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Thermal creep behaviour of the EUROFER 97 RAFM steel and two European ODS EUROFER 97 steels

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Abstract

Thermal creep tests have been conducted on the EUROFER 97 reduced activation ferritic/martensitic (RAFM) steel and two kinds of oxide dispersion strengthened (ODS) EUROFER 97 steels, which were manufactured using slightly different powder metallurgy techniques at the Centre of Research in Plasma Physics, Switzerland and at the CEA Grenoble, France, respectively. Thermal creep experiments were conducted under constant stress at temperatures between 450 and 750 °C, in an argon flow, up to rupture. Creep exponents as well as activation energies have been determined and evaluated.

Keywords: Reduced activation ferritic/martensitic (RAFM); Oxide dispersion strengthened (ODS) steels; EUROFER 97

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels have proven to be a good alternative to austenitic steels as structural materials in fusion applications for their higher swelling resistance, lower damage accumulation and improved thermal properties. However, irradiated RAFM steels exhibit a low temperature hardening and an increase in the ductile-to-brittle transition temperature, which imposes a severe restriction on the reactor applications at temperatures below about 350 °C [1]. Furthermore, a high density of small cavities (voids or helium bubbles) has been recently evidenced in specimens irradiated with a mixed spectrum of neutrons and protons at about 300 °C to a dose of 10 dPa, which could affect their fracture properties at intermediate temperatures [2]. The upper temperature of use of RAFM steels is presently limited by a drop in mechanical strength at about 550 °C. However, It has been shown that it can be significantly increased by the addition of a fine dispersion of strong particles [3].

The present paper relates to the creep properties of the EUROFER 97 RAFM steel and two oxide dispersion strengthened (ODS) steels, with the EUROFER 97 RAFM steel as matrix material and 0.3 wt.%

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Y_2O_3 particles as reinforcement material, that have been recently developed by two European laboratories within the European Fusion Development Agreement (EFDA) programme.

2. Experimental procedure

2.1. Materials

The EUROFER 97 steel was developed in Europe within the EFDA Programme [4]. Its chemical composition is the following: 8.93 wt.% Cr, 1.07 wt.% W, Mn, V, Ta, Si and C below 1 wt.% in sum total, and Fe for the balance. It was normalized at 980 °C for 0.5 h and tempered at 760 °C for 1.5 h.

Both ODS and EUROFER 97 steels were prepared by powder metallurgy methods at the Centre of Research in Plasma Physics (CRPP, Switzerland) [5] and at the CEA Grenoble, France [6], respectively.

In the case of the CRPP batch, atomised EUROFER 97 has been sieved to particles smaller than 45 μm and mixed with 0.3 wt.% Y_2O_3 particles, 10–30 nm in size, by ball milling for 24 h in an Ar atmosphere. Then, the powder was pre-pressed uniaxially under 35 MPa at 1100 °C for about 7 min, in a rough vacuum (150 mbar) to close as much as possible the porosity, and cooled down. Final compaction was made by hot isostatic pressing (HIPping) under 180 MPa at 1000 °C for 1 h.

In the case of the CEA batch, atomised EUROFER 97 has been sieved to particles smaller than 45 μm and mixed with 0.3 wt.% Y_2O_3 particles by ball milling for 80 h. Compaction was performed by HIPping at 1100 °C for 2 h. The batch was then annealed at 950 °C for 1 h, water quenched and heat-treated at 750 °C for 2 h.

2.2. Creep specimens

Two kinds of creep specimens have been cut out by spark erosion from the three different materials: cylindrical DIN specimens with a diameter of 3 mm and a gauge length of 20 mm, in case of the EUROFER 97 alloy, and cylindrical specimens with a diameter of 2 mm and a gauge length of 7.6 mm in case of both ODS steels, as more restrained quantities of the latter materials were available. Both kinds of specimens had 45° shoulders.

In the case of the EUROFER 97 alloy, creep specimens were cut out from a 25 mm-thick plate (Heat E83697) with their gauge length along the rolling direction, as it is well known that the creep life is shorter if the tensile direction is perpendicular to the rolling direction because of the texture induced by the rolling process. In the case of both ODS steels, specimens were cut out from the prepared batches without any preferential orientation.

2.3. Creep machine

A creep machine was designed to investigate the thermal creep properties of the EUROFER 97 alloy and ODS steels under constant stress using a cam lever (a lever arm with curved profile) that allows accounting for the continuous decrease in cross-sectional area with increasing specimen elongation. The creep machine can be operated between room temperature and 800 °C.

2.4. Creep experiments

Creep experiments were performed in an argon flow, at 550 °C and at various temperatures ranging between 450 and 750 °C in case of the EUROFER 97 alloy and ODS steels, respectively. Three creep tests, corresponding to three different applied stresses, were conducted at each temperature.

3. Results and discussion

3.1. EUROFER 97

Fig. 1 shows creep curves that have been obtained at 550 °C for applied stresses of 285 and 300 MPa. The creep curves appear to be composed of a very short primary stage, characterized by a decreasing strain rate with increasing time, followed by an extended steady state stage and a tertiary stage characterized by an increasing strain rate with time. Then, the rupture takes place. The creep life was found to be about 2 h at 550 °C for an applied stress of 285 MPa. The strain rate corresponding to the steady state stage was found to be about $1 \times 10^{-5}\,s^{-1}$ under 300 MPa. It clearly increases with applied stress. All deformed specimens of EUROFER 97 exhibit necking and were observed to fail in a ductile manner.

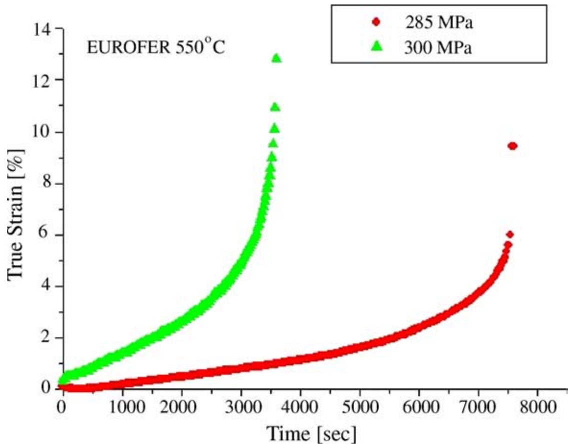

Fig. 1. Examples of creep curves at 550 °C for the EUROFER 97.

3.2. ODS EUROFER 97 steels

Figs. 2 and 3 show examples of creep curves that have been obtained for the CRPP batch and the CEA batch, respectively. Like for the EUROFER 97, they appear composed of a primary stage followed by a steady state stage and a tertiary stage. The primary stage is here better defined. Like for the EUROFER 97 alloy, the strain rate increases with applied stress and temperature and extension of the steady state stage diminishes strongly with increasing applied stress and temperature. For instance, for the CRPP ODS batch, at 650 °C, the creep life was found to be about 29 h (respectively 9 h) for an applied stress of 160 MPa (respectively 200 MPa). The corresponding strain rate associated with the steady state stage was found to be about $6 \times 10^{-7}\,s^{-1}$ (respectively $1.45 \times 10^{-6}\,s^{-1}$).

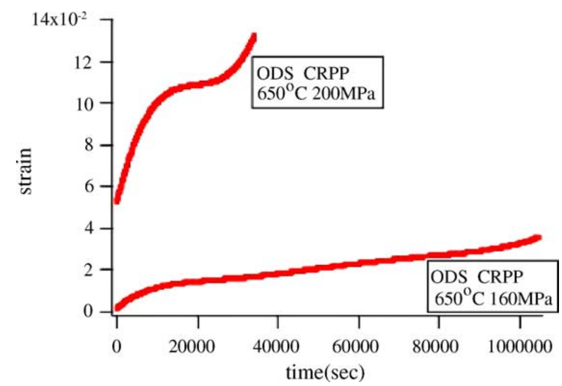

Fig. 2. Examples of creep curves at 650 °C for the CRPP ODS batch.

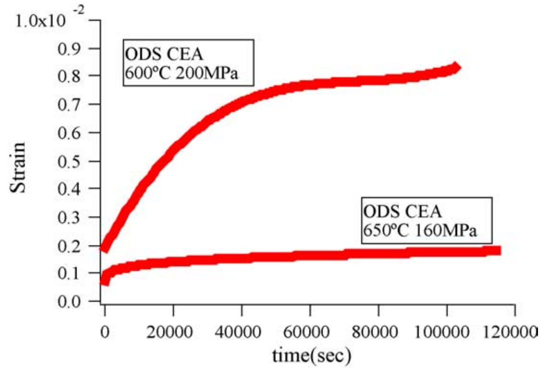

Fig. 3. Examples of creep curves at 650 °C for the CEA ODS batch.

3.3. Comparative analysis

The Larson–Miller parameter, P, was determined for the three sets of data, namely those obtained for the EUROFER 97 alloy and both ODS steels, by using the following formula:

$$P = T_k(30 + \log t_m) \times 10^{-3} \quad (1)$$

where T_k is the test temperature (in Kelvin) and t_m is the creep life (in hours). Results versus applied stress are reported in Fig. 4. It can be seen in Fig. 4 that the creep strength of the CRPP ODS batch is quite similar to that of the CEA ODS batch. The creep strength of both ODS steels is much higher than that of the EUROFER 97 alloy. For a given applied stress value, the Larson–Miller parameter is larger, about 1.5 for

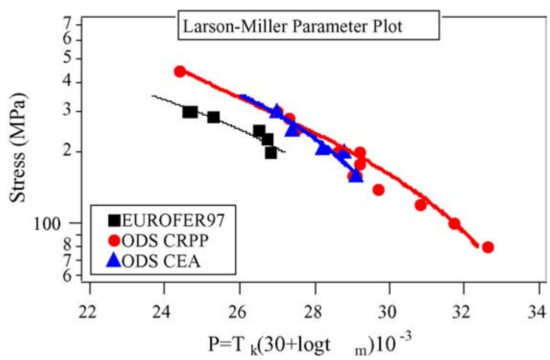

Fig. 4. Larson–Miller parameter (P) plot for the EUROFER 97 alloy and both ODS steels.

both ODS steels with respect to the EUROFER 97 alloy, which corresponds to a temperature increase of about 65 °C for a given rupture time and indicates that both ODS steels could be used up to a maximum temperature that is about 65 °C higher than for the EUROFER 97 alloy. The results obtained in the present study for the EUROFER 97 are in good agreement with the creep strength behaviour, previously evidenced for the same material using a creep machine operating in air under constant load [3]. However, the creep strength of both ODS steels appears slightly lower than that of the ODS steel of same composition produced previously by Forschungszentrum Karslruhe (FZK) in collaboration with Plansee company, Austria [3].

Creep exponents, n, have been determined using the classical Norton creep law that relates the creep rate, $\dot{\varepsilon}$, to the applied stress, σ:

$$\dot{\varepsilon} = A\sigma^n \quad (2)$$

where A is a function of material and temperature. A creep exponent of about 14 was found for the EUROFER 97 alloy tested at 550 °C, while creep exponents in the range 3.9–5.5 were found for both ODS steels tested between 500 and 750 °C. The latter values disagree with the large value of $n = 50$ that has been found for the recrystallized MA957 ODS steel (Fe–14Cr–1Ti–0.3Mo–0.3 wt.% Y_2O_3) tested at 650–700 °C [7] and the values of $n > 15$ and $n = 11$–13, that have been found for the DT2203Y05 ODS steel (Fe–13Cr–2.2Ti–1.5Mo–0.5 wt.% Y_2O_3) tested at 600–700 °C and in the range 500–700 °C, respectively [8,9]. However, these latter materials have been prepared by hot extrusion and exhibit a textured microstructure composed of coarse or at least intermediate-sized, elongated grains of a few tens microns, and creep exponent values are known to be strongly dependent on the grain size [10] in addition to the imposed creep conditions in terms of stress and temperature [8]. In the present study, the grain size was about 5 μm in the case of the CRPP ODS batch [5] and in the range 2–5 and 0.2–0.5 μm (bimodal distribution) in the case of the CEA ODS batch [6].

Values of 4–6 can be expected when creep occurs by the generation and movement of dislocations in the ferrite matrix [8]. So, the values in the range 3.9–5.5 that were obtained in the present study correlate well with a dislocation creep mechanism. The low values obtained also indicate that the strain rate sensitivity to the applied stress of both ODS steels is much lower than that of the EUROFER 97 alloy, which is beneficial to their future use.

The activation energy, Q, has been determined for the CRPP ODS batch using the following equation:

$$\dot{\varepsilon} = A'\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (3)$$

where A' is a constant. An activation energy value of about 0.38 eV was found for the CRPP ODS batch. This value is close to the activation energy for lattice self-diffusion and suggests the occurrence of dislocation climb at obstacles, i.e. the Y_2O_3 particles.

4. Summary

Thermal creep experiments were performed at various temperatures and applied stresses on the EUROFER 97 reduced activation ferritic/martensitic steel and two European oxide-dispersion-strengthened RAFM steels, with the EUROFER 97 RAFM steel as matrix material and 0.3 wt.% Y_2O_3 as reinforcement material, using a laboratory-made creep machine operating under constant stress in an argon flow. It was found that both ODS EUROFER 97 steels exhibit significantly higher creep strength than the EUROFER 97 alloy. Creep exponents in the range 3.9–5.5 were found for both ODS steels and an activation energy of 0.38 eV was measured for the CRPP ODS batch. These values are the characteristic of a dislocation creep mechanism associated with dislocation climb at obstacles, i.e. the Y_2O_3 particles. A creep exponent of about 14 was found for the EUROFER 97 alloy, which indicates that the strain rate sensitivity to the applied stress is much lower for both ODS EUROFER 97 steels than for the EUROFER 97 alloy, which is beneficial to their future use.

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