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Metallurgical properties of reduced activation martensitic steel Eurofer'97 in the as-received condition and after thermal ageing

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Abstract

This paper describes the microstructural studies and the mechanical testing (hardness, tensile and charpy tests) performed on the Eurofer'97 steel in the as-received condition and after thermal ageing treatments up to 600 °C. In addition, fracture toughness tests on the as-received condition have been carried out in order to determine the Master Curve. During the thermal ageing treatments studied (500 °C/5000 h and 600 °C/1000 h) the general microstructure of the steel (tempered martensite with $M_{23}C_6$ and MX precipitates) remained stable. Only a slight growth of the particles has been observed. In terms of mechanical properties, the Eurofer'97 steel exhibited similar values of tensile properties (tensile and yield strength) and ductile–brittle transition temperature regardless of the material condition studied.

1. Introduction

Within the framework of the European Fusion Development Agreement a new experimental reduced activation ferritic/martensitic steel, named Eurofer'97, has been developed as possible first wall and breeder blanket structural material for fusion applications.

According to the criteria adopted in the development of the reduced activation materials, the nominal composition of Eurofer'97 (Fe–9Cr–1WVTa) has been optimized in order to improve the metallurgical properties of these alloys and also to reduce, as low as possible, the concentration of highly radioactive elements (such as Mo, Nb and Ni) from a point of view of waste management considerations and irradiation effects. For this reason, the qualification of this alloy implies an exhaustive knowledge of their microstructural and mechanical properties in the initial metallurgical condition (normalized plus tempered) and after thermal ageing at relevant temperatures for fusion applications.

This paper presents the metallurgical properties (microstructural and mechanical behaviour) of the Eurofer'97 in the as-received condition and after thermal ageing treatments performed in the range 400–600 °C up to 5000 h.

2. Experimental procedure

The material investigated is the reduced activation ferritic/martensitic steel Eurofer'97, with the following chemical composition (wt%): 0.11C, 8.7Cr, 1W, 0.10Ta, 0.19V, 0.44Mn, 0.004S, balance Fe (Ciemat analyses). The steel was supplied as plates in the normalized (980 °C/27′) plus tempered (760 °C/90′/air-cooled) condition, denominated in this paper as the as-received state. To evaluate the metallurgical properties after thermal ageing treatments, the Eurofer'97 was thermally aged in the range of temperatures between 400 and 600 °C during periods up to 5000 h.

Microstructure was characterized by optical microscopy and scanning electron microscopy (SEM).

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Transmission electron microscopy (TEM) investigations of thin foils and carbon extraction replicas were also performed using a 200 kV JEOL device equipped with an X-ray energy dispersive spectrometer (EDS).

Tensile tests were carried out on the as-received and aged materials at 400 and 500 °C up to 1000 and 5000 h, using cylindrical specimens of 5 mm diameter with a gauge length of 25 mm. The as-received specimens were tested at room temperature, 400, 500 and 600 °C. However, in the aged materials apart from the room temperature, the test temperature was the same as that of the ageing treatments. Vickers hardness measurements at 30 kg load were carried out at room temperature on the as-received and aged materials (400 and 500 °C for 1000 and 5000 h). Charpy impact tests were conducted on the as received and aged material at 500 °C for 5000 h, using V-notched specimens according to ASTM Standard E-23. Impact curve was determined over the temperature range from −100 °C to room temperature. The ductile–brittle transition temperature (DBTT) was estimated as 50% brittle fracture mode. It means fracture appearance transition temperature. Fracture toughness tests were performed with 1/2 TCT (1/2″ thickness) specimens following ASTM Standard E 1921-97 in the transition region in order to determine K_{JC} values.

3. Results and discussion

3.1. Microstructural characterization

The microstructure of the Eurofer'97 (Fig. 1) consists of laths of tempered martensite, about 0.5 ± 0.2 μm wide, within fine prior austenite grains. The average prior austenite grain size was ASTM 10–11.5 (6.7–11 μm). A detailed microstructural characterization by optical and SEM of the Eurofer'97 steel on the as-received condition is described in Ref. [1].

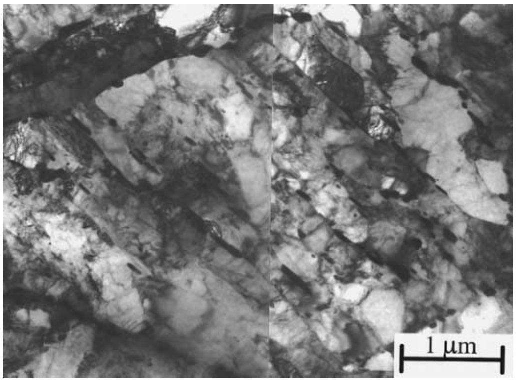

Fig. 1. General microstructure by TEM of the Eurofer'97 steel on the as-received condition.

The tempering treatment produced large amounts of carbide precipitation distributed preferentially along grain and lath boundaries but precipitates appear also in the bulk of the martensite laths. The main precipitation consists of $M_{23}C_6$ precipitates of variable size (∼25–210 nm) located preferentially along grain and lath subgrain boundaries. Their morphology varied from globular to plates or to irregular geometrical shapes, and their typical atomic concentration, obtained from extraction replicas, was 66 ± 1Cr/31 ± 1Fe/1.9 ± 0.2 W, though in some analyses Ta and/or V were found to replace W. Furthermore, in other analyses V was also detected in addition to Cr, Fe and W.

In the as-received condition other types of precipitates (MX type) rich in Ta or V, with a size in the range from ∼8 to 40 nm, have been identified. They are mainly located inside the subgrains. It is well known that MX type precipitates are considered to be very useful for long term creep resistance at elevated temperatures. Three types of MX morphologies have been identified in the steel after tempering (Fig. 2). Type I is a spherical Ta or V rich MX, this type being the most numerous. Type II is a fine V-rich precipitate with a plate shape, and Type III represents a specific morphology that is formed by secondary V precipitation at expense of the Ta rich particles, so called V-wing. Abe and co-workers have observed these three types of MX morphologies in CrWNb steels after tempering [2]. These authors also

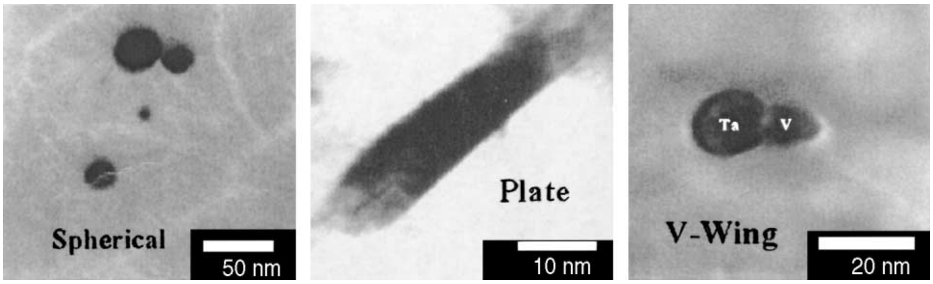

Fig. 2. Morphologies of the MX precipitates in the Eurofer'97 steel.

report that the Nb(C,N) formed during the normalization treatment has a region rich in V on the surface and consequently these precipitates can act as nucleation sites during tempering, growing the VX like a wing.

EDS analyses performed on carbon extraction replicas in the as-received condition showed that Ta rich precipitates contain about 60–80%Ta and 20%V (at.%). In the case of V rich precipitates, V concentration is ~70% and Ta ~15% (at.%). Precipitates with equal amounts of Ta and V (~45 at.%) were also observed. In addition, Fe and Cr were generally detected in these analyses.

After thermal ageing treatments at 500 °C/5000 h and 600 °C/1000 h no significant microstructural changes were observed by TEM. The average width of the martensite laths was 0.6 ± 0.2 μm for the aged material at 500 °C and 0.5 ± 0.2 μm for the aged material at 600 °C. The morphology of each type of precipitates, $M_{23}C_6$ and MX, was the same than in the as-received condition. However, a slight growth of both types of precipitates was observed (Fig. 3(a) and (b)). The range of $M_{23}C_6$ particle diameter varied from ~40 to 260 nm for 500 °C/5000 h and from ~40 to 300 nm for 600 °C/1000 h. In the case of MX precipitates the range size was ~10–60 nm and ~10–100 nm for 500 °C/5000 h and 600 °C/1000 h, respectively.

The effects of the ageing in the chemical composition of the precipitates were as follows. At 500 °C/5000 h the $M_{23}C_6$ composition was similar to that found in the as-received condition, with the same elements and concentrations. However, in the case of the material aged at 600 °C/1000 h the $M_{23}C_6$ carbides presented a higher Cr and W concentrations (~71 and ~3.5 at.% respectively) than in the previous conditions and a lower Fe content (~26 at.%). Similar trends in the Cr and Fe values of the carbides has been observed by other authors [3,4] as a result of ageing treatments at 550 and 600 °C for long times. Regarding the MX type precipitates, the most important finding was that after the ageing treatments at 500 and 600 °C Ta pure particles were found that were not seen in the as-received condition.

3.2. Mechanical properties

3.2.1. Tensile properties and hardness measurements

Fig. 4 shows the tensile properties of the Eurofer'97 steel for the as-received condition and after ageing at 400 °C and 500 °C for times of 1000 and 5000 h. Tensile tests were conducted at room temperature and ageing temperature for all samples. The ultimate tensile strength and the yield strength at room temperature were 662 and

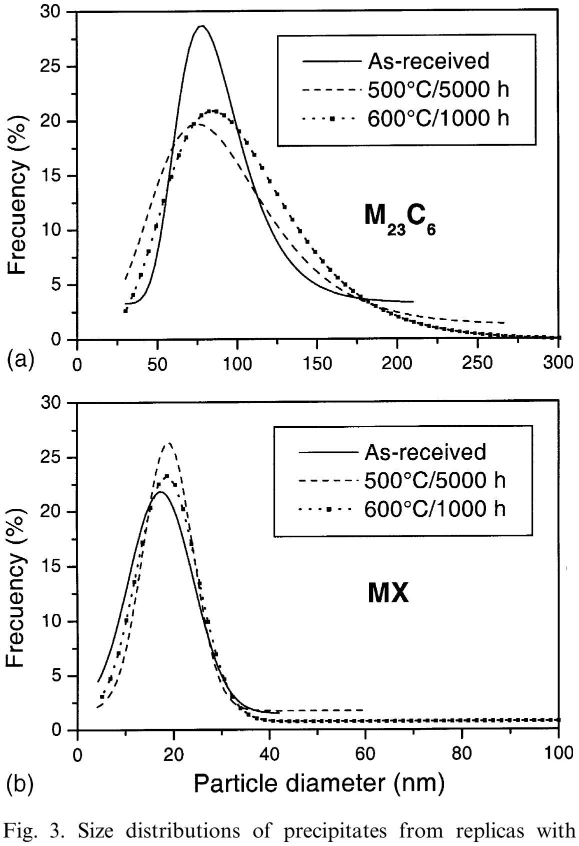

Fig. 3. Size distributions of precipitates from replicas with differentiation of particles and material conditions (a) $M_{23}C_6$ and (b) MX.

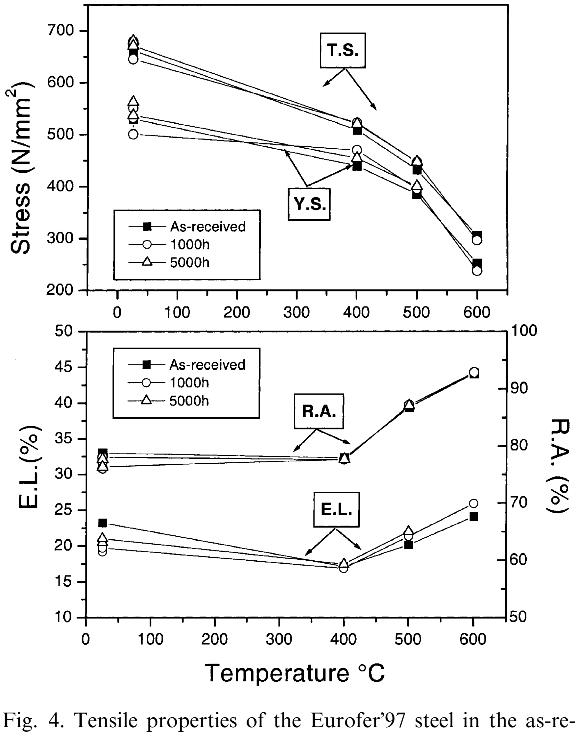

Fig. 4. Tensile properties of the Eurofer'97 steel in the as-received condition and after ageing at 400 and 500 °C.

530 MPa respectively. The Eurofer'97 exhibits adequate strength and ductility levels, comparable to other reduced activation ferritic/martensitic steels such as JLF-1 [5] and the F-82H modified steel [4]. As shown in the figure, the main result obtained in these tests was that for the same test temperature no degradation of tensile properties (ultimate tensile strength and yield strength) on aged material was detected, comparing to the as-received state. Similar behaviour after thermal ageing treatments was also observed in the reduced activation F-82H modified steel [4]. In addition, it can be observed in the graphs that time of ageing has no influence in these mechanical properties.

The Eurofer'97 steel showed a Vickers hardness value of 210 ± 3 (HV30) in the as-received condition. After ageing treatments (400 and 500 °C for times of 1000 and 5000 h) this value did not change significantly 212 ± 4 (HV30). These hardness results together with the microstructural stability observed can be considered the main reasons of the tensile behaviour of the Eurofer'97 after ageing.

3.2.2. Impact tests

Charpy impact tests were conducted on the as-received and the thermally aged material at 500 °C for 5000h in order to determine full impact curves, Fig. 5. In the as-received state the Eurofer'97 exhibited a DBTT, estimated at the 50% brittle fracture mode, of about −51 °C and an upper shelf energy (USE) of 266J. As shown in Fig. 5, the DBTT stayed constant after ageing and the USE decreased, approximately 30 J. The Eurofer'97 exhibits a better toughness after tempering and ageing than other reduced activation ferritic/martensitic steel, like F-82H modified [1], which exhibits values of DBTT around −25 °C. The DBTT differences between the two alloys are basically attributable to the finer prior austenite grain size of the Eurofer'97 due to the Ta (0.10%) and V (0.19%) concentration compared to the F-82H modified (0.005% Ta and 0.14% V). Another important element from a point of view of impact properties is the W concentration. It is known [6,7] that the optimum impact values are obtained with W concentration around 1%, as in the case of Eurofer'97 and in contrast to the F-82H modified, whose W content is 2%.

3.2.3. Fracture toughness tests

Fracture toughness tests have been performed in the Eurofer'97 on the as-received condition in order to determine the T_0 reference temperature. The Master Curve approach was developed by VTT [8] and it is based on the statistical analysis of cleavage fracture data. Following this approach, dependence of fracture toughness with temperature can be described by

$$K_{JC(med)} = 30 + 70\exp(0.019(T - T_0)),$$

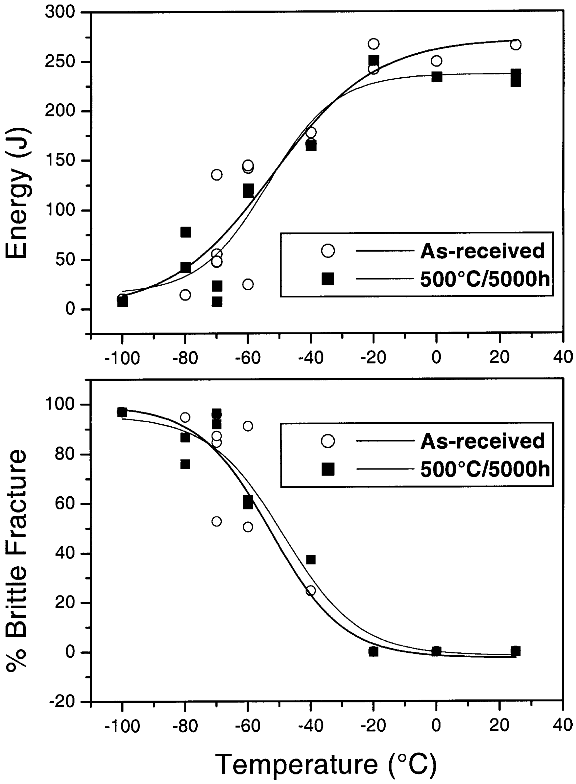

Fig. 5. Impact curves for the Eurofer'97 steel in the as-received condition and after ageing at 500 °C for 5000 h.

where $K_{JC(med)}$ is the median value of the Weibull distribution that describe the scatter of fracture toughness results. T_0 is the reference temperature at which $K_{JC(med)}$ has a value of 100 MPa $\sqrt{m}$ for a 1″ specimen thickness. Master Curve has been extensively used for ferritic materials [9–11] and recently it is also used for ferritic/martensitic steels [12]. T_0 reference temperature for Eurofer'97 has been determined following the multi-temperature technique included in ASTM E1921 May 2000 Draft. Experimental fracture toughness results and Master Curve is presented in Fig. 6. The scatter of fracture toughness tests is the normal found in this type of test. It is not so clear that the slope of the Master Curve can fit the data points. Due to the scarce tests at high and low temperature, a clear conclusion can not be established and more tests should be performed.

However, Odette [12] assumes that Master Curve could be applied for static loads with fracture in or near the small scale yielding (SSY) regime for the F82H steel. This assumption could be applied to Eurofer'97 fracture toughness tests as they have been performed with static load and with a geometry (1/2 TCT, $a/W = 0.5$) that would assure SSY regime.

In order to clarify if the Master Curve developed for ferritic steels could be applied to martensitic steels,

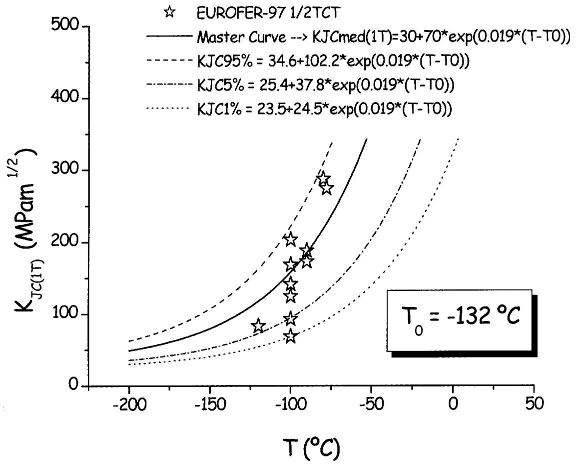

Fig. 6. Multi-temperature Master Curve of the Eurofer'97 steel in the as-received condition.

CIEMAT is performing fracture toughness tests with other martensitic steel (EM10 steel). Fracture toughness tests are being carried out with the same specimen geometry and loading rate than the Eurofer'97 tests. Analysing a similar number of testing performed on the same temperatures range, the tendency for EM10 is similar to that seen in this paper for the Eurofer'97 steel, that is the slope of the fracture toughness tests seems to be steeper that the predicted by the Master Curve. However, when more fracture toughness tests are performed at higher temperatures, always in the transition region, the Master Curve fits well the fracture toughness in the transition region for EM10 steel. These results (to be published) suggests that more fracture toughness tests at higher temperature, in the transition region, with the Eurofer'97 steel should be performed in order to confirm that the Master Curve can be applied.

4. Summary and conclusions

The Eurofer'97 in the material conditions investigated is a fully martensitic steel with $\sim 0.5 \pm 0.2$ μm wide martensite laths. Two kinds of precipitates with different morphologies, namely $M_{23}C_6$ and MX (Ta or V rich), have been detected on the as-received condition as well as in the aged material conditions studied (500 °C/5000 h and 600 °C/1000 h). After the ageing treatments a slight growth of both types of precipitates has been observed.

The ageing treatments studied in this work have no influence in the tensile properties and DBTT.

The slope of fracture toughness versus temperature for the Eurofer'97 steel seems to be steeper than the predicted with the Master Curve.

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