



# Effect of neutron irradiation at low temperature on the embrittlement of the reduced-activation ferritic steels

V.V. Rybin \*, I.P. Kursevich, A.N. Lapin

*Central Research Institute of Structural Materials "Prometey", 49 Shpalernaja Str., 193015 St. Petersburg, Russian Federation*

## Abstract

Effects of neutron irradiation to fluence of  $2.0 \times 10^{24}$  n/m<sup>2</sup> ( $E > 0.5$  MeV) in temperature range 70–300°C on mechanical properties and structure of the experimental reduced-activation ferritic 0.1%Cr–(2.5–12)%Cr–(1–2)%W–(0.2–0.7)%V alloys were investigated. The steels were studied in different initial structural conditions obtained by changing the modes of heat treatments. Effect of neutron irradiation estimated by a shift in ductile-brittle transition temperature ( $\Delta$ DBTT) and reduction of upper shelf energy ( $\Delta$ USE) highly depends on both irradiation condition and steel chemical composition and structure. For the steel with optimum chemical composition (9Cr–1.5WV) after irradiation to  $2 \times 10^{24}$  n/m<sup>2</sup> ( $E \geq 0.5$  MeV) at 280°C the  $\Delta$ DBTT does not exceed 25°C. The shift in DBTT increased from 35°C to 110°C for the 8Cr–1.5WV steel at a decrease in irradiation temperature from 300°C to 70°C. The CCT diagrams are presented for several reduced-activated steels. © 1998 Elsevier Science B.V. All rights reserved.

## 1. Introduction

Development of fast induced-radioactivity decay (FIRD) structural alloys for fusion reactor application is considered an urgent task of radiation materials science. The high-chromium tungsten–vanadium steels, containing no long-lived radioactive isotopes (Ni, Co, Mo, Nb and so on) are considered as very promising materials [1,2].

Almost all published data refer to irradiation in the fast reactors at a temperature more than 350°C. The data, which relate to irradiation at temperature lower than 300°C have been very scant [3].

In this paper the experimental data are given on the effects of radiation strengthening and embrittlement of some experimental FIRD steels with (7–11)%Cr, (0–1.5)%W; (0.2–0.7)%V (designated 7Cr–1.5WV; 8Cr–1.5WV; 9Cr–1.5WV; 9Cr–V; 9Cr–WV; 11Cr–1.5WV) containing the modifying additions (the rare earth elements) and without them and differing in phosphorus and copper impurities content. In addition the influence of the cooling rate and tempering modes of these steels

upon their microstructure, phase composition and mechanical properties are described.

## 2. Experimental procedure

The experimental 2.5Cr–WV and (7–11) Cr–WV FIRD steels with various content of main (Cr, W, V) and impurity (P, Cu) elements as well as the 2.5Cr–MoV and 11Cr–1MoV steels were investigated. The alloys containing molybdenum were selected as reference one.

The chemical composition of materials is given in Table 1. Experimental heats of FIRD steels and the 2.5Cr–MoV and 11Cr–1MoV steels were melted in open induction 100-kg furnace and poured in 16-kg ingots. These ingots were forged within the temperature range 1050–900°C into sheet billets 40 × 80 × 150 mm and rods of 10–15 mm diameter. Sheet billets were rolled out into plates (15 × 20 × 400 mm) from which transverse Charpy V-notched specimens (5 × 5 × 27.5 mm) were cut out. Tensile cylindrical specimens with gauge of 3 mm diameter and 15 mm length, as well as cylindrical specimens of 3 mm diameter and 10 mm length to obtain continuous cooling transformation diagrams were cut out of the rods.

\* Corresponding author. Tel.: +7 812 2741227; fax: +7 812 2741707; e-mail: root@rybin.spb.su.

Table 1  
Chemical composition of steels

Steel designation	Concentration (wt%) <sup>a</sup>									
	C	Si	Cr	Mn	W	V	S	P	Cu	Others
2.5Cr–0.7MoV <sup>c</sup>	0.18	0.23	2.38	0.48	–	0.30	0.004	0.006	<0.10	0.73Mo
2.5Cr–1.4WV	0.18	0.20	2.40	0.34	1.3	0.24	0.004	0.007	<0.10	<0.03Mo
7Cr–1.5WV	0.09	0.10	7.0	0.23	1.65	0.21	0.012	0.015	<0.10	
7Cr–1.5WVP	0.10	0.10	6.7	0.20	1.6	0.21	0.013	0.035	<0.10	
7Cr–1.5WVCu	0.13	0.13	7.0	0.25	1.6	0.24	0.012	0.020	0.15	
8Cr–1.5WV	0.15	0.19	8.2	0.35	1.4	0.26	0.006	0.018	<0.10	RE <sup>b</sup>
9Cr–V	0.09	0.33	9.1	1.30	–	0.70	0.012	0.006	<0.10	RE
9Cr–WV	0.10	0.31	9.1	1.20	0.5	0.40	0.012	0.005	<0.10	RE
9Cr–1.5WV	0.11	<0.20	9.0	0.59	1.28	0.24	0.013	<0.005	<0.10	RE
11Cr–1.5WV	0.13	0.29	10.6	0.37	1.35	0.26	0.007	0.018	<0.10	RE
11Cr–1MoV <sup>c</sup>	0.09	0.28	10.9	0.47	–	0.25	0.007	0.015	<0.10	0.81Mo + RE

<sup>a</sup> Balance iron.

<sup>b</sup> Rare-earth addition.

<sup>c</sup> The steels are presented as reference one.

Heat treatment of steels was carried out according to typical regime accepted for the FIRD steels: austenitization at 1000–1050°C, cooling in air, and tempering at 650–750°C for 0.5–3.0 h.

Irradiation was carried out in the core of experimental reactor WWR-M at temperature 70°C (in cooling water) and within temperature range 240–300°C (in inert gas), with a fluence from  $1.2 \times 10^{24}$  up to  $2 \times 10^{24}$  n/m<sup>2</sup> ( $E \geq 0.5$  MeV). The irradiation temperature was measured by means of thermocouples installed directly on the specimens along the assembly height. The impact tests were carried out by means of the impact testing machine 2121 KM-0.05 with maximum energy 50 J. The tensile tests were carried out in air at room temperature at a strain rate of  $3 \times 10^{-3}$  s<sup>-1</sup>.

### 3. Results and discussion

#### 3.1. Initial structure

The initial structure and mechanical properties of ferritic steels depends not only on chemical composition but on heat treatment conditions. In Fig. 1 are shown the thermo-kinetic diagrams of phase transformations in 2.5Cr–1.4WV; 2.5Cr–0.7MoV; 8Cr–1.5WV; 9Cr–V, and 9Cr–WV steels after their continuous cooling with different rates from the austenitizing temperature 1050°C. The thermo-kinetic diagrams showed that it is not always necessary that steels containing 2.5 and (8–9)%Cr should have a martensitic structure. If the cooling rate after austenitizing is lower than a critical value ( $V_{cr}$ ), the steel will undergo a ferritic-carbide transformation and as a result its structure will be not single phase of martensite but a complete ferrite phase which is characterized by large grains containing no fragments and

dislocations inside. The value  $V_{cr}$  is determined by the chemical composition of steel.

The results of the phase transformation investigation correlate with the mechanical properties of these steels obtained after cooling with different rates from the austenitization temperature. In 9Cr–V and 9Cr–WV steels, the yield stress exceeds 1000 MPa for the martensitic structure, and is 520–640 and 340 MPa for partial and 100% ferritic structures.

According to preliminary estimations the dimension of shells corresponding to a critical cooling rate for the 8Cr–1.5WV steel will be about 300 mm, and ~650 mm for the 9Cr–V; 9Cr–WV; 2.5Cr–1.4WV and 2.5Cr–0.7MoV steels.

#### 3.2. Influence of neutron irradiation

The data on the influence of neutron irradiation with fluence  $(1.2–2.0) \times 10^{24}$  n/m<sup>2</sup> within the temperature range 70°C and 240–300°C on the yield stress and the Charpy impact properties are given in Table 2 and Figs. 2 and 3. Analysis of the obtained data shows that the reduction of the irradiation temperature down to 70°C, 240–300°C did not cause an anomalous large shift in DBTT which is stay typical for martensitic-ferritic Cr–Mo–Nb steels after the irradiation at a temperature lower than 300°C ( $\Delta DBTT > 110^\circ\text{C}$ ) [4], although the fluence is rather small in our experiment,  $(1.2–2.0) \times 10^{24}$  n/m<sup>2</sup>. According to Rieth et al. [5]  $\Delta DBTT$  saturation takes place at a fluence of  $10^{24}$  n/m<sup>2</sup> at a lowered irradiation temperature ( $T_{irr} \approx 300^\circ\text{C}$ ).

As shown in Table 2, the shifts in DBTT of steels 8Cr–1.5WV, and 11Cr–1.5WV under irradiation are in the range of 15–35°C. As for steels 9Cr–V and 9Cr–WV the  $\Delta DBTT$  is consist of 40–60°C. The more considerable shifts in DBTT were observed for steels 7Cr–1.5WV

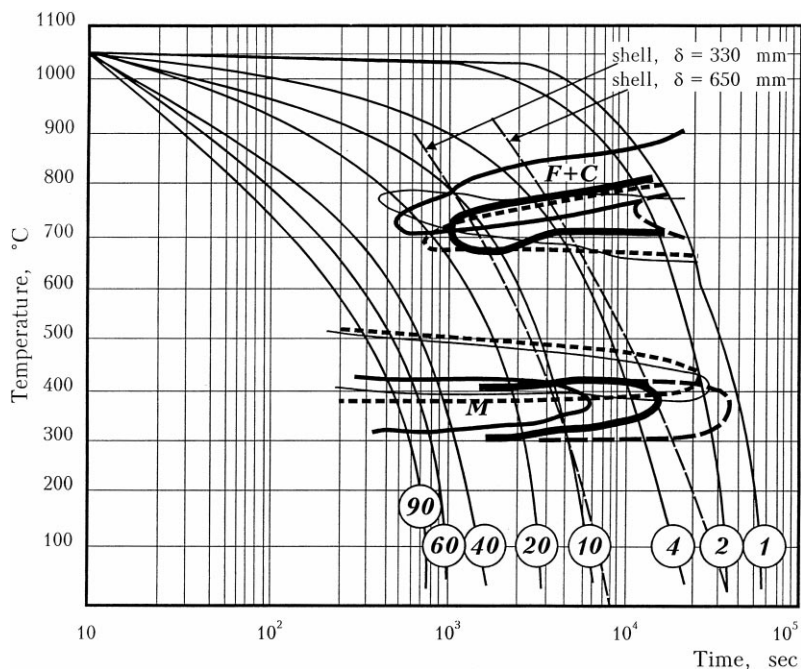


Fig. 1. Continuous-cooling transformation diagrams of FIRD steels: — 8Cr–1.5WV; — 9Cr–V; – – 9Cr–WV; - - - 2.5Cr–1.4WV; 2.5Cr–0.7MoV; (⊙) – cooling rate (°C/min).

(100°C), 7Cr–1.5WVCu (102°C) and 7Cr–1.5WVP (128°C). It should be noted that these steels, besides a smaller chromium concentration, also contain a high phosphorous and copper concentrations and have not of the rare earth additions in contrast to the 8Cr–1.5WV; 9Cr–V; 9Cr–WV; 9Cr–1.5WV and 11Cr–1MoV steels (Table 1). The beneficial role of the rare earth additions is consist in the refining of the ferritic steels [3].

These results indicate the importance of technological factors during melting to ensure high radiation resistance of FIRD steels.

Taking into account the fact that the materials used in this study were melted in the open induction furnace with constituent materials of usual quality, the obtained data should be considered as a conservative estimation of their mechanical properties both in the initial state and after irradiation. It should be noted that the results published earlier concerning irradiation embrittlement of the FIRD 9Cr–2WV steels [1–3] were obtained on electric slag remelt metal which was provided for good metallurgical qualities.

When the behavior of steels 11Cr–1MoV and 11Cr–1.5WV is compared (Fig. 3) it becomes evident that molybdenum replacement by tungsten in an approximately equiatomic relation exerts a favourable influence upon the radiation resistance of steel and causes a reduction of the  $\Delta$ DBTT. It should be noted that both steels after normalization and tempering are characterized by a martensitic-ferritic structure with about 20% ferrite content.

As shown in Table 2 data for the 2.5Cr–1.4WV steel are very promising because the yield stress at 20°C and DBTT are equal 680 MPa and –75°C consequently for the unirradiated condition.

In conclusion it should be noticed that the present investigations demonstrate all the advantages of the developed FIRD steels Cr–W–V in application to the low temperature neutron irradiation (less than 300°C) with fluence up to  $2 \times 10^{24}$  n/m<sup>2</sup>.

According to our estimation the obtained results can be related to large size products (up to 650 mm thickness). It means that these results are applicable to the first wall and blanket of fusion reactor as well as to pressure vessels and in-reactor equipment of nuclear power fission reactors.

#### 4. Conclusion

1. Chromium tungsten–vanadium FIRD ferritic steels reveal a high resistance to radiation embrittlement at irradiation temperatures 70°C, 240–300°C. The shifts in DBTT for the 8Cr–1.5WV; 9Cr–1.5WV, and 11Cr–1.5WV steels under irradiation to  $(1.3–2) \times 10^{24}$  n/m<sup>2</sup> ( $E \geq 0.5$  MeV) at a temperature 240–300°C are in the range of 15–35°C.
2. The more considerable shifts in DBTT (100–128°C) are observed for the 7Cr–1.5WV; 7Cr–1.5WVCu,

Table 2  
Effect of irradiation on hardening and embrittlement of the reduced-activation steels

Steel heat treatment	$F \times 10^{24}$ n/m <sup>2</sup> ( $E \geq 0.5$ MeV)	$T_{irr}$ (°C)	$\sigma_y^{20^\circ\text{C}}$ (MPa)		$\Delta\sigma_y$ (MPa)	DBTT(°C) <sup>a</sup>		$\Delta\text{DBTT}$ (°C)	USE(J)		$\Delta\text{USE}$ (%)
			Unirr	Irrad		Unirr	Irrad		Unirr	Irrad	
2.5Cr-1.4WV			680			-75			27		
1000°C/1h/ac+680°C/10h											
2.5Cr-0.7MoV			640			-65			26		
1000°C/1h/ac+680°C/10h											
7Cr-1.5WV	1.2	70	540	825	285	0	130	130	16	10	35
1050°C/1h/ac+700°C/3h	1.2	240	510	775	265	0	100	100	16	13	19
7Cr-1.5WVP	1.3	300	510	775	265	-18	110	128	19	12	37
1050°C/1h/ac+750°C/3h											
7Cr-1.5WVCu	1.4	240	495	750	255	-17	85	102	15	12	20
1050°C/1h/ac+750°C/3h											
8Cr-1.5WV	1.2	70	550	870	320	-20	90	110	18.5	15	19
1050°C/1h/ac+700°C/3h	1.3	300	550	790	240	-20	15	35	18.5	17	8
9Cr-1.5WV	2.0	280	700	850	150	20	45	25	13	11	15
1030°C/1h/ac+700°C/2h											
9Cr-V	1.2	70	520	770	220	-45					
1000°C/1h/ac+650°C/2h	1.4	260	520	710	190	-45	0	45	20	17	15
	1.2	70	465	740	275	-65	40	105	21.5	15	33
1000°C/1h/ac+700°C/2h	1.4	260	465	640	175	-65	-10	55	21.5	19	12
9Cr-WV	1.2	70	570	770	200	-40					
1000°C/1h/ac+650°C/2h	1.4	260	570	750	180	-40	0	40	17	15.5	9
	1.2	70	500	760	260	-70	10	80	22	14	36
1000°C/1h/ac+700°C/2h	1.4	260	500	710	210	-70	-10	60	22	15	32
11Cr-1.5WV	1.2	70	570	850	280	20					
1050°C/1h/ac+700°C/3h	1.4	240	570	820	250	20	35	15	15	13	13
11Cr-1MoV	1.2	70	720	990	270	0					
1050°C/1h/ac+700°C/0.5h	1.3	300	720	880	160	0	60	60	13	12	8

<sup>a</sup> Calculated from 1/2 USE values.

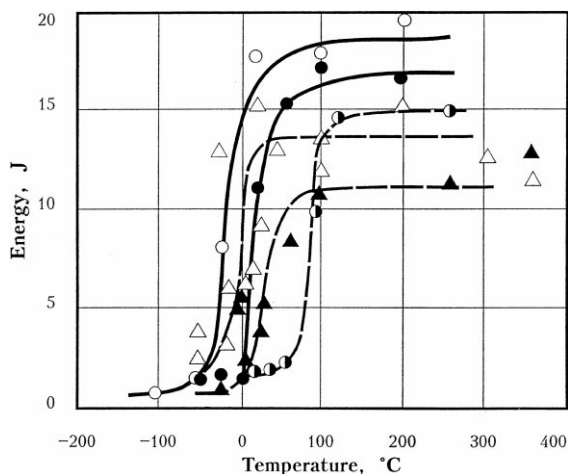


Fig. 2. Impact toughness of 8Cr-1.5WV (○,●,●) and 9Cr-1.5WV (△,▲) steels: (△,○) unirradiated; (▲,●,●) irradiated; (●)  $F = 1.3 \times 10^{24}$  n/m<sup>2</sup>;  $T_{irr.} = 300^\circ\text{C}$ ; (●)  $F = 1.2 \times 10^{24}$  n/m<sup>2</sup>;  $T_{irr.} = 70^\circ\text{C}$ ; (▲)  $F = 2.0 \times 10^{24}$  n/m<sup>2</sup>;  $T_{irr.} = 280^\circ\text{C}$ .

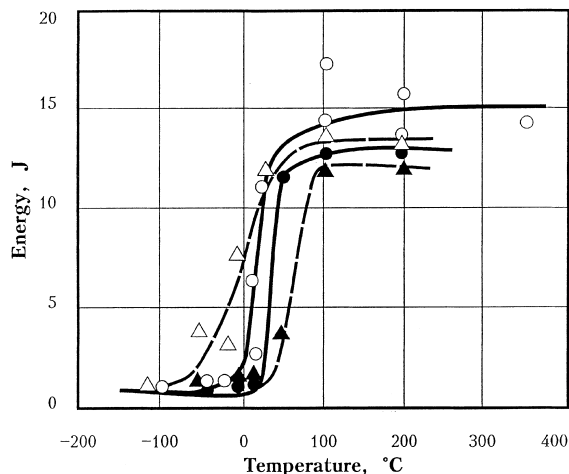


Fig. 3. Impact toughness of 11Cr-1.5WV (○,●) and 11Cr-1MoV (△,▲) steels with 20% ferrite: (△,○) unirradiated (▲,●) irradiated; (●)  $F = 1.4 \times 10^{24}$  n/m<sup>2</sup>;  $T_{irr.} = 240^\circ\text{C}$ ; (▲)  $F = 1.3 \times 10^{24}$  n/m<sup>2</sup>;  $T_{irr.} = 300^\circ\text{C}$ .

and 7Cr-1.5WVP steels. However, these steels contain more high P and Cu-impurities and have not of the rare earth additions.

3. Ferritic phase with volume content up to 100% can be formed in the steels of (2.5–9)%Cr-1WV type at cooling rates lower than 1°C/min.
4. The results obtained give the grounds to consider 2.5%Cr-WV; (8–9)%Cr-WV and of 11%Cr-WV steels as future advantageous FIRD materials from the standpoint of their further study.

## References

- [1] R.L. Klueh, J. Miner. Met. Mater. Soc. 4 (1992) 20.
- [2] Fujio Abe et al., J. Iron Steel Inst. Japan 2 (1986) 170.
- [3] V.V. Rybin et al., Embrittlement of fast induced radioactivity decay martensitic-ferritic steels irradiated with neutrons within the temperature range 240–300°C. In: ICFRM-7, Obninsk, September 1995 (Abstracts).
- [4] R.L. Klueh, D.J. Alexander. J. Nucl. Mater. 179–181 (1991) 733.
- [5] M. Rieth et al., Fusion Eng. Design 29 (1995) 365.