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Effect of heat treatment and irradiation temperature on mechanical properties and structure of reduced-activation Cr–W–V steels of bainitic, martensitic, and martensitic–ferritic classes

I.V. Gorynin, V.V. Rybin *, I.P. Kursevich, A.N. Lapin, E.V. Nesterova, E.Yu. Klepikov

Central Research Institute of Structural Materials 'Prometey', 49 Shpalernaja Street, 193015 St. Petersburg, Russian Federation

Abstract

Effects of molybdenum replacement by tungsten in steels of the bainitic, martensitic, and martensitic–ferritic classes containing 2.5%, 8% and 11% Cr, respectively, were investigated. The phase composition and structure of the bainitic steels were varied by changing the cooling rates from the austenitization temperature (from values typical for normalization up to $V = 3.3 \times 10^{-2}$°C/s) and then tempering. The steels were irradiated to a fluence of 4×10^{23} n/m^2 ($\geqslant$0.5 MeV) at 270°C and to fluences of 1.3×10^{23} and 1.2×10^{24} n/m^2 ($\geqslant$0.5 MeV) at 70°C. The 2.5Cr–1.4WV and 8Cr–1.5WV steels have shown lower values of the shifts in ductile–brittle transition temperature (DBTT) under irradiation in comparison with corresponding Cr–Mo steels. Radiation embrittlement at elevated irradiation temperature was lowest in bainitic 2.5Cr–1.4WV steel and martensitic–ferritic 11Cr–1.5WV steel. The positive effect of molybdenum replacement by tungsten at irradiation temperature ~300°C is reversed at $T_{irr} = 70$°C.

1. Introduction

Investigations of radiation damage resistance of reduced-activation Cr–W–V steels for fusion reactor applications have generally been carried out on materials irradiated in fast reactors to high doses (>10 dpa) at irradiation temperature $T_{irr} \geqslant 360$°C [1–4]. However, more recently researchers have studied radiation resistance of these materials under conditions of neutron irradiation at lower temperatures and doses [5,6].

It has been shown [5] that the replacement of molybdenum by tungsten on an approximately atom-for-atom basis improves the radiation resistance of the two-phase 11Cr–1.5WV steel in comparison with 11Cr–0.8MoV steel at a fluence of $\sim 1.4 \times 10^{24}$ n/m^2 (T_{irr} = 240–300°C). These results suggest that the replacement of molybdenum by tungsten may have a favorable influence not only on the reduced-activation characteristics, but also on the radiation resistance of other heat-resistant chromium steels. This paper presents the results of molybdenum replacement by tungsten on the structure and mechanical properties of reduced-activation Cr–W–V-steels containing 2.5%, 8–9% and 11% Cr under low dose irradiation conditions at different temperatures.

2. Experimental procedure

Reduced-activation Cr–W–V-steels containing ~2.5%, ~8%, and 11% Cr and designated as 2.5Cr–1.4WV, 8Cr–1.5WV, 11Cr–1.5WV were investigated. The corresponding molybdenum-containing reference materials have nominal compositions of 2.5Cr–0.7MoV, 8Cr–0.8MoV and 11Cr–0.8MoV. Molybdenum was replaced by tungsten approximately on an atom-for-atom

* Corresponding author. Tel.: +7-812 274 1227; fax: +7-812 274 1707.

E-mail address: rybin@prometey2.spb.su (V.V. Rybin).

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Table 1
Chemical composition of steels

Steel designation	Concentration (wt%)[a]										
	C	Si	Cr	Mn	W	V	S	P	Cu	Y	Mo
Bainitic steels											
2.5Cr–1.4WV	0.18	0.20	2.40	0.34	1.3	0.24	0.004	0.007	<0.10	–	<0.03
2.5Cr–0.7MoV[b]	0.18	0.23	2.38	0.48	–	0.30	0.004	0.006	<0.10	–	0.73
Martensitic steels											
8Cr–1.5WV	0.15	0.19	8.2	0.35	1.4	0.26	0.006	0.018	<0.10	0.01	–
8Cr–0.8MoV[b]	0.10	0.18	8.4	0.48	–	0.26	0.007	0.015	<0.15	–	0.85
Martensitic–ferritic steels											
11Cr–1.5WV	0.13	0.29	10.6	0.37	1.35	0.26	0.007	0.018	<0.10	0.01	–
11Cr–0.8MoV[b]	0.09	0.28	10.9	0.47	–	0.25	0.007	0.015	<0.10	0.01	0.81

[a] Balance iron.
[b] Reference steels.

basis in the reduced-activation versions. Yttrium was added to the 8% and 11% Cr steels to modify and clear grain boundaries. The chemical compositions of the materials are given in Table 1. The steels containing 2.5% Cr are bainitic, and the 8% and 11% Cr steels are martensitic and martensitic–ferritic (about 20% of delta-ferrite), respectively.

The reduced-activation Cr–W–V and reference Cr–Mo–V steels were melted in an open induction, 100 kg furnace with the same charge, and were poured as 16 kg ingots. The ingots were forged at temperatures between 900°C and 1050°C into rods 10–15 mm diameter and sheet billets $40 \times 80 \times 150$ mm^3. Sheet billets were rolled into plates ($15 \times 20 \times 400$ mm^3). The rods and the plates were heat treated as shown in Table 2. Small charpy specimens ($5 \times 5 \times 27.5$ mm^3) with a V-notch 0.5 mm wide and 0.5 mm deep were cut out of the plates along the rolling direction (L–T orientation). Cylindrical tensile specimens with a 3 mm diameter and 15 mm long gauge section were cut from the rods. The structure of the steels was investigated by both optical and transmission electron microscopy methods.

Specimens of the bainitic steels were irradiated in the core of a VVER-440 power reactor at the coolant temperature (270 ± 10°C) to a fluence of $\sim 4 \times 10^{23}$ n/m^2 ($\geqslant 0.5$ MeV). Irradiation of the high chromium steels was carried out in the core of the WWR-M experimental reactor at a temperature of 70°C (cooling water) to fluences of 1.3×10^{23} and 1.4×10^{24} n/m^2 ($\geqslant 0.5$ MeV). The reference 8Cr–0.8MoV steel was irradiated in the core of the WWR-M reactor at ~ 300°C (in helium) together with the steels reported earlier [5].

Impact tests of the unirradiated and irradiated specimens were carried out on a remote pendulum-type impact machine with a maximum energy of 50 J.

The experimental data were fit to a hyperbolic tangent function to permit the ductile–brittle-transition temperature (DBTT) and the upper shelf energy (USE) to be evaluated. In this case the DBTT was defined at the absorbed energy halfway between the upper and lower shelves and it is indicated below as $T_{0.5}$. A DBTT (T_{6J}) was also evaluated at 6 J of impact energy [7].

The tensile tests were carried out on a remote testing machine in air at room temperature at a strain rate of 3×10^{-3} s^{-1}.

3. Results and discussion

3.1. Bainitic steels

The 2.5Cr–1.4WV and 2.5Cr–0.7MoV steels were investigated in four structural conditions obtained by the following heat treatments:
1. austenitized 1 h at 1000°C, air cooled (normalized);
2. normalized with the subsequent tempering 10 h at 680°C;
3. austenitized 1 h at 1000°C, cooling at 3.3×10^{-2}°C/s;
4. #3 with subsequent tempering for 10 h at 680°C.

The structure of both steels after normalization is practically identical and characterized by a non-uniform distribution of ferrite (up to 10%) and granular bainite. The granular bainite consists of a ferrite matrix with a high dislocation density ($\rho = 10^{15}$ m^{-2}) and martensite-austenite islands located both at grain boundary triple points and on subgrain boundaries.

The mechanical properties of these steels after normalization are also similar: yield stress $\sim$925 MPa, $T_{0.5}$ -21°C, USE $\sim$ 14 J (Table 2).

The structure of both steels cooled at a rate of 3.3×10^{-2}°C/s differs greatly from the structure of the normalized steel. The ferrite quantity is $\sim$80% and $\sim$70% for tungsten- and molybdenum-containing steels, respectively. The ferrite grains are characterized by a low dislocation density ($\rho = 10^{13}$ m^{-2}), and they contain a uniform distribution of carbides that are spherical with a

Table 2
Effect of heat treatment on radiation hardening and embrittlement in steels of bainitic, martensitic and martensitic–ferritic classes

Steel heat treatment[a]	Structure[b]	$F \times 10^{24}$ n/m^2 ($E \geqslant 0.5$ Mev)	T_{irr} (°C)	$\sigma_{YS}^{20°C}$ (MPa)		$\Delta\sigma_{YS}$ (MPa)	DBTT[c] ($T_{0.5}/T_{6\,J}$) (°C)		ΔDBTT ($T_{0.5}/T_{6\,J}$) (°C)	A_F[d]	USE (J)		Ref.
				Unirr	Irrad		Unirr	Irrad			Unirr	Irrad	
Bainitic steels													
2.5Cr–1.4WV, B1	10% F + 90% B	–	–	925	–	–	21/0	–	–	–	14.5	–	
2.5Cr–1.4WV, B2	10% F + 90% B	–	–	685	–	–	−78/−98	–			26	–	
		0.4	270	–	735	50	–	−66/−91	12/7	3.5	–	30	
2.5Cr–1.4WV, B3	80% F + 20% PP	–	–	300	–	–	−45/−54	–	–	–	20	–	
		0.4	270	–	370	70	–	−21/−46	24/8	7	–	28	
2.5Cr–1.4WV, B4	80% F + 20% PP	–	–	325	–	–	−64/−89	–	–	–	23	–	
		0.4	270	–	360	35	–	−38/−58	26/31	8	–	25	
2.5Cr–0.7MoV, B1	10% F + 90% B	–	–	925	–	–	21/−8	–	–	–	14	–	
2.5Cr–0.7MoV, B2	10% F + 90% B	–	–	670	–	–	−79/−105	–	–	–	24	–	
		0.4	270	–	685	15	–	−49/−69	30/36	9	–	25	
2.5–0.7MoV, B3	70% F + 30% PP	–	–	350	–	–	−36/−55	–	–	–	18	–	
		0.4	270	–	390	40	–	3/−5	39/50	11.5	–	28	
2.5Cr–0.7MoV, B4	70% F + 30% PP	–	–	330	–	–	−53/−75	–	–	–	25	–	
		0.4	270	–	395	65	–	−24/−41	29/34	8.5	–	30	
Martensitic steels													
8Cr–1.5WV, M1	TM	–	–	550	–	–	−20/−25	–	–	–	18.5	–	
		0.13	70	–	760	210	–	61/59	81/84	35	–	15	
		1.2	70	–	870	320	–	90/86	110/111	23	–	15	
		1.3	300	–	790	240	–	15/13	35/28	7	–	17	[5]
8Cr–0.8MoV, M1	TM	–	–	530	–	–	−30/−36	–	–	–	15	–	
		1.3	300	–	780	250	–	52/51	82/87	16.5	–	12	
Martensitic–ferritic steels													
11Cr–1.5WV, M–F1	TM	–	–	570	–	–	20/8	–	–	–	15	–	
		1.2	70	–	850	280		125/118	145/126	30	–	14	
		1.4	240	–	820	250	–	35/47	15/39	3	–	13	[5]
11Cr–0.8MoV, M–F2	TM	–	–	720	–	–	0/−6	–	–	–	13	–	
		1.2	70	–	990	270	–	97/97	97/103	20	–	12	
		1.3	300	–	880	160	–	60/59	60/65	12	–	12	[5]

[a] B1 – 1000°C/1 h/ac; B2 – 1000°C/1 h/ac + 680°C/10 h; B3 – 1000°C/1 h/c.r. 3.3×10^{-2}°C/s; B4 – 1000°C/1 h/c.r. 3.3×10^{-2}°C/s + 680°C/10 h; M1 – 1050°C/1 h/ac + 700°C/3 h; M–F1 – 1050°C/1 h/ac + 700°C/3 h; M–F2 – 1050°C/1 h/ac + 700°C/1.5 h.

[b] F–ferrite; B–bainite; PP–pseudo-pearlite; TM–tempered martensite.

[c] DBTT($T_{0.5}$) and DBTT($T_{6\,J}$)-ductile–brittle-transition temperature evaluated at an energy level halfway between the upper and lower shelves and energy level 6 J, respectively.

[d] – Calculated by the ratio $\Delta T_{0.5} = A_F \cdot F^{1/3}$ [9].

diameter ~100 nm. In addition, a pearlitic structure with regularly located layers of cementite was observed. The respective mechanical properties of the 2.5Cr–1.4WV and 2.5Cr–0.7MoV steels were: yield stress ~300 and 350 MPa, $T_{0.5}$ −45°C and −36°C, and USE −20 and 18 J.

Tempering the normalized steels resulted in polygonization of their dislocation structure and dissociation of the martensite and residual austenite into ferrite and large ($d \approx 400$ nm) cementite carbides located on boundaries of the subgrains. The respective properties of the tungsten- and molybdenum-containing steels were: yield stress 685, 670 MPa; $T_{0.5}$ −78°C, −79°C; and USE 26, 24 J.

Tempering the steels after slow cooling resulted in some enlargement of the cementite layers. In this case, the yield stress of the 2.5Cr–1.4WV steel increased to ~325 MPa and the yield stress of the 2.5Cr–0.7MoV steel decreased to 330 MPa. The $T_{0.5}$ of steels decreased to −64°C and −53°C and the USE rose up to 23 and 25 J, respectively (Table 2).

Thus, replacement of molybdenum by tungsten did not affect the structure and mechanical properties of the bainitic steels in the normalized and normalized-and-tempered conditions. Even for the slowly cooled steels, which differed from normalized steels in structure and properties, replacement of molybdenum by tungsten did not change the properties.

Table 2 shows the effects of irradiation at a fluence of 4×10^{23} n/m^2 at 270°C upon mechanical properties of the bainitic steels. Radiation hardening for the various heat treatments of the 2.5Cr–1.4WV steel is within the range 35–70 MPa and for 2.5Cr–0.7MoV steel is within the range 15–65 MPa. The uniform and total relative elongation was not much affected by irradiation. These elongations decreased less than 2.5%. The replacement of molybdenum by tungsten in both steels affected mostly the shift in DBTT. For the 2.5Cr–1.4WV steel in the normalized-and-tempered condition the shift in $T_{0.5}$ was 12°C, while for molybdenum-contained steel it was 30°C. Similarly, the tungsten-containing steel has the lower DBTT after both slow cooling without tempering (regime 3) and with subsequent tempering (regime 4) compared with the molybdenum-containing steel.

In Table 2, it is also seen that the upper shelf energy does not decrease after irradiation but increases slightly. For normalized-and-tempered tungsten- and molybdenum-containing steels, this increase was 1–4 J; for the slowly cooled steels without tempering, 8–10 J; and for slowly cooled steels with tempering, 2–5 J. These shifts are consistent with the degree of hardening and the small decrease in elongation. Because of the increase of the USE after irradiation, the value of $T_{0.5}$ had an additional component of increase. Accordingly, the T_{6J} shifts were appreciably lower for the reduced-activation steel compared to the reference steels in the normalized-and-tempered condition. Thus, the 2.5Cr–1.4WV reduced-activation steel showed better radiation resistance than that of 2.5Cr–0.7MoV steel in all of the conditions.

3.2. Martensitic and martensitic–ferritic steels

The positive influence of replacing molybdenum by tungsten on radiation resistance also occurs for the martensitic steels containing 8% Cr. After irradiation to a fluence of 1.3×10^{24} n/m^2 ($T_{irr} = 300$°C), the 8Cr–1.5WV and 8Cr–0.8MoV steels in the normalized-and-tempered condition had shifts in $T_{0.5}$ of 35°C and 82°C, and shifts in T_{6J} −28°C and 87°C, respectively. In both cases, the increase in yield stress was 240–250 MPa.

However, the 11Cr–1.5WV tungsten-containing steel loses its advantage in radiation resistance as compared with 11Cr–0.8MoV molybdenum-contained steel at low irradiation temperature. After irradiation to fluence of 1.2×10^{24} n/m^2 at 70°C, the shifts in $T_{0.5}$ of these steels were 145°C and 97°C, and the shifts in T_{6J} were 126°C and 103°C. This effect was theoretically predicted in the previous work [8].

The data obtained in this study indicate a strong influence of neutron irradiation at low temperature (70°C).

Comparison with previous data [5] shows decreasing radiation effects for martensitic and martensitic–ferritic steels with increasing irradiation temperature. Increasing the irradiation temperature from 70°C to 300°C (a fluence is $(1.2–1.3)\times10^{24}$ n/m^2) result in decreases of $\Delta T_{0.5}$ from 110°C to 35°C for the martensitic 8Cr–1.5WV steel (Table 2). The decrease in shift with increasing irradiation temperature from 70°C to 240°C is also significant for the martensitic–ferritic 11Cr–1.5WV steel (Table 2).

One may try to estimate quantitatively the sensitivity to neutron irradiation of the bainitic steels for which the changes in DBTT within the range of neutrons fluence 10^{23}–10^{24} n/m^2 are described by the following equation [9]:

$$\Delta T_{0.5} = (A_F)F^{1/3}, \qquad (1)$$

where A_F is a coefficient of radiation embrittlement and F is the neutron fluence in units of 10^{22} n/m^2. For molybdenum-containing steels of the 2.5Cr–0.7MoV grade at an irradiation temperature 270°C, the coefficient A_F is in the range of values 8–12 [9]. For the reference steel of 2.5Cr–0.7MoV grade studied here, A_F for different heat treatment conditions is in the range of values 9–11.5, which correspond well with previous data [9], and can be considered as a reference value for comparison. For the tungsten-containing steel, A_F for different heat treatment conditions is within the range 3.5–8. This is again indicative that the reduced-activation tungsten-containing steels are less sensitive to radiation embrittlement than the reference steels.

Likewise the A_F values at irradiation temperatures in the range 240–300°C for 8Cr–1.5WV and 8Cr–0.8MoV steels are 7 and 16.5, and for the 11Cr–1.5WV and 11Cr–0.8MoV steels are 3 and 12, respectively.

At low temperature, the sensitivity of the steels to irradiation is much higher: A_F values of 8Cr–1.5WV and 11Cr–1.5WV tungsten-containing steels are within the range 23–35, while A_F value of 11Cr–0.8MoV molybdenum-containing steel is equal to 20.

Thus, among tungsten-containing steels, the 2.5Cr–1.4WV and 11Cr–1.5WV steels have shown the least sensitivity to radiation embrittlement at $T_{irr} = 240$–300°C; the A_F coefficient for these steels was 3.5 and 3, respectively.

4. Conclusions

1. Replacing molybdenum by tungsten in 2.5Cr–1.4WV and 2.5Cr–0.7MoV bainitic steels improve radiation resistance at $T_{irr} = 270°C$. At a fluence of $\sim 4 \times 10^{23}$ n/m^2, the shift in DBTT ($T_{0.5}$) for these steels in the normalized-and-tempered condition was 12°C and 30°C, respectively. In the unirradiated condition, these steels have a similar level of mechanical properties for a range of heat treatment variations.
2. Replacing molybdenum by tungsten in 8Cr–1.5WV and 8Cr–0.8MoV martensitic steels also improves them. After irradiation to a fluence of $\sim 1.3 \times 10^{24}$ n/m^2 ($T_{irr} = 300°C$), the shifts in DBTT ($T_{0.5}$) of these steels were 35°C and 82°C, respectively.
3. For 11% Cr martensitic–ferritic steels (11Cr–1.5WV and 11Cr–0.8MoV), the replacing molybdenum by tungsten result in lower shifts at higher irradiation temperature. At $T_{irr} = 240$–300°C (a fluence of $\sim 1.3 \times 10^{24}$ n/m^2) the shifts in DBTT ($T_{0.5}$) in these steels were 15°C and 60°C, while after irradiation at 70°C these shifts were 145°C and 97°C, respectively.

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