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# Mechanical properties of 9Cr martensitic steels and ODS-FeCr alloys after neutron irradiation at 325 °C up to 42 dpa

A. Alamo [a,*], J.L. Bertin [a], V.K. Shamardin [b], P. Wident [a]

[a] Commissariat à l'Energie Atomique, CEA-Saclay, Service de Recherches Métallurgiques Appliquées (SRMA), 91191Gif-sur-Yvette, France
[b] SSC-RF Research Institute of Atomic Reactors (RIAR), 433510 Dimitrovgrad, Russia

**Abstract**

Reduced activation ferritic/martensitic steels (F82H, JLF-1, 9Cr2WTaV and EUROFER-9Cr1WTaV), advanced ODS Fe–14%Cr alloys, standard and modified 9Cr1Mo conventional martensitic steels were irradiated as specimens for mechanical tests in different neutron irradiation experiments performed in the range 300–325 °C. The objective of this paper is to present the tensile and impact properties as well as the irradiation creep of these materials after irradiation to high doses (32–42 dpa) in BOR60. The evolution of tensile properties is discussed as a function of the dose up to 42 dpa including results from irradiations carried out in SM2 and OSIRIS reactors. In general, RAFM steels presented a lower level of hardening and embrittlement compared to 9Cr1Mo conventional steels. ODS-Fe–14%Cr exhibited the lowest hardening and a relatively high residual ductility at 42 dpa.


## 1. Introduction

Reduced activation martensitic/ferritic (RAFM) steels with 9 wt%Cr and ODS-FeCr alloys are considered the main candidates for internal structures of future fusion reactors. A large program of neutron irradiations was carried out to study the susceptibility of materials to irradiation-induced hardening and embrittlement at low temperatures ($T < 400$ °C) where these phenomena could reach a high detrimental level [1,2].

The aim of this paper is to summarize recent data on tensile, impact properties and irradiation-creep obtained after irradiation in BOR60 fast-reactor (RIAR) for doses ranging from 32 to 42 dpa. The 'EUROFER' European reference and experimental 9Cr2WTaV RAFM steels, 9Cr–1Mo and 9Cr–1MoVNb conventional steels and the advanced ODS-Fe–14%Cr alloy were irradiated in this experiment.

The evolution of tensile properties at irradiation-temperatures of 300–325 °C is analyzed for doses up to 42 dpa including data obtained from experiments performed in OSIRIS (CEA-Saclay) and SM2 (RIAR) mixed-spectrum reactors, which covered dose ranges of 1–9 dpa and 8–14 dpa, respectively.

* Corresponding author. Tel.: +33 1 6908 2517; fax: +33 1 6908 5795.
E-mail address: ana.alamo@cea.fr (A. Alamo).

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## 2. Experimental

The irradiation in the BOR60 reactor was performed at 325 ± 5 °C for doses ranging from 32.5 to 42.3 dpa. The following materials were irradiated as specimens for mechanical tests: RAFM steels EUROFER (9Cr–1W–0.18V–0.15Ta) and 9Cr2WTaV (9Cr–2W–0.24V–0.08Ta), the advanced alloy ODS-MA957 (Fe–14Cr–1Ti–0.3Mo–0.25$Y_2O_3$), and conventional martensitic steels, 9Cr–1Mo and 9Cr–1MoVNb. Tensile tests were performed at 20 and 325 °C with a strain-rate of $1.4 \times 10^{-3}\ s^{-1}$ on specimens of 2 mm diameter and 12 mm gauge length. Impact properties were determined with Charpy-V subsize specimens of $3 \times 4 \times 27\ mm^3$ with T–L orientation. Pressurized tubes of 6.55 mm in diameter, 0.45 mm thick and 55 mm total length were included to estimate the irradiation-creep. Argon pressure was adjusted to induce a hoop-stress of 150 and 220 MPa at the irradiation-temperature. The measurement of the external diameter was performed by profilometry to accurately determine the strain in the central region of tubes.

F82H (7.5Cr–2W–0.15V–0.02Ta) and JLF-1 (9Cr–2W–0.18V–0.08Ta), both RAFM steels manufactured in Japan, and 9Cr1Mo steel were irradiated as tensile specimens in the SM2 reactor at 300 ± 5 °C with doses ranging from 8 to 14 dpa. Specimens (as described above) were tested at 20 °C and at the irradiation temperature with a strain rate of $3 \times 10^{-4}\ s^{-1}$.

Details of the irradiation performed in OSIRIS reactor at 325 °C/1–9 dpa are given in [3,4]. F82H RAFM-steel, 9Cr1Mo-(EM10), 9Cr1MoVNb and ODS-Fe14%Cr were irradiated as tensile specimens.

The martensitic steels were provided in the normalized-and-tempered condition; ODS-Fe14%Cr fully-ferritic alloy was investigated in the fine-grain non-recrystallised-condition [5].

## 3. Evolution of tensile properties with the dose

The irradiation experiments performed in OSIRIS, SM2 and BOR60 reactors permit evaluating the effects of dose on the evolution of tensile properties up to 42 dpa.

Table 1 shows the tensile strength values of different materials determined at 300–325 °C before and after irradiation for the maximum dose-level achieved in different experiments.

As expected, all materials exhibit an increase of the tensile strength. The dose dependence of irradiation-induced hardening, measured by the increase of the yield-stress, is shown in Fig. 1. A very rapid

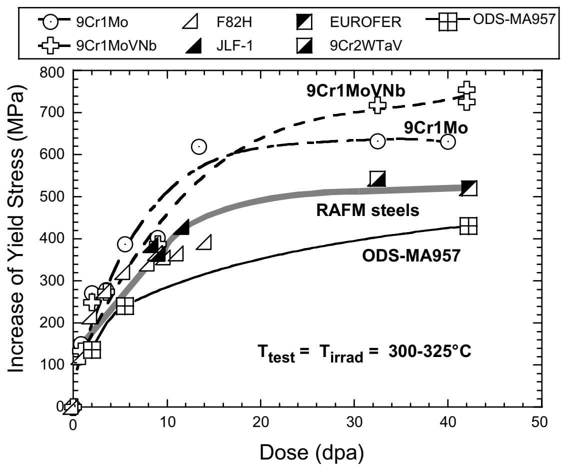


Fig. 1. Evolution of the irradiation-induced hardening at 300–325 °C, measured by the increase of the yield stress, as a function of the dose. Data obtained from experiments performed in OSIRIS (1–9 dpa), SM2 (8–14 dpa) and BOR60 (32–42 dpa) reactors.

Table 1
Tensile strength of unirradiated and irradiated materials measured at 300–325 °C

| Material | Unirradiated | | Irradiated | | Dose (dpa) | Irradiation Experiment |
|---|---|---|---|---|---|---|
| | 0.2% YS (MPa) | UTS (MPa) | 0.2% YS (MPa) | UTS (MPa) | | |
| F82H | 445 | 510 | 865 | 891 | 9 | Osiris [3,4] |
| | | | 838 | 839 | 14.2 | SM2 |
| JLF-1 | 427 | 511 | 856 | 857 | 11.8 | SM2 |
| EUROFER | 456 | 533 | 976 | 993 | 42.3 | BOR60 |
| 9Cr2WTaV | 450 | 522 | 992 | 999 | 32.5 | BOR60 |
| 9Cr1Mo | 447 | 562 | 1066 | 1072 | 13.4 | SM2 |
| | | | 1077 | 1084 | 40.0 | BOR60 |
| 9Cr1MoVNb | 453 | 563 | 1208 | 1219 | 42.2 | BOR60 |
| ODS-MA957 | 895 | 976 | 1326 | 1372 | 42.2 | BOR60 |

increase of hardening is observed for all materials up to about 10 dpa with a continuous decrease of the hardening-rate; beyond this value there could be a tendency to saturation in the case of 9Cr1Mo steel.

Data obtained at high dose (32.5–42.3 dpa) for EUROFER and 9Cr2WTaV seem to follow the same trend of F82H and JLF-1 at low dose (<15 dpa). Therefore, the behavior of all RAFM steels is represented by a unique curve.

As shown in Fig. 1, the magnitude of hardening depends on the material. The highest hardening-level at 325 °C and 40–42 dpa was reached for 9Cr1MoVNb (750 MPa), 630 MPa for 9Cr1Mo, whereas a lower hardening (520–540 MPa) was found for RAFM-steels, EUROFER and 9Cr2WTaV, in the investigated dose range. These 9%Cr martensitic materials presented quite similar tensile properties before irradiation (see Table 1), but they evolved in a different manner during irradiation.

Hardening measured at room temperature displays the same features and the magnitude is about 10–20% higher than that determined at 325 °C. The yield stress and ultimate tensile strength (UTS) reached nearly the same values after irradiation indicating a reduction of the strain-hardening capability of materials.

The increase of tensile strength is associated with a ductility drop given by the decrease of total and uniform elongation as well as the reduction-in-area values. As shown in Fig. 2, the total elongation of RAFM steels for low doses decreases much faster than that of the 9Cr1Mo standard. Above 15 dpa the ductility loss of RAFM steels seems to be stabilized. The total elongation of 9Cr1Mo continues to decrease to reach very poor values (<2%), especially for tests performed at 20 °C, even a nearly saturated hardening is detected for 9Cr1Mo by 10 dpa. Regarding the uniform elongation, very low levels in the range 0.3–0.5% were obtained at both test temperatures for RAFM and 9Cr1MoVNb steels since 2 dpa, but 9Cr1Mo preserves higher values of uniform elongation up to 10 dpa.

It is worthwhile to notice the interesting behavior of the ODS-Fe14%Cr ferritic alloy. This material exhibited the lowest hardening (430 MPa) and the highest values of total and uniform elongation at 42 dpa confirming the trend previously observed at lower doses in OSIRIS experiments [5].

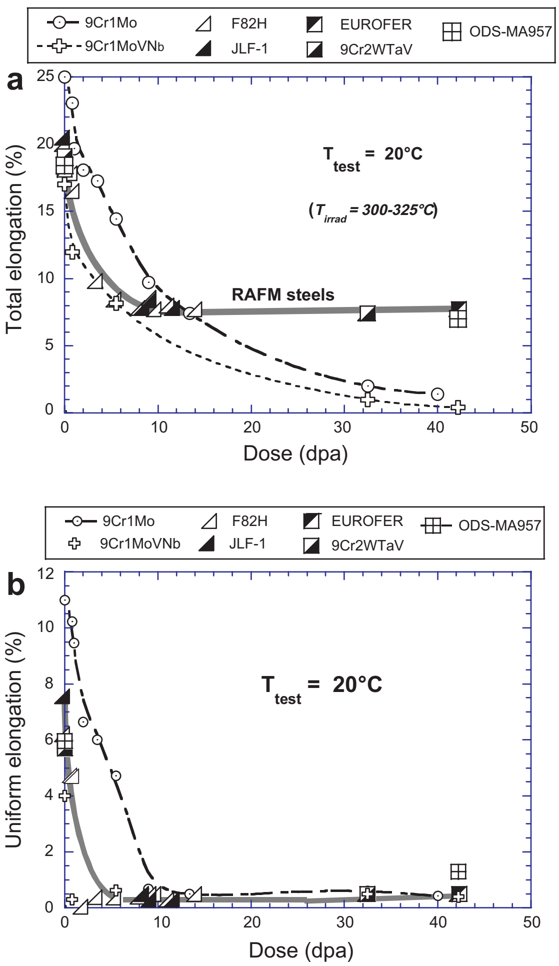


Fig. 2. Evolution of the total (a) and uniform (b) elongation as a function of the dose from tests performed at room temperature.

## 4. Impact properties of 9Cr martensitic steels

The irradiation-effects on impact properties were investigated on EUROFER, 9Cr2WTaV and 9Cr1Mo irradiated in BOR60. Energy transition curves, determined from Charpy tests, are shown in Fig. 3 for irradiated and unirradiated conditions. Before irradiation, the ductile–brittle transition temperature (DBTT) was about −100 °C/−80 °C for these materials.

After irradiation at 325 °C, an important degradation of impact properties, i.e., an increase of the DBTT associated with a significant decrease of the Upper Shelf Energy (USE) values, is observed for all materials. The DBTT-shift is about 150 °C for 9Cr2WTaV (32.5 dpa), 200 °C for EUROFER (42 dpa), about 250 °C for 9Cr1Mo (40 dpa). The last material also exhibits a very huge degradation

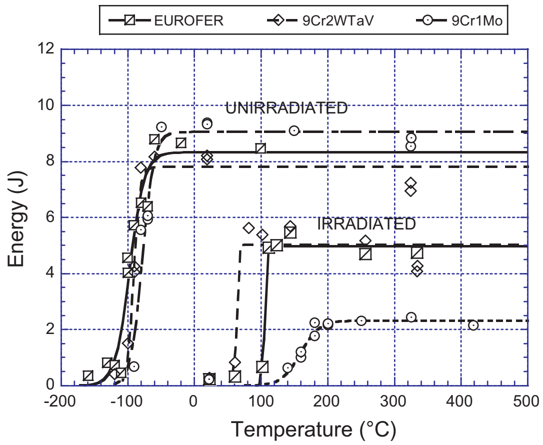


Fig. 3. Comparison of impact properties of EUROFER, 9Cr2WTaV and 9Cr1Mo steels in the unirradiated condition and after irradiation in BOR60 reactor at 325 °C for 32–42 dpa.

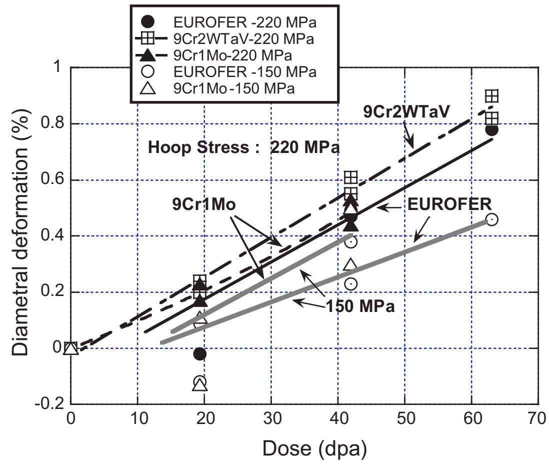


Fig. 4. Diametral strain of EUROFER, 9Cr2WTaV and 9Cr1Mo steels as a function of the dose for hoop-stresses of 150 and 220 MPa.

of the USE. Similar behavior was found for 9Cr1MoVNb steel.

RAFM steels present a lower degree of irradiation-induced embrittlement compared to 9Cr1Mo-type conventional steels in agreement with the behavior of tensile properties.

## 5. Irradiation creep

Pressurized tubes of 9Cr1Mo-(EM10), EUROFER and 9Cr2WTaV steels were irradiated to determine the deformation due to irradiation-creep as a function of the dose and the applied stress.

The measurements were performed before irradiation and for doses of 19.3, 41.9 and 63 dpa; the last one obtained for tubes re-irradiated also in BOR60 at the same temperature. The pressurized specimens were weighted at each step to check the maintaining of argon-content.

The average diametral-strains determined for each material are shown in Fig. 4. The strains are very low ($\leqslant$0.9%) after 63 dpa, confirming that 9Cr martensitic steels display rather good dimensional stability at 325 °C as already determined at higher irradiation temperatures (400–550 °C) and equivalent doses on several conventional ferritic–martensitic steels [6].

The irradiation-creep-modulus was estimated using the relation: $\varepsilon_\theta = 0.75$ A $\phi t \sigma_\theta$, where $\varepsilon_\theta$ is the diametral strain, $\phi t$ the dose, $\sigma_\theta$ the average hoop stress and A the irradiation-creep-modulus. This was estimated assuming that only irradiation-creep is contributing to the diametral deformation because swelling is expected to be very low [6] and thermal creep is negligible at 325 °C.

9Cr1Mo, 9Cr2WTaV and EUROFER present nearly the same average value of the irradiation-creep-modulus, which is $A = (0.7 \pm 0.1) \cdot 10^{-6}$ (dpa MPa)$^{-1}$.

## 6. Discussion

Comparative results regarding the irradiation-behavior at 300–325 °C of 9Cr-RAFM martensitic steels and ODS-Fe14%Cr were obtained up to high doses and for low irradiation temperatures ($T <$ 400 °C) where existing data are quite limited.

As expected, all materials exhibited a degradation of mechanical behavior in the irradiated condition. However, the magnitude of hardening and embrittlement was found to be different for 9Cr-materials, which exhibited similar mechanical properties before irradiation. As shown in Fig. 1, 9Cr1Mo and 9Cr1MoVNb steels seem to harden faster compared to RAFM tungsten-containing alloys like EUROFER-9Cr1WTaV and 9Cr2WTaV. The irradiation in SM2-reactor showed that the hardening-level of 9Cr1Mo steel at 13.4 dpa is nearly the same than that obtained at 40 dpa. This fact should indicate some trend to saturation of hardening. Nevertheless, no tendency to saturation of ductility-loss is observed for both 9Cr1Mo steels; their elongation values decrease continuously with the dose and reach nearly zero when measured at 20 °C after 40–42 dpa. In contrast, RAFM steels maintain reasonable ductility up to this dose.

Very attractive results were obtained concerning the irradiation performances of the ODS-Fe14%Cr irradiated at 325 °C up to 42 dpa. Due to the high Cr-content, a significant fraction of embrittling-precipitation could be expected [5]. However, this material exhibited the lowest hardening and the highest uniform elongation after irradiation, compared to RAFM and conventional martensitic steels.

The evolution of impact properties confirms the results of tensile tests. The most important DBTT shift and a pronounced decrease of USE values were obtained for 9Cr1Mo steels. However, RAFM steels exhibited after irradiation a DBTT value of about +100 °C that means an increase of around 200 °C.

On the other hand, the present results illustrate the huge effect of irradiation-temperature on the irradiation behavior of materials. Fig. 5 shows the values of the 0.2% yield-stress of 9Cr1Mo-(EM10) conventional steel, measured at the irradiation temperature, from experiments performed in this work and PHENIX-reactor [7]. As shown, the hardening-magnitude of 9Cr1Mo strongly increases with the decreasing temperature below 400 °C, in contrast to the high stability of mechanical properties observed in the range 400–550 °C, where the DBTT of 9Cr1Mo and 9Cr1MoVNb steels were lower than room temperature after irradiation at high doses (70–100 dpa) [7].

Consequently, these results put forward the dominant effect of the irradiation-temperature on materials behavior and show the important degree of induced-hardening and embrittlement that could be expected for temperatures lower than 400 °C,

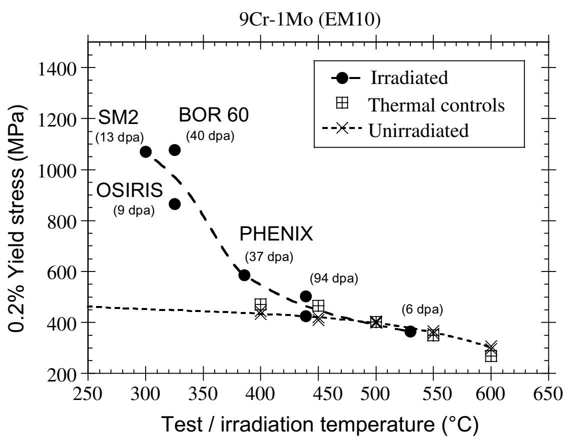


Fig. 5. Effects of the irradiation temperature on the irradiation-induced hardening determined for 9Cr1Mo-(EM10) martensitic steel.

as previously observed at low doses [3,8,9]. This fact is an important concern for applications of 9Cr martensitic steels in this low range of temperatures.

RAFM-9CrWTaV steels present improved performances and showed delayed irradiation-effects compared to 9Cr1Mo materials irradiated in the same conditions. Further investigation is needed to explain the reasons for this enhanced behavior and elucidate the contributions of chemistry, micro/nano-structure and irradiation-induced defects. One reason is certainly related to the more restricted chemical composition-specification and a better control of impurities during manufacturing the 9CrWTaV experimental martensitic steels.

It is also important to notice the high dimensional-stability of 9Cr-RAFM and 9Cr1Mo steels after irradiation. Very low strains were measured at high doses (63 dpa) for 150 and 220 MPa hoop-stress. All materials presented the same irradiation-creep-modulus and this value is in good agreement with those already determined at higher temperatures (400–550 °C) for 9Cr1Mo and other ferritic–martensitic steels [6].

## 7. Conclusions

The irradiation behavior of EUROFER-9Cr1WTaV, other RAFM martensitic steels, 9Cr1Mo conventional steels and ODS-Fe–14%Cr was studied after neutron irradiations performed at 300–325 °C in various reactors and in particular in BOR60 fast-reactor for high doses (32–42 dpa). The main conclusions are:

- Significant irradiation-induced hardening was obtained for all materials after irradiation at 300–325 °C up to 42 dpa, the highest level was reached for 9Cr1Mo-type steels.
- At high dose, EUROFER and 9Cr2WTaV RAFM-steels preserved a significant total elongation level. But, a continuous ductility loss with the dose was observed for 9Cr1Mo steels.
- Irradiation at 32–42 dpa induced a very important DBTT-shift for all materials, 150–200 °C for RAFM steels, 250 °C for 9Cr1Mo alloys, associated with a decrease of USE-level.
- In general, a better behavior was found for RAFM-9CrWTaV martensitic steels, which displayed lower level of hardening and embrittlement compared to conventional steels.
- EUROFER, 9Cr2WTaV and 9Cr1Mo martensitic steels presented a very low irradiation-creep

deformation (less than 1%) after irradiation for 63 dpa at 325 °C.
- For temperatures lower than 400 °C, the hardening and embrittlement of 9Cr-steels are strongly determined by the irradiation-temperature. However, their magnitude seems to depend also on the chemistry and the metallurgical condition of steels.
- In spite of its high-Cr content, ODS-Fe14%Cr presented the lowest hardening-level and high residual ductility after irradiation at 42 dpa.

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