



# Mechanical behavior of reduced-activation and conventional martensitic steels after neutron irradiation in the range 250–450°C

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## Abstract

The objective of this work is to examine the susceptibility to hardening and embrittlement of Fe7.5/11CrW-TaV reduced-activation (RA) and conventional 9/12Cr–Mo martensitic steels as a function of fluence up to 10 dpa and irradiation temperature in the range of 250–450°C. For this purpose, materials were irradiated in the Osiris Reactor (Saclay) at 325°C for various doses ranging from 0.8 dpa to a maximum dose of 8–9 dpa. Available data concern the evolution of tensile properties for doses from 0.8 to 3.4 dpa. On the other hand, RA-steels were irradiated as Charpy V and tensile specimens in the high flux reactor (HFR) at Petten at temperatures ranging from 250°C to 450°C with a dose of about 2.4 dpa. © 2000 Elsevier Science B.V. All rights reserved.

## 1. Introduction

Reduced-activation (RA) martensitic steels of the Fe7.5/11CrW-TaV type are promising candidates for structural components of future fusion reactors [1–3]. The assessment of RA-materials needs the qualification of their irradiation behavior in the relevant temperature range for fusion reactors, in particular for temperatures lower than 400°C where ferritic-martensitic steels are sensitive to irradiation-induced hardening and embrittlement [1,4]. The main goal of this paper is to discuss the irradiation behavior of RA-martensitic steels and compare them to conventional FeCrMo steels. For this purpose, irradiation experiments were conducted for different irradiation tem-

peratures in the range 250–450°C and various dose levels.

## 2. Materials

RA-martensitic steels investigated in this work are: F82H (7.5Cr–2W-TaV), two heats of 9Cr–0.7W with different Ta contents, i.e., LA12LC (0.01%Ta) and LA12TaLC (0.10%Ta), as well as high-Cr LA4Ta (11Cr–0.7W) and high-W LA13Ta (9Cr–3W) alloys. Details of chemical compositions are given in [3]. F82H steel was supplied as plates in the normalized-tempered (N&T) condition [5]. Experimental heats (referenced as LA...) were produced as plates in the N&T-10% cold worked condition (N&T-CW) [3].

Conventional martensitic steels examined here are commercial alloys with different contents of Cr (9–12%), Mo and stabilizing elements (V, Nb), i.e., 9Cr–1Mo, 9Cr–1MoVNb, Manet II (10.4Cr–0.6MoVNb) and HT9 (12Cr–1Mo–0.5WNbV). In general, these materials were produced as plates in the N&T condition, except for 9Cr–1Mo, which was also obtained in the N&T-CW condition.

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### 3. Experimental

Materials were irradiated in Osiris Reactor at 325°C as plate tensile specimens 2 mm wide, 1 mm thick and 8 mm in gauge length. Tensile tests were performed at the irradiation temperature with a strain rate of  $3 \times 10^{-4} \text{ s}^{-1}$ .

Irradiation in HFR have been performed at five irradiation temperatures, i.e., 250°C, 300°C, 350°C, 400°C and 450°C. Two tensile and seven Charpy specimens of each RA-material were irradiated at each irradiation temperature. Tensile tests were conducted at the irradiation temperature on cylindrical specimens 3 mm in diameter and 18 mm in gauge length, using a strain rate of  $1.8 \times 10^{-4} \text{ s}^{-1}$ . Impact properties were determined with Charpy V subsize specimens of  $3 \times 4 \times 27 \text{ mm}^3$  with a notch depth of 1 mm obtained along the rolling direction of the plates (L-T orientation). The ductile–brittle transition temperature (DBTT) was determined at the level midway between the upper and the lower shelf energies.

### 4. Results

#### 4.1. Effects of the dose

Effects of the fluence level were studied from the irradiation experiment performed in the Osiris Reactor at 325°C. Partial unloading provided 5 doses ranging from 0.8 to 8–9 dpa [6]. Table 1 compares the tensile properties of RA and conventional steels before and after irradiation at 325°C to a dose of 3.4 dpa (data available up to now).

In all the cases, an increase of tensile strength with fluence is observed, which ranges from 150 to 450 MPa depending on the chemical composition and the metallurgical condition of the steels. Ductility values decrease simultaneously with increasing strength.

RA steels exhibit a lower amount of irradiation-induced hardening at 325°C for both N&T and N&T-CW metallurgical conditions as shown in Fig. 1(a) and (b). In particular, LA12LC (9Cr–0.7W), LA4Ta (11Cr–0.7W–Ta) and LA13Ta (9Cr–3W–Ta) obtained in the cold-worked condition display a lower increase of strength compared to the N&T F82H. A saturation of hardening is observed for N&T-CW RA-steels and 9Cr–1Mo beyond 2 dpa. In contrast, the conventional steels HT9 and Manet II steels display a continuous strength increase up to 3.4 dpa and the most important irradiation-induced hardening. They also show a greater decrease of reduction in area values, which goes down to 25–35% after 3.4 dpa.

At 3.4 dpa, N&T 9Cr–1Mo steel shows a high level of all ductility parameters, i.e., total/uniform elongations and reduction in area. F82H shows comparable values of tensile strength and area reduction to 9Cr–1Mo steel,

Table 1  
Tensile properties at 325°C of RA and conventional martensitic steels determined before and after irradiation (3.4 dpa) in Osiris Reactor

Metallurgical condition	0.2% yield stress (MPa)		UTS (MPa)		Total elongation (%)		Uniform elongation (%)		Reduction in area (%)	
	Unirr.	Irrad.	Unirr.	Irrad.	Unirr.	Irrad.	Unirr.	Irrad.	Unirr.	Irrad.
<i>RA-steels</i>										
F82H	456	734	513	734	15.0	8.3	2.9	0.2	73	59
LA12LC	541	700	559	701	13.5	7.6	0.7	0.1	71	61
LA4Ta	645	807	646	809	12.0	6.3	0.8	0.3	63	51
LA13Ta	690	819	691	832	9.9	6.4	1.1	0.7	60	47
<i>Conventional steels</i>										
9Cr–1Mo	462	741	584	783	17.0	12.1	7.7	4.2	64	58
9Cr–1Mo	612	938	623	955	12.5	7.9	1.1	1.1	66	52
9Cr–1MoVNb	667	941	688	943	11.0	6.4	0.7	0.3	59	45
Manet II	591	990	677	1005	13.5	5.5	2.7	0.7	65	36
HT9	485	932	691	1013	17.0	8.6	9.0	5.0	51	25

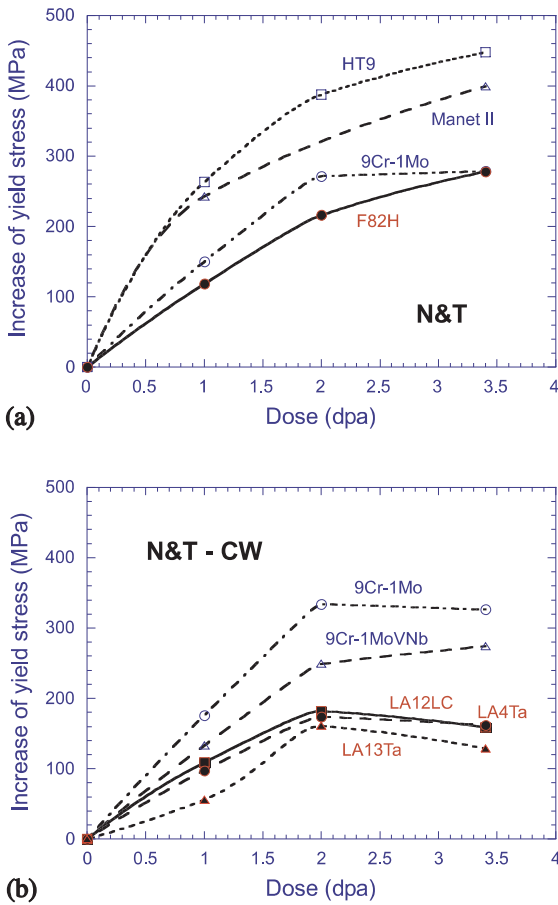


Fig. 1. Increase of yield stress with the dose for reduced-activation and conventional steels in (a) normalized-tempered (N&T) and (b) normalized-tempered-cold worked (N&T-CW) conditions. Irradiation experiment performed in Osiris reactor.  $T_{test} = T_{irr} = 325^{\circ}\text{C}$ .

but exhibits a loss of strain hardening capacity as shown in Table 1 and previous work [7].

Correlation of the irradiation hardening with the chemical composition is not easy to establish. As shown in Fig. 1, for N&T-steels the increasing sensitivity to hardening could be related to Cr-content (F82H (7.5Cr), 9Cr-1Mo, Manet II (10.4Cr), HT9 (12Cr)). But, in the case of N&T-CW steels, 9Cr-1Mo hardens much faster than LA12LC (9Cr-0.7W) and the same strength increase is detected for LA4Ta (11Cr-0.7W) and LA13Ta (9Cr-3W). Consequently, hardening seems to be determined not only by the chemical composition but also by the initial metallurgical condition.

On the other hand, it is important to note that materials, which show low values of reduction in area (<50%) like HT9, Manet II and 9Cr-1MoVNb, should exhibit poor impact properties. Such behavior can be anticipated according to previous CEA work, where a

linear relation was established between the upper shelf energy (USE) of transition curves and the reduction in area determined from tensile tests for several FeCrW RA-steels [8] and FeCr-Mo [6] conventional ferritic-martensitic steels including different metallurgical conditions (N&T, N&T-CW, unaged and thermal aged).

#### 4.2. Effects of the irradiation temperature

The irradiation experiments performed in HFR in the range 250–450°C show the influence of the irradiation temperature ( $T_{irr}$ ). Fig. 2 shows the evolution of the yield stress with  $T_{irr}$  for LA12TaLC and LA12LC RA-steels and 9Cr-1Mo (N&T) conventional steel. The dose level reached 2.4 dpa for RA-steels and 0.8 dpa for 9Cr-1Mo alloy.

In the case of RA-steels, both materials show the same behavior. The irradiation-induced hardening reaches the higher values at 250°C and 300°C. In the range 350–450°C, the tensile strength of irradiated specimens is very close to the level obtained on thermal controls. Note that the same behavior was observed for UTS values and that no important effects of  $T_{irr}$  are detected on the total elongation and reduction in area.

As shown in Fig. 2, the conventional steel 9Cr-1Mo exhibits nearly the same qualitative behavior as a function of  $T_{irr}$ . The main difference is related to the fact that the irradiation-induced hardening is produced over a wide range from 250°C to 400–450°C.

The hardening induced by irradiation, measured by the increase in yield stress, is reported in Fig. 3 for materials investigated here and compared with data obtained from the literature for F82H [9] and 9Cr-1Mo irradiated in the Phenix Reactor at high doses [10] for

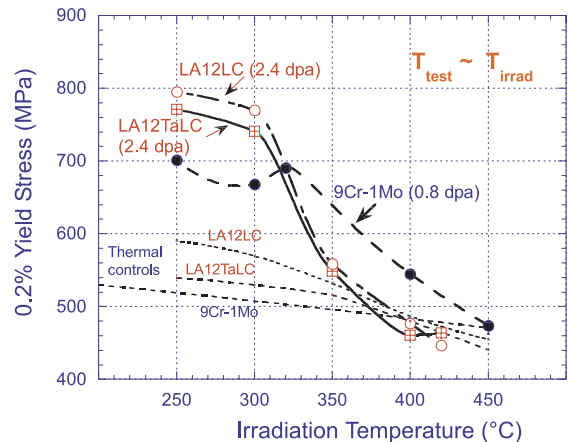


Fig. 2. Evolution of yield stress with the irradiation temperature for LA12LC and LA12TaLC reduced-activation steels irradiated at 2.4 dpa and 9Cr-1Mo conventional steel irradiated at 0.8 dpa. Irradiation experiment performed in HFR reactor.

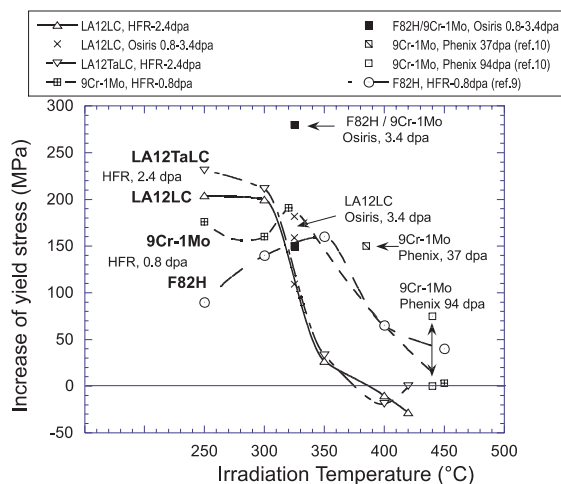


Fig. 3. Increase of the yield stress as a function of the irradiation temperature for LA12LC, LA12TaLC, F82H [9] reduced-activation and 9Cr-1Mo conventional steels. Comparison of data obtained from HFR, Osiris and Phenix [10] irradiation experiments.  $T_{\text{test}} = T_{\text{irr}}$ .

the same metallurgical condition (N&T). F82H and 9Cr-1Mo irradiated at the same conditions and dose (HFR-0.8 dpa), show the same dependence with  $T_{\text{irr}}$  and equivalent hardening as observed in the Osiris experiment. Their maximum hardening seems to be extended up to 320–350°C. Consequently, the dependence of increasing strength with dose determined in the Osiris experiment should approximately correspond to the evolution of the maximum hardening of both materials. So, a significant increase of strength is expected for higher doses as shown in Fig. 3.

In the case of LA12LC and LA12TaLC, the maximum hardening occurs at 250–300°C. The low increase of strength for LA12LC detected at 325°C in Osiris corresponds in fact to the temperature where hardening decreases significantly with  $T_{\text{irr}}$ . These results suggest that materials in the cold-worked condition harden in a lower temperature range than materials in the N&T condition. To explain these results, it could be argued that the higher dislocation density of cold-worked steels should act as effective sinks for defect annihilation resulting in a recovery of irradiation damage at lower  $T_{\text{irr}}$  compared to N&T materials.

Impact properties as a function of the irradiation temperature were determined only for LA12LC and LA12TaLC RA-steels for a dose of 2.4 dpa. The evolution of their DBTT is presented in Fig. 4. In contrast to tensile properties, a different evolution of the DBTT-shift ( $\Delta\text{DBTT}$ ) with the irradiation temperature is found for both materials. USE level exhibits nearly the same decrease (<20%) for all  $T_{\text{irr}}$  and both materials.

In the case of LA12TaLC alloy, the highest increase of DBTT occurs at 250°C, where the maximum hard-

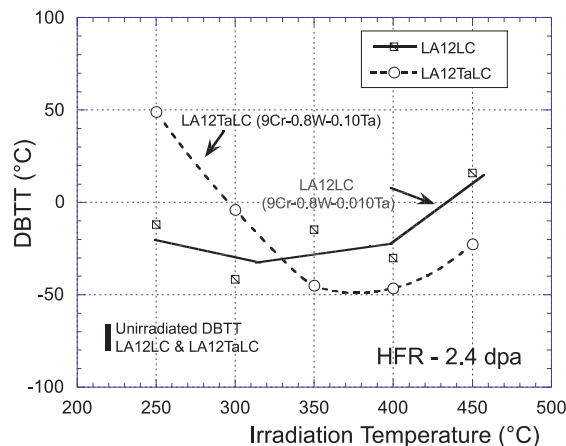


Fig. 4. Evolution of the DBTT with the irradiation temperature corresponding to LA12LC and LA12TaLC reduced-activation steels irradiated in HFR reactor.

ening is observed. The lower DBTT values are found at 350–400°C and DBTT increases again at 450°C as observed for other RA-steels irradiated in the same conditions [11,12]. For LA12LC steel, the most important shift is detected at 450°C as shown in Fig. 4 and no correlation was found between hardening and  $\Delta\text{DBTT}$ .

The main difference between LA12LC and LA12TaLC steels is the Ta-content, which also induces a difference in the prior austenite grain size. At 450°C, the lower embrittlement is obtained for the material with the higher Ta-content. This seems to increase the resistance to embrittlement at  $T_{\text{irr}} > 400^\circ\text{C}$ , as proposed in [12]. However, this assumption cannot be applied for  $T_{\text{irr}} = 250^\circ\text{C}$ , where the minimum DBTT-shift is observed for the material having the lower Ta-content. A great deal of work is still necessary to explain compositional and microstructural effects on embrittlement mechanisms occurring in martensitic steels.

## 5. Conclusions

Several FeCrW reduced-activation and FeCrMo conventional martensitic steels have been irradiated at different doses from 0.8 to 3.4 dpa and various irradiation temperatures in the range 250–450°C. The main results enable us to draw the following conclusions:

- For dose levels and irradiation temperatures used in the present work, 9Cr and in particular the reduced-activation variants present a better resistance to irradiation-hardening compared with conventional high Cr steels.
- In the range 250–450°C, the hardening and embrittlement is strongly determined by the irradiation temperature and seems to vary with the chemical composition and the metallurgical condition of steels.

- The irradiation-induced hardening is shown to decrease rapidly with increasing irradiation temperature and disappears at  $T_{\text{irr.}} = 350^{\circ}\text{C}$  for cold-worked RA-steels and at  $T_{\text{irr.}} = 450^{\circ}\text{C}$  for N&T alloys.
- Different behavior of impact properties were found for 9Cr–0.7W–0.1C(Ta) RA-steels after irradiation at 2.4 dpa. The lower DBTT shift is observed at  $250^{\circ}\text{C}$  for the steel with the lower Ta-content, while at  $450^{\circ}\text{C}$  the higher Ta-content steels show higher resistance to embrittlement.

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