ELSEVIER

Journal of Nuclear Materials 273 (1999) 146–154

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

The effect of tantalum on the mechanical properties of a 9Cr–2W–0.25V–0.07Ta–0.1C steel [1]

R.L. Klueh [a,*], D.J. Alexander [b], M. Rieth [c]

[a] Oak Ridge National Laboratory, P.O. Box 2008, MS 6376, Oak Ridge TN 37831-6376, USA

[b] Los Alamos National Laboratory, Los Alamos NM 87545, USA

[c] Staff Research Scientist, Forschungszentrum Karlsruhe Institut für Materialforschung II, P.O. Box 3640, D-76021 Karlsruhe, Germany

Received 13 November 1998; accepted 1 February 1999

Abstract

An Fe–9Cr–2W–0.25V–0.07Ta–0.1C (9Cr–2WVTa) steel has excellent strength and impact toughness before and after irradiation in the Fast Flux Test Facility (FFTF) and the High Flux Reactor (HFR). The ductile–brittle transition temperature (DBTT) increased only 32°C after 28 dpa at 365°C in FFTF, compared to a shift of ≈60°C for a 9Cr–2WV steel the same as the 9Cr–2WVTa steel but without tantalum. This difference occurred despite the two steels having similar tensile properties before and after irradiation. The 9Cr–2WVTa steel has a smaller prior-austenite grain size, but otherwise microstructures are similar before irradiation and show similar changes during irradiation. The irradiation behavior of the 9Cr–2WVTa steel differs from the 9Cr–2WV steel and other similar steels in two ways: (1) the shift in DBTT of the 9Cr–2WVTa steel irradiated in FFTF does not saturate with fluence by ≈28 dpa, whereas for the 9Cr–2WV steel and most similar steels, saturation occurs at <10 dpa, and (2) the shift in DBTT for 9Cr–2WVTa steel irradiated in FFTF and HFR increased with irradiation temperature, whereas it decreased for the 9Cr–2WV steel, as it does for most similar steels. The improved properties of the 9Cr–2WVTa steel and the differences with other steels were attributed to tantalum in solution.

1. Introduction

A nominally Fe–9Cr–2W–0.25V–0.07Ta–0.1C (9Cr–2WVTa) steel (all compositions are in wt%) has been developed for possible use in fusion power plants [1]. A major problem of such ferritic/martensitic steels for fusion applications is the embrittlement caused by irradiation hardening below ≈400°C. Irradiation hardening is measured as an increase in yield stress ($\Delta\sigma_y$), and embrittlement is measured in a Charpy impact test as a shift in ductile–brittle transition temperature (ΔDBTT) caused by the irradiation. The 9Cr–2WVTa steel has shown excellent resistance to such embrittlement after irradiation at 365°C in the Fast Flux Test Facility (FFTF) [2–5] and the High Flux Reactor (HFR) [6,7].

Several irradiation experiments have been conducted that compared the behavior of the 9Cr–2WVTa steel with a 9Cr–2WV steel, which is the same as the 9Cr–2WVTa but without tantalum [2–5,8]. The steels showed similar hardening when irradiated at 365°C, but the 9Cr–2WVTa showed considerably less embrittlement. There were also other differences in the behavior of the 9Cr–2WVTa steel relative to the 9Cr–2WV steel and steels such as 9Cr–1MoVNb (modified 9Cr–1Mo) and 12Cr–1MoVW (Sandvik HT9).

In this paper, observations from the irradiation experiments in FFTF that contained the 9Cr–2WV and 9Cr–2WVTa steels and from HFR experiments that contained the 9Cr–2WVTa steel will be reviewed, and the combined results will be analyzed to try to understand better the behavior of the 9Cr–2WVTa steel

* Corresponding author. Tel.: +1-423 574 5111; fax: +1-423 574 0641; e-mail: ku2@ornl.gov

[1] Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-960R22464 with Lockheed Martin Energy Research Corp.

0022-3115/99/$ – see front matter
PII: S0022-3115(99)00035-5

during irradiation and how the properties of this type of steel might be improved.

2. Experimental procedure

The 9Cr–2WV and 9Cr–2WVTa steels are nominally Fe–9Cr–2W–0.25V–0.1C without and with 0.07% Ta, respectively. They were prepared as 18 kg electro-slag remelted heats by Combustion Engineering, Chattanooga, Tennessee. In addition to the Cr, W, V, C, and Ta, elements normally found in such steels (e.g., Mn, Si, etc.,) were adjusted to levels typical of commercial steel processing practice. Melt compositions have been published [3].

The steels were normalized-and-tempered prior to irradiation. Normalization involved an austenitization treatment of 0.5 h at 1050°C in a helium atmosphere, followed by cooling in flowing helium gas. The steels were tempered for 1 h at 750°C.

Tensile specimens were machined from a 0.76 mm sheet. The tensile specimens were 25.4 mm long and had a reduced gage section 7.62 mm long by 1.52 mm wide by 0.76 mm thick. Tensile tests were conducted at 365°C in vacuum on a 120-kN Instron universal test machine at a nominal strain rate of $\approx 1 \times 10^{-3}$ s^{-1}.

Charpy specimens for the FFTF irradiations were one-third size V-notch specimens measuring $3.3 \times 3.3 \times 25.4$ mm^3 with a 0.51 mm deep 30°C V-notch and a 0.05–0.08 mm root radius that were machined from normalized-and-tempered 15.9 mm plates [2]. Impact specimens for the HFR irradiations were European subsize standard specimens, $3 \times 4 \times 27$ mm^3 with a notch depth of 1 mm. Specimens were machined from the normalized-and-tempered plate along the rolling direction with the notch transverse to the rolling direction (L–T orientation) [8]. The DBTT was determined at an energy level midway between the upper and lower shelf energies. Details on the test procedure for the subsize impact specimens have been published [6,9].

Two tensile specimens and six Charpy specimens of each heat were irradiated in the Materials Open Test Assembly of FFTF over the range 7–28 dpa at 365°C [2–5] and Charpy specimens were irradiated to $\approx$14 dpa at 393°C [8]. Charpy specimens of the 9Cr–2WVTa steel were also irradiated in the High Flux Reactor (HFR) in the Netherlands to 0.8 and 2.5 dpa at 250°C, 300°C, 350°C, 400°C and 450°C [6,7]. Details on the irradiation conditions were given in the previous publications [2–8].

3. Results

Three irradiation experiments will be discussed: (1) irradiation of the 9Cr–2WV and 9Cr–2WVTa steels in FFTF at 365°C to $\approx$7, 14, 21, and 28 dpa [2–5], (2) irradiation of 9Cr–2WV and 9Cr–2WVTa steels in FFTF at 393°C to $\approx$14 dpa [8], and (3) irradiation of 9Cr–2WVTa steel in HFR to 0.2, 0.8 and 2.5 dpa at 250°C, 300°C, 350°C, 400°C and 450°C [6,7].

3.1. Irradiation in FFTF at 365°C

Tensile tests were conducted on the 9Cr–2WV and 9Cr–2WVTa steels irradiated to three different fluence levels in FFTF at 365°C (Table 1). There was little difference in the strength (yield stress and ultimate tensile strength) of the two steels either before or after irradiation (Fig. 1(a)). The strength increase saturated with fluence. Likewise, there was little difference in the uniform and total elongation up to $\approx$15 dpa, although there appeared to be a slight divergence at the highest fluence, especially for the total elongation (Fig. 1(b)).

Charpy specimens of the two steels were irradiated in FFTF to four fluences at $\approx$365°C Table 2. The 9Cr–2WVTa steel had a lower transition temperature prior to irradiation, and it improved the superiority during irradiation by developing a smaller ΔDBTT (Table 2 and Fig. 2). There was also a definite difference in the effect of the continued irradiation on the two steels.

Table 1
Tensile properties of Cr–W steels irradiated in FFTF at 365°C [a]

Steel	Fluence (dpa)	Strength (MPA)		Elongation (%)	
		Yield	Ultimate	Uniform	Total
9Cr–2WV	0	549	659	4.7	12.3
	7.7	710	764	3.5	10.2
	16.7	697	745	2.3	9.0
	27.6	705	756	2.3	8.7
9Cr–2WVTa	0	544	652	4.3	12.3
	6.4	669	734	3.9	11.1
	15.4	699	765	2.9	9.7
	27.2	710	769	3.5	12.0

[a] Values are the average of two tests.

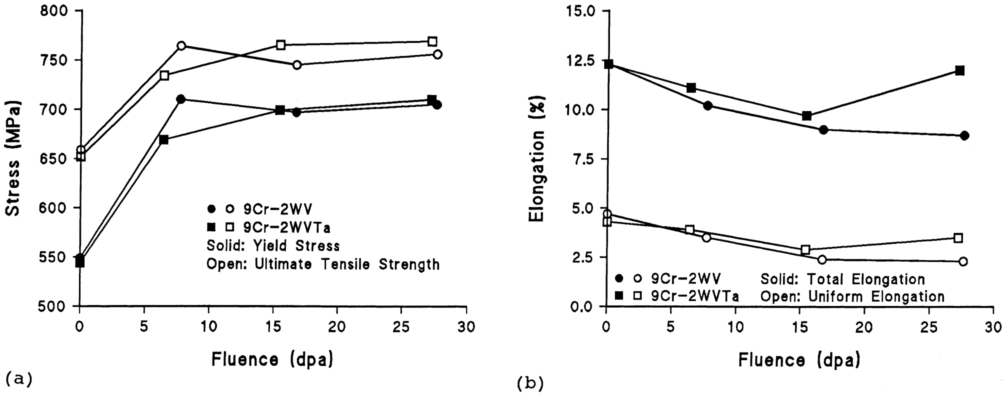

Fig. 1. The (a) yield stress and ultimate tensile strength and (b) the uniform and total elongation for 9Cr–2WV and 9Cr–2WVTa steels irradiated at 365°C in FFTF.

Table 2
Charpy impact properties of Cr–W steels irradiated in FFTF at 365°C [a]

Steel	Temp. (°C)	Fluence (dpa)	DBTT (°C)	ΔDBTT (°C)	USE (J)
9Cr–2WV	Unirr	0	−60		8.4
	365	7.7	8	68	6.4
	365	16.7	−32	28	6.3
	365	23.9	−8	52	6.3
	365	27.6	1	61	6.6
	393	14	−14	46	8.1
	393	14	−28	32	8.0
9Cr–2WVTa	Unirr	0	−88		11.2
	365	6.4	−84	4	8.6
	365	15.4	−74	14	8.5
	365	22.5	−67	21	9.6
	365	27.6	−56	32	8.1
	393	14	−53	35	8.4
	393	14	−45	43	8.9

[a] Evaluated at an energy level halfway between the upper and lower shelves.

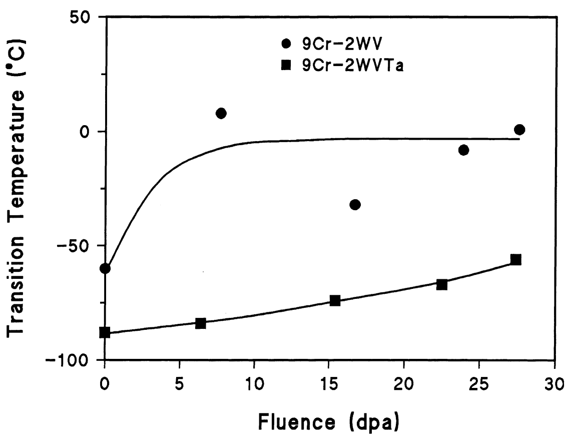

Fig. 2. The transition temperature as a function of fluence for 9Cr–2WV and 9Cr–2WVTa steels irradiated at 365°C in the FFTF.

The 9Cr–2WV steel developed a large ΔDBTT (68°C) after the 7.7 dpa irradiation, and the ΔDBTT values after 23.9 dpa (52°C) and 27.6 dpa (61°C) were similar. The ΔDBTT after the 16.7 dpa irradiation was only 28°C. However, based on the observation of the saturation in yield stress with fluence (Fig. 1(a)), it was concluded that the data point at 16.7 dpa was spurious, and the curve in Fig. 2 was drawn to indicate a saturation in the ductile–brittle transition temperature (DBTT) with fluence, as is noted for most steels.

Although the yield stress for the 9Cr–2WVTa steel also showed a saturation with fluence (Fig. 1(a)), the DBTT did not give an indication of saturation with fluence (Fig. 2). Rather, it appeared to increase continuously with fluence up to 27.6 dpa. However, even without saturation, the DBTT after 27.6 dpa for the 9Cr–2WVTa was still only −56°C with a ΔDBTT of only 32°C.

3.2. Irradiation in FFTF at 393°C

Two sets of Charpy specimens of 9Cr–2WV and 9Cr–2WVTa steels were irradiated to 14 dpa at 393°C in FFTF (Table 2) [8]. For the 9Cr–2WV steel, the DBTT values (−14°C and −28°C) at 393°C were lower than the saturation value at 365°C (about 0°C). The saturation at 365°C was chosen for comparison rather than the value at 16.7 dpa (closer to 14 dpa), because, as discussed above, the yield stress saturated with fluence, and the DBTT at 16.7 dpa appeared to be in error relative to the other three values, which were similar and indicative of a saturation with fluence.

Contrary to the observation on the 9Cr–2WV steel, the DBTT values for the 9Cr–2WVTa steel after irradiation to 14 dpa at 393°C (−53°C and −45°C) were higher than the DBTT observed after irradiation at 365°C to 15.4 dpa (−74°C). Although the DBTTs for the 9Cr–2WVTa steel were below those for the 9Cr–2WV steel after irradiation at 393°C, the ΔDBTTs for the 9Cr–2WVTa steel and the 9Cr–2WV steel were quite similar (46°C and 32°C for the 9Cr–2WV and 34°C and 43°C for the 9Cr–2WVTa steel) (Table 2) [8].

3.3. Irradiation in the HFR

Rieth et al. irradiated six steels, including the 9Cr–2WVTa steel, in the HFR at 250°C, 300°C, 350°C, 400°C and 450°C to 0.2, 0.8, and 2.4 dpa [6,7]. The steels included two conventional Cr–Mo steels, MANET I (nominally Fe–11Cr–0.8Mo–0.2V–0.9Ni–0.16Nb–0.06Zr–0.14C) and MANET II (Fe–10Cr–0.6Mo–0.2V–0.7Ni–0.14Nb–0.4Zr–0.1C), and four low-activation steels, OPTIFER Ia (Fe–9.3Cr–1W–0.25V–0.07Ta–0.1C), OPTIFER II (Fe–9.4Cr–1.1Ge–0.3V–0.13C), F82H (Fe–8Cr–2W–0.2V–0.02Ta–0.1C), and 9Cr–2WVTa. Melt compositions have been published [6].

Fig. 3 from Rieth et al. [6] shows the DBTT for the different steels after irradiation to 0.8 dpa. Of interest for this discussion is the superior behavior of the 9Cr–2WVTa steel (labeled ORNL) between 250°C and 400°C. However, between 350°C and 450°C, the DBTT of the 9Cr–2WVTa increases, which is contrary to the other five steels. The data after irradiation at 0.2 and 2.4 dpa will be discussed below.

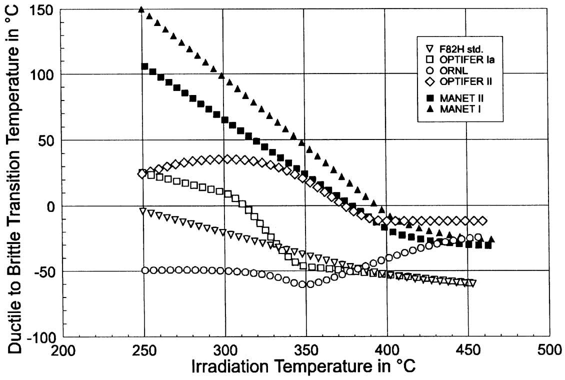

Fig. 3. Transition temperature as a function of irradiation temperature for six steels irradiated in the HFR to 0.8 dpa. Taken from Rieth et al. [6].

3.4. Microstructures of 9Cr–2WV and 9Cr–2WVTa steels

A detailed TEM examination indicated the microstructures of 9Cr–2WV and 9Cr–2WVTa steels were similar both before and after irradiation in FFTF at 420°C to ≈36 dpa [10]. Microstructures were typical for tempered martensite. Lath size (width) was estimated to be ≈0.3 μm for both steels before irradiation, and it increased during irradiation to 0.4 and 0.45 μm for the 9Cr–2WV and 9Cr–2WVTa, respectively. The major microstructural difference in the two steels was a smaller prior-austenite grain size of the 9Cr–2WVTa steel (22 μm) compared to the 9Cr–2WV steel (32 μm) [2].

Although the size and number density of precipitates differed slightly in the two steels, the amounts were similar before and after irradiation. Most of the precipitate was $M_{23}C_6$ with lesser amounts of MC (Table 3) [10]. Analysis of the MC by X-ray energy dispersive spectroscopy indicated it was mainly vanadium rich, but some of it was tantalum rich in the 9Cr–2WVTa steel. It was estimated that less than half of the tantalum in the steel was present in the MC [10]. Subsequent atom probe analysis of the 9Cr–2WVTa indicated that over 90% of the tantalum remained in solution in the normalized-and-tempered steel [11]. No atom probe studies were conducted on irradiated specimens. There were slight differences in size and number density of precipitates for

Table 3
Precipitates in 9Cr–2WV and 9Cr–2WVTa steels before and after irradiation

Steel	Ppt	Before irradiation		After irradiation	
		Density (m^{-3})	Avg diam (nm)	Density (m^{-3})	Avg diam (nm)
9Cr–2WV	$M_{23}C_6$	5.9×10^{19}	125	3.2×10^{19}	160
	MC	1.2×10^{18}	54	1.1×10^{18}	60
9Cr–2WVTa	$M_{23}C_6$	4.5×10^{19}	136	4.1×10^{19}	143
	MC	7.5×10^{18}	29	5.6×10^{18}	36

9Cr–2WV and 9Cr–2WVTa (Table 3), but the amounts were similar. A slight increase in precipitate size and a corresponding reduction in number density occurred for both steels during irradiation [10].

The major observed microstructural change that occurred during irradiation of the 9Cr–2WV and 9Cr–2WVTa steels was the formation of dislocation loops. Again, no measurable difference was detected in the loop number density and size of the loops for the two steels [10]; loops were estimated to have an average size of ≈50 nm and a number density of $\approx 3 \times 10^{21}$ m^{-3} for both steels irradiated in FFTF to 36 dpa at 420°C. Total dislocation line density was estimated at 5×10^{14} m^{-2} for both steels [10].

4. Discussion

The 9Cr–2WVTa steel has excellent impact toughness, as measured by a low DBTT before irradiation. It also has excellent resistance to irradiation embrittlement, as demonstrated by a small shift in DBTT after irradiation at 250–450°C [2–8]. To try to understand the origin of the irradiation resistance of the 9Cr–2WVTa steel and the difference in properties with other similar steels, the properties of the steel will first be compared with those of the 9Cr–2WV, which differs from the 9Cr–2WVTa by not containing 0.07% Ta.

Irradiation hardening, as measured by an increase in yield stress, occurs for martensitic steels, such as the conventional 9Cr–1MoVNb and 12Cr–1MoVW steels, when irradiated at 425°C [12,13]. Hardening generally saturates with fluence, and the tensile properties of the 9Cr–2WV and 9Cr–2WVTa saturated by 7–10 dpa when irradiated at 365°C in FFTF (Fig. 1) [5]. Irradiation hardening causes the increase in DBTT, and just as hardening saturates with fluence, the DBTT and ΔDBTT also saturate for the 9Cr–1MoVNb and 12Cr–1MoVW steels [12,14]. Saturation in ΔDBTT apparently occurred for the 9Cr–2WV steel by 7 dpa at 365°C in FFTF, but this did not occur for the 9Cr–2WVTa steel (Fig. 2) [5].

The initial tensile and Charpy results obtained on the 9Cr–2WV and 9Cr–2WVTa steels after irradiation to 7 and 13 dpa were interpreted to show a linear relationship between ΔDBTT and $\Delta\sigma_y$ [3], indicating that the shift in DBTT was directly related to the irradiation hardening. However, this interpretation was subsequently rejected [4,5] after specimens irradiated to higher fluences showed that although hardening saturated with fluence for both steels, the ΔDBTT saturated only for the 9Cr–2WV, while it continued to increase for the 9Cr–2WVTa. Thus, although hardening is still thought to cause the embrittlement, something within the 9Cr–2WVTa changes with fluence to cause the increase in ΔDBTT in the absence of a change in $\Delta\sigma_y$.

The observation of similar tensile properties for 9Cr–2WV and 9Cr–2WVTa steels at 365°C is consistent with their relatively similar microstructures [10], although in some previous tests, the 9Cr–2WVTa steel was 4–10% stronger than the 9Cr–2WV steel over the range room temperature to 600°C (the smallest difference occurred at the highest test temperatures) [1]. The major microstructural difference in the two steels appears to be the prior-austenite grain size, which could account for part or all of the difference in Charpy impact transition temperature in the normalized-and-tempered condition. Tantalum, like niobium, is known to inhibit austenite grain growth, although the effect is generally concluded to be caused by carbides [15]. For the 9Cr–2WVTa steel, tantalum in solution or a very small amount of TaC must cause the smaller grain size, because most of the tantalum was in solution after the normalizing and tempering treatment [10,11]. Grain size can affect tensile behavior, but it did not appear to do so for the 365°C tests (Fig. 1(a)).

Aside from some relatively minor changes in the precipitate size and precipitate number density, which were similar for the 9Cr–2WV and 9Cr–2WVTa steels (Table 3), the major effect of irradiation on microstructure was the formation of dislocation loops; the number and size of loops formed and the final dislocation density in the two steels was the same after irradiation [10]. Similar microstructural changes resulted in similar changes in tensile properties, as might be expected (Fig. 1). Thus, the only significant difference observed in the 9Cr–2WV and 9Cr–2WVTa steels before and after irradiation was the prior-austenite grain size and the tantalum in solution.

Prior-austenite grain size could perhaps explain the difference in the Charpy impact properties of the normalized-and-tempered steels. However, after irradiation, the 9Cr–2WVTa showed two types of behavior that differed from what was observed for similar steels either conventional Cr–Mo steels (e.g., 9Cr–1MoVNb and 12Cr–1MoVW) or Cr–W steels (e.g., 9Cr–2WV, F82H, etc.). These differences were: (1) a continuous, albeit slow, increase in DBTT with increasing fluence (up to ≈28 dpa at 365°C) for the 9Cr–2WVTa, compared with a saturation in DBTT with fluence for other steels and (2) an increase in DBTT with increasing irradiation temperature for the 9Cr–2WVTa compared with a decrease with irradiation temperature for most other steels [12,14].

The increase in DBTT with fluence for 9Cr–2WVTa was not large, but it appeared to be continuous from ≈7 to 28 dpa (Fig. 2) (ΔDBTT increased from 4°C to 32°C). This was different from the behavior of 9Cr–2WV steel (Fig. 2). With the exception of the scatter in the 9Cr–2WV steel data due to the result at 16.7 dpa, the data gave a clear indication of a saturation of ΔDBTT with fluence. The reader will notice for the curve for

9Cr–2WV in Fig. 2 that if instead of ignoring the datum point at 16.7 dpa the one at 7.7 dpa is ignored, then a curve is obtained that shows an increasing DBTT with fluence, similar to the one for the 9Cr–2WVTa steel. This alternative was rejected based on the observation of the saturation in fluence for the tensile data, the observation that most steels show a saturation in the shift in DBTT with fluence, and the other observations (discussed here) of differences between the 9Cr–2WVTa and 9Cr–2WV and between the 9Cr–2WVTa and other steels.

The difference in behavior of the 9Cr–2WV and 9Cr–2WVTa steels cannot be explained by the difference in prior-austenite grain size, because prior-austenite grain size does not change during irradiation [8]. The lath size changes, but the change for the two steels was similar (the lath size of 9Cr–2WVTa was estimated to be slightly larger than that of the 9Cr–2WV after irradiation) [10]. As a possible explanation, it has been proposed that tantalum in solution causes the improved impact properties of the 9Cr–2WVTa, and tantalum then precipitates from solution during irradiation to cause the deterioration in properties [4]. Since no new precipitates were observed after irradiation [10], the tantalum is presumably incorporated in existing precipitates, although this still needs to be confirmed.

Irradiation hardening decreases with increasing irradiation temperature. Therefore, the ΔDBTT should also decrease with increasing irradiation temperature. This is what is observed for most steels [6,8,12]. At 400–425°C, hardening generally ceases [12,13], and ΔDBTT decreases to a small value or zero [12]. This has been observed for 9Cr–1MoVNb [12,13], 12Cr–1MoVW [12,13], and MANET [16] steels and, as seen in Fig. 3 for the 7–10% Cr steels irradiated to 0.8 dpa in HFR, the DBTT decreased with increasing irradiation temperature for all but the 9Cr–2WVTa steel [6]. Above ≈400°C where hardening ceases, the DBTT for all but the 9Cr–2WVTa steel appeared to approach a constant low value, and the ΔDBTT approached a small value. In contrast to these observations, the DBTT of the 9Cr–2WVTa at 450°C was larger than at 350°C and 400°C.

Contrary to the 9Cr–2WVTa steel, the DBTT of the 9Cr–2WV steel decreased with increasing temperature for irradiations at 365°C and 393°C (Table 2). A similar observation was made for the following steels irradiated in the same experiment [8]: Fe–2.25Cr–0.25V–0.1C, Fe–2.25Cr–1W–0.25V–0.1C, Fe–2.25Cr–2W–0.1C, Fe–2.25Cr–2W–0.25V–0.1C, and Fe–12Cr–2W–0.25V–0.1C.

The difference in the effect of temperature for the 9Cr–2WVTa steel and other steels in HFR and FFTF, especially the 9Cr–2WV, must again be sought in the tantalum in solution, since this is the major difference between 9Cr–2WV and 9Cr–2WVTa. The increase in DBTT with increasing irradiation temperature follows if tantalum is being removed from the solution during irradiation. Diffusion increases with irradiation temperature, and then at the higher temperature, diffusion accelerates the precipitation of tantalum over that precipitated at a lower temperature. Likewise, an increase in fluence (dpa) will allow for more irradiation-accelerated diffusion and more tantalum precipitation.

The ductile–brittle transition of a steel is explained in terms of the variation in flow stress with temperature and the relation of the flow stress to the fracture stress for cleavage, as shown schematically in Fig. 4 [17]. Flow stress increases with decreasing temperature, and as temperature decreases, it eventually exceeds the cleavage fracture stress, at which temperature a brittle cleavage fracture occurs (the DBTT). Irradiation causes an increase in flow stress, which causes an increase in DBTT, assuming that fracture stress is unchanged (Fig. 4). The observed effect of tantalum can be attributed to tantalum in solution either causing an increase in the fracture stress and/or a change in the flow stress behavior.

No detailed measurements of flow stress with temperature and strain rate (to determine if tantalum affects the behavior when the strain rate is increased from that for a tensile test to that of a Charpy test) exist for the 9Cr–2WV and 9Cr–2WVTa steels, although tensile tests over the range room temperature to 600°C were made on the normalized-and-tempered specimens [1]. The yield stress of the 9Cr–2WVTa steel was 4–10% greater than that for 9Cr–2WV steel, with the difference decreasing with increasing temperature, which is in accordance with the grain-size difference of the two steels. However, control specimens tested at 365°C for the

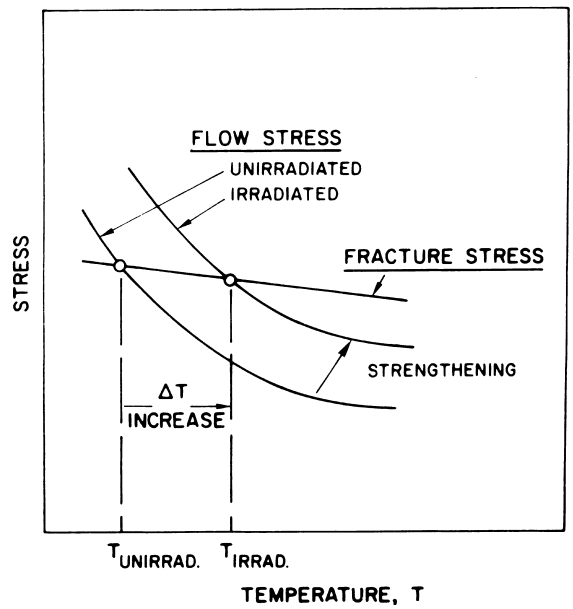

Fig. 4. Schematic illustration of the effect of irradiation on the transition temperature.

365°C irradiations in FFTF indicated less than 1% difference in the yield stress of the two steels as normalized-and-tempered (Fig. 1(a)) [5]. Likewise, after thermal aging up to 20 000 h at 365°C and testing at 365°C, there was less than 4% difference in yield stress, and after ≈28 dpa at 365°C in FFTF, tests at 365°C showed a difference of less than 1% (Fig. 1) [5]. These results give little indication of differences in flow stress behavior for the two steels that could explain the large effect of tantalum. Tests to determine the effect of temperature and strain rate on the flow curves are planned.

For this discussion, tantalum in solution is assumed to increase the fracture stress and/or change the flow curve behavior, and then during irradiation, precipitation of the tantalum causes the enhanced irradiation resistance of the 9Cr–2WVTa to deteriorate [8]. Assuming this hypothesis is correct, tantalum should precipitate in the 9Cr–2WVTa until equilibrium is achieved, after which the change in DBTT should saturate with fluence. This should also occur for the other tantalum-containing steels shown in Fig. 3, namely, F82H (0.02% Ta) and OPTIFER Ia (0.07% Ta). Fig. 3 shows data after irradiation to 0.8 dpa and no increase in DBTT at the highest irradiation temperature is observed for these two steels. Rieth et al. [7] recently published the combined data for the steels irradiated to 0.2, 0.8, and 2.5 dpa in HFR, and Fig. 5 shows the results for F82H, OPTIFER Ia and 9Cr–2WVTa – the tantalum-containing steels. After 2.5 dpa, the DBTT at 450°C for all three steels is higher than it is at 350°C and 400°C. The behavior of the three tantalum-containing steels contrasted with that of MANET I, MANET II and OPTIFER II – the steels with no added tantalum. With one exception, the steels without tantalum additions do not show a DBTT after 2.5 dpa at 450°C that is above that at 350°C and 400°C. The only exception is MANET II, where the DBTT at 450°C is slightly higher than at 400°C, but it is well below that at 350°C.

It is recognized that Charpy data can contain considerable scatter. Nevertheless, the trend of these data for the tantalum-containing F82H and OPTIFER Ia appear to agree with the suggestion that tantalum causes the inverse temperature effect for the DBTT of the 9Cr–2WVTa steel. A loss of tantalum from solution during irradiation can explain these results, although the actual loss of tantalum needs to be confirmed. Atom probe analysis is probably the best method to study this, given the small amount of tantalum in the steels.

Figs. 5 and 6 show some other interesting effects relative to the above discussion. First, saturation was not established for any of the steels at the lowest temperature by 0.8 dpa (the DBTT subsequently increased during the 2.5 dpa irradiation). Whether saturation was achieved by 2.5 dpa can only be determined by higher dose experiments. Several of the steels give an indication of saturation at 300°C, and at 350°C saturation appears

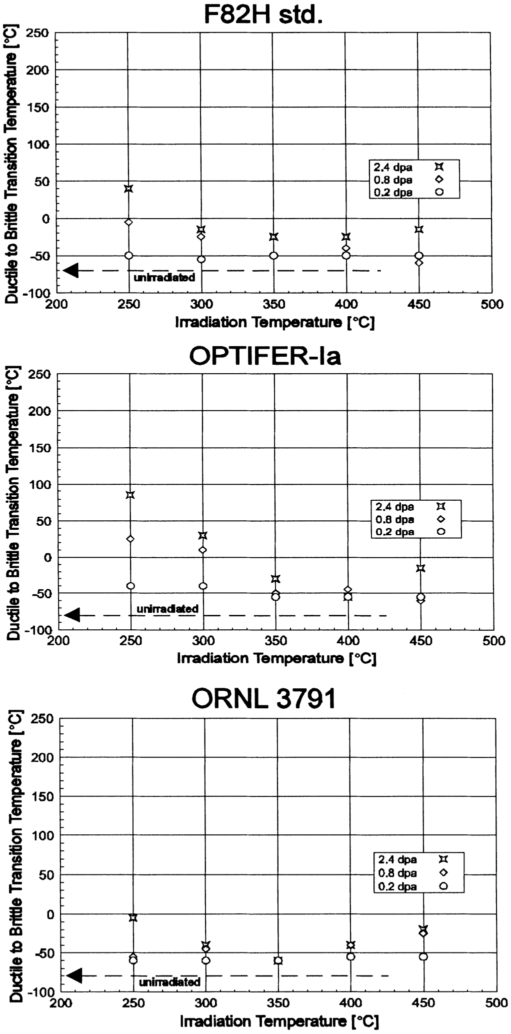

Fig. 5. Ductile–brittle transition temperature as a function of irradiation temperature for the F82H, OPTIFER Ia, and 9Cr–2WVTa steels.

to have been reached for most of the steels by 0.8 dpa. This should be a metastable saturation for the tantalum-containing steels, since with time (fluence), tantalum should precipitate at temperatures below 450°C and have the same effect at the lower temperatures as at 450°C.

These results raise the question about the usefulness of tantalum in the 9Cr–2WVTa steel. The increase in DBTT with fluence should continue until tantalum equilibrium is reached. If it is assumed that at equilibrium essentially all of the tantalum is removed from solution, the matrix of the 9Cr–2WVTa will be similar to that of the 9Cr–2WV (the only difference will be the few tantalum-containing precipitates in the 9Cr–2WVTa, which do not appear to affect the strength). Similar

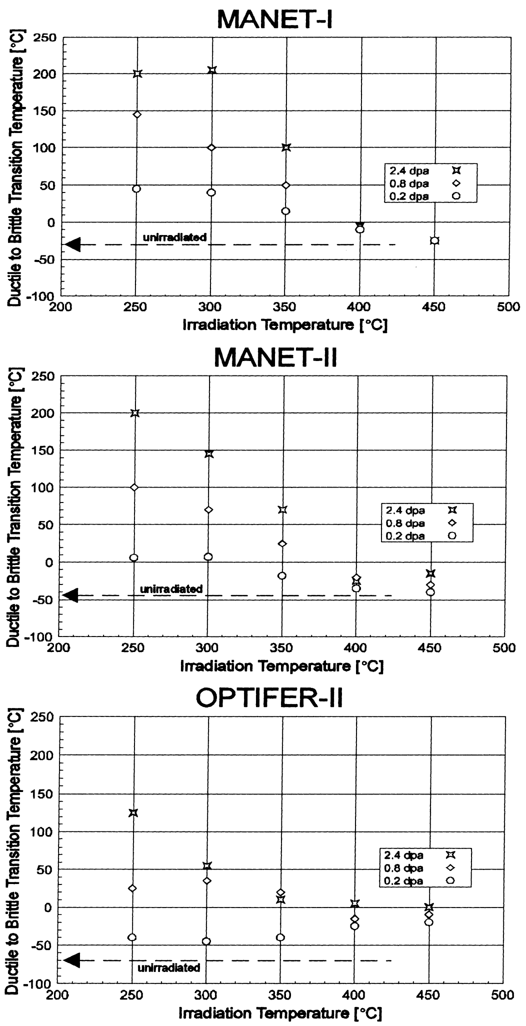

Fig. 6. Ductile–brittle transition temperature as a function of irradiation temperature for the MANET I, MANET II, and OPTIFER II steels.

microstructures imply similar ΔDBTTs. This would seem to indicate that, although the ΔDBTT of the 9Cr–2WVTa increases with dose, it should be similar to that of the 9Cr–2WV when the tantalum in solution reaches equilibrium. This implies that the 9Cr–2WVTa will still have an advantage over the 9Cr–2WV, because it had the lowest DBTT in the normalized-and-tempered condition due to tantalum in solution and the smaller prior-austenite grain size. The latter will be maintained after irradiation.

That this conclusion might be correct is seen from the observations on the 9Cr–2WV and 9Cr–2WVTa steels after irradiation in FFTF at 393°C (Table 2). At this temperature, the ΔDBTT values for the two tests for each steel were, respectively, 34°C and 43°C for 9Cr–2WVTa compared to 46°C and 34°C for 9Cr–2WV; thus, the large advantage for the 9Cr–2WVTa steel at 365°C is no longer there at 393°C. With allowances for data scatter, the shifts can be concluded to be similar. Because of the lower DBTT of the 9Cr–2WVTa before irradiation, this steel still has a lower DBTT than the 9Cr–2WV after the 393°C irradiation.

If the above analysis is correct, the advantage for the 9Cr–2WVTa steel after precipitation of the tantalum is attributed to the smaller prior-austenite grain size in the tantalum-containing steel. Therefore the tantalum addition is beneficial. However, if this analysis is correct, it should be possible to get similar results in the absence of tantalum if the grain size of the 9Cr–2WV steel could be reduced, say by an appropriate heat treatment. Properties could be further improved by alloying with an element that changes the fracture stress and/or the flow stress similar to the change caused by tantalum, but unlike tantalum, it remains in solution during irradiation.

5. Summary and conclusions

An Fe–9Cr–2W–0.25V–0.07Ta–0.1C (9Cr–2WVTa) steel shows excellent resistance to irradiation embrittlement as manifested by the small shift in DBTT after irradiation to ≈7–28 dpa at 365°C in FFTF. Tantalum plays an important role in the improved irradiation resistance of the 9Cr–2WVTa, because the ΔDBTT for the 9Cr–2WVTa steel after ≈28 dpa at 365°C (32°C) is considerably smaller than for the 9Cr–2WV steel – the same steel but without tantalum – also irradiated to ≈28 dpa at 365°C (ΔDBTT ≈60°C). The 9Cr–2WVTa steel also had excellent irradiation resistance relative to other steels when irradiated in HFR at 250–450°C.

The 9Cr–2WVTa steel displayed differences in behavior from other steels. First, the DBTT of the 9Cr–2WVTa steel increased continuously with fluence when irradiated at 365°C in FFTF, compared to the 9Cr–2WV steel and most other such steels that show a saturation in ΔDBTT with fluence. Contrary to most other steels of this type, the 9Cr–2WVTa steel also showed a larger DBTT and increase in DBTT at 400–450°C than at the lower irradiation temperatures (250–350°C) in the HFR and higher values at 393°C than 365°C in FFTF.

The increased resistance to irradiation-induced embrittlement of the 9Cr–2WVTa steel was proposed to be due to tantalum in the solution causing either an increase in the fracture stress or a change in the flow behavior. Further work is required to determine which of these possibilities causes the observed effect. Precipitation of tantalum from solution during irradiation was hypothesized to cause the difference in behavior of a tantalum-containing steel compared to the one without tantalum when fluence or temperature is increased. The precipitation of the tantalum during irradiation still needs to be verified.

Acknowledgements

We wish to thank R.L. Swain and E.T. Manneschmidt for conducting the impact tests and M.A. Sokolov and S.J. Zinkle for reviewing the manuscript.

References

[1] R.L. Klueh, Metall. Trans. A 20A (1989) 463.
[2] R.L. Klueh, D.J. Alexander, P.J. Maziasz, J. Nucl. Mater. 186 (1992) 185.
[3] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 736 (1994) 212.
[4] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 336 (1996) 233.
[5] 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, 1999, p. 911.
[6] M. Rieth, B. Dafferner, H.D. Röhrig, J. Nucl. Mater. 351 (1996) 233.
[7] M. Rieth, B. Dafferner, H.D. Röhrig, J. Nucl. Mater. 1147 (1998) 258.
[8] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 265 (1999) 262.
[9] 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.
[10] J.J. Kai, R.L. Klueh, J. Nucl. Mater. 230 (1996) 116.
[11] R. Jayaram, R.L. Klueh, Met. and Matls. Trans. A 29A (1998) 1551.
[12] W. Hu, D.S. Gelles, in: F.A. Garner, C.H. Henager Jr., N. Igata (Eds.), Influence of Radiation on Material Properties: 13th International Symposium (Part II), ASTM STP 956, American Society for Testing and Materials, Philadelphia, 1987, p. 83.
[13] R.L. Klueh, J.M. Vitek, J. Nucl. Mater. 182 (1991) 230.
[14] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 1269 (1998) 258.
[15] G. Krauss, Steels: Heat Treatment and Processing Principles, ASM International, Materials Park, OH, 1990, p. 199.
[16] M. Rieth, B. Dafferner, H.D. Röhrig, C. Wassilew, Fusion Eng. Design 29 (1995) 365.
[17] J.R. Hawthorne, Treatise Mater. Sci. Tech. 25 (1983) 461.