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Cyclic instability of martensite laths in reduced activation ferritic/martensitic steels

A.F. Armas [a], C. Petersen [b,*], R. Schmitt [b], M. Avalos [a], I. Alvarez [a]

[a] *Instituto de Física Rosario, CONICET-UNR, Bv. 27 de Febrero 245 Bis, 2000 Rosario, Argentina*

[b] *Forschungszentrum Karlsruhe in der Helmholtz-Gemeinschaft, Institut für Materialforschung II, P.B. 3640, 76021 Karlsruhe, Germany*

Abstract

Low cycle fatigue tests were performed in the temperature range between room temperature and 550 °C in low activation quenched and tempered steels. After the first few cycles a pronounced cyclic softening that continues up to failure is observed for all these steels. Although this softening is coincident with similar behaviour observed in commercial martensitic alloys, reduced activation steels present some remarkable different features. Almost all cyclic curves never reach a saturation stress and after few cycles, dependent on the total strain range, they converge in a common softening stage independent of the strain range. The mechanism governing this softening stage is almost independent of temperatures below 450 °C. The cyclic softening observed in the reduced activation steels is more pronounced than for MANET II and commercial martensitic steels. This fact is related to the difference in the Martensite Start temperature.

1. Introduction

Structural metallic materials subjected to cyclic strain situations may either soften or harden. These phenomena illustrate the inadequacy of using tensile properties only in fatigue based design. The initial high strength of hardened steels can be seriously compromised even after a low number of loading cycles, reducing their original strength and load carrying capabilities. Normalized and tempered 9–12% Cr ferritic/martensitic steels exhibit attractive tensile strength properties up to approximately 600 °C. These properties, in addition to their ability to resist the effects of high doses of irradiation, focused the interest on these alloys for first-wall and blanket structure components of fusion reactors. Much of the good performance is based on high dislocation densities in a fine, well dispersed precipitate distribution. But alloys with such characteristics are prone to softening during cyclic loading.

Due to the long radioactive decay times for some of the activated alloying elements in these steels (especially Ni, Mo and Nb), programs were conducted worldwide to produce variants in which the critical elements have been substituted either completely or partly.

The present work is an examination of the fatigue response of reduced activity ferritic/martensitic (RAF/M) alloys in the temperature range between RT and 550 °C with particular attention to the tendency of cyclic softening. The purpose of this work was also to compare with the cyclic behaviour of MANET in the second variant, MANET II, the commercial Nb-free 12% Cr steel DIN 1.4923 and AISI 410 in order to make evident the difference in the observed cyclic softening.

2. Experimental details

The materials used in this study were the reduced-activation ferritic/martensitic steels F82H (Heat 9741), OPTIFER IV (Heat 986779) and EUROFER 97 (Heat E83697). Table 1 shows the chemical composition of the steels. In the table are also represented the chemical composition of MANET II, DIN 1.4923 and AISI 410.

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* Corresponding author. Tel.: +49-7247 823267; fax: +49-7247 823826.

E-mail address: claus.petersen@imf.fzk.de (C. Petersen).

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Table 1
Chemical composition of the ferritic/martensitic steels (wt%)

	C	N	Si	Mn	Ni	Cr	Mo	Al	V	Ti	Nb	Cu	W	Ta
F82H	0.09	0.007	–	0.16	0.019	8.16	0.0018	–	0.15	–	0.01	–	2.2	0.03
EUROFER 97	0.12	0.018	0.06	0.47	0.022	8.93	0.0015	0.008	0.2	0.01	–	0.004	1.07	0.14
OPTIFER IV	0.1	0.06	–	0.6	–	9.35	–	–	0.23	–	–	–	1.2	0.1
MANET II	0.10	0.03	0.14	0.75	0.65	10.3	0.57	–	0.19	–	0.14	–	–	–
AISI 410	0.11	–	0.47	0.70	0.38	13	–	–	–	–	–	–	–	–
DIN 1.4923	0.21	–	0.37	0.50	0.42	11.2	0.83	<0.05	0.21	<0.05	<0.05	0.1	–	–

The F82H steel was normalised at 1040 °C for 0.5 h and tempered at 750 °C for 2 h, and OPTIFER IV and MANET II were normalised at 1075 °C for 0.5 h and tempered at 750 °C for 2 h. The EUROFER 97 steel and the commercials DIN 1.4923 and AISI 410 steels were normalised at 980 °C for 0.5 h and tempered at 760 °C for 1.5 h. Specimens were examined by an transmission electron microscope (TEM) operating at 100 kV. Transversal disks were electrolytically polished and finally thinned for the observations. The microstructure consists of tempered laths of martensite with a substantial dislocation structure [1]. But high density of dislocations produced during quenching still remained after tempering.

Low cycle fatigue (LCF) tests have been performed with a MTS servohydraulic and an INSTRON electromechanical testing machine operating under a strain-controlled condition using a triangular wave form. Total strain ranges were controlled at 0.5%, 0.6%, 1.0% and 1.5% with a strain rate of 3×10^{-3} s^{-1}. Specimens of 77 mm length and 8.8 mm diameter in the cylindrical gauge were used. The gauge length of the axial extensometer was 21 mm.

3. Results and discussion

Cyclic softening curves were obtained upon testing EUROFER 97 specimens at RT, 250, 450 and 550 °C with total strain ranges of 0.6% and 1.0% as shown in Fig. 1. It is important to remark that similar trends were found in this work on OPTIFER IV, and in a previous work [2], on F82H. But, low activation steels show, as observed in Fig. 1, 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 (in a log–log scale) stage follows an analytical expression of the type:

$$\boldsymbol{\sigma} = \mathbf{A} \times \mathbf{N}^{-\mathbf{S}}$$

with **σ**: peak tensile stress; **N**: number of cycles; **A**: pre-exponential factor and **S**: cyclic softening coefficient. As was observed [2] for F82H, at each temperature this relationship is almost independent of the total strain amplitude.

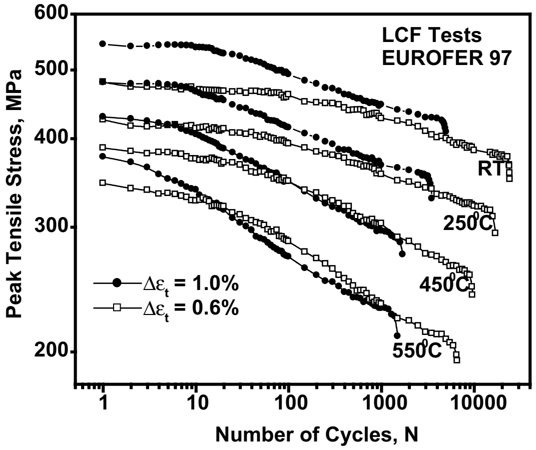

Fig. 1. Double logarithmic plot of Peak Tensile Stress vs. Number of Cycles for EUROFER 97 under LCF for two strain amplitudes and different temperatures.

Previous workers [3] have reported the microstructural stability of normalized and tempered 9–12% Cr modified martensitic steels during subsequent annealing times up to 1100 h at 550 °C. However, this apparent microstructural stability could be destabilized under cyclic strain conditions as was shown by Jones [4] in modified 9Cr–1Mo steel. The apparently stable lath martensite structure is strongly unstable under cyclic conditions being gradually replaced by the development of a cell structure. The growth of laths/subgrains suggests that the migration and/or annihilation of lath boundaries occur during cycling. The evolution of the typical martensite lath structure of low-carbon alloy steels to a cell structure was reported [5] to be already established after few cycles. The partial dissolution of a martensite lath wall at the beginning of a test can be observed in Fig. 2. This micrograph also shows a high dislocation density inside the subgrains and carbides along the martensite lath boundaries. The evolution of the microstructure of a sample that was cycled up to

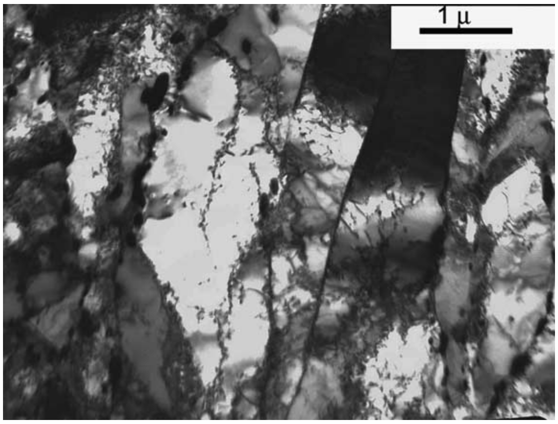

Fig. 2. Partial annihilation of martensite lath boundaries after cycling EUROFER 97 up to $N = 100$ cycles at 450 °C with $\Delta\varepsilon_t = 0.6\%$.

failure at 450 °C with total strain range 0.6% is observed in Fig. 3. Wider laths/subgrains with low dislocation density in their interior are clearly observed in the micrograph. The presence of carbides inside the subgrains which are lined up indicates the position of previous laths boundaries that were completely dissolved during cycling.

Commercial steels also show, similar to modified 9Cr–1Mo martensitic steels, at room [6,7] and high temperatures [7,8], cyclic softening prior to fracture initiation. Much of the softening occurs during the initial few percent of the fatigue lives. In these steels a saturation stress was always reached essentially after ≈20% of the fatigue lives. The saturation stress was different for the different total strain range at each test temperature. This behaviour was also found in the actual work in quenched and tempered DIN 1.4923 and AISI 410 commercial steels.

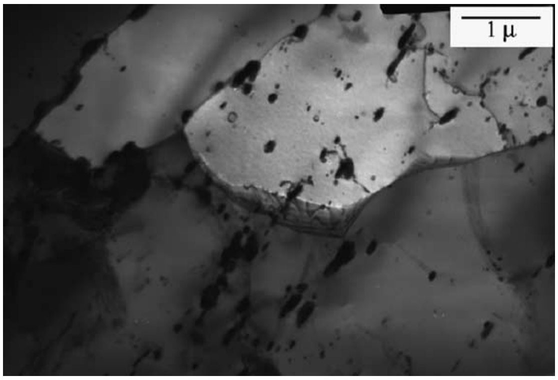

Fig. 3. TEM micrograph showing the microstructure resulting after cycling EUROFER 97 up to rupture $N_f = 9000$ cycles at 450 °C with total strain range 0.6%.

In this work, some remarkable features were found in reduced activation steels, including MANET II, that differ with that observed in commercial steels. These are:

(1) At each temperature and after a certain number of cycles, dependent on the total strain amplitude, the cyclic curves gather together in a common linear (in a log–log diagram) softening stage with the same slope and independent of the plastic strain amplitude.
(2) The softening slope of the linear stage strongly depends on temperature but only above 450 °C.
(3) No saturation stress was evident from the curves, but for the highest strain range, above 1.0%, and the lowest temperature, RT and 250 °C. The linear cyclic softening stage leads directly to the rupture of the samples.

However, previous workers [3–5,8] have related the mechanism leading to a break up or partitioning of the lath structure to a sufficiently high fatigue temperature. As can be seen in Fig. 1 cyclic softening of reduced activation steels seems to follow the same trend independent of the temperature of the test. At each temperature, the first stage depends on the total strain range being shorter for the higher total strain range. Curves with lower total strain range approach or intersect the curve belonging to the higher strain range. After the approach or intersection both curves converge in a common curve. As can be seen in Fig. 4, the linear softening stage seems to be governed by a mechanism independent of the plastic strain range imposed to the sample. Since this behaviour is also observed at room temperature we are suggesting that the mechanism is athermal. Equiaxed subgrains with carbides lined up in their interior are also evident from TEM examinations. Coarsening and spheroidization of the $M_{23}C_6$ carbide is expected to occur at temperatures above 450 °C, especially if these particles are responsible for the pinning of the lath boundaries. Such coarsening, accelerated by cycling at elevated temperature, would be expected to improve the progressive breakdown of the lath morphology and the development of an equiaxed substructure (Fig. 3). More pronounced cyclic softening should be observed at higher temperatures as it was shown in Fig. 1.

The most striking result of the present work was observed comparing the cyclic softening coefficient, **S**, of the reduced activation steels with that of commercial steels. The cyclic softening behaviour of some martensitic steels is compared in Fig. 5, where F82H steel was selected as representative of reduced activation steels. The cyclic softening coefficients measured for EUROFER 97 and OPTIFER IV have almost the same value than that of F82H. In the figure it is evident the more

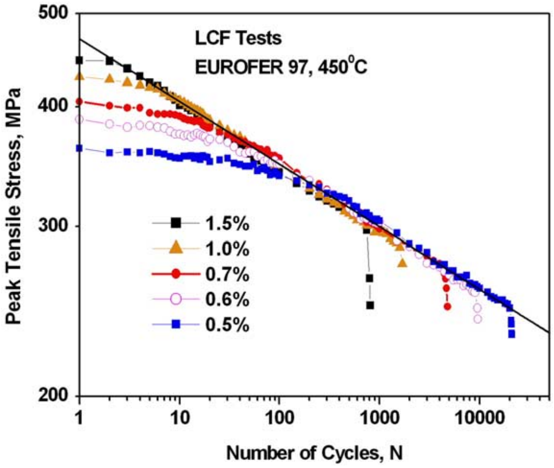

Fig. 4. Double logarithmic plot of peak tensile stress vs. number of cycles for EUROFER 97 under LCF for different strain amplitudes at 450 °C.

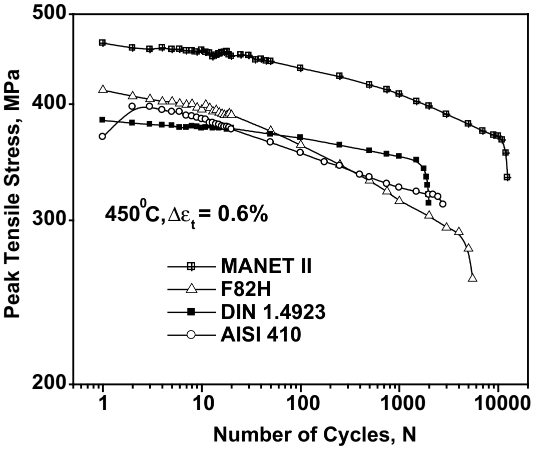

Fig. 5. Double logarithmic plot of peak tensile stress vs. number of Cycles for several martensitic steels under LCF.

pronounced softening exhibited by F82H. In this work, it is proposed that the cyclic softening rate observed in martensitic steels depends on the M_S (martensite start temperature). A lath martensite structure with a high density of tangled dislocations within their laths is characteristic of normalized and tempered steels with high M_S temperature [9]. Chemical composition is the main factor affecting the M_S temperature of the steel. RAF/M steels such as EUROFER 97, OPTIFER IV and F82H with a lower amount of C, Ni, Mo, Nb are steels with a high M_S temperature. Table 2 summarizes the M_S temperature and the cyclic softening coefficient, **S,** corresponding to the steels of Fig. 5. The M_S temperature was calculated on the basis of the equation proposed by Steven and Haynes [10] and **S** was obtained from the linear stage preceding the rupture of the samples. On cycling, martensite laths with the higher dislocation density (higher M_S) could quickly become untidy loosing and releasing a significant amount of free dislocations per cycle causing faster cyclic softening. Nevertheless, more experiments will be done in order to confirm this proposal.

Table 2
Martensite start temperatures and cyclic softening coefficient (450 °C, $\Delta\varepsilon_t = 0.6\%$)

	M_S	**S**
F82H	370	0.060
Manet II	290	0.044
AISI 410	260	0.037
1.4923	230	0.020

4. Conclusions

Reduced activation steels present cyclic softening behaviour that differ with that observed in commercial steels. Independent of temperature the cyclic curves converge on a common linear stage. It is proposed that the mechanism of lath boundaries annihilation is, principally, athermal and independent of the strain amplitude. As a consequence of their high martensite start temperature low activation steels show a more pronounced cyclic softening.

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