

Thermal and mechanical behaviour of the reduced-activation-ferritic-martensitic steel EUROFER

R. Lindau *, A. Möslang, M. Schirra

Forschungszentrum Karlsruhe, Institut für Materialforschung I, P.O. Box 3640, 76021 Karlsruhe, Germany

Abstract

Reduced activation ferritic/martensitic (RAFM) steels are being considered for structural application in potential fusion energy systems. Based on the substantial experience with RAFM developmental steels of OPTIFER type, an industrial 3.5 tons batch of a 9CrWVTa-RAFM steel, called EUROFER 97 had been specified and ordered. A characterisation programme has been launched to determine the relevant mechanical and physical-metallurgical properties in order to qualify the steel for fusion application. The hardening, tempering and transformation behaviour of EUROFER is in good agreement with that of other RAFM-steels like OPTIFER and the Japanese industrial scale heat F82H mod. Tensile tests, performed between RT and 750 °C, show comparable strength and ductility values that are not strongly affected by different heat treatments and ageing at 580 and 600 °C up to 3300 h. Impact bending tests indicate a superior ductile to brittle transition temperature (DBTT) of EUROFER in the as-received condition compared with that of F82H mod. Creep tests between 450 and 650 °C up to test times of 15 000 h reveal a creep strength similar to other RAFM steels like OPTIFER and F82H mod. EUROFER shows a good low-cycle fatigue behaviour with longer lifetimes than F82H mod. The deformation and softening behaviour is similar.

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Keywords: Reduced activation ferritic martensitic (RAFM); OPTIFER; EUROFER; Tensile; Charpy; Creep tests; LCF tests

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels are being considered for structural application in potential fusion energy systems. Based on the substantial experience with RAFM develop-

mental steels of OPTIFER type, produced as laboratory melts of 25 kg, a RAFM steel of the 9CrWVTa type, called EUROFER 97 had been specified and ordered. BÖHLER EDELSTAHL GmbH in Austria was finally selected as manufacturer for about 3.5 tons of semi-finished products (plates, forgings, filler wire material and tubes). Apart from different technological goals the question of transferability of properties from laboratory to industrial scale melts is of major interest.

* Corresponding author. Tel.: +49-7247-82-6402; fax: +49-7247-82-4567

E-mail address: rainer.lindau@imf.fzk.de (R. Lindau).

2. Experimental

A characterisation programme has been launched to determine the relevant mechanical and physical-metallurgical properties in order to qualify EUROFER for fusion application. Table 1 gives for EUROFER 97 and other relevant alloys the main alloying element content as well as the radiologically undesired tramp elements that are responsible for the long-term activation behaviour. The effect of the undesired impurities is clearly shown in Fig. 1, which gives the surface gamma dose rate in dependence of the time after irradiation. Currently developed RAFM steels like EUROFER and F82H mod. reach the remote recycling level in about 100 years and could be stored as low level waste. The difference between these two alloys (hatched area) is due to the lower Nb (1 ppm) content of F82H mod. compared with 10 ppm for EUROFER. The developmental goal, a real low activation steel (EUROFER ref.) with very low impurity contents indicated as ‘theoretical’ in Table 1, [1], is not achievable with the momentary industrial steel making processes nor can these small amounts be analysed in every case. On the basis of far-reaching experience with precursor RAFM-alloys of OPTIFER type, for EUROFER the contents of Cr, Ta, W were specified carefully. Since the ductile to brittle transition temperature (DBTT) in impact tests reaches a minimum at 9% Cr and in order to achieve a better corrosion resistance, 9% Cr was regarded to be appropriate. Ta stabilises the grain size by carbide formation and improves DBTT and strength. A higher content than chosen has no advantage [2]. 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. To achieve reduced activation behaviour the radiologically undesired elements, such as Nb, Mo, Ni, Cu, Al and others, have to be limited to contents in the wppm-range. Except for Ni, all contents were lower than the specified values. The reasons for the deviation have been identified and can be avoided in future [3].

3. Results and discussion

3.1. Hardening-, tempering- and transformation-behaviour

For practical applications the transformation-, hardening- and tempering behaviour of ferritic/martensitic steels are of great importance. A continuous-cooling-transformation-diagram (CCT-diagram) has been created, which gives relevant parameters such as transformation temperatures and critical cooling rates, i.e. the minimum cooling rate to achieve a full martensitic transformation. Using air-cooling, a value of 5 K/min corresponds to a cross-section of approximately 220 mm in diameter, where an overall martensitic transformation is achieved. The transformation behaviour of EUROFER is in good agreement with that of other RAFM-alloys like OPTIFER and F82H mod. [4]. The hardening and tempering behaviour of EUROFER corresponds to that of the 25 kg laboratory melts of OPTIFER-type. The higher Ta-content leads to more stable and smaller grain size ($11\text{--}21\text{ }\mu\text{m} \approx \text{ASTM } 10.5\text{--}8$) in the relevant temperature region between 900 and 1050 °C compared with F82Hmod ($27\text{--}51\text{ }\mu\text{m} \approx \text{ASTM } 7.5\text{--}5.5$) [5]. Ageing experiments in the temperature region from 550 to 750 °C up to 3300 h indicate the good structural stability of EUROFER and an equal or even better ageing resistance compared with OPTIFER and the Japanese RAFM steel F82H mod., respectively.

3.2. Tensile tests

Cylindrical tensile specimens of EUROFER 97 with 5 mm in diameter and 25 mm gauge length were machined from the \varnothing 100 mm forged bar and the 14 mm plate material and subjected to tensile tests in the temperature range between RT and 750 °C. Fig. 2 gives the ultimate tensile strength R_m and total elongation A_5 of EUROFER 97 in different heat treatments.

The ultimate tensile strength of the as-received materials show the highest strength values. The strength values coincide very well with those of the precursor alloys of OPTIFER-type and are comparable to those of F82Hmod. [6]. Increasing the

Table 1
Content of main alloying elements and radiologically undesired tramp elements of examined steels in mass% or ppm, respectively

Alloy	C (mass- %)	Cr (mass-%)	W (mass- %)	V (mass- %)	Ta (mass-%)	Mn (mass-%)	Si (µg/g (ppm))	Nb (µg/g (ppm))	Mo (µg/g (ppm))	Ni (µg/g (ppm))	Cu (µg/g (ppm))	Al (µg/g (ppm))	Co (µg/g (ppm))
EUROFER Specified	0.090 12	8.50 9.50	1.0 1.2	0.15 0.25	0.05 0.09	0.20 0.60	< 500	< 10	< 50	< 50	< 50	< 100	< 50
EUROFER Range ^a	0.10 0.12	8.62 8.96	1.07 1.15	0.18 0.20	0.13 0.14	0.38 0.49	400 500	2 7	10 32	75 200 ^b	15 200 ^b	50 90	30 60
OPTIFER V	0.12	9.48	0.985	0.245	0.061	0.39	600	< 10	< 15	48	21	< 4	< 15
F82H mod.	0.09	7.7	1.94	0.16	0.02	0.16	1100	1 4	30	200	100	30	50
Theoretical							< 400	< 0.01	< 0.50	< 10	< 10	< 1	< 10

^a Range of all products.

^b Reasons identified.

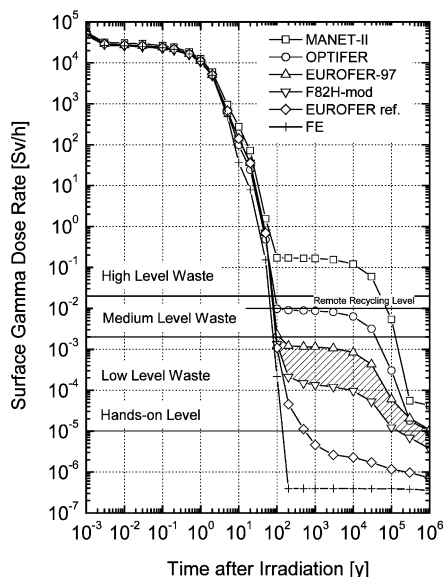


Fig. 1. Calculated γ -surface dose rate of pure Fe and (RA)FM steels after irradiation in a first wall DEMO spectrum (12.5 MWa/m²).

austenitising temperature to 1075 °C leads to lower strength values, most pronounced at lower test temperatures, whereas the ductility is less affected. This loss in strength is accompanied by higher ductile to DBTT in the impact test. Ageing at 580 and 600 °C up to 3300 h causes only a marginal decrease in tensile strength. It is worthwhile to mention that the strength of the 14 mm plate material matches very well the values of the bar material. The total elongation A_5 , representing the ductility, is similar in appearance for all alloys. This applies also for the uniform elongation A_u and reduction of area Z , which are not indicated here (nomenclature according to European standard EN 10 002). A complementary and expanded description of tensile results can be found in [2,5,6].

3.3. Impact bending tests

Standard ISO-V Charpy-specimens were machined from the 14 mm plate and 100 mm bar material. The specimen orientation was transverse or in one case longitudinal with respect to the final roll of the plates. The specimens machined from

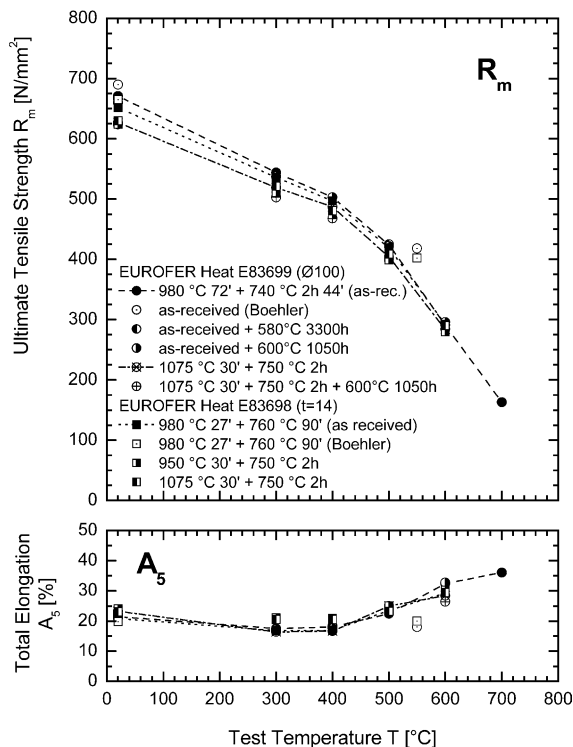


Fig. 2. Ultimate tensile strength and total elongation of EUROFER 97 subjected to different heat treatments as a function of the test temperature.

the bar were taken along or transverse to the rod axis. The upper shelf energy of both materials with varying austenitising temperatures (980, 1050 and 1075 °C for 30 min) and a tempering temperature of 750 °C (2 h) is roughly between 240 and 300 J. The DBTT, which is an important measure for the suitability of RAFM alloys for structural applications in a fusion reactor, is for all EUROFER variants with values between -78 and -54 °C remarkably better than for the Japanese steel F82H mod. (-20 °C) in the as-received condition (1040 °C 30 min + 750 °C 1 h). The values of the longitudinal specimens taken from the bar are comparable to the results obtained from the plate, which shows similar results independent from the specimen direction whereas the transverse specimens taken from the bar show distinctly lower values. This finding can be attributed to the different production route of the semi-finished products. The 100 mm bars are produced by

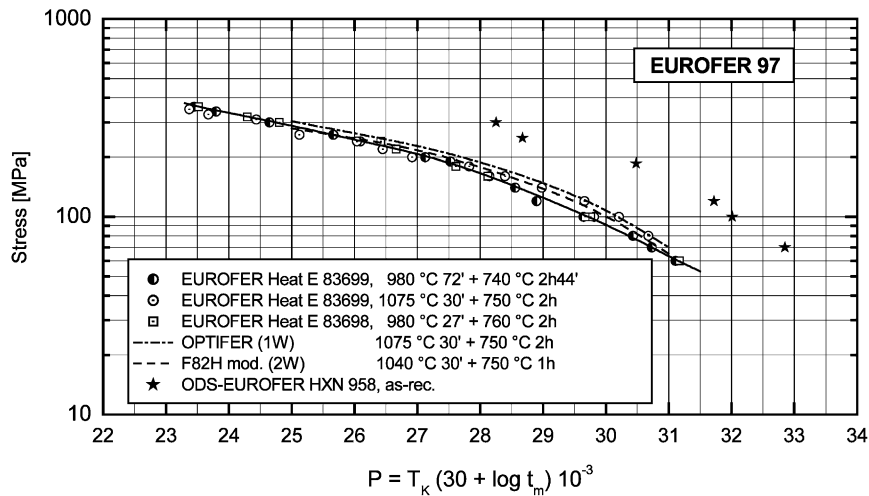


Fig. 3. Larson–Miller-Plot for EUROFER 97 bar and plate material with different heat treatments in comparison with OPTIFER developmental alloy, F82H mod. and ODS-EUROFER.

forging whereas BÖHLER BLECHE applies the so-called cross rolling technology to achieve isotropic mechanical and physical properties, which has been verified here in an excellent way. One reason for the higher DBTT (−20 °C) of F82H mod. is the larger grain size (55 μm), which depends on the austenitisation temperature. Another reason could be the higher oxygen content (124 ppm) [2] compared with 10 ppm for EUROFER which for the as-received condition (980 °C 27 min + 760 °C 90 min) exhibits a DBTT of −70 °C. Increasing the austenitising temperature to 1050 °C does not alter the DBTT (−73 °C) significantly although the grain size increases to 26 μm. A further increase to 1075 °C, however, increases the DBTT to −56 and −57 °C for the transverse and longitudinal specimens, respectively. This shift in DBTT can be related to a further increase of the grain size (45 μm). The observed step-like shape of the energy–temperature-curve [7] in the transition region between upper and lower shelf energy was confirmed in statistical tests with five specimens per temperature. The reasons for that behaviour are still unclear and subject of further investigations. A more detailed description can be found in [6,7].

3.4. Creep behaviour

Creep tests at temperatures between 450 and 650 °C and test times up to 15 000 h, using the same specimen type as for the tensile tests, have been conducted on EUROFER bar and 14 mm plate material in different heat treatment conditions. The results are presented in Fig. 3 in the form of a so-called Larson–Miller-Plot, which

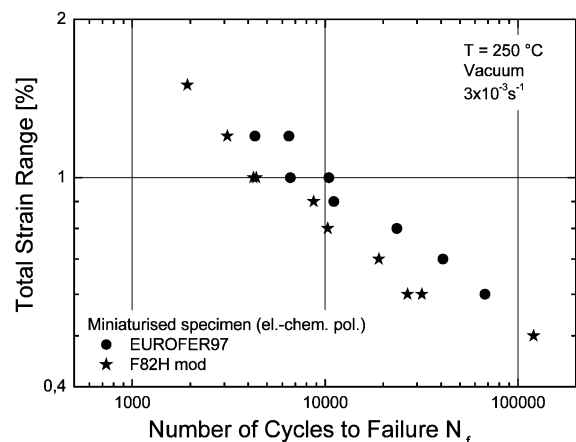


Fig. 4. Low-cycle fatigue behaviour of EUROFER 97 compared with F82H mod.

allows a good comparison and extrapolation of creep strength to different temperatures and times. The creep strength of EUROFER (solid line) in the as-received condition with an austenitising temperature of 980 °C is identical for both, bar (circles) and plates (squares). If the austenitisation temperature is raised to the same value as for OPTIFER (1075 °C) the creep strength at lower stress levels is improved and reaches the curve for OPTIFER and surpasses F82H mod. which is austenitised at 1040 °C. At high stress levels, the creep strength is decreased as compared with OPTIFER and F82H mod. A re-inforcement of EUROFER with Yttria particles (0.5 wt.%) introduced by mechanical alloying can increase the creep strength (stars) substantially, an applied stress of e.g. 100 MPa would lead at 650 °C to a rupture time of 50 000 h compared with 500 h for EUROFER [8].

3.5. Low-cycle fatigue behaviour

Strain-controlled low-cycle fatigue tests were performed under vacuum in the temperature range between 250 and 550 °C applying total strain amplitudes between 0.5 and 1.5%. The used (small specimen test technology) SSTT specimen type with 2 mm diameter and a gauge length of 7.6 mm has been especially developed for the use in IFMIF and has proven its equivalence to macroscopic specimen types [9].

Fig. 4 gives a comparison of the fatigue lives of EUROFER and F82H mod. The shorter lifetime of F82H mod. can be attributed to accelerated crack initiation at precipitates of mixed composition Ta, Al(O,C) near the surface. The deformation and softening behaviour of both steels is almost identical.

4. Conclusions

The results of the presented characterisation work shows that it is possible to transfer the good

mechanical and structural properties of precursor laboratory melts to an industrial scale melt. Although progress has been made in order to achieve low contents of undesirable elements, a further reduction into the sub-ppm-range is necessary on the way from reduced to real low activation alloys.

References

- [1] K. Ehrlich, S.W. Cierjacks, S. Kelzenberg, A. Möslang, The Development of Structural Materials for Reduced Long-term Activation, vol. 1270, ASTM Special Technical Publications, STP, 1996, pp. 1109–1122.
- [2] M. Schirra, S. Heger, H. Kempe, M. Klotz, H. Zimmermann, J. Lapeña, Untersuchungen zu physikalischen und mechanischen Eigenschaften der OPTIFER-Legierungen, FZKA report 6167, April 1999, ISSN 0947-8620.
- [3] R. Schneider, P. Würzinger, G. Lichtenegger, H. Schweiger, Metallurgie an den technischen Grenzen höchster Reinheitsgrade und niedrigster Spurenlementgehalte, Berg- und Hüttenmännische Monatshefte BHM 145 (2000) 199-203, ISSN 0005-8912.
- [4] H. Finkler, M. Schirra, Transformation behaviour of high temperature martensitic steels with 8–14% chromium, Steel Research 67 (8) (1996) 328–342.
- [5] R. Lindau, M. Schirra, Erste Ergebnisse zur Charakterisierung einer Großcharge des niedrigaktivierbaren ferritisch-martensitischen Stahls EUROFER 97, Annual Meeting On Nuclear Technology 2000 of the Kerntechnische Gesellschaft, 23–25 May, Bonn, ISSN 0720-9207, pp. 613–616.
- [6] M. Schirra, A. Falkenstein, P. Graf, S. Heger, H. Kempe, R. Lindau, H. Zimmermann, Results of the characterisation of physical and mechanical properties of the martensitic 9% Cr-steel EUROFER 97, FZKA report 6707, in print (partly in German).
- [7] R. Lindau, M. Schirra, First results on the characterisation of the reduced-activation-ferritic-martensitic steel EUROFER, Fusion Eng. Des. 58–59 (2001) 781.
- [8] R. Lindau, A. Möslang, M. Schirra, P. Schlossmacher, M. Klimenkov, Mechanical and microstructural properties of a hipped RAFM ODS-steel, International Conference on Fusion Reactor Material (ICFRM-10), Baden-Baden, 2001, J. Nucl. Mater., in press.
- [9] E. Diegele, A. Möslang, Development of small scale specimens for creep fatigue testing, International Conference on Fusion Reactor Material (ICFRM-10), Baden-Baden, 2001, J. Nucl. Mater., in press.