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MECHANICAL PROPERTIES OF TWO IMPROVED EUROFER ODS STEELS BEFORE AND AFTER LOW DOSE NEUTRON IRRADIATION AT 300 °C

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Within the European Fusion Development Agreement (EFDA) Long Term Programme activities on Material Research, several versions of EUROFER ODS (Oxide-Dispersion Strengthened) have been produced and characterized. The most promising ones to date are the so-called "2nd generation" ODS (HIPped, hot rolled and thermomechanically treated) and the "EU batch" (produced by Plansee in the form of hot rolled plates and extruded bars). These two materials have been mechanically characterized in the unirradiated condition at SCK•CEN in collaboration with other European institutes by means of tensile, impact and fracture toughness tests. The same characterization has been performed at SCK•CEN on the two materials after low dose irradiation at 300 °C in the BR2 test reactor (1.5-1.7 dpa). The results are compared with available data from early versions of EUROFER ODS and conventional (i.e. non-ODS) EUROFER, unirradiated and irradiated under similar conditions. It is confirmed that even the most advanced ODS steels show higher tensile strength than the base material, but also significantly worse fracture toughness properties. On the other hand, the "EU batch" irradiated to 1.52 dpa shows comparatively limited irradiation sensitivity.

I. INTRODUCTION

Within the European Union, the two main breeding blanket concepts are the helium-cooled pebble bed (HCPB) and the helium-cooled lithium lead (HCLL) blankets. In both cases, operating temperatures range from 250 to 550 °C for the conservative approach based on reduced activation ferritic/martensitic (RAFM) steels and from 250 to 650 °C for the more advanced approach which considers oxide-dispersion strengthened (ODS) steels.

The European reference RAFM steel is denominated EUROFER; its nominal composition is 8.9Cr, 1.1W, 0.2V, 0.14Ta, 0.42Mn, 0.06 Si, 0.11 C and Fe for the balance.[1] This steel was chosen for the production of two variants of ODS steel with different Y_2O_3 contents (0.3% and 0.5%), in order to achieve a substantial increase of about 100 °C for the operating temperature. Activation calculations showed that addition of yttria would not impair long-term activation properties.[2-4]

Early versions of EUROFER ODS were manufactured by inert gas atomisation of EUROFER by STARCK, followed by mechanical alloying in industrial ball mills of attritor type by PLANSEE. Hot Isostatic Pressing (HIP) was used as consolidation process for the production of four bars of each heat, 60 mm in diameter and 300 mm in length.[5]

Part of this material, which is commonly referred to as "1st generation" ODS or ODS-1, was made available to several European labs for testing.[6] Characterization tests showed, in comparison with conventional EUROFER, a considerable increase (~35%) in tensile properties and a higher creep strength.[6,7] On the other hand, impact properties were particularly disappointing with respect to conventional EUROFER (DBTT values over 150 °C higher, USE reduced by almost 50%).[8,9]

In order to overcome the problems related to ductility and impact toughness, two groups at CEA and CRPP tried to optimize the fabrication route of ODS-EUROFER (with 0.3% yttria). CEA produced ODS material in a more conventional manner by mechanical alloying and hipping, with special emphasis on the influence of the initial powders and the hipping parameters. CRPP produced mechanically alloyed powder in an attritor mill, which was consolidated in two steps by hot compaction and a subsequent hipping process without canning.

However, in both cases the performance at low temperature was not satisfactory; additional research was required in order to optimize the ODS composition and/or the fabrication parameters. The main issues remained the low fracture toughness properties and the relatively high DBTT (Ref. 10).

Subsequent developments concentrated on more advanced blanket concepts, such as HCLL, consisting of a EUROFER structure with SiCf/SiC flow channel inserts in the self-cooled Pb-17Li breeding zone, which serve as thermal and electrical insulators, and a first wall which is plated with a 2-3 mm thick ODS-layer to withstand the high thermal and mechanical loads.[11] In this framework, a sheet of ODS-EUROFER steel was produced in cooperation between FZK and PLANSEE; the production route included compaction of the mechanically alloyed steel powder by HIP, using 0.3% Y_2O_3, followed by hot rolling using a cross-rolling technique, which was expected to provide homogeneous in-plane properties. Different thermo-mechanical treatments were applied to study their effect on mechanical properties.

Mechanical characterization of this material (which was designated "2nd generation" ODS, or ODS-2), carried out by FZK, showed:[1,12]

- tensile and creep strength comparable to ODS-1;
- significantly increased total and uniform elongation at higher temperatures, generally equal or even higher than conventional EUROFER in the range RT-700 °C;
- remarkable improvements in the impact behaviour, with DBTT values in the range -80 °C to -40 °C and upper shelf energy increased by about 40% with respect to ODS-1 (Fig. 1).

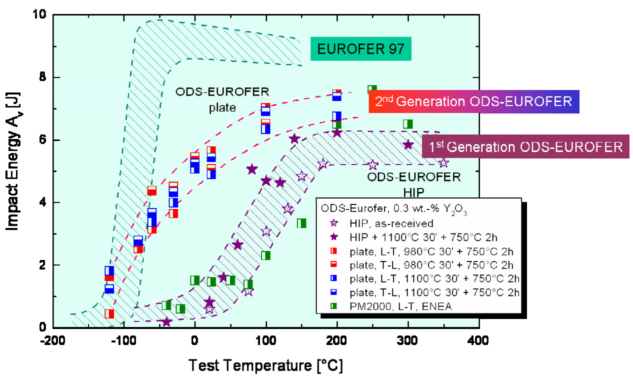

Fig. 1. Impact test results obtained from conventional EUROFER, ODS-1 and ODS-2 (Ref. 1); results obtained by ENEA on the commercially available ODS alloy PM2000 are also included.

ODS-2 material was irradiated in the BR2 reactor at SCK•CEN in Mol at 300 °C up to a nominal dose of 2 dpa in the framework of the IRFUMA-IV experiment (the acronym IRFUMA stands for *IRradiation of FUsion MAterials*). The irradiation campaign and the results of post-irradiation mechanical testing have been described in detail in a previously published paper.[13]

A new batch of EUROFER ODS, presently designated as "EU batch" or ODS-EU, was produced by PLANSEE in the form of hot-rolled plates and extruded rods. The material was delivered to FZK in March 2005. Qualification impact tests performed at FZK on KLST subsize Charpy specimens showed that the impact toughness of ODS-EU is somewhat worse than ODS-2 but still significantly better than the "first generation" ODS.[1]

The characterization of the mechanical properties of ODS-EU (tensile, impact, fracture toughness and creep) in the unirradiated condition was performed in several European laboratories in the framework of the European Fusion Development Agreement (EFDA) Long-Term programme on High Performance Steels.

At SCK•CEN, ODS-EU was characterized in both the unirradiated condition and after irradiation at 300 °C in BR2 up to 1.52 dpa (IRFUMA-5M experiment).[14]

This paper deals with the pre- and post-irradiation mechanical properties of ODS-EU and the comparison with ODS-1, ODS-2 and conventional EUROFER.

II. UNIRRADIATED CONDITION

II.A. Tensile Properties

Twenty-two sub-size cylindrical tensile specimens (2.4 mm diameter of the reduced section, total length 27 mm) were tested between -150 and 650°C. The results obtained (yield strength in Fig. 2 and uniform elongation in Fig. 3) show that tensile strength and ductility of ODS-EU are comparable to previous ODS versions and significantly higher than conventional EUROFER up to 650°C. Note also in Fig. 3 that ODS-EU exhibits much higher uniform elongation that ODS-2.

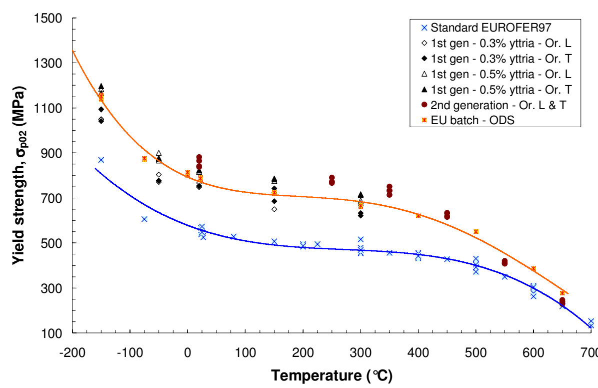

Fig. 2. Yield strength of various EUROFER ODS materials and comparison with conventional EUROFER.

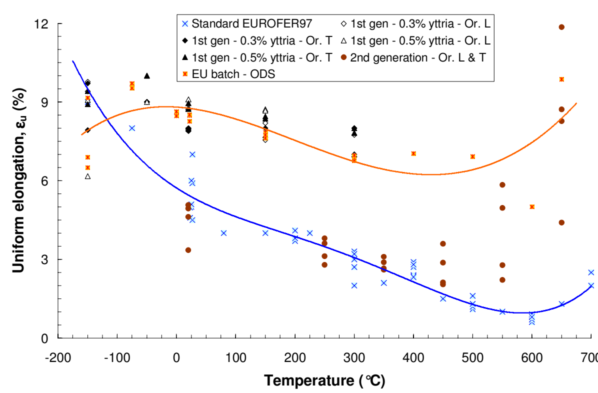

Fig. 3. Uniform elongation of various EUROFER ODS materials and comparison with conventional EUROFER.

II.B. Fracture Toughness Properties

Fifteen sub-size (KLST) fatigue precracked Charpy specimens were tested in the ductile-to-brittle transition region (-130 to 0°C) in order to determine the Master Curve reference temperature T_0 according to ASTM E 1921-05. Results obtained allowed defining the trend of fracture toughness as a function of test temperature (Fig. 4) and yielded a value of T_0 just above room temperature (32°C). This value is much lower than the reference temperature (102°C) reported for a 12% Cr ferritic ODS steel denominated 12YWT (Ref. 15). The comparison with the Master Curve measured at SCK•CEN on the unirradiated conventional EUROFER in a previous investigation[16], shown in Fig. 3, shows a large difference (144 °C) in terms of normalized fracture toughness ($K_{Jc,1T}$) at the 100 MPa√m level, demonstrating that the current European reference ODS material has, in the unirradiated state, significantly worse toughness properties than the corresponding non-ODS steel.

Further comparisons with other ODS versions are not possible, since fracture toughness properties have not been measured on any other EUROFER ODS material.

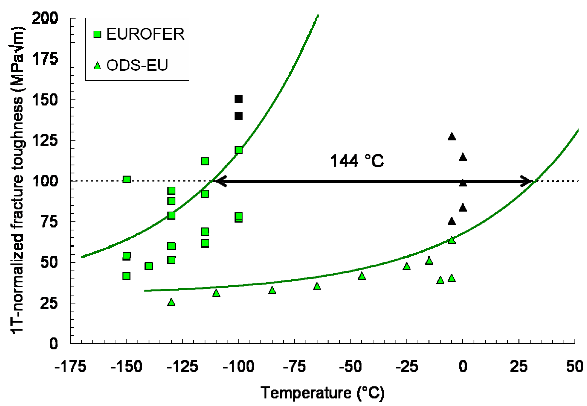

Fig. 4. Fracture toughness properties in the ductile-to-brittle transition region (Master Curve analysis) for conventional EUROFER and ODS-EU.

III. IRRADIATION CONDITIONS

As mentioned above, the two most promising EUROFER ODS materials, ODS-2 and ODS-EU, were irradiated in the BR2 test reactor in the framework of the IRFUMA-IV (Ref. 17) and IRFUMA-5M (Ref. 14) campaigns. For both experiments, irradiation temperature was 300 ± 5°C; the specimens were in direct contact with the coolant (water).

For both materials, subsize tensile and KLST Charpy specimens (partly notched and partly precracked) were irradiated. The mean accumulated dose, estimated by means of extensive dosimetry, was 1.74 dpa for ODS-2 and 1.52 dpa for ODS-EU.

IV. POST-IRRADIATION TESTING

IV.A. Tensile properties (radiation hardening)

300°C is the most critical temperature in terms of radiation-induced hardening for RAFM steels (Ref. 18). Fig. 5 shows the increase of yield strength measured for ODS-EU after irradiation, ranging from 152 MPa at room temperature to 107 MPa at 300°C.

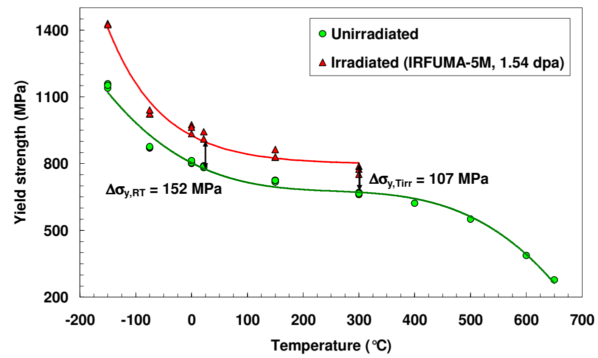

Fig. 5. ODS-EU yield strength.

Uniform elongation was observed to decrease moderately at room temperature, RT (from 8.4% to 6.9%) and negligibly at 300°C (from 6.9% to 6.5%). Total elongation also showed limited degradation (from 18.4% to 16.9% and from 16.2% to 14.2% respectively).

The comparison between engineering stress-strain curves (Fig. 6) at RT shows that ODS-EU retains considerable strain hardening after irradiation, unlike ODS-2 and conventional EUROFER irradiated under similar conditions (temperature and dose).[13,19] Note also the much higher hardening (~400 MPa) and ductility loss observed for ODS-2.

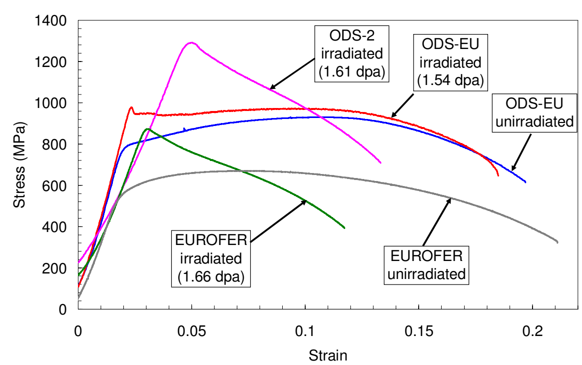

Fig. 6. RT engineering stress-strain curves for conventional EUROFER, ODS-2 and ODS-EU.

IV.B. Impact properties (radiation embrittlement)

For ODS-EU, we cannot formulate a meaningful assessment of the radiation-induced increase of ductile-to-brittle transition temperature (DBTT) and variation of upper shelf energy (USE), since KLST Charpy data in the unirradiated condition are not available for the same product form (6 mm plate) and the same orientation (T-L). However, Fig. 7 shows that DBTT for ODS-EU at 1.47 dpa remains well below RT and is slightly lower than for ODS-2 irradiated at 1.76 dpa (Ref. 13); on the other hand, even considering the difference in accumulated dose, the USE for ODS-EU is significantly higher than for ODS-2.

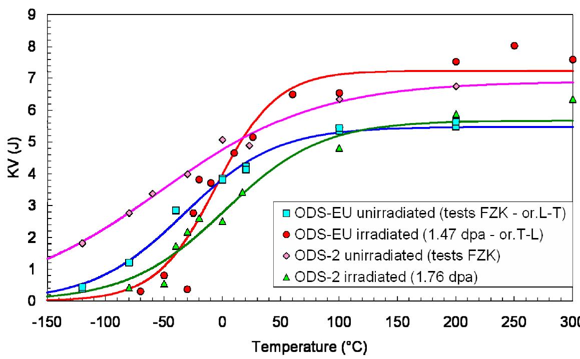

Fig. 7. KLST Charpy energy curves for ODS-EU and ODS-2 in the unirradiated and irradiated conditions.

IV.C. Fracture toughness properties (radiation embrittlement)

Several investigations[16,19-21] have shown that for RAFM steels, and high Cr steels in general, the embrittlement measured from Charpy tests strongly underestimates the degradation of actual fracture toughness. Therefore, in order to accurately and safely assess radiation-induced embrittlement, fracture mechanics specimens have to be tested and analyzed. For ODS-EU irradiated up to 1.54 dpa, fifteen KLST specimens were fatigue precracked and tested in order to obtain the reference temperature T_0 at 100 MPa√m according to the Master Curve approach (ASTM E 1921-05). With respect to the unirradiated condition, the radiation-induced shift of T_0 is 41°C, less than half the increase observed for conventional EUROFER irradiated under similar conditions[16] (98°C, Fig. 8). However, due to the large difference in toughness between the two materials in the unirradiated condition, even after irradiation ODS-EU is significantly less tough (~60°C) that the base material.

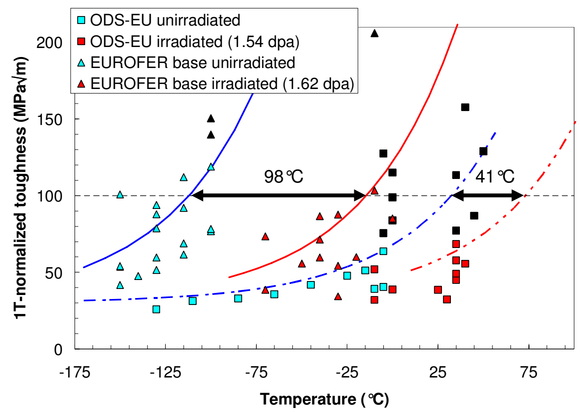

Fig. 8. Irradiation effects on the fracture toughness of conventional EUROFER and EU-ODS.

No meaningful comparison with ODS-2 after irradiation can be made, since (as detailed in Ref. 13) for the latter material no fracture toughness value above 75 MPa√m was measured and therefore the Master Curve analysis could not be performed nor a value of T_0 defined. However, it's fair to state that a significant improvement in post-irradiation fracture toughness was achieved for ODS-EU with respect to ODS-2.

V. DISCUSSION: RADIATION SENSITIVITY

Fig. 9 shows that the two investigated ODS materials have a clearly different hardening rate, and also that ODS-2 and ODS-EU harden significantly faster and slower than conventional EUROFER respectively. This is also confirmed by the direct comparison of $\Delta\sigma_{p02}$ (yield strength increase) values in Fig. 10.

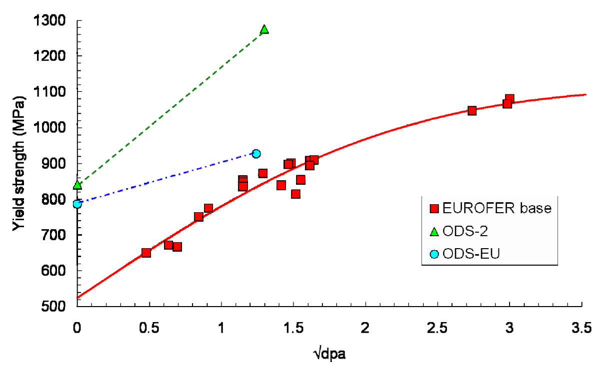

Fig. 9. Yield strength for conventional EUROFER, ODS-2 and ODS-EU as a function of √dpa.

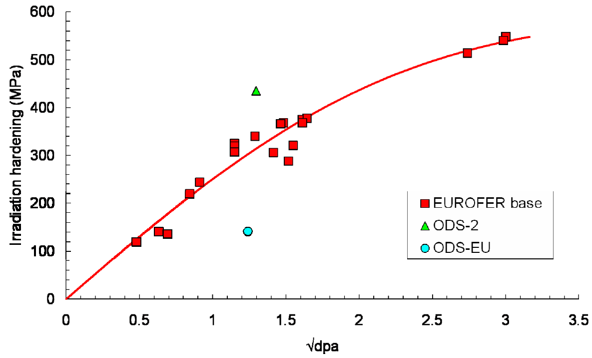

Fig. 10. Irradiation hardening for conventional EUROFER, ODS-2 and ODS-EU as a function of √dpa.

EUROFER data and fitting curves shown in Figs. 8 and 9 are taken from (Ref. 18).

Similar considerations for ODS-EU (worse toughness in the unirradiated condition but lower embrittlement rate) can be formulated for the Master Curve reference temperature T_0, as shown by Figs. 11 and 12 (EUROFER data and fitting curves are taken from Ref. 21).

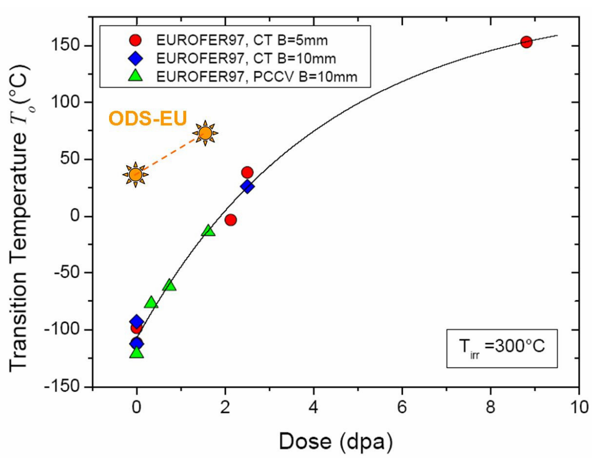

Fig. 11. Master Curve reference temperature for conventional EUROFER and ODS-EU as a function of dose.

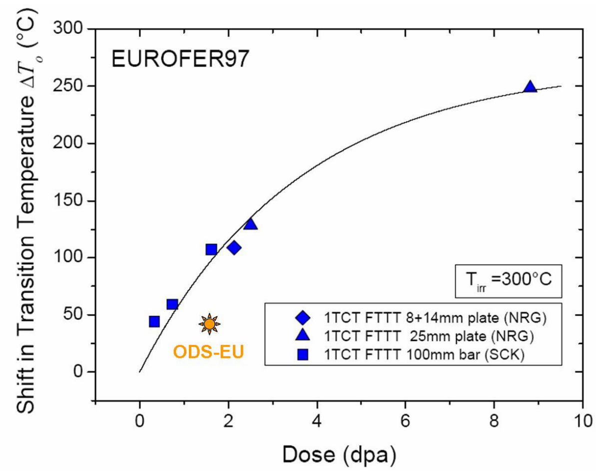

Fig. 12. Irradiation embrittlement for conventional EUROFER and ODS-EU as a function of dose. (FTTT = Fracture Toughness Transition Temperature, i.e. T_0.)

VI. CONCLUSIONS

Pre- and post-irradiation mechanical investigations (tensile, impact, fracture toughness) were conducted on the two most promising Oxide Dispersion Strengthened (ODS) EUROFER materials, the "2nd generation" and the "EU batch". The relationship between the two ODS materials and the conventional (non-ODS) EUROFER has emerged clearly from the investigations performed.

1. In the unirradiated condition, the tensile properties of the two ODS materials are equivalent, except for uniform elongation, which is much better for ODS-EU. With respect to conventional EUROFER, ODS-EU has higher mechanical strength, equivalent or better ductility but is worse in terms of quasi-static fracture toughness.
2. After irradiation at 300°C up to 1.5-1.7 dpa, the mechanical properties of ODS-2 and ODS-EU significantly diverge, with the "EU batch" showing less hardening, better ductility and less embrittlement than ODS-2. Although in absolute terms ODS-EU remains harder and less tough than EUROFER, the rate of hardening and embrittlement seems significantly lower. Conversely, ODS-2 appears to harden faster than conventional EUROFER.

The promising irradiation response of the "EU batch" needs to be confirmed by additional irradiations up to higher doses.

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