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Present development status of EUROFER and ODS-EUROFER for application in blanket concepts

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Abstract

Within the European Union, the two major breeding blanket concepts presently being developed are the helium cooled pebble bed (HCPB), and the helium cooled lithium lead (HCLL) blankets. For both concepts, different conceptual designs are being discussed with temperature windows in the range 250–550 °C for conservative approaches based on reduced activation ferritic–martensitic (RAFM) steels, and in the range 250–650 °C for more advanced versions, taking into account oxide dispersion strengthened (ODS) steels. As a final result of a systematic development of RAFM-steels in Europe, the 9% CrWVTa alloy EUROFER was specified and produced in an industrial scale with a variety of product forms. A large characterisation program is being performed including irradiation in materials test reactors between 60 and 450 °C ($\leq$15 dpa), and in a fast breeder reactor at 330 °C up to 30 dpa. EUROFER is resistant to high temperature ageing, and the existing creep-rupture data ($\sim$30,000 h, 450–600 °C) indicate long-term stability and predictability.

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The ODS variant of EUROFER shows superior tensile and creep properties compared to EUROFER. Applying a new production route has diminished the problem of lower ductility and inferior impact properties. A reliable joining technique for ODS and RAFM steels employing diffusion welding was successfully developed.

Keywords: Reduced activation ferritic–martensitic (RAFM); Oxide dispersions strengthened (ODS); EUROFER; Tensile; Charpy; Creep tests; Irradiation behaviour

1. Introduction

Within the European Union, the two major breeding blanket concepts presently being developed are the helium cooled pebble bed (HCPB), and the helium cooled lithium lead (HCLL) blankets. For both concepts, different conceptual designs are being discussed with temperature windows in the range 250–550 °C for conservative approaches based on reduced activation ferritic–martensitic (RAFM) steels, and in the range 250–650 °C for more advanced versions, taking into account oxide dispersion strengthened (ODS) steels.

Ferritic–martensitic steels show reasonably good thermo-physical and mechanical properties, a low sensitivity to radiation-induced swelling and helium embrittlement under (fission) neutron irradiation and good compatibility with major cooling and breeding materials [1]. In recent years, reduced activation versions of this type of steels have been developed in Japan and Europe in laboratory scale and tested with equivalent or even better mechanical properties. In Japan, industrial batches of such a RAFM steel, F82H mod, were produced and tested by IEA partners [2,3]. As a result of a systematic development of RAFM-steels in Europe, the 9% CrWVTa alloy EUROFER was specified, and industrial batches have been produced in a variety of different semi-finished product forms.

2. Manufacturing of EUROFER 97

RAFM steels are modified compositions of conventional ferritic–martensitic 8–12% CrMoVNb steels, mainly by exchanging Mo, Nb and Ni with W and Ta in order to obtain low activation capability [4]. The European 9% CrWVTa steel EUROFER 97 was specified, and industrial batches of 3.5 and 7.5 tonnes have been produced by two different EU companies (Böhler, Austria and Saarschmiede, Germany) with 10 different heats and 18 different semi-finished, quality-assured product forms (forged bars, plates, tubes wires) in various dimensions. Table 1 shows the chemical compositions of EUROFER 97 and the Japanese reference RAFM steel F82H mod with upper limits of radiologically undesired elements. The chemical composition has been optimised according to experience with RAFM laboratory heats in Europe, Japan and US [5–9]. The Cr content of EUROFER was raised to 9% to achieve better corrosion resistance. In addition, the shift in ductile-to-brittle transition-temperature (DBTT) after irradiation is minimised at Cr contents around 9% [9]. Ta stabilises the grain size by carbide formation and improves DBTT and strength. About 1% W represents a good compromise regarding low activation, DBTT, tensile strength, ductility and creep strength. The tritium breeding ratio is higher for lower W content. The levels of radiologically undesired elements, such as Nb, Mo, Ni, Cu, Al and others, were specified according to the current capabilities of steel making industry in Europe, applying raw materials selection and appropriate clean steel making technologies. Although no scrap was used to avoid incorporation of undesired elements, the achieved compositions deviated in some cases (Table 1). The impurity increases can be explained by pick-up from small residues left in the equipment used for conventional steel compositions. The effect of the undesired impurities is shown in Fig. 1 that gives the surface gamma dose rate in dependence of the time after irradiation. It clearly shows the progress in RAFM development. First RAFM steels like OPTIFER (70 ppm Nb) reached the remote recycling level at about 100 years after irradiation, while EUROFER and F82H mod could be stored as low-level waste after the same period of time after reactor shut-down. The difference between the latter two alloys (hatched area) is due to the lower Nb (1 ppm) content

Table 1
Chemical composition of EUROFER 97 compared to F82H mod, OPTIFER V and MANET II

	Radiologically desired (ppm)	EUROFER 97 specified (mass%)	EUROFER 97 achieved[a] (mass%)	F82H mod Heat 9741[a] (mass%)	OPTIFER V Heat 735[a] (mass%)	MANET II Heat 50804[a] (mass%)
(A) Main alloying elements (mass%)						
C		0.09–0.12 [0.11]	0.11–0.12	0.09	0.12	0.11
Cr		8.5–9.5 [9.0]	8.82–8.96	7.7	9.48	10.3
W		1.0–1.2 [1.1]	1.07–1.15	1.94	0.985	–
Mn		0.20–0.60 [0.40]	0.38–0.49	0.16	0.39	0.78
V		0.15–0.25	0.18–0.20	0.16	0.245	0.2
Ta		0.10–0.14 [0.12]	0.13–0.15	0.02	0.061	–
N_2		0.015–0.045 [0.030]	0.018–0.034	0.006	0.0225	0.031
P		<0.005	0.004–0.005	0.002	0.0035	0.003
S		<0.005	0.003–0.004	0.002	0.0025	0.004
B		<0.001	0.0005–0.0009	0.0002	0.0002	0.0073
O_2		<0.01	0.0013–0.0018	0.01	0.006	
(B) Radiologiocally undesired elements (mass% and μg/g = ppm)						
Nb	<0.01	[<10]	2–7	1	70	1400
Mo	<1	[<50]	10–32	30	50	6100
Ni	<10	[<50]	70–280[b]	200	50	6800
Cu	<10	[<50]	15–220[b]	100	50	100
Al	<1	[<100]	60–90	30	70	40
Ti	<200	<100	50–90	100	70	
Si	<400	<500	400–700	1100	60	1900
Co	<10	[<50]	30–70	50		

Target values are in brackets.
[a] Analyses by manufacturers and Ch. Adelhelm FZK EUROFER 97: range over 10 heats, 11 tonnes of 18 different product forms (forged bar, plates, tubes, wire).
[b] Reasons identified.

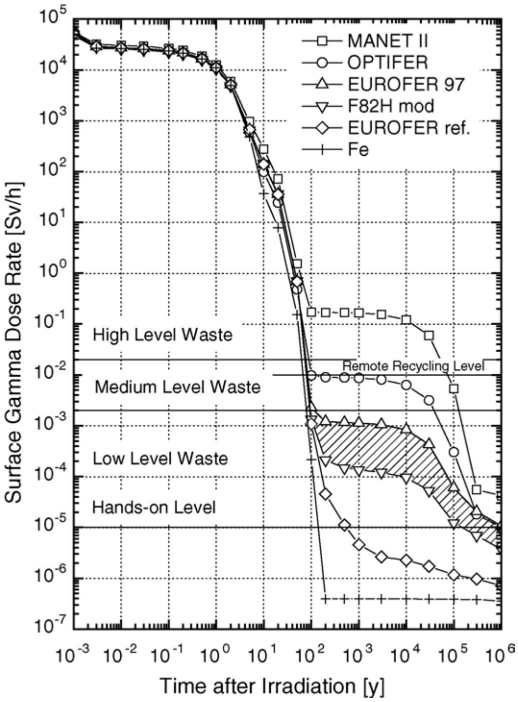

Fig. 1. Calculated decay of γ-surface dose rate in iron and ferritic–martensitic steels after irradiation (12.5 MWa/m^2) in a first wall DEMO spectrum, according to Ref. [4].

of F82H mod compared to 10 ppm for EUROFER. EUROFER ref. stands for an alloy composition containing the theoretical values of undesired elements. In order to move into the hands-on-level domain the activation level has to be reduced by two orders of magnitude. This seems to be technically feasible; it requires increased feedstock control and production lines reserved for low activation steel only.

3. Characterisation of EUROFER 97

3.1. Mechanical properties and microstructural stability

A large characterisation program is being performed including microstructural, mechanical properties, corrosion and compatibility experiments [6–8,10].

Yield strength $R_{p0.2}$ and ultimate tensile strength R_m of EUROFER 97 measured on different heats, product forms and orientations in the temperature range between *RT* and 750 °C are similar to those of F82H mod and OPTIFER steels. Charpy impact tests using ISO-V specimens show a DBTT around −70 °C for EUROFER. The slightly higher DBTT of F82H mod at −40 °C can be attributed to the larger grain size and oxygen content of this material [7]. Ageing between 500 and 600 °C up to 10,000 h does not significantly influence the tensile and impact properties, while at higher temperatures (700 °C, 2000 h) it decreases tensile strength and causes a shift in DBTT [7,10–12].

Continuous cooling transformation (CCT) diagrams have been analysed in detail together with the transformation, long-term ageing, hardening and tempering behaviour. Due to a minimum cooling rate for full martensitic transformation of about 5 K/min, a wide range of EUROFER component thicknesses (<220 mm) can be heat treated. The prior austenite grain size of EUROFER 97 is quite stable (ASTM 10.5–8) in the relevant normalisation temperature range between 900 and 1050 °C [7,12–14] and does not change significantly during ageing at 600 °C for 10,000 h and shows high microstructural stability.

In the design-relevant temperature and stress range, broad-based creep-rupture experiments between 450 and 650 °C up to 30,000 h creep rupture time have been executed on EUROFER 97 heats in two EU laboratories, FZK and CIEMAT. Creep behaviour, stress exponents, 1% yield limits and rupture times showed no deviations from expected behaviour, confirming long-term stability and predictability [7]. Emphasis is presently given on the safety relevant low-stress range (100 MPa and below).

3.2. Irradiation behaviour

Irradiation programs in materials test reactors have been performed between 60 and 450 °C (≤15 dpa), and in a fast breeder reactor at 330 °C up to 30 dpa. 75 dpa data will become available in about two years. Irradiation induced hardening and shift of DBTT after irradiation at temperatures below about 350 °C are the most critical issues for ferritic–martensitic steels. The impact energy of unirradiated steel and samples, irradiated in the mixed-spectrum HFR reactor in Petten, show the dependence on the test temperature (Fig. 2) [15]. KLST specimens (DIN 50115) with a size of 3 mm × 4 mm × 27 mm, 1 mm notch depth, 0.1 mm notch root radius, and 60° notch angle were

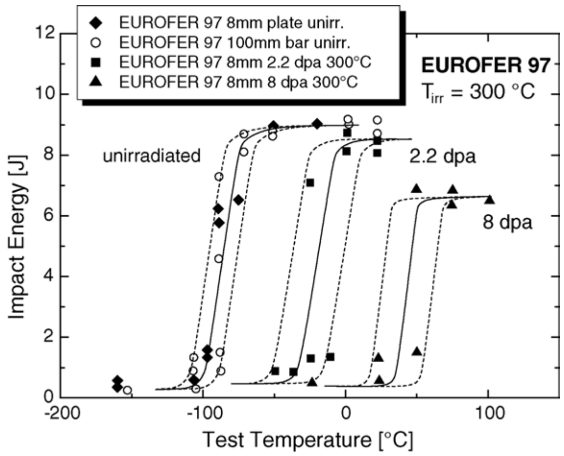

Fig. 2. Impact energy of EUROFER 97 (KLST specimens) after 300 °C irradiation, from Ref. [15].

used in these irradiation experiments. The DBTT in the as-received condition of about −90 °C is increased after irradiation at 300 °C to 8 dpa to about +40 °C. The upper shelf energy is decreased from 9 to about 7 J, which is about 0.3 J per dpa. The absolute shift of DBTT at 300 °C irradiation temperature does not increase linearly with irradiation dose; it seems to move towards a saturation level. Fracture toughness tests reveal that the shift of the reference temperature T_0 after irradiation is more pronounced than the shift in DBTT. T_0 is shifted from –121 °C for unirradiated EUROFER 97 to −14 °C irradiated to 1.6 dpa at 300 °C in the BR2 reactor in Mol. This shift is consistent with data measured on F82H mod irradiated at comparable conditions [16].

Although irradiated EUROFER specimens have been examined only at 10 dpa and below, it can be stated that irradiation-induced hardening, ductility reduction and fracture toughness degradation are highly superior to irradiated conventional ferritic–martensitic steels like EM10, HT9, MANET and T91.

4. High performance steel ODS-EUROFER

A replacement of presently considered RAFM steels by suitable ODS alloys would allow a substantial increase of the operating temperature from ∼550 °C to about 650 °C. Several European labs have elaborated ODS variants of EUROFER 97 [17–20]. The consolidation of the mechanically alloyed powder was mainly done by hot isostatic pressing (HIP), following the idea to fabricate near-net-shape blanket structures by hipping; one laboratory also investigated extrusion [20]. The consolidated products have yield and tensile strength that are about 35% higher than plain EUROFER 97 up to 700 °C [17,20] as shown in Fig. 3. The ductility of the hipped products was not satisfying above 350 °C; only the extruded material showed total elongations comparable to standard EUROFER. Creep tests on ODS-EUROFER have been performed under vacuum at temperatures between 600 and 700 °C up to rupture times of 10,000 h. The results are given in the Larson–Miller plot in Fig. 4, which compares the creep strength of ODS-EUROFER in different product forms, orientations and heat treatments with standard RAFM steels EUROFER and F82H mod. All ODS-EUROFER variants exhibit clearly higher creep strength than the RAFM steels. ODS-EUROFER shows the same creep strength as EUROFER but at temperatures about 100 K higher.

Advanced blanket concepts like the helium cooled lithium lead blankets consist of a EUROFER structure with SiC_f/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 load [21]. Therefore, a sheet of ODS-EUROFER steel was produced in cooperation of FZK and PLANSEE. The production route included compaction of the mechanically alloyed steel powder (0.3 wt.% Y_2O_3) by HIP and subsequently hot rolling using a cross rolling technique, which should provide homogeneous in-plane properties. Different heat treatments were applied to study their effect on the mechanical properties. From Fig. 3, it can be seen that the thermo-mechanically treated samples have a tensile strength comparable to the hipped and extruded ODS-variants. The total and uniform elongation of the ODS plate are notedly increased at higher temperatures and are equal or even higher compared to the base material over the whole temperature range (*RT*, 700 °C). This is in contrast to the first generation of ODS-EUROFER and a commercial ferritic ODS-alloy (PM 2000), where the total elongation above 400 °C was lower.

The biggest progress was made concerning the impact behaviour. Impact tests on sub-size KLST

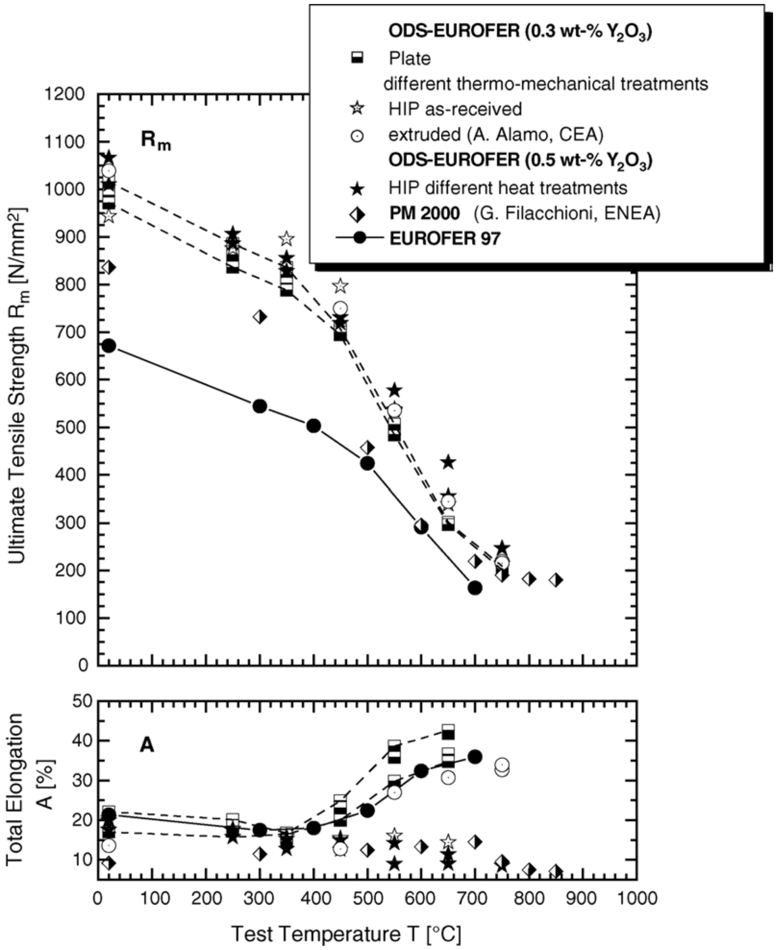

Fig. 3. Ultimate tensile strength and total elongation of different ODS steels compared to standard EUROFER 97 as a function of the test temperature.

specimens show an improved DBTT which could be shifted from values between +60 and +100 °C for hipped ODS-Eurofer of the first generation to values between −40 and −80 °C as shown in Fig. 5. The upper-shelf energy (USE) was increased by about 40%. Nevertheless, thermo-mechanically treated ODS-EUROFER does not reach the very low DBTT and high USE of EUROFER 97.

First results of creep tests in the temperature range between 600 and 700 °C are very encouraging. Samples of the rolled plate material reach at lower stress levels and higher temperatures the values of the hipped ODS-EUROFER, which has a higher content (0.5 wt.%) of Yttria. Due to the cross roll of the plates, there is no significant difference between samples taken from longitudinal and transverse direction of the plate.

The use of ODS as plating of a first-wall or in divertor structures requires also the development of joining techniques. It was successfully demonstrated that it is possible to fabricate joints of ODS/ODS (CEA) and ODS-EUROFER/EUROFER (FZK) by diffusion welding. Tensile tests show good strength of the joints. Fracture always occurs outside the welded area in the EUROFER part. In the case of the dissimilar ODS-EUROFER/EUROFER joints, the results of first impact tests are very promising. The upper shelf energy is depending on HIP and post-HIP treatment up to 80% of USE of EUROFER; DBTT is about −50 °C,

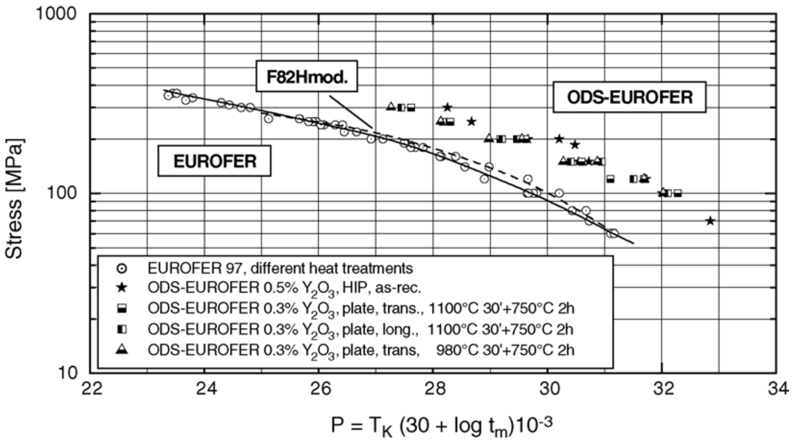

Fig. 4. Larson–Miller plot for EUROFER 97 with different heat treatments in comparison with oxide dispersion strengthened ODS-EUROFER and F82H mod (- · · -).

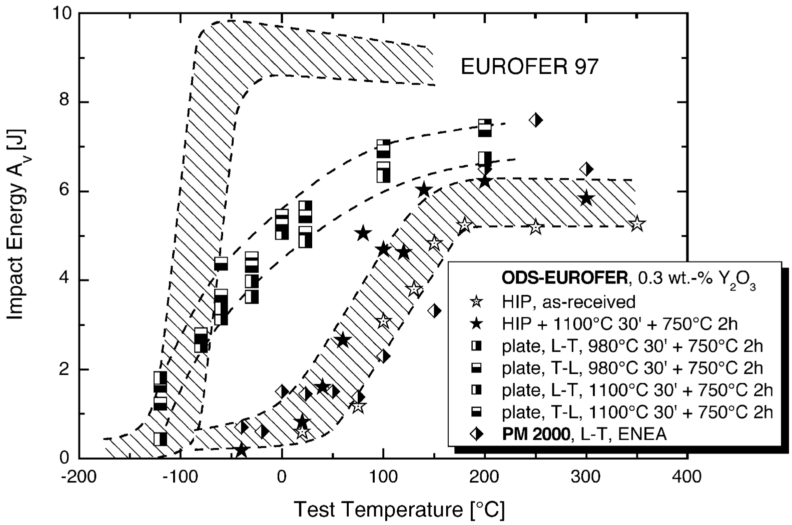

Fig. 5. Test temperature dependence of total absorbed energy of different ODS-EUROFER steels in comparison with EUROFER 97 (KLST specimens).

which is well below DBTT of the hipped ODS material [22].

5. Conclusions

It has been shown that the RAFM steel EUROFER 97 can be manufactured in industrial scale with reproducible properties, which meet the requirements of the test blanket modules for ITER. ODS variants of this steel are under development with promising properties, which would allow a further increase of the operational temperature. The feasibility of joining ODS-EUROFER and EUROFER has been demonstrated successfully.

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