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Mechanical properties of the European reference RAFM steel (EUROFER97) before and after irradiation at 300 °C

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Abstract

EUROFER97 is the European candidate RAFM steel for use as a structural material in fusion energy systems. It is presently under investigation by several European laboratories, within the long term programme of EFDA (European Fusion Development Agreement). This paper presents the outcome of the mechanical characterization of this steel that has been carried out at SCK • CEN (Belgian Center for Nuclear Studies), in the unirradiated condition and after irradiation during three different campaigns in the BR2 reactor (300 °C, doses between 0.3 and 1.6 dpa). Tensile, Charpy impact, and fracture toughness specimens have been irradiated and tested, in order to obtain an experimental assessment of the main effects of neutron exposure on tensile and toughness properties; namely, irradiation hardening (increase of mechanical resistance and loss of ductility) and irradiation embrittlement (shift of ductile-to-brittle transition temperature and degradation of upper shelf energy). Comparisons with another well-known IEA reference RAFM steel, F82H, are provided.

1. Introduction

High Chromium ferritic/martensitic steels, with Cr content between 9% and 12%, were developed more than 50 years ago and have been used for a long time in the power-generation industry as boiler and turbine materials, as well as for other applications. In the early 1970's, they were considered for fast breeder fission reactors and later for fusion applications, mainly on account of their better resistance to swelling as compared to austenitic stainless steels [1].

In the last 15 years of the past century and up to present, fusion material programs in Europe, Japan and US have been focusing on the development of Reduced Activation Ferritic/Martensitic (RAFM) steels, which would reduce the environmental impact of the irradiated steel after the service lifetime of a fusion reactor. This was achieved by selectively replacing chemical elements which, in a fusion neutron spectrum, would transmute into high-energy radiation emitters with long half-life.

In the European union, within the long term programme of EFDA, remarkable efforts are being spent by several institutes for the characterization and optimization of a reference RAFM steel, which was given the name of EUROFER97 [2]. This was modelled after the conventional T91 alloy and exhibits a tempered martensitic microstructure which allows operation at relatively high temperatures (up to 500–550 °C); presently, attempts at extending the temperature operational range are being investigated through the application of alternative manufacturing routes, such as ODS (oxide dispersion strengthening) [3].

Since 2000, SCK • CEN (Belgian Center for Nuclear Studies) has been investigating the mechanical properties of EUROFER97 (tensile, impact, fracture toughness) after neutron exposure. Three irradiation campaigns have been carried out in the BR2 experimental reactor in Mol (IRFUMA-I, II and III), up to nominal accumulated doses of respectively 0.25, 1 and 2.25 dpa; in all cases, irradiation temperature was 300 °C. For every condition, tensile, instrumented Charpy

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and fracture toughness tests have been performed and evaluated. In parallel, a thorough mechanical characterization of the unirradiated condition was carried out in order to assess the changes in the material's properties caused by neutron irradiation. In addition, comparisons will be proposed with other RAFM steels, and particularly F82H (the first alloy approved by IEA for a collaborative test program in the early 90's, [4,5]).

2. Experimental procedure

EUROFER97 is a RAFM steel with nominal composition 9Cr-1.1W-0.2V-0.07Ta-0.1C (Fe balance, all wt%). The steel, used in the normalised and tempered condition, was produced by Böhler in Germany and manufactured in four different product forms: plates with thickness 8, 14 and 25 mm and bars with diameter 100 mm. Normalisation was performed at 980 °C and tempering, followed by air cooling, was done at 760 °C for 1.5 h for the plates and at 740 °C for 3.7 h for the bars. SCK • CEN received from FZK Karlsruhe in 1999 a section of one of the bars.

Although mechanical data in the unirradiated condition for the plates were already available from other institutions participating in the EFDA long-term programme, a complete mechanical characterization of the bar in the baseline condition has been performed at SCK • CEN (tensile, Charpy impact and fracture toughness data).

Tensile, Charpy impact and precracked Charpy samples were irradiated in the BR2 Test Reactor in Mol, during three consecutive irradiation campaigns, all of them at 300 °C:

- IRFUMA-I (2000): 1 reactor cycle, target dose = 0.25 dpa;
- IRFUMA-II (2001): 4 reactor cycles, target dose = 1 dpa;
- IRFUMA-III (2001-2002): 9 reactor cycles, target dose = 2.25 dpa.

In all cases, extensive dosimetry allowed calculating the dose associated to each individual specimen. In the first experiment (IRFUMA-I), the EUROFER97 samples were located around the mid-plane section of the reactor and the relevant fluence was rather uniformly distributed among the different specimens (average dose = 0.32 ± 0.060 dpa). However, in the two subsequent irradiations (IRFUMA-II and III), samples from other experiments occupied the central portion of the rig and the EUROFER97 samples were located in the lateral sections of the rig, which are characterized by a steep fluence gradient (cosine shape); as a consequence, the actual average dose was lower than the target value and the scatter of the individual samples was much too large to consider the data sets as homogeneous from the point of view of the irradiation: 0.55 ± 0.278 (51%) dpa for IRFUMA-II and 1.28 ± 0.530 (41%) dpa for IRFUMA-III.

This circumstance was accounted for in the analysis of the mechanical test results as follows:

- In the case of tensile tests, where each individual test delivers a complete set of information about the tensile properties, each sample was treated individually and the analysis was performed by grouping specimens only on the basis of the test temperature;
- In the case of Charpy impact and fracture toughness tests, where the required information (Ductile-to-Brittle Transition Temperature, Upper Shelf Energy, Reference Temperature) is only obtained from the analysis of a complete data set, samples have been grouped according to their effective dose rather than their corresponding IRFUMA campaign; three subgroups have been obtained, as will be detailed in the relevant test results sections.

Individual specimen doses reported in this study are average measured values, which are typically associated to an uncertainty of ±10%.

3. Results and discussion

3.1. Tensile tests

Tensile tests have been performed on unirradiated and irradiated sub-size cylindrical specimens, with nominal gage length $L_o = 15$ mm and diameter $D_o = 3$ mm, at the following temperatures: −150, −75 °C, room temperature, 150, 225 °C and irradiation temperature (300 °C). Tests were conducted at a strain rate of approximately 2×10^{-4} s^{-1}.

As a consequence of irradiation hardening, increase of yield (R_y) and tensile (R_m) strengths as well as loss of ductility (elongation and reduction of area) have been observed. Measured values of yield strength and total elongation are shown in Figs. 1 and 2, respectively.

If the yield strength increase at a given temperature ΔR_y is normalised to the corresponding unirradiated R_y value, a rather consistent trend for all test temperatures can be observed (Fig. 3); these data can be fitted as a function of accumulated dose, obtaining the empirical relationship:

$$\frac{\Delta R_y}{R_y} = 0.6012 \cdot \mathrm{dpa} - 0.1624 \cdot (\mathrm{dpa})^2 \quad (1)$$

for $0 \leqslant \mathrm{dpa} \leqslant 2$ and with coefficient of determination $R^2 = 0.91$. Eq. (1) can be used to reasonably estimate the

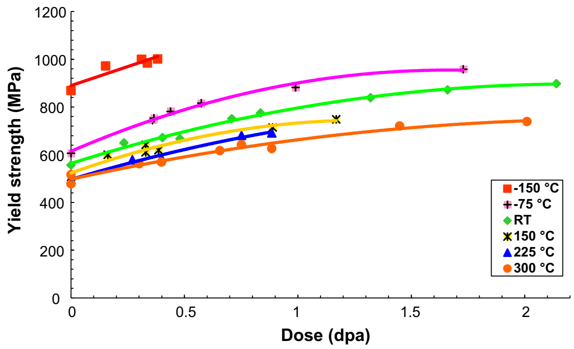

Fig. 1. Yield strength values measured from tensile tests as a function of dose.

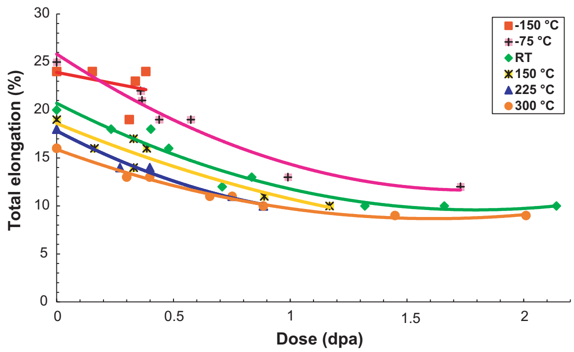

Fig. 2. Total elongation values measured from tensile tests as a function of dose.

yield strength corresponding to a given dose if the unirradiated value is known at the same temperature.

With respect to published data on F82H [4,6], EUROFER97 shows similar tensile properties both in the as-received condition and after irradiation at 300 °C in the range 0.1–10 dpa.

3.2. Charpy impact tests

Charpy tests have been performed in the unirradiated and irradiated condition using a 300 J instrumented pendulum equipped with a 2-mm striker, at temperatures ranging from −130 °C to irradiation temperature (300 °C). Full transition curves for absorbed energy (KV), lateral expansion (LE) and shear fracture appearance (SFA) have been obtained. In order to increase the number of available data points and therefore improve the reliability of the results, several new Charpy specimens have been obtained by reconstituting previously broken samples [7].

The following specimen sub-groups have been considered in the analyses:

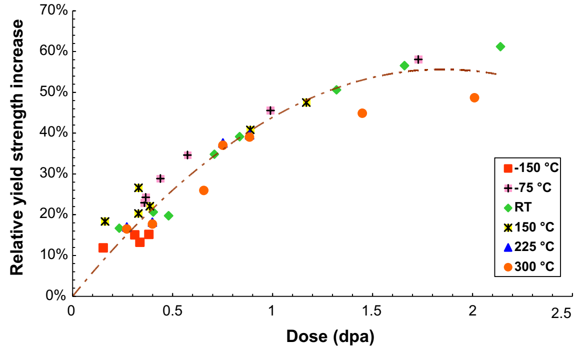

Fig. 3. Relative yield strength increase measured from tensile tests as a function of dose.

- Sub-group 1 (specimens from IRFUMA-I and II): 0.33 ± 0.045 (14%) dpa;
- Sub-group 2 (specimens from IRFUMA-II and III): 0.70 ± 0.150 (21%) dpa;
- Sub-group 3 (specimens from IRFUMA-III): 1.57 ± 0.328 (21%) dpa.

As a consequence of neutron irradiation, embrittlement of the material is observed in terms of a shift of the DBTT (Ductile-to-Brittle Transition Temperature) towards higher temperatures. Data points and fitting curves for absorbed energy are illustrated in Fig. 4, which also shows that USE (Upper Shelf Energy) is only affected for the highest dose, and to a minor extent (7.5% decrease with the respect to the baseline).

Published data for F82H and several other RAFM steels [4], all irradiated at 300 °C, show that EUROFER97 yields the lowest DBTT shifts for doses below 1 dpa, whereas at 1.6 dpa the shift seems similar to that of F82H (Fig. 5). A substantial invariancy of the USE up to 2.5 dpa has also been reported for F82H [6,8].

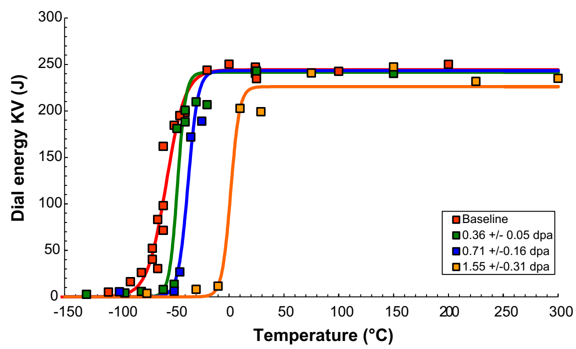

Fig. 4. Transition curves of absorbed energy measured from Charpy impact tests.

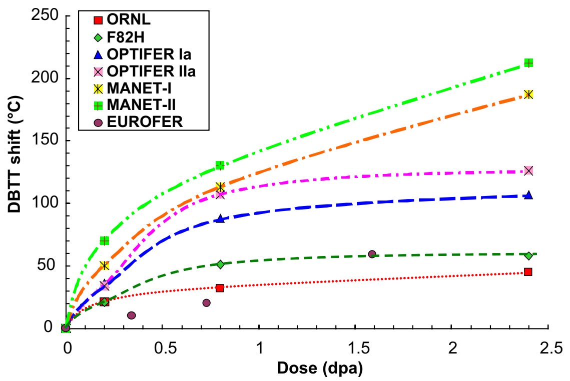

Fig. 5. Comparison between different RAFM steels irradiated at 300 °C [4] and EUROFER97 in terms of Charpy-based *DBTT* shift.

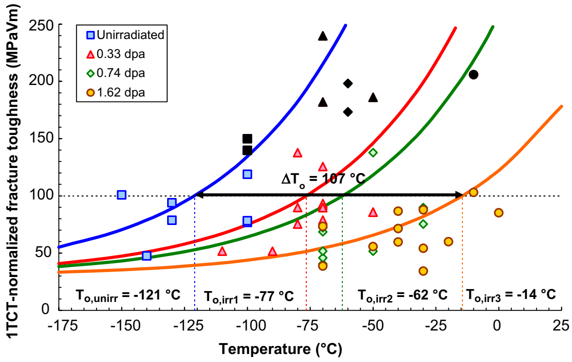

Fig. 6. Fracture toughness results and corresponding Master Curves measured in the unirradiated and irradiated conditions (closed points correspond to invalid data according to ASTM E1921-02).

3.3. Fracture toughness tests

Toughness tests have been performed in the unirradiated and irradiated condition on precracked Charpy (PCCv) specimens, in order to obtain the reference temperature T_o in accordance with the ASTM E1921-02 standard (the so-called 'Master Curve' methodology). Tests have been carried out at several temperatures within the transition region, using plain-sided (non-sidegrooved) specimens.

As in the case of Charpy tests, additional specimens were reconstituted from broken ones and eventually tested to improve the accuracy of the calculated T_o values; the following sub-groups have been considered in the analyses:

- Sub-group 1 (specimens from IRFUMA-I and II): 0.33 ± 0.034 (10%) dpa;
- Sub-group 2 (specimens from IRFUMA-II and III): 0.74 ± 0.191 (26%) dpa;
- Sub-group 3 (specimens from IRFUMA-III): 1.62 ± 0.333 (21%) dpa.

Experimental data points and corresponding Master Curves for the unirradiated and irradiated conditions are shown in Fig. 6, where the progressive embrittlement of the material with increasing dose can be appreciated.

T_o shifts measured on EUROFER97 are consistent with data published for F8H2 irradiated under comparable conditions (same temperature but higher doses) [5,8]; however, if we compare such shifts with those of the Charpy-based *DBTT* for both steels, we observe that the former ones are consistently and significantly larger (Fig. 7). This entails that, as previously stated by other authors [8,9], relying solely on Charpy tests for assessing the irradiation embrittlement of EUROFER97 or F82H might lead to overestimation of material performance.

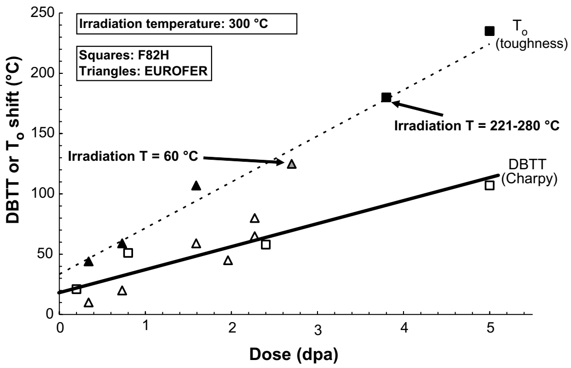

Fig. 7. Irradiation-induced *DBTT* and T_o shifts for F82H and EUROFER97, showing that the latter ones are consistently larger. A point corresponding to an irradiation performed at 60 °C [9] is also included.

4. Conclusions

A comprehensive mechanical characterisation of the properties of the European candidate RAFM steel (EUROFER97) has been performed, both in the as-received condition and following irradiation at 300 °C up to accumulated doses in the range 0.25–2.15 dpa.

Neutron exposure produces both hardening and embrittlement. In the range between ambient and irradiation temperature, we measured yield strength increases of 50–60% for doses corresponding to approximately 2 dpa, with a loss of ductility of 7–10% in terms of total elongation; the trend is consistent with published data on F82H.

As far as the degradation of toughness is concerned, both Charpy and fracture toughness tests show an increase of ductile-to-brittle transition temperatures with accumulated dose; however, the shifts evaluated from fracture toughness tests in terms of T_o (fracture toughness tests) are consistently and significantly larger than those of the *DBTT* measured from Charpy impact tests (as observed by other authors for F82H). Charpy results show no degradation of USE with respect to the unirradiated condition up to 0.7 dpa and only a moderate decrease (7.5%) at 1.6 dpa.

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