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Irradiation resistance of Eurofer97 at 300 °C up to 10 dpa

J. Rensman [a,*], E. Lucon [b], J. Boskeljon [a], J. van Hoepen [a],
R. den Boef [a], P. ten Pierick [a]

[a] *NRG, P.O. Box 25, 1755 ZG Petten, The Netherlands*
[b] *SCK • CEN, Boeretang 200, 2400 Mol, Belgium*

Abstract

Eurofer97 has been irradiated in material test reactors in Mol, Belgium and Petten, The Netherlands at a temperature of 300 °C from 0.2 to 10 dpa. Tensile tests show a continuous logarithmic hardening trend for this dose range of 280 MPa per decade dpa. Impact tests on miniature Charpy-type specimens prove the superiority of Eurofer97 over F82H-mod. when DBTT shift is considered. DBTT shift in degree centigrade is about one fourth the hardening in MPa for the former. The upper shelf energy drops 0.3 J per dpa. Eurofer97 25 mm plate shows worse impact properties than the other product forms of Eurofer97.

1. Introduction

Eurofer97 is the primary candidate steel in the fifth framework programme of the European Fusion Development Agreement for breeding blanket mock-ups in ITER. This particular class of materials is used in a tempered martensitic state, where it should combine good post irradiation mechanical properties with other specific benefits, like abundant H/He-trapping sites and reduced activation properties. In succession, the Manet, F82H-mod., and now Eurofer97 steels have shown a continuous improvement of irradiation resistance at the critical hardening irradiation temperatures of 300 °C and below [1,2].

2. Experimental

The base alloy composition of Eurofer97 [2], 9Cr1W0.2VTa, has been modelled after commercial steels with a tempered martensitic microstructure. This microstructure allows operation at relatively high temperatures up to 500 °C and offers good dimensional stability under high neutron dose level conditions. Developments in the ferritic/martensitic steels for nuclear applications tend toward a fine microstructure with small prior austenite grains and a low number of inclusions to achieve an optimum combination of toughness, strength and high temperature properties [3]. The material has been characterised by various European institutes in unirradiated condition [4–6].

An extensive irradiation campaign has been performed by the Dutch nuclear research centre NRG in the High Flux Reactor (HFR) in Petten. Materials irradiated were Eurofer97 25, 14, and 8 mm plate materials in the as-received condition, a lab-scale Eurofer97-clone, and a 9Cr2W0.2VTa heat. Target temperature has been 300 °C and the target dose level for the irradiations has been 2.5 dpa, with one irradiation up to 10 dpa. The irradiation sample holders, with acronyms (for identification only) SUMO and SOSIA, were filled with Na and actual measured specimen irradiation temperature has been within ±15 °C maximum of the target.

In the BR2 reactor at the Belgian nuclear research centre SCK • CEN in Mol, three irradiation experiments were performed with acronyms (for identification only)

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[*] Corresponding author. Tel.: +31-224 56 4857; fax: +31-224 56 8883.
E-mail address: rensman@nrg-nl.com (J. Rensman).

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IRFUMA-I, II, and III. The irradiation temperature was 300 ± 5 °C nominally, with achieved dose averages for the tensile specimens of 0.34, 0.59, and 1.31 dpa, respectively. The material in these experiments was Eurofer97 100 mm bar in the as-received condition [7,8].

All tensile tests have been performed in accordance with ASTM E8M with the note that no extensometer is used. The tensile specimens have a diameter *D* of 4 mm for NRG and 3 mm for SCK • CEN, both with a gauge length of 5D.

NRG has done impact tests on DIN-standard mini Charpy-type (so-called *kl*ein*st* KLST) specimens with a 50 J instrumented impact tester with an ISO striker at an impact speed of 3.85 m s^{-1}. Temperature correction is applied for the travel time from conditioning chamber to time of impact.

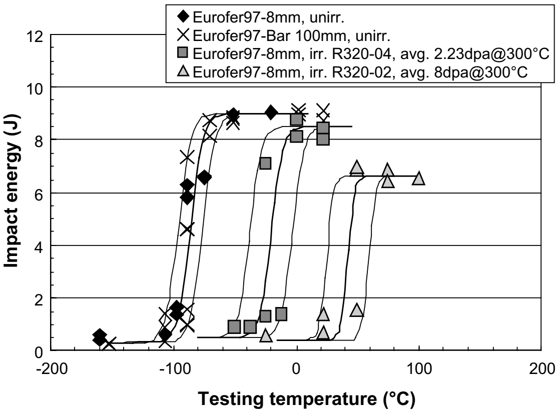

Fig. 2. KLST impact transition curves for Eurofer97 8 mm plate after 300 °C irradiation.

3. Results

In Fig. 1, a typical example is given of the tensile response of Eurofer97 after three different NRG irradiations. The results from the 60 °C irradiation have been reported before [9]. It is evident that at 300 °C irradiation no saturation has occurred at 2.4 dpa. Irradiation at 60 °C does not give the same hardening for equivalent dose. In addition, the hardening present at room temperature decreases rapidly at temperatures of 100 °C and over by annealing when compared to the 300 °C irradiations. The yield stress hardening for the latter irradiations to 2.4 and 8.4 dpa irradiation decreases only slightly when going from 27 to 300 °C testing temperature.

In Fig. 2, an example is shown of the shift in both ductile-to-brittle-transition-temperature (DBTT) and upper shelf energy (USE) with dose for 8 mm Eurofer97 plate. It has been noted before [4] that the impact behaviour in unirradiated condition of Eurofer97 100 mm bar, and 8 and 14 mm plate is identical with a DBTT of around −90 °C. The deviation of 25 mm plate has of yet not been explained.

When the DBTT is plotted vs. dose, as has been done in Fig. 3, one can see that the deviant behaviour of the 25 mm plate is continued after irradiation. The error bars indicate the range in dose for one impact series and the 'width' of the transition curve, respectively. The Eurofer97 8 and 14 mm plate and both NRG lab heats have the lowest KLST-impact transition temperatures: below 60 °C at doses up to 9 dpa.

Some of the F82H results at 2.5 dpa in Fig. 3 have been obtained in the fourth framework programme in the CHARIOT irradiation experiment series [10]. They are considered representative of 250–275 °C irradiation temperature, slightly less severe than the 300 °C condition. This is reflected in the USE. In CHARIOT-4,

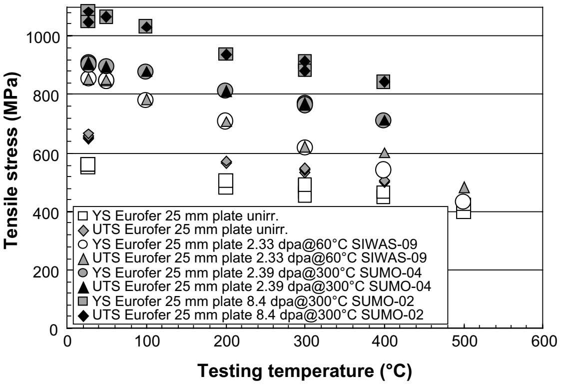

Fig. 1. Yield stress and ultimate strength of 300 °C irradiated Eurofer97.

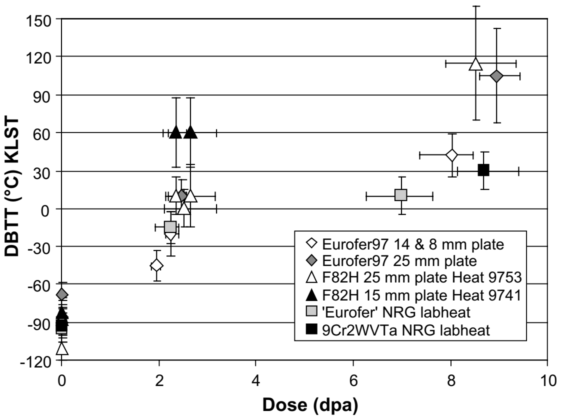

Fig. 3. DBTT of various RAFM heats vs. dose level acquired at 300 °C.

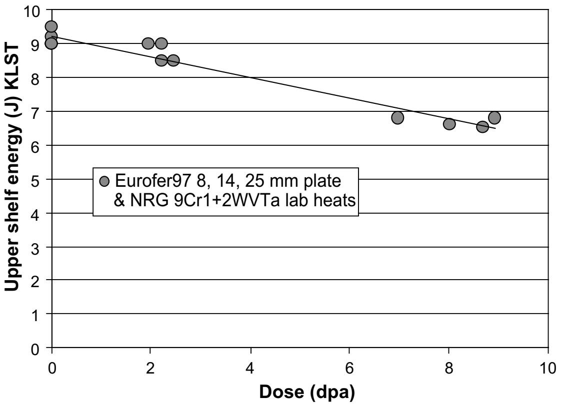

Fig. 4. Variation of the USE vs. the dose level acquired at 300 °C.

F82H-mod. 25 mm plate retained its 10 J USE from the unirradiated state after 2.50 dpa, whereas in SUMO-04, it dropped to 9 J after 2.34 dpa, with the DBTT's not significantly different. The USE of Eurofer97 decreases with dose acquired at 300 °C, as shown in Fig. 4. The slope is approximately 0.3 J per dpa for the dose range studied.

4. Discussion

Although the absolute yield stress of the various heats in this study varied more than 40 MPa, the yield stress increase ΔYS, or hardening, was found to be very consistent with dose. The ΔYS has been analysed using the data supplied by SCK • CEN with doses below 2 dpa and the data of NRG with doses ranging from 2 to 10 dpa. A procedure was applied to compare ΔYS for all available tests up to the irradiation temperature of 300 °C to generate more statistics for the evaluation of the evolution of ΔYS with irradiation dose.

The ΔYS at temperature has been analysed and for three very narrow dose ranges: 0.80 ± 0.09, 2.35 ± 0.12, and 9.07 ± 0.15 dpa. For these dose ranges it was found that the yield stress hardening decreases slightly with increasing testing temperature with the same percentage, suggesting a simple rule for all 300 °C doses up to 10 dpa. This allows a simple recalculation of the ΔYS at temperature to the room temperature reference for any temperature from 27 °C up to the irradiation temperature of 300 °C. When this calculated ΔYS is plotted vs. dose, the result is a graph like Fig. 5.

Several observations can be made. The hardening for Eurofer97 irradiated at 300 °C between 0.2 and 10 dpa is continuous and increases with 280 MPa per decade (power of 10) of dose level. No sign of genuine saturation is observed, contrary to the observations made in [11]. The hardening can be described with a logarithmic function

$$\Delta \text{YS (MPa) at } 27\ °\text{C} = 122 \times \text{Ln(dose)} + 230, \qquad (1)$$

for $0.20 \leqslant \text{dose} \leqslant 10$ (dpa).

With present confidence in the temperature control in NRG's Na-filled experiments, the first author judges the He-filled 5 dpa irradiation in [11] to have been irradiated at a temperature of 10–25 °C above the 300 °C target. The hardening is the same for all tested heats of Eurofer97 including the NRG lab heats and seems to describe the behaviour of F82H-mod. as well.

The question then remains if the shift in DBTT correlates with hardening in the same manner for all materials. The answer to that can be derived from Fig. 6, and must be a negative.

The F82H-mod. heats show considerably more shift in DBTT than the Eurofer97 heats. The former have

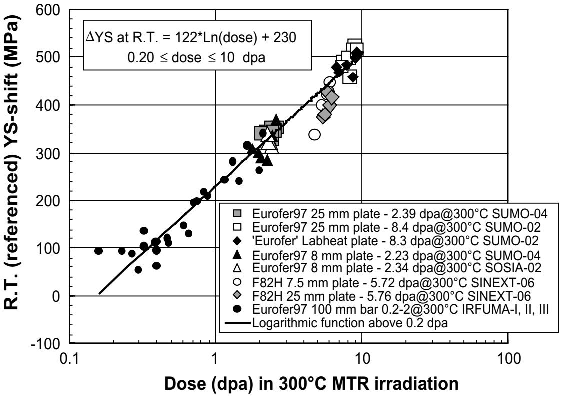

Fig. 5. Variation of the room temperature (referenced) YS-hardening vs. dose level at 300 °C.

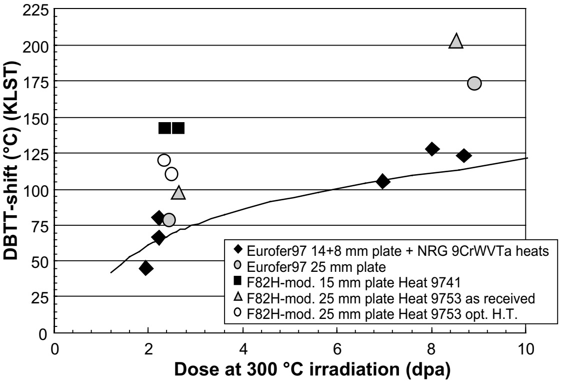

Fig. 6. Shift of the DBTT for Eurofer97 vs. dose compared with that of F82H-mod.

about twice the ΔDBTT at 2.5 dpa, even if a special heat treatment proposed by FzK is applied of normalisation: 0.5 h/920 °C + temper: 2 h/730 °C [3] that gives optimum impact properties in the unirradiated state.

Eurofer97 25 mm plate challenges this conclusion to a degree. This heat has the same microstructure as the other Eurofer97 heats, although NRG has not performed a TEM-scale analysis. Although the ΔDBTT may be similar at 2.5 dpa for all Eurofer97 heats, the higher DBTT of Eurofer97 25 mm plate in unirradiated state is a sign that the carbide/precipitate structure is different. It is shown in Fig. 6 that the fracture properties' response to irradiation can thus be very different. It demonstrates that sub-microstructural features, not detected by gross yielding tests like tensile or hardness tests, influence the fracture behaviour of normalised and tempered 9Cr-steels significantly. A number of investigations into this subject have been performed [12–16] focusing mostly on ageing. No quantitative relationship between (the morphology of) the precipitates and the DBTT (shift) has yet emerged.

The development of the microstructure with irradiation must also be different for Eurofer97 25 mm plate compared to the thinner plates, as the impact properties deviate even more after irradiation. SANS experiments are being planned to gain more insight.

5. Conclusions

The yield stress of 300 °C neutron irradiated Eurofer97 increases continuously with a logarithmic trend for dose levels between 0.2 and 10 dpa of 280 MPa per decade. F82H-mod. seems to follow the same yield stress hardening in tensile tests. However, the KLST ΔDBTT in degree centigrade of Eurofer97 8 and 14 mm plate and the NRG lab heats is approximately one fourth of their hardening in MPa, whereas for F82H this is about one half. The KLST USE of Eurofer97 decreases approximately linearly with dose at a rate of 0.3 J per dpa.

Compared to 300 °C irradiation, 60 °C irradiation produces lower hardening for equivalent dose, as does 325 °C irradiation. Irradiation at 250 °C gives less reduction in USE.

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