CYCLIC SOFTENING OF EUROFER 97 AT ROOM TEMPERATURE-MECHANICAL AND MICROSTRUCTURAL BEHAVIOUR

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ABSTRACT

The quenched and tempered reduced-activation ferritic/martensitic steel EUROFER 97 subjected to cycling at room temperaure has been studied. Under Low-Cycle Fatigue (LCF) test this steel shows, after the first few cycles, a pronounced cylic softening accompanied by microstructural changes such as the decrease of the dislocation density inside the subgrain. During LCF tests, the softening seems to be governed by a mechanism independent of the plastic strain range imposed to the specimen. From the analysis of the peak tensile stress of the hysteresis loops and its respective correlation with the transmission electron microscopy observations can be concluded that the cyclic softening observed at room temperature could be attriuted to the progressive annihilation of dislocactions located in the interior of the subgrains.

Keywords: Cyclic softening, Ferritic/Martensitic Steels, Low-cycle fatigue, Room temperature, Microstructure.

ABLANDAMIENTO CICLICO DE EUROFER 97 A TEMPERATURA AMBIENTE-COMPORTAMIENTO MECANICO Y MICROESTRUCTURAL

RESUMEN

El acero ferrítico/martensítico de activación reducida templado y revenido EUROFER 97 sujeto a ciclado a temperatura ambiente fue estudiado. Bajo ensayos de fatiga de bajo número de ciclos (LCF) este acero presenta, después de los primeros ciclos, un pronunciado ablandamiento cíclico acompañado por cambios microestructurales como la disminución en la densidad de dislocaciones en el interior de los subgranos. El ablandamiento cíclico durante los ensayos de LCF aparenta estar gobernados por un mecanismo independiente del rango de deformación plástica aplicado a las muestras. Del análisis del pico de tensión de tracción de los lazos de histéresis y su respectiva correlación con las observaciones en el microscopio electrónico de transmisión se concluye que el ablandamiento cíclico observado a temperatura ambiente podría ser atribuido a la progresiva aniquilación de las dislocaciones pertenecientes al interior de los subgranos.

Palabras claves: Ablandamiento cíclico, Aceros ferríticos/martensíticos, Fatiga de bajo número de ciclos, Temperatura ambiente, Microestructura.

INTRODUCTION

The fast decay characteristics of induced radioactivity imposed on candidate steels of fusion reactors have led to the development of the reduced activation ferritic/martensitic (RAFM) steels. The Fe-(7-9%)Cr ferritic/martensitic steels like the European heat EUROFER 97 is one of the most promising alloys because of their better irradiation resistance as well as a high creep-rupture strength. Cyclic straining processes can lead to microstructural changes causing cyclic hardening-softening of the material. This effect could become a significant engineering problem affecting creep, swelling and segregation phenomena during irradiation.

Although there are several works in the literature about low cyclic fatigue of martensitic stainless steels at high temperature revealing a cyclic softening trend [1-7], relatively little information has been found about low-cyclic fatigue of martensitic stainless steels at room temperature. A micromechanical model has been proposed for predicting both microstructure evolution and macroscopic softening of such materials [3] cycled at high temperature. This model was based on the disappearance of Low-angle Boundaries (LaBs) due to annihilation between mobile impinging dislocations and LaB dislocations. From a microstructural point of view, it has been observed that the subgrain size increases during cyclic test [5–7]. However, the present paper reports the importance of the dislocation arrangement evolution during cycling in the interior of the subgrains. In addition, this paper shows that there are no appreciable differences in the subgrain size between the different samples, which were cycled at room temperature and with plastic strain ranges 0.2%, 0.3% and 0.6%.

The aim of this work is to investigate the microstructural evolution during Low-Cycle Fatigue (LCF) tests at room temperature and its effect on the cyclic behaviour of the RAFM steels EUROFER 97.

MATERIAL AND METHODS

The material used in this study was EUROFER 97. Table 1 shows its chemical composition. The final applied heat treatment consisting in: austenitizing at 980^{0}C for 30 min, followed by air cooling and tempering at 760^{0}C for 90 min, and subsequent air cooling.

LCF tests have been performed at both room temperature and 450^{0}C in an electromechanical INSTRON testing machine, operating under plastic strain control using a triangular wave form. The high temperature test was performed in order to compare its behavior during LCF with the behavior registered in the room temperature tests. The selected plastic strain ranges were 0.2%, 0.3% and 0.6% with a total strain rate of, approximately, $3x10^{-3}s^{-1}$. Cylindrical specimens were machined with a diameter of 8.8 mm and a length of 77 mm to be tested in the INSTRON machine. The gage length of the axial extensometer is 21mm.

Specimens were examined by transmission electron microscopy (TEM) using a Philips EM 300 microscope operating at 100kV. Transversal disks were electrolytically polished and finally thinned for the electron microscopy observations. Thin foils of 2-mm diameter were prepared using double jet-polishing technique.

Table 1. Chemical composition of the ferritic/martensitic steel Eurofer 97.

ALLOYING ELEMENT	WEIGHT PERCENT
C	0.12
N	0.018
Si	0.06
Mn	0.47
Ni	0.022
Cr	8.93
Mo	0.002
Al	0.01
V	0.2
Ti	0.01
Nb	0.0022
Cu	0.004
W	1.1
Ta	0.14

RESULTS AND DISCUSSION

Mechanical test results:

Figure 1 shows the cyclic hardening-softening curves, one of them cycled at room temperature and the other one at 450ºC, both with plastic strain range of 0.2%. For a better and easier comparison, the high temperature curve was shifted toward the room temperature one, represented in the figure by an arrow. As can be seen in this figure, the test performed at 450ºC requires lower tensile stresses. However, in the semi-log diagram both curves present quite similar behavior. That is, a transitional stage corresponding to the first part of the fatigue life followed by an almost linear second stage. After this linear stage both curves show a stabilization period before the failure of the sample.

The most important feature of Figure 1 is that, the linear stage of each curve runs almost parallel to the other one. As a result, the linear softening stage seems not to be highly dependent on the temperature during the low-cyclic fatigue tests. Because of this, it can be rationalized that the principal cyclic softening mechanisms of this steel is independent of the temperature.

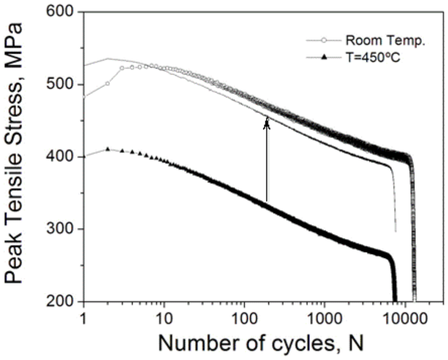

Fig. 1. Evolution of the Peak Tensile Stress versus Number of Cycles during LCF testing of EUROFER 97 cycled at room temperature and at 450ºC with plastic strain range of 0.2%.

Figure 2 shows the cyclic hardening-softening curves obtained at room temperature with plastic strange ranges of 0.2%, 0.3% and 0.6%. In the semi-log diagram, all the curves show also similar trends with a transitional first stage that is followed by the secondary linear stage. After this linear stage, the peak tensile stress of the hysteresis loops reachs an almost stable stage that continuous up to the failure of the sample. The first stage depends on the plastic strain range being shorter for the higher plastic strain range.

The striking feature of Figure 2 is that the linear stage of each curve, before the satabilization stage, runs almost parallel to the other ones. Consequently, the linear softening stage seems to be governed by a mechanism independent of the plastic strain range imposed to the specimen.

TEM observations:

Previous workers [8] have reported the microstructural stability of normalized and tempered 9 – 12% Cr modified martensitic steels during subsequent annealing times up to 1100 hours at 550^{0}C. However, this apparent microstructural stability could be destabilized under cyclic strain conditions as was shown by Armas et al. [9] in modified 9Cr-1Mo steels. The apparently stable lath martensite structure is strongly unstable under cyclic conditions being gradually replaced by the development of a cell structure. The evolution of the typical martensite lath structure of low-carbon alloy steels to a cell structure was reported [10] to be already established after few cycles.

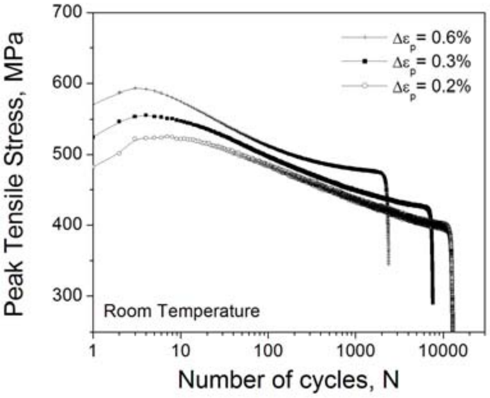

Fig. 2. Evolution of the Peak Tensile Stress versus Number of Cycles during LCF testing of EUROFER 97 cycled at RT with plastic strain ranges of 0.2%, 0.3% and 0.6%.

Figure 3 shows the as received material. As can be seen, prior austenitic grains are decorated by lines of carbides, the structure is composed mainly of sub micrometric equiaxed subgrain and the dislocation density inside the subgrains is high.

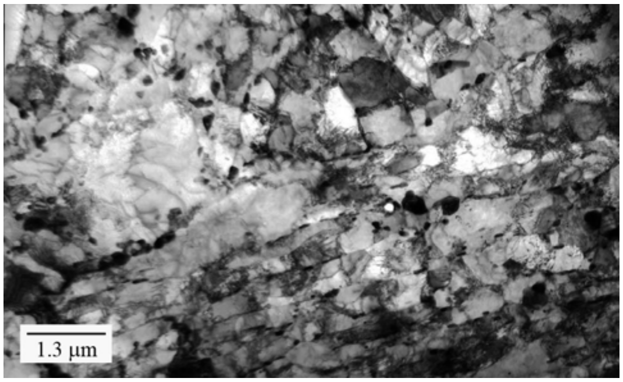

Fig. 3. TEM micrograph of the as received material.

Figure 3 documents that each micro subgrain contains dislocation. However, dislocation densities can vary, not only between different subgrains but also within one subgrain. Elongated martensitic laths are rather rare. There exist also larger grains, up to several microns in diameter, which are not fractioned into clearly developed subgrains or cells. There are at least two types of precipitates: fine ones which are more or less regularly distributed in the grains and large ones found mainly on the boundaries or more commonly on the triple points.

Transmission electron microscopy observations show that the original tempered martensite lath structure evolved to a recovered subgrain structure of lower dislocation density. Figures 4(a), (b) and (c) are the characteristic microstructures developed at room temperature at plastic strain ranges of 0.2%, 0.3% and 0.6%, respectively. Cycling at room temperature induces two visible microstructural evolutions. First and the most evident, total dislocation density decreases during cycling (compare Fig. 3 with Figs. 4(a), (b) and (c)). Second, some low-angle boundaries disappear during cycling. This microstructural evolution is similar to previous results mentioned in the literature obtained for high temperature [5].

It should be noted that subgrains (i.e. grains with the misorientation comparing to their neighbors smaller than 5º, formed during the thermal treatment of the material) and fatigue cells (cells created by the dislocation activity on several crystallographic planes during the cyclic plastic deformation, resulting into small almost equiaxed volumes separated by dislocation walls having a small angle of disorientation with neighbors cells) are essentially the same objects, it is not possible to track their origin and therefore the designation of these objects is only a question of terminology.

As can be seen in these micrographs, the density of the dislocations located in the interior of the subgrains had a significant decline. Dislocations may be observed mainly in small grains, suspected not to be deformed plastically, or inside some grains attached and pinned by precipitates.

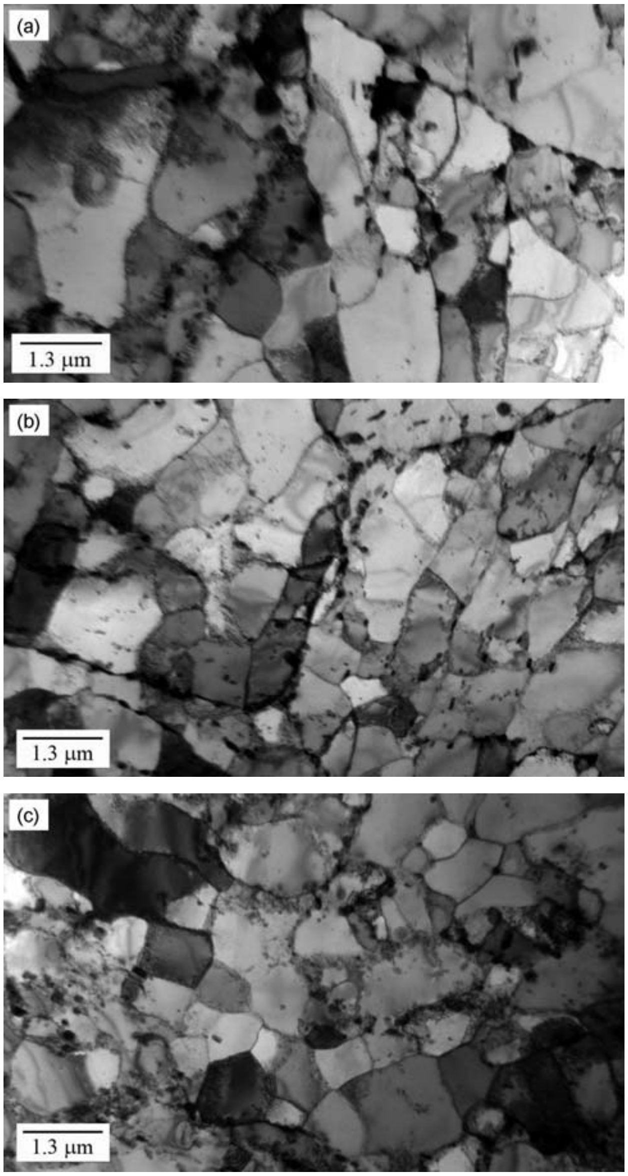

Fig. 4. TEM micrographs after a test up to rupture performed at room temperature under plastic strain ranges of 0.2% (a), 0.3% (b) and 0.6% (c).

Despite the differences in plastic deformation, there are no appreciable differences between the samples neither in the subgrain size nor in the dislocation density in the interior of the subgrains, at least subgrains do not increase their size. The fact that the different micrographs in Figure 4 show a similarity related to the absence of dislocations inside the subgrains and the subgrain sizes is consistent with the mechanical results, in which all the curves (Fig. 2) show similar trends. As expected, the fact that the samples do not show significant differences in the LCF tests is reflected in the microstructure itself. From these results it can be concluded that the cyclic softening rate observed in this steel is almost independent of the plastic strain range.

Comparing Figures 3 and 4, it can be concluded that the major effect produced by cycling at room temperature is a "cleaning out" of the dislocations inside the subgrains. According to this result, the cyclic softening observed at room temperature could be attributed to the progressive annihilation of dislocations located in the interior of the subgrains.

Unlike most workers who refer to the elimination of the subgrain boundaries or LaBs as the main cause of cyclic softening, in this paper is proposed as the main cause of softening to the progressive annihilation of dislocations inside the subgrains. This mechanism could be independent of the temperature and the plastic strain range imposed to the specimen, at least up to 450ºC.

CONCLUSIONS

Reduced activation steel EUROFER 97 presents a cyclic softening behaviour which differ from the behaviour observed in commercial steels. In order to study the microstructural changes during room temperature fatigue and its influence on the softening, low cycle fatigue tests were perfomed with three different plastic strain ranges. The main results obtained are the following:

1) The cyclic softening seems to be independent of the temperature during the low-cyclic fatigue tests.

2) The major effect produced by cycling at room temperature is a "cleaning out" of the dislocations inside the subgrains.

3) Cyclic softening rate observed in this steel is almost independent of the plastic strain range.

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