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Mechanical properties of irradiated 9Cr–2WVTa steel with and without nickel

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Abstract

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 h 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.

1. 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 9Cr and 12Cr steels have been irradiated in a mixed-spectrum reactor such as the 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}Ni and the thermal neutrons in the mixed-neutron spectrum. This results in the simultaneous production of displacement damage and helium in the steel matrix, thus simulating what will happen in a fusion reactor 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 $\lesssim$300 °C with caution. These hardening effects were well below temperatures where nickel-doped steels were used to indicate helium effects [1].

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To more fully understand the effect of nickel, tensile and Charpy properties were determined 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.

2. Experimental procedure

The nominal composition for the 9Cr–2WVTa steel in wt% is: Fe–9Cr–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 [4].

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 $20.3 \times 1.52 \times 0.76$ 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 4×10^{-4} s^{-1}.

One-third-size Charpy specimens measuring $3.3 \times 3.3 \times 25.4$ 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 [5–7].

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

3. Results

The yield stress (Fig. 1(a)) for 9Cr–2WVTa and 9Cr–2WVTa–2Ni steels given different tempers before and after irradiation indicated that irradiation caused little or no hardening for the 9Cr–2WVTa with the 700 °C temper and the 9Cr–2WVTa–2Ni for both tempers. The yield stress of 9Cr–2WVTa tempered at 750 °C increased after irradiation, but for both tempers, ultimate tensile strength showed little change. For 9Cr–2WVTa–2Ni, both 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 9Cr–2WVTa decreased after irradiation for both tempering conditions, but total elongation for 9Cr–2WVTa–2Ni

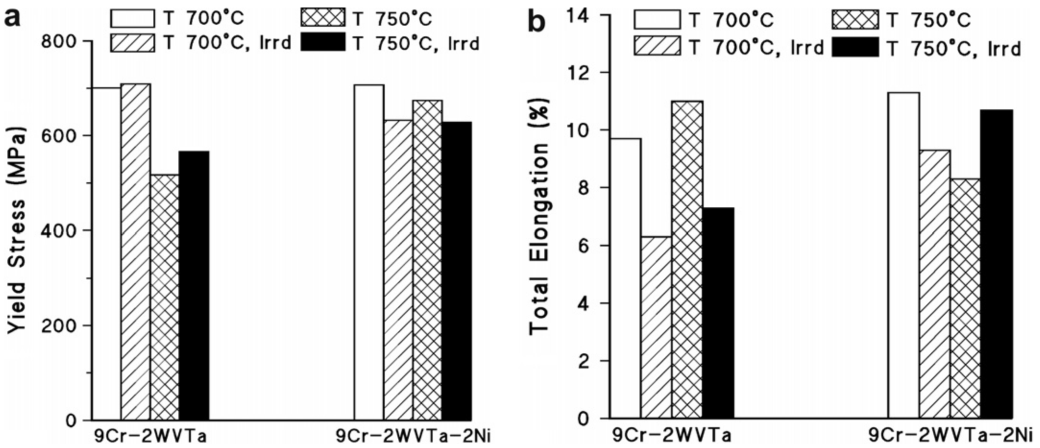

Fig. 1. (a) The yield stress and (b) total elongation of 9Cr–2WVTa and 9Cr–2WVTa–2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

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

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

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 ≈60% (Table 1).

Nickel has a beneficial effect on the Charpy transition temperature prior to irradiation, and in both heat-treated conditions, the 9Cr–2WVTa–2Ni steel had the lowest value (Table 2 and Fig. 2). For the two steels tempered at 700 °C, the 9Cr–2WVTa steel had the larger shift in DBTT after irradiation; note, however, that the 9Cr–2WVTa was irradiated at a lower temperature than 9Cr–2WVTa–2Ni. For the steels tempered at 750 °C, the DBTT of 9Cr–2WVTa decreased 17 °C 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.

Before irradiation, the 9Cr–2WVTa and 9Cr–2WVTa–2Ni had relatively similar USE values (Fig. 2(b)) for similar tempers, with the values after

Table 2
Charpy data for unirradiated and irradiated steels

Steel	Temper (°C)	Irradiation temperature (°C)	Dose (dpa)	Unirradiated DBTT (°C)	Irradiated DBTT (°C)	DBTT shift (°C)	Unirradiated USE (J)	Irradiated USE (J)
9Cr–2WVTa	700	376	23	−77	−26	51	9.6	7.1
	750	390	33	−81	−98	−17	14.0	9.0
9Cr–2WVTa–2Ni	700	404	26	−97	−81	16	9.1	7.6
	750	390	33	−125	−86	39	12.8	7.1

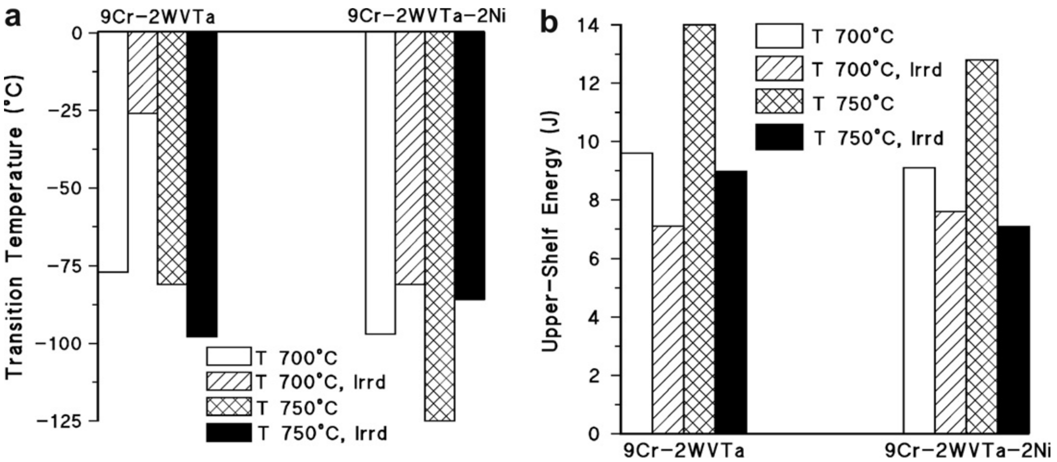

Fig. 2. (a) Transition temperature and (b) upper-shelf energy of 9Cr–2WVTa and 9Cr–2WVTa–Ni steels in unirradiated and irradiated conditions.

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.

4. Discussion

It is well known that adding nickel, an austenite stabilizing element, to ferritic steel causes a reduction in Ac_1, the temperature where ferrite begins to transform to austenite when the steel is heated. If the tempering temperature of the 9Cr steels of this experiment was above Ac_1, 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 Ac_1 of modified 9Cr–1Mo (9Cr–1MoVNb) and Sandvik HT9 (12Cr–1MoVW) below 750 °C [8]. Because of the similarity of the commercial and reduced-activation steels, it was assumed Ac_1 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 temper 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 of the 9Cr–2WVTa and 9Cr–2WVTa–2Ni are somewhat different from previous experiments where steels with and without nickel were irradiated at ≈400 °C [9–15]. Hardening at around 400 °C generally increases with dose and reaches a saturation by <10 dpa [9,14,16–18]. There are also observations of a peak in irradiation hardening with increasing fluence for reduced-activation steels [19,20] and for commercial-type Cr–Mo steels [13,20–22]. The previous results indicated that the reduction in irradiation hardening with increasing dose begins to approach the unirradiated value near 30 dpa for irradiations near 400 °C [19,20], 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 [23].

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 the 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}Ni and thermal neutrons produces much higher helium concentrations [1]. Previous irradiations of 9Cr–1MoVNb and 12Cr–1MoVW steels with and without nickel in mixed-spectrum [13–15] and fast [24] reactors indicated that there was no increased hardening or enhanced shift in DBTT for the steel with nickel compared to one without nickel when irradiated in FFTF, a fast reactor [24]. However, when these steels were irradiated in HFIR, where over 200 appm He formed, a larger DBTT shift was observed for the nickel-containing steel. The additional DBTT shift was attributed to helium [1]. In agreement with the previous results for irradiation in a fast reactor [24], the results of the present fast-reactor irradiations indicated that nickel did not cause increased hardening or embrittlement in reduced-activation 9Cr–2WVTa steel.

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 an 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 [24] 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 [1,24].

5. Summary and conclusion

ORNL 9Cr–2WVTa and this 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 small amount of hardening and change in Charpy properties may be an indication of a maximum in hardening with irradiation dose observed in ferritic steels that has been attributed to softening due to irradiation-enhanced recovery offsetting irradiation hardening.

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References

[1] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 187 (1992) 60.
[2] R. Kasada, A. Kimura, H. Matsui, M. Narui, J. Nucl. Mater. 258–263 (1998) 1199.
[3] A. Kimura, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf. 2001-007, p. 348.
[4] R.L. Klueh, P.J. Maziasz, Metall. Trans. 20A (1989) 373.
[5] D.J. Alexander, R.K. Nanstad, W.R. Corwin, J.T. Hutton, in: A.A. Braun, N.E. Ashbaugh, F.M. Smith (Eds.), Applications of Automation Technology to Fatigue and Fracture Testing, ASTM STP 1092, American Society for Testing and Materials, Philadelphia, 1990, p. 83.
[6] 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, 1990, p. 179.
[7] 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, 1996, p. 384.
[8] R.L. Klueh, J.M. Vitek, M.L. Grossbeck, in: Effects of Radiation on Materials, ASTM STP 782, American Society for Testing and Materials, Philadelphia, 1982, p. 648.
[9] R.L. Klueh, D.J. Alexander, M. Rieth, J. Nucl. Mater. 273 (1999) 146.
[10] R.L. Klueh, J.M. Vitek, J. Nucl. Mater. 132 (1986) 27.
[11] R.L. Klueh, J.M. Vitek, J. Nucl. Mater. 137 (1986) 44.
[12] R.L. Klueh, D.J. Alexander, in: Effects of Radiation on Materials: 18th International Symposium, ASTM STP 1325, American Society for Testing and Materials, Philadelphia, 1999, p. 911.
[13] R.L. Klueh, P.J. Maziasz, J. Nucl. Mater. 187 (1992) 43.
[14] R.L. Klueh, J.M. Vitek, J. Nucl. Mater. 150 (1987) 272.
[15] R.L. Klueh, P.J. Maziasz, J.M. Vitek, J. Nucl. Mater. 141–143 (1986) 960.
[16] T. Lechtenberg, J. Nucl. Mater. 133&134 (1985) 149.
[17] A. Alamo, M. Horsten, X. Averty, E.I. Materna-Morris, M. Rieth, J.C. Brachet, J. Nucl. Mater. 283–287 (2000) 353.
[18] A. Kimura, T. Morimura, M. Narui, H. Matsui, J. Nucl. Mater. 233–237 (2004) 243.
[19] Y. Kohno, A. Kohyama, T. Hirose, M.L. Hamilton, M. Narui, J. Nucl. Mater. 271&272 (1994) 145.
[20] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.
[21] S.A. Maloy, M.R. James, T.J. Romero, M.B. Toloczko, R.J. Kurtz, A. Kimura, J. Nucl. Mater. 341 (2005) 141.
[22] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 253–258 (1998) 1269.
[23] V.S. Khabarov, A.M. Dvoriashin, S.I. Porollo, J. Nucl. Mater. 233–237 (1996) 236.
[24] W.R. Corwin, J.M. Vitek, R.L. Klueh, J. Nucl. Mater. 149 (1987) 312.