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Mechanical and microstructural behaviour of isothermally and thermally fatigued ferritic/martensitic steels

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Abstract

Isothermal low cycle fatigue (LCF) and thermal low cycle fatigue results in the temperature range between room temperature and 823 K of the quenched and tempered reduced activation ferritic/martensitic steels F82H mod. and EUROFER 97 are reported. Under these test conditions both steels show, after the first few cycles and for both types of tests, a pronounced cyclic softening up to failure. The softening during LCF tests described by a simple empirical relationship is dependent on temperature but independent of the total strain amplitude of the tests. From the analysis of the hysteresis loops and corroborated by electron microscopy observations it can be concluded that the cyclic softening is produced by the softening observed in the internal stress as a consequence of the evolution of the microstructure. During cycling, the martensitic lath structure with high dislocation density and carbides along the lath interfaces evolves to a softer dislocation subgrain structure. This conclusion could be correlated with transmission electron microscopy observations.

1. 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 (RAF/M) steels. The Fe–(7–9)Cr ferritic/martensitic steels like the Japanese F82H mod. and the European heat EUROFER 97 are the most promising alloys because of their better irradiation resistance as well as high creep-rupture strength. Creep-rupture studies on tempered martensitic steels have shown that these materials can be microstructurally unstable, particularly when they are used for extended periods of time at temperatures of 823 K or higher [1,2]. During operation first wall structural materials would be subjected to cyclic loading at high temperatures. Therefore, in order to use these steels for that purpose, fatigue properties must be better understood. Cyclic straining processes can lead to microstructural changes causing cyclic hardening and/or softening of the material. This effect could become a significant engineering problem affecting creep, swelling and segregation phenomena during irradiation. While the occurrence of cyclic softening in quenched-and-tempered 9–12% Cr steels has been well documented [3–5], the origin of the effect and the kinetics of the softening behaviour are not well understood.

The purpose of the present research is to investigate the microstructural instability during thermal (TCF) and isothermal low cycle fatigue (LCF) tests and its effect on the cyclic behaviour of the RAF/M steels F82H mod. and EUROFER 97. The study is performed on tests carried out at temperatures up to 823 K.

2. Experimental details

The materials used in this study were two RAF/M steels: F82H mod. and EUROFER 97. Table 1 shows

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Table 1
Chemical composition of the ferritic/martensitic steels

	C	N	Si	Mn	Ni	Cr	Mo	Al	V	Ti	Nb	Cu	W	Ta
EUROF 97	0.12	0.018	0.06	0.47	0.022	8.93	0.002	0.01	0.2	0.01	0.0022	0.004	1.1	0.14
F82H mod.	0.09	0.007	0.11	0.16	0.019	8.16	0.002	0.02	0.15	0.002	0.0001	0.006	2.2	0.02

the chemical composition of both steels. The F82H mod. steel was normalised at 1313 K and tempered at 1023 K for 2 h, and EUROFER 97 was normalised at 1253 K and tempered at 1033 K for 1.5 h. The microstructure consists of tempered martensitic laths with a substantial dislocation structure [2]. Carbides, presumably $M_{23}C_6$ ranging from 0.05 to 0.5 μm in size, were distributed preferentially along lath and prior austenitic grain boundaries. It should be noted that a high density of dislocations produced during quenching of the steel remains after tempering. LCF tests have been performed with a servohydraulic MTS as well as an electromechanical INSTRON testing machine, operating under strain-controlled conditions using a triangular wave form. Total strain ranges were performed at 0.5%, 0.6%, 1.0% and 1.5% with a total strain rate 3×10^{-3} s^{-1}. Solid specimens of 77 mm length and 8.8 mm diameter in the cylindrical gauge length have been deformed. The gauge length of the axial extensometer was 21 mm.

The TCF test rig consists of a stiff load frame for mechanical clamping of the sample which is directly heated by the digitally controlled ohmic heating device. Hollow specimens were used with similar outer dimensions as that described above, but with a wall thickness of 0.4 mm. The tests were performed by cycling the temperature of the specimen between T_L (lower temperature) and T_H (higher temperature). The temperature rate was kept constant at 5.8 K/s for all thermal conditions. Afterwards, specimens were examined by transmission electron microscopy using a 100 kV microscope. Transversal disks and shells cut from the solid and hollow specimens, respectively, were electrolytically polished and finally thinned for the electron microscopy observations.

3. Results and discussion

Four sets of cyclic softening curves obtained upon LCF testing of F82H mod. specimens at RT, 523, 723 and 823 K are shown in Fig. 1. For clarity only curves obtained with total strain range 0.6–1.0% are shown at each temperature. In the log–log diagram all the sets show similar tendencies, that is, a transitional stage corresponding to the first part of the fatigue life followed by a linear second stage that occupies the main part of the life, after which the failure occurs. The linear or main stage of the curves is almost independent of the

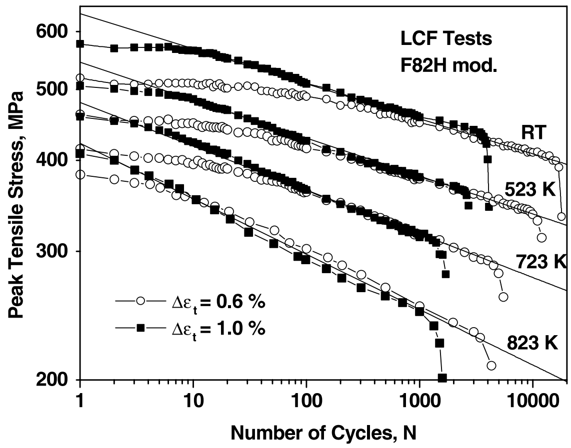

Fig. 1. Evolution of the peak tensile stress versus number of cycles during LCF testing of F82H mod.

total strain range at each test temperature. This stage follows an analytical expression of the type:

$$\sigma = AN^{-S}, \tag{1}$$

where σ is the peak tensile stress of the hysteresis loop, N the number of cycles, S the cyclic softening coefficient and A is a constant that depends on temperature. S, equal to the slope of the linear stage in the log–log diagram, represents the cyclic softening.

The first stage, occupying only a few cycles of the specimen life (less than 3%), depends on the total strain range. Indeed, the higher the strain amplitude of the test, the higher the stress values and the shorter the stage duration. Therefore, it can be concluded that the tempered martensitic steel will soften gradually during cyclic loading and eventually reach a saturation condition dependent only on the test temperature. This saturation condition will be reached in a degree of approximation that is independent of the strain amplitude.

Cyclic softening curves obtained after LCF loading of EUROFER 97 steel specimens at 723 and 823 K are shown in Fig. 2. Similar behaviour as in F82H mod. is observed in this steel. In comparison with F82H mod., a higher number of cycles to failure can be detected.

The second stage of each curve set of Figs. 1 and 2 was approximated by an expression similar to Eq. (1). The pre-exponential factor A and the cyclic softening coefficient S for both steels cycled at the referred temperature are listed in Table 2.

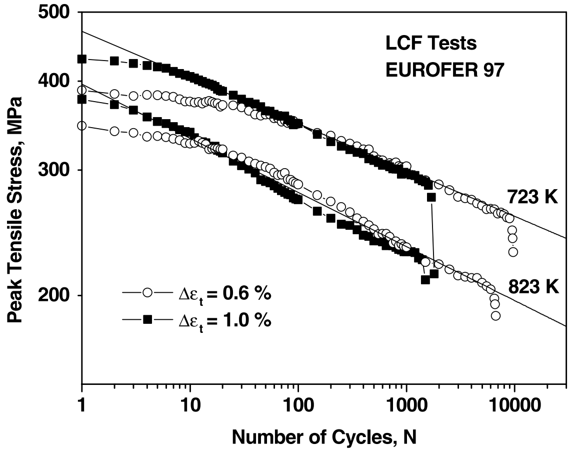

Fig. 2. Evolution of the peak tensile stress versus number of cycles during LCF testing of of EUROFER 97.

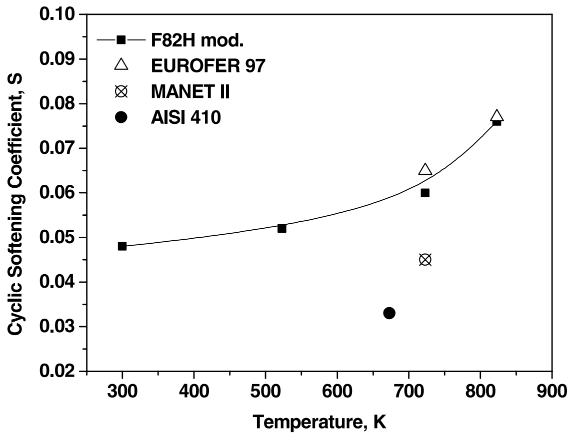

Fig. 3. Cyclic softening coefficient versus temperature for several ferritic/martensitic steels.

Table 2
Pre-exponential factor A and cyclic softening coefficient S

	RT	523	723	823
F82H mod.				
A	635	545	480	422
S	0.048	0.052	0.060	0.076
EUROF 97				
A			470	396
S			0.065	0.077

The cyclic softening coefficient, S, for F82H mod. and EUROFER 97 are summarised graphically in Fig. 3. The data indicate that for temperatures up to 723 K the cyclic softening taking place on the tempered steels is only slightly dependent on temperature. Above 723 K the softening coefficient increases dramatically, indicating that synergetic effects of cycling and temperature are becoming important. Besides, it can be seen that EUROFER 97 and F82H mod. present almost similar cyclic softening behaviour.

The analysis of the hysteresis loops of F82H mod. at 723 K has shown that the internal stress is responsible for the strong cyclic softening observed in this steel [6]. Cottrell [7] has proposed that the behaviour of the internal stress is the result of an interaction of strong athermal obstacles to the dislocation movement. Therefore, it can be concluded that for these steels long range obstacles become weaker during cycling.

In these steels with a high dislocation density and carbides distributed along lath boundaries, the various mechanisms that have been proposed to rationalise cyclic softening are [8]:

(a) Re-solution by which the metastable strengthening precipitates completely dissolve in the matrix after being cut by dislocations to a size smaller than the critical size for particle nucleation.
(b) Over-aging, which leads to the growing of precipitates into coarsely distributed stable ones.
(c) Rearrangement of the transformation-induced dislocation substructure into a dislocation subgrain structure of lower internal stress.

It is difficult to imagine that the rather large and highly incoherent carbide particles produced during tempering are cut and dissolved to give cyclic softening. Transmission electron microscopic observations failed to show a re-solution of precipitates on fatigued specimens at all temperatures. Depending on the temperature of the cyclic test, the original tempered martensitic lath structure evolved to a recovered subgrain structure of lower dislocation density or also to a recovered subgrain structure but with larger carbides along their boundaries (Fig. 4). In this work the mechanisms (b) and (c) are proposed to rationalise the cyclic softening observed in the steels under examination.

It is well known [9] that the quenched-and-tempered structure of low-carbon steels, such as F82H mod. and EUROFER 97, consists of martensitic laths with a high internal dislocation density. Carbide particles, principally of the type $M_{23}C_6$, occupy the tempered martensitic lath boundaries [2,10] as they do with the prior austenitic grain boundaries. Although pronounced aging did not produce marked changes in tempered martensitic lath structures of similar 9% Cr steels [3,10], it is evident from the present results that continuos cycling produces changes in the microstructure and generates a marked cyclic softening.

The cyclic softening observed at temperatures below 723 K in Fig. 3 is considered to be caused by the rearrangement of the initial high dislocation density of these steels to a dislocation cell structure of lower internal

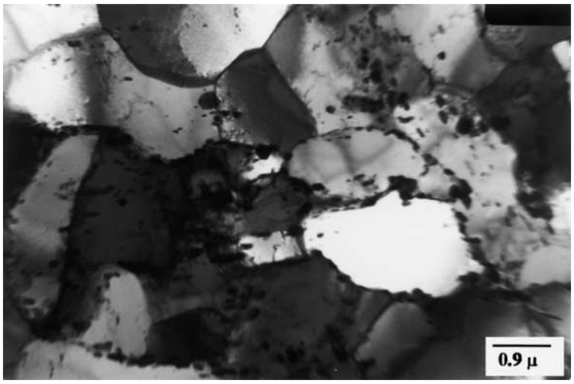

Fig. 4. Equiaxed subgrain structure of EUROFER 97 tested up to rupture under LCF-loading at 823 K and with $\Delta\epsilon_t = 1.5\%$

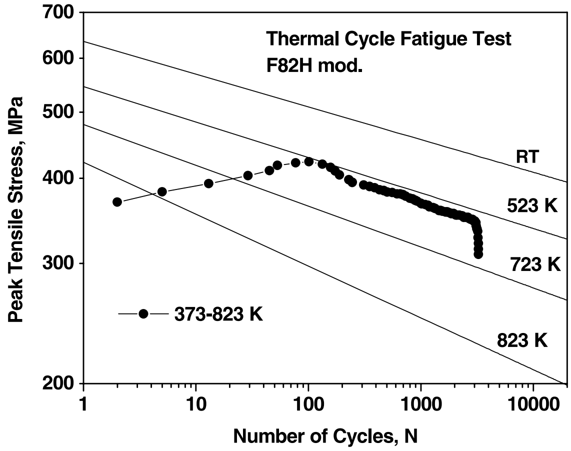

Fig. 5. Evolution of the peak tensile stress versus number of cycles for F82H mod. Under 373–823 K thermal fatigue test.

stresses. Increasing temperature will assist this rearrangement producing a recovery of the ferritic matrix and hence a lower dislocation density. Above 723 K coarsening of the carbides promotes an over-aging process leading to a marked recovery as well as recrystallisation of the microstructure as a result of the inability of precipitates to pin the martensitic lath boundaries. On the other hand, between RT and 723 K the cyclic softening coefficient is only slightly dependent on temperature. This dependence could be attributed to the decrease of the elastic modulus and the accelerated recovery of the dislocations in the lath boundaries as temperature increases.

It is also observed in Fig. 3 that the cyclic softening is more pronounced above 723 K. The mobility of the lath interfaces is expected to increase as the coarsening and spheroidization of the $M_{23}C_6$ carbides proceed, especially if these particles are responsible for the pinning of the lath boundaries. Such coarsening, accelerated by cycling at elevated temperatures, would be expected to promote the progressive breakdown of the lath morphology and the development of an equiaxed substructure of subgrains with precipitates along their boundaries. This is consistent with the substructure observed in EUROFER 97 cycled at 823 K with a total strain range of 1.5% (Fig. 4). In Fig. 3 are also represented cyclic softening coefficients obtained in previous tests carried out in MANET II at 723 K and in a commercial AISI 410 steel at 673 K. The lower cyclic softening observed in these steels can be rationalised taking into account that these two steels have a higher carbon concentration in comparison with EUROFER 97 and F82H mod. Lower carbon steels with a higher martensitic start temperature, M_S, have a higher dislocation density than higher carbon steels [9]. Consequently, a structure with higher dislocation density that softens with a dislocation rearrangement mechanism will show a higher cyclic softening coefficient in correspondence with the results of Fig. 3.

Preliminary TCF tests were also carried out on F82H mod. specimens. Fig. 5 shows the peak tensile stress versus the number of cycles for a test performed with a temperature change of 373–823 K. In addition, it shows the four straight lines representing the main stage of each of the four sets of LCF curves obtained for this steel. Similar to LCF tests on this steel a pronounced cyclic softening is also observed for this type of test. A relationship such as Eq. (1) could also be proposed for TCF tests. In this case, it is interesting to note that the slope corresponding to the main stage of the TCF test with temperature change 373–823 K lies between those of LCF tests carried out at 523 and 723 K.

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