tempered at 700°C, the 9Cr-2WVTa steel showed the larger shift in DBTT after irradiation; note, however, the 9Cr-2WVTa was irradiated at a lower temperature than the 9Cr-2WVTa-2Ni. For the steels tempered at 750°C, the 9Cr-2WVTa showed a 17°C decrease in DBTT after irradiation, while 9Cr-2WVTa-2Ni showed an increase of 39°C. The final DBTT values for the 9Cr-2WVTa and 9Cr-2WVTa-2Ni were quite similar (-98 and -86°C), because of the lower DBTT of the latter steel in the unirradiated condition.

Table 2. Charpy data for unirradiated and irradiated steels

Steel	Temper	Irrad. Temp	Dose	Uirrad. DBTT	Irrad. DBTT	DBTT Shift	Uirrad. USE	Irrad. USE
9Cr-2WVTa	700°C	376°C	23 dpa	-77°C	-26°C	51°C	9.6 J	7.1 J
	750°C	390°C	33 dpa	-81°C	-98°C	-17°C	14.0 J	9.0 J
9Cr-2WVTa-2Ni	700°C	404°C	26 dpa	-97°C	-81°C	16°C	9.1 J	7.6 J
	750°C	390°C	33 dpa	-125°C	-86°C	39°C	12.8 J	7.1 J

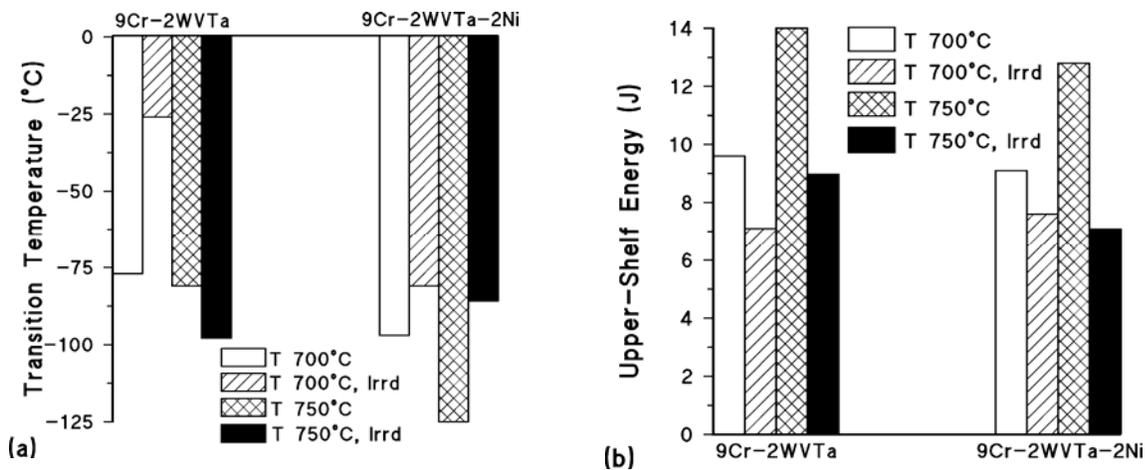


Fig. 2. (a) Transition temperature and (b) upper-shelf energy of 9Cr-2WVTa and 9Cr-2WVTa-Ni steels in unirradiated and irradiated conditions.

Before irradiation, the 9Cr-2WVTa and 9Cr-2WVTa-2Ni had relatively similar USE values [Fig. 2(b)] for similar tempers, with the values after the 750°C temper being higher than after the 700°C temper.

Irradiation caused a decrease in the USE, with the largest decreases occurring for the steels with the 750°C temper. After irradiation, there was relatively little difference in the USE of the steels with and without nickel for similar tempers.

## Discussion

It is well known that adding nickel, an austenite stabilizing element, to ferritic steel causes a reduction in  $A_{c1}$ , the temperature where ferrite begins to transform to austenite when ferritic steel is heated. If the tempering temperature of the 9Cr steels is above  $A_{c1}$ , then any austenite formed during tempering will transform to martensite during cooling, and the “normalized-and-tempered” steel will contain untempered martensite. It is known that 2% Ni lowers the  $A_{c1}$  below 750°C for modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) [9], and because of the similarity of the commercial and reduced-activation steels, it is assumed  $A_{c1}$  for 9Cr-2WVTa-2Ni is below 750°C. This was verified in the tensile tests (Table 1), where the presence of untempered martensite after the 750°C treatment can be inferred from the unirradiated strength of the 9Cr-2WVTa-2Ni by comparing the strengths of this steel after the 700 and 750°C tempers with the strengths of the steel without nickel [Fig. 1(a)].

The strengths after irradiation of 9Cr-2WVTa-2Ni tempered at 700 and 750°C are less than the unirradiated strength (Table 1). There was no change in strength for the 9Cr-2WVTa steel due to irradiation after the 700°C temper, but for this steel given the 750°C temper, the yield stress increased slightly, with essentially no change in the ultimate tensile strength. In addition to the effect of nickel on strength, the effect on ductility is remarkable, especially for the steel tempered at 750°C, where both uniform and total elongation increased after irradiation. This occurred despite the presence of untempered martensite in the microstructure of 9Cr-2WVTa-2Ni. As a further indication of the apparent beneficial effect of nickel, the uniform and total elongation in the nickel-containing steel with the 700°C temper are significantly less affected by irradiation than the steel without nickel tempered at 700°C.

The relatively minor hardening—and actual softening—was unexpected from previous experiments where steels with and without nickel were irradiated at  $\approx 400^\circ\text{C}$  [10-12]. An explanation may be found in results from previous studies that found a peak in irradiation hardening with increasing fluence for reduced-activation steels [13] and for commercial-type Cr-Mo steels [14,15]. The previous results indicated that the reduction in irradiation hardening with increasing dose begins to approach the unirradiated value near 30 dpa, which is similar to the dose of the specimens irradiated in the present experiment. An explanation for the strength peak is that irradiation-enhanced recovery offsets irradiation hardening [15].

At first glance, it appears the Charpy properties of the 9Cr-2WVTa-2Ni are better than those of 9Cr-2WVTa after the 700°C temper. However, this can probably be attributed to the higher irradiation temperature for 9Cr-2WVTa-2Ni—404°C compared to 376°C for 9Cr-2WVTa. The irradiation resistance of 9Cr-2WVTa-2Ni was not as good as 9Cr-2WVTa after the 750°C temper, but even then, the DBTT for the nickel-containing steel was still -86°C. Thus, after irradiation, not only was the 9Cr-2WVTa-2Ni with the 750°C temper significantly stronger than 9Cr-2WVTa given a similar temper, it also had better ductility and excellent impact toughness relative to 9Cr-2WVTa. Further investigation of the effect of the untempered martensite in the steel is required.

As discussed earlier, nickel has been added to steels to study helium effects by comparing irradiation effects with and without the nickel addition in a fast reactor, such as EBR-II where very little helium forms, and in a mixed-spectrum reactor, such as HFIR where a transmutation reaction with  $^{58}\text{Ni}$  and thermal neutrons produces much higher helium concentrations [1]. Previous irradiations of 12Cr-1MoVW steels with and without nickel indicated that there was no increased hardening or enhanced shift in DBTT for the steel with nickel when irradiated in FFTF, a fast reactor [16]. However, when irradiated in HFIR where over 200 appm He formed, a larger DBTT shift with little difference in hardening was observed for the nickel-containing steel. The additional DBTT shift was attributed to helium [1]. The results of the present fast-reactor irradiations indicated that the nickel did not cause increased hardening or embrittlement in reduced-activation 9Cr-2WVTa steels.

In recent years, data obtained from irradiations at 170–270°C indicated the addition of nickel to 9Cr steel could produce excess hardening and a larger shift in DBTT, even in a reactor where no significant helium formed [2,3]. There was neither indication that the nickel-containing steel in the present experiment irradiated near 400°C hardened excessively, nor that there was a larger DBTT shift caused by the presence of nickel. It appears that irradiation test temperature may be the key cause of the difference in hardening behavior in tests that showed no hardening effect caused by nickel when irradiated in a fast reactor [16] and the tests that showed hardening [2,3]. The low-temperature nickel-enhanced irradiation hardening was attributed to finer defect clusters in the nickel-containing steels [3]. It is probable that such clusters are not stable at the higher temperatures of the present and previous tests [1,16].

### Summary and Conclusions

ORNL 9Cr-2WVTa and the steel with 2% Ni were irradiated at 376–404°C in EBR-II to 23–33 dpa (3–6 appm He). The steels showed little hardening in a temperature regime where hardening is normally observed. The limited hardening resulted in only minor changes in DBTT. Results indicated that there were no adverse effects of nickel, which is contrary to recent low-temperature irradiations that showed nickel causes excess hardening. Differences with these previous investigations are probably due to differences in irradiation temperatures (< 300°C in previous work and 376–404°C in present investigation). The minor hardening and minor change in Charpy properties may be an indication of the maximum in hardening with dose observed in ferritic steels that has been attributed to softening due to irradiation-enhanced recovery offsetting irradiation hardening.

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