Available online at www.sciencedirect.com
SCIENCE DIRECT®
Fusion Engineering and Design 75–79 (2005) 1003–1008
Fusion Engineering and Design
www.elsevier.com/locate/fusengdes

Creep strength of reduced activation ferritic/ martensitic steel Eurofer'97

P. Fernández [a,*], A.M. Lancha [a], J. Lapeña [a], R. Lindau [b], M. Rieth [b], M. Schirra [b]

[a] *Ciemat, Avda. Complutense No. 22, 28040 Madrid, Spain*
[b] *Forschungszentrum Karlsruhe, Institut für Materialforschung I, P.O. Box 3640, 76021 Karlsruhe, Germany*

Available online 25 July 2005

Abstract

Creep rupture strength of tempered martensitic steel Eurofer'97 has been investigated. Different products form (plate and bar) have been tested in the temperature range from 450 °C to 650 °C at different loads. No significant differences in the creep rupture properties have been found between the studied product forms. The Eurofer'97 has shown adequate creep rupture strength levels at short creep rupture tests, similar to those of the F-82 H mod. steel. However, for long testing times (>9000 h) the results available up to now at 500 °C and 550 °C seem to indicate a change in the creep degradation mechanism.

Keywords: Ferritic/martensitic steels; Creep properties; Fusion reactor

1. Introduction

Since several years, a new European reference structural material for fusion power systems, the Eurofer'97 steel, is being intensively investigated. The continuous development and qualification of reduced activation tempered martensitic steels for fusion applications require an exhaustive understanding of their microstructure and mechanical properties. Of special relevance is the behaviour of these materials under long term loading conditions at the high temperatures of fusion reactor operation. Consequently, the creep properties of these steels and their microstructural evolution during creep must be studied since the maximum operating temperature of the fusion power plants will be determined, among others, by the creep characteristics [1]. The aim of this work is to evaluate the creep rupture strength properties of the as-received Eurofer'97 steel in different product forms (plate and bar).

2. Experimental

The material investigated is the reduced activation tempered martensitic steel Eurofer'97 which chemical composition is (wt%): 0.11 C, 8.7 Cr, 1 W, 0.10 Ta, 0.19 V, 0.44 Mn and 0.004 S (Ciemat analyses).

* Corresponding author. Tel.: +34 91 3466613; fax: +34 91 3466661.
E-mail address: pilar.fernandez@ciemat.es (P. Fernández).

0920-3796/$ – see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.fusengdes.2005.06.085

The steel was supplied as plates of 14 mm thickness and bars of 100 mm diameter in the normalized plus tempered condition (980 °C/27 min plus 760 °C/90 min followed by air-cooled for plates and 980 °C/110 min plus tempering at 740 °C/220 min/air-cooled for bars).

Constant load creep tests up to rupture were performed in the as-received state at different stress levels and temperatures ranging from 450 °C to 650 °C. The majority of the FZK tests at 600 °C and 650 °C were performed under vacuum in order to avoid high oxidation of the material. Some tests are still running (230 MPa at 500 °C, 150 MPa at 550 °C, 90 MPa at 600 °C and 50 MPa at 650 °C).

Microstructural investigations were performed by TEM in some selected specimens. On the other hand, phase extraction was performed by anodic dissolution of the matrix, both in the gauge length and in the head of the specimens in order to provide data on the influence of ageing under stress during the creep tests in comparison with the ageing without stress. The identification of second phase precipitates in the extracted residues was performed by EDS (SEM) and by X-ray diffraction (XRD).

3. Results

3.1. Creep behaviour

The variation of the minimum creep rate ($\dot{\varepsilon}_{\min}$) with the applied stress (σ) is generally described by a power law so-called Norton equation [2]: $\dot{\varepsilon}_{\min} = k\sigma^n$. This equation describes the creep deformation characteristics because a change of the Norton stress exponent (n) is indicative of a change in deformation mechanism. According to the Norton equation the n exponent of the Eurofer'97 steel was determined for each test temperature, Fig. 1. The examination of the results does not show a creep dependence on the product form (plate or bar) and seems to indicate that at 500 °C and the lowest stress a creep behaviour change has taken place. In addition, the stress-time to rupture curve at 500 °C suggests a slope change at the lowest stress that might be an indication of a sigmoidal creep behaviour. However, additional results at lower stresses would be necessary to determinate the n exponent and to corroborate that the Eurofer'97 steel exhibits two regions of different behaviour. On the other hand, the variation of the n exponent versus $1/T$ appears to be linear

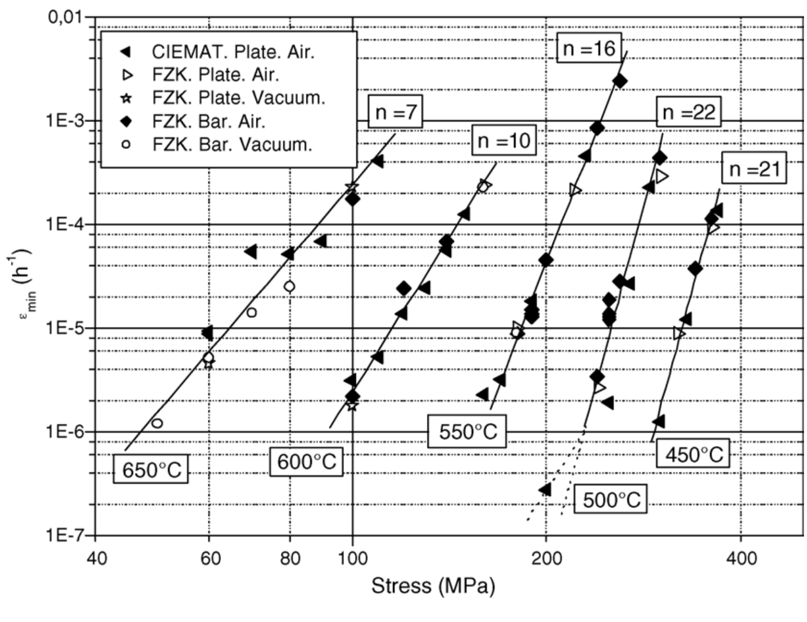

Fig. 1. Minimum creep rate as function of the applied stress for the Eurofer'97 steel.

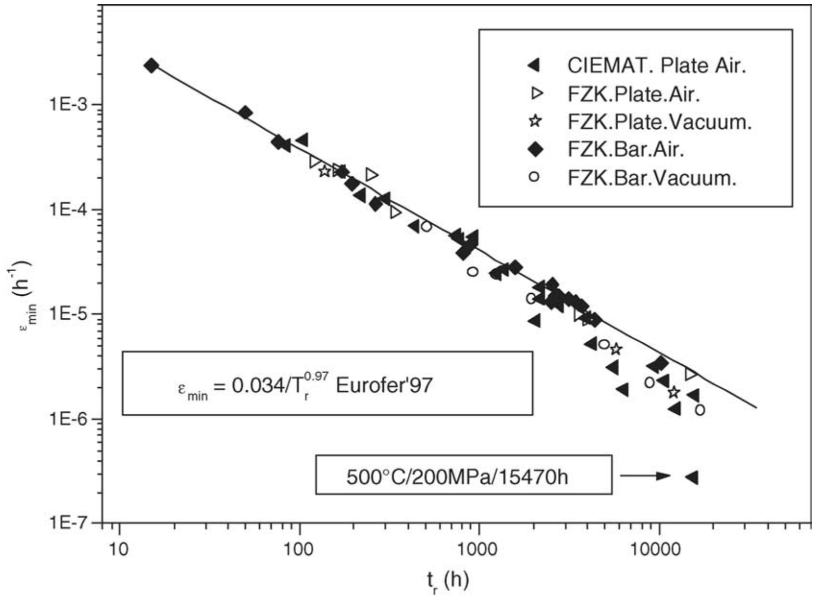

Fig. 2. Monkman–Grant equation plot for the Eurofer’97 steel.

over the temperature range 500–650 °C while the data point corresponding to 450 °C is well out the linear trend of the higher temperatures. Complementary tests at 450 °C are be performing that will permit to confirm this observation.

The relation between the minimum creep rate ($\dot{\varepsilon}_{\min}$) and the time to rupture (t_r) is usually described by the Monkman–Grant relationship [3] in the generalized form: $\dot{\varepsilon}_{\min} = K/t_r^b$. The constant b is usually in the range 0.8–1.2 [4]. The Monkman–Grant equation for the Eurofer’97 steel (Fig. 2) was determined fitted all the creep tests data except the value of 500 °C/200 MPa/15,470 h. In general, for testing times higher than 4500 h, independently of the test temperature, the data do not describe the Monkman–Grant equation with accuracy. The results seem to suggest again a slope change as a consequence of different creep degradation mechanism.

The creep master curve over the Larson–Miller parameter $P = T(C + \log t_r)$ [5] for the Eurofer’97 steel and the F-82H mod. steel for comparison are shown in Fig. 3. As can be seen in the graph, the creep rupture strength properties of both alloys can be considered equivalent. However, it is worth mentioning that all F-82H results correspond to testing times <3000 h.

3.2. Microstructural characterization

The normalizing (980 °C/27 min) plus tempering (760 °C/90 min/air-cooled) heat treatments yielded a fully martensite structure within the fine prior austenite grain (6.7–11 μm size). Two types of precipitates were detected: $M_{23}C_6$ and MX. Detailed microstructural investigations of the Eurofer’97 plates in the as-received condition and after simulated service conditions are described in [6,7]. On the other hand, optical and SEM investigations of the different products forms of the Eurofer’97 steel (plates of 8 mm, 14 mm and 25 mm thickness and bars of 100 mm diameter) did not show any significant microstructural differences [8].

Representative creep ruptured samples tested at 500 °C (270 MPa/1370 h and 200 MPa/15,470 h), at 550 °C (170 MPa/9360 h), at 600 °C (130 MPa/1250 h) and at 650 °C (70 MPa/938 h) were selected for phase extraction and X-ray diffraction studies. The amount of extracted residue in the stressed areas, as well as in the unstressed zones (specimens head), was in general slightly higher (from 2.6 wt% to 2.9 wt%) than in the as-received condition (2.5 wt%), except in the case of the sample tested at 500 °C/200 MPa/15,470 h. In the stressed area of this sample, a percentage of

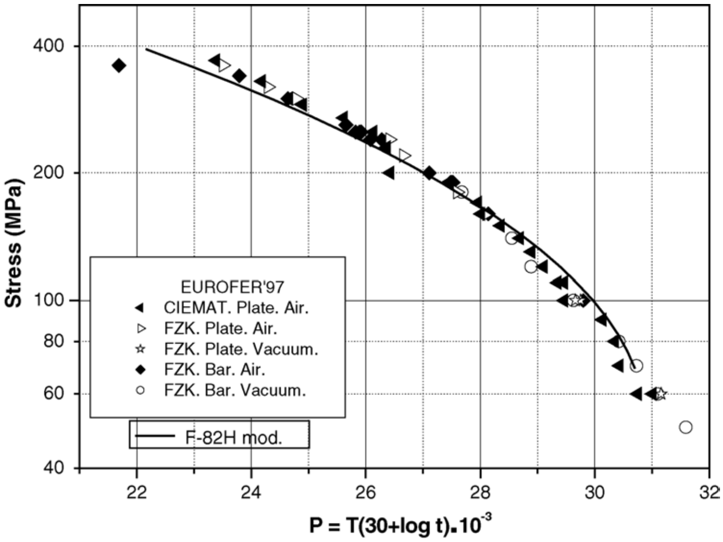

Fig. 3. Comparative Larson–Miller parameter of the Eurofer'97 steel and the F-82H mod. steel in the as-received condition.

extracted residue of 3.9 wt% was obtained. This result suggests that a new nucleation and/or growth of precipitates have occurred during the creep test. The X-ray diffraction patterns of the extracted residues showed the same type of precipitates to those detected in the as-received state, e.g. $M_{23}C_6$ as predominant carbides and precipitates with the same structure than TaC for all specimens [6], excepted for the samples tested at 500 °C/200 MPa/15,470 h and at 550 °C/170 MPa/9360 h, in which besides of these precipitates weak peaks of Fe_3W_3C (M_6C) were also detected. In order to clarify the results of the phase extraction and X-ray diffraction, TEM investigations on thin foils were carried out in the stressed areas of the longest testing times (500 °C/200 MPa/15,470 h and 550 °C/170 MPa/9360 h). These studies revealed the presence of new particles, Fig. 4, tentatively identified according to the X-ray diffraction results as M_6C type (Fe_3W_3C), mainly precipitated at the sub-grain boundaries and in the majority of the cases associated to the $M_{23}C_6$ carbides. Typical EDS spectra of these M_6C and $M_{23}C_6$ carbides are shown in Fig. 4. As it can be seen, the analyses show high Fe and W concentrations in the M_6C type particles. Further microstructural studies are being performing in order to elucidate the influence of the temperature and stress in the precipitation of M_6C particles.

4. Discussion

The Eurofer'97 steel presents adequate creep rupture strength levels similar to the ones of the F-82H mod. steel for short creep rupture tests (<3000 h). Two creep strengthening mechanisms (solid solution strengthening and precipitation strengthening) are generally considered. Regarding to the precipitation strengthening it is accepted [9] that in order to obtain good creep properties, the primary requirement is to obtain a suitable dispersion of the fine particles within the grains of the matrix, because these particles can act as barriers to the movement of dislocation. This seems to be the case of the Eurofer'97 steel whose microstructure contain $M_{23}C_6$ and fine MX particles against to the F-82H mod. with only $M_{23}C_6$ precipitates in its structure. From the point of view of solid solution strengthening mechanism, it is also well established that in these steels the presence of W in the solid solution in concentrations ≥2 wt% [10], as it is the case of the F-82H mod. steel, exerts a strong effect on solid solution creep strengthening. Therefore, both materials have a strengthening mechanism contribution resulting in similar creep rupture properties.

The evaluation of the Eurofer'97 data available up to now at 500 °C and 550 °C by the Norton law suggests that for long testing times (>9000 h) a change

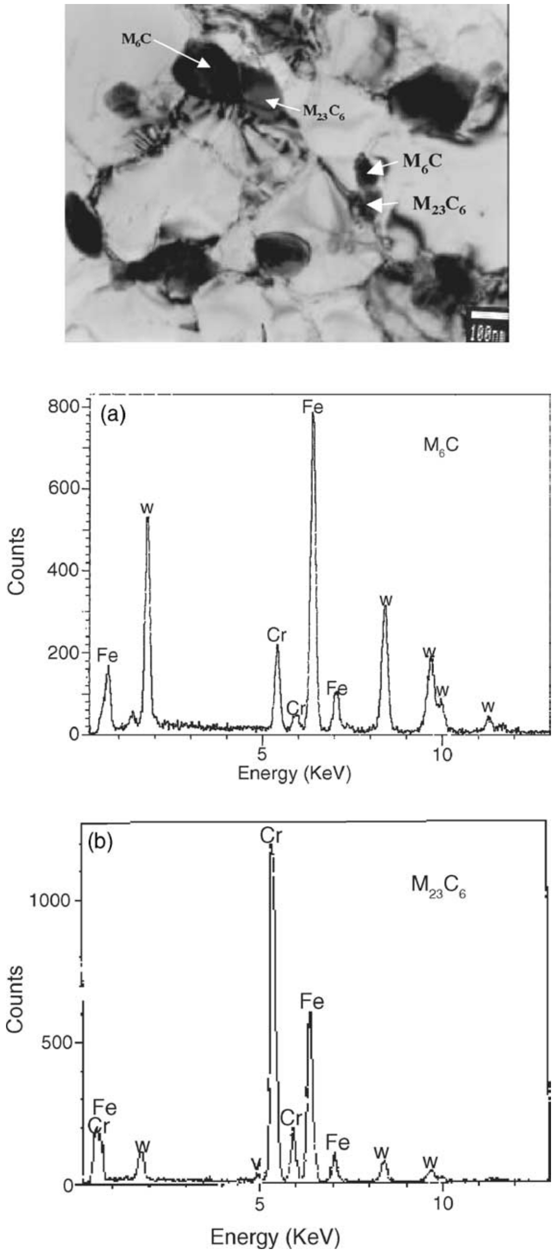

Fig. 4. TEM photomicrograph of $M_{23}C_6$ and M_6C particles in creep specimen tested at 500 °C/200 MPa/15,470 h, and EDS analyses of (a) M_6C and (b) $M_{23}C_6$ precipitates.

in the deformation mechanism could be taking place. The precipitation of M_6C type particles, with high W content, could be the reason of the creep strength degradation of Eurofer'97 steel for long testing times. As W is a solid solution strengthening element, the formation of new precipitates rich in W during the creep tests produces a depletion of W in the matrix. The result is a loss of solid solution hardening that contributes to the deterioration of the creep properties. Similar precipitation of M_6C carbides during creep at 550 °C and long test times has been observed in several casts of 12CrMoVNb steels [11]. The authors indicate that the precipitation of M_6C phase plays an important role in the decrease of creep rupture strength of these steels. These observations are in good agreement with the results obtained in this work for the Eurofer'97 steel.

The long testing results of the Eurofer'97 obtained up to now could question the widely used Larson–Miller parameter as assessment to predict the creep rupture strength of this steel for long times. This parameter assumes that temperature and time can be interchanged, provided no important microstructural changes occur during the creep. Several studies [4,12] performed in 9% Cr steels seem to indicate that the extrapolation through the Larson–Miller parameter is more realistic when rupture times above 3000 h are considered. Ennis and Czyrska-Filemonowicz [4] suggests that it is better for the extrapolation to use the Monkman–Grant equation together with the Norton law for the 9% Cr steels. From the Monkman–Grant plot, the minimum creep rate for a given rupture life can be seen, and from the Norton equation plot the stress which gives rise to this creep can be determined. In the case of Eurofer'97, the results obtained up to now are not enough to perform this estimation with accuracy, clearly additional creep results at low stresses and microstructural investigations are needed.

5. Summary and conclusions

- The creep rupture strength of the Eurofer'97 steel in the normalized plus tempered condition seems to be independent of the product form (plate or bar) in the range of temperatures (450–650 °C) and loads (370–50 MPa) investigated.
- The Eurofer'97 shows adequate creep rupture strength levels at short creep rupture tests (<3000 h). However, for long testing times (>9000 h) the results available up to now at 500 °C and 550 °C point to a change in the creep degradation mechanism.

- The microstructural investigations performed in some selected samples have shown the presence of a new phase rich in W and Fe precipitated during creep, tentatively identified as M_6C type. The formation of these particles seems to be the reason of the creep properties deterioration at 500 °C (200 MPa, 15,470 h) and 550 °C (170 MPa, 9360 h).

References

[1] R.L. Klueh, D.R. Harries, High Chromium Ferritic and Martensitic Steels for Nuclear Applications. ASTM Stock Number: Mono 3, 2001, p. 1.

[2] F. H. Norton, McGraw Hill Publishing Co. Ltd., 1929, p. 67.

[3] F.C. Monkman, N.J. Grant, ASTM 56 (1956) 593.

[4] P.J. Ennis, A. Czyrska-Filemonowicz, Creep resistant steels for power plant, OMMI 1 (1) (2002) 21.

[5] F.R. Larson, J. Miller, Trans. ASME 74 (1952) 765.

[6] P. Fernández, A.M. Lancha, J. Lapeña, M. Hernández-Mayoral, Fusion Eng. Des. 58–59 (2001) 787.

[7] P. Fernández, M. García-Mazario, A.M. Lancha, J. Lapeña, J. Nucl. Mater. 329–333 (2004) 273.

[8] E.W. Schuring, H.E. Hofmans. ECN Report. ECN-C-00-108, October, 2000.

[9] R. Ishii, Y. Tsuda, M. Yamada, M. Miyazaki, Advanced heat resistant steels for power generation, in: R. Viswanathan, J. Nutting (Eds.), IOM Communications, 1999, p. 277.

[10] F. Abe, S. Nakazawa, Mater. Sci. Technol. 13 (1992) 1063.

[11] A. Strang, V. Vodarek, Microstructural stability of creep resistant alloys for high temperature plant applications, in: A. Strang, J. Cawley, G.W. Greenwood (Eds.), IOM Book, vol. 682, 1998, p. 117.

[12] W. Bendick, K. Haarmann, M. Ring, M. Zschau, Proceedings of the Conference 9th International Symposium Creep Resistant Metallic Materials, Hradec and Moravici, Czech Republic, 23–26 September, 1996.