On the Cyclic Softening Mechanisms of Reduced Activity Ferritic/Martensitic Steels

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Low-cycle fatigue tests were performed in three different ferrite/martensite steels, i.e., the European RAFM steel EUROFER 97 and the commercials AISI 410 and AISI 420, at room temperature (RT) and at 550°C. After the first few cycles, a cyclic softening that continues up to failure is observed for all these steels. The cyclic softening exhibited by AISI 420 is less pronounced than for the other two steels. The comparison between the mechanical responses of the materials was based on the study of the flow stress components, i.e., the friction and the back stresses, and their correlation with the microstructure evolution. In most cases, the strong cyclic softening observed is produced by the decreasing stress values exhibited by both stress components. However, at RT, for AISI 420, the back stress does not present variation during cycling. The decrease of the free dislocation density inside the subgrains and the growth of the mean subgrain size represent the main microstructural evolution.

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels are leading candidates for blanket/first-wall structures of future fusion reactors. It is evident from previous results[1–4] that continuous cycling produces changes in the microstructure and a marked cyclic softening that could deteriorate their mechanical properties. Although the origin of the effect and the kinetics of the softening behavior are not well understood, it was, mainly, attributed to the gradual elimination of obstacles, such as dislocation segments, precipitates, and grain or lath boundaries, to the motion of dislocations.[5] Particularly in the RAFM steels, a pronounced cyclic softening rate and an independence of this softening rate on the strain amplitude were also reported. In the literature, some authors[6] have rationalized this behavior as a consequence of the higher martensite start temperature of this type of steels.

To study in detail the decrease of the observed cyclic stress, the flow stress was decomposed from the hysteresis loops into its components: the friction stress, σ_f, and the back stress, σ_b. The former, is related to short-range interaction obstacles and the latter is related to long-range interaction obstacles. This partition was proposed first by Cottrell[7] based on the work of Seeger[8] for monotonic loadings. The key issue of this method is the understanding of the role played by the microstructural changes on the cyclic softening. Fournier et al.[9] showed that during fatigue tests the most influential factor in the softening for martensitic steels is the annihilation of long-range obstacles.

The aim of this work is to study the cyclic behavior, at room temperature (RT) and at 550°C, of the commercial steels AISI 410 and AISI 420 and to compare them with previous results obtained for EUROFER 97.[10] The discussion is focused on the correlation of the friction and the back stresses behaviors and the evolution of the dislocation microstructure.

2. Materials and Methods

The materials studied in this investigation are EUROFER 97 RAFM steel, the commercials AISI 410 (similar amount of carbon as EUROFER 97) and AISI 420 (with 0.28% C). This last steel was chosen as representative of steel with lower martensite start temperature. **Table 1** shows the chemical composition of these steels. The thermal treatments consisted in normalization at 980°C for 30 min for EUROFER 97 and at 1000°C for 40 min for AISI 410 and AISI 420, followed by tempering at 760°C for 90 min for EUROFER 97

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	C	Cr	Ni	Mo	V	Si	Mn	Ti	Cu	Ta	B	W	N
EUROFER 97	0.12	8.93	0.022	0.0015	0.2	0.06	0.47	0.01	0.004	0.14	<0.001	1.07	0.018
AISI 410	0.11	13	0.38	–	–	0.47	0.7	–	–	–	–	–	–
AISI 420	0.27	12.3	0.13	0.07	0.04	0.36	0.35	–	–	–	–	–	–

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

and at 730°C for 120 min for the commercial steels. Two different thermal treatments were applied in order to obtain similar microstructures on the three materials.

Low-cycle fatigue tests have been performed under plastic strain control with an electromechanical INSTRON testing machine. Cylindrical specimens of 8.8 mm diameter and 21 mm in gauge length were manufactured. Tests were carried out at RT and at 550°C using a plastic strain range $\Delta\varepsilon_p = 0.2\%$. The total strain rate was $\dot{\varepsilon} = 2 \times 10^{-3}\,s^{-1}$. The dislocation structures of the different steels were studied in thin foils taken perpendicular to the specimen axis at rupture. In addition, for EUROFER 97 the observations were also made for interrupted tests at 10 cycles and 500 cycles. The observations were performed using two different microscopes, Philips EM 300 operating at 100 kV, for general structure, and Philips CM 200 operating at 200 kV, for detailed microstructure studies.

3. Results

3.1. Low-Cycle Fatigue Tests

The cyclic curves subjected to constant plastic strain range of 0.2% at 20 and 550°C are shown in **Figure 1**. After the first

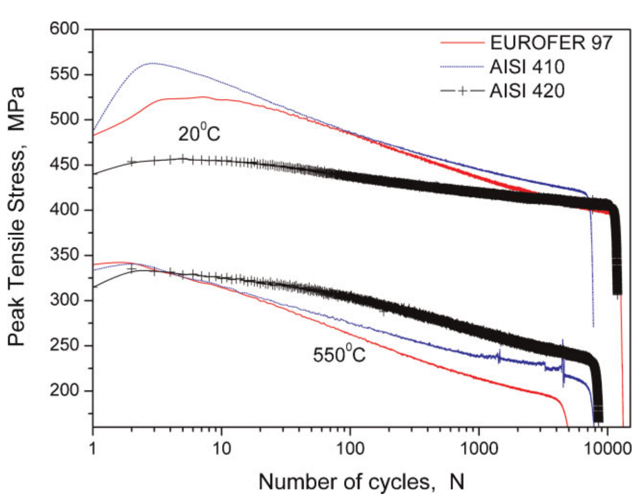

Figure 1. Cyclic softening curves obtained at RT and 550°C, with 0.2% plastic strain range.

cycles, a continuing cyclic softening can be observed in all the steels up to fracture. Whereas EUROFER 97 and AISI 410 exhibit a very pronounced cyclic behavior, it is clearly more attenuated in AISI 420. On the other hand, at high temperature, 550°C, all the steels soften under cyclic loading but, as at RT, for AISI 420 this behavior is less noticeable.

The evolution of the flow stress during cycling was studied by analyzing the stress components, the back and the friction stresses, obtained from the data provided by the hysteresis loops (**Figure 2**). The method produces a reasonable scatter band of 5% of the stress values. Figure 2 represents theses values and clearly shows the trends of the curves. At RT (Figure 2a), the cyclic softening of EUROFER 97 and AISI 410 is produced mainly by the decrease observed in the friction stress while for AISI 420, the friction stress seems to be the only component responsible for the observed softening.

At high temperature, the main contribution to the cyclic softening of the materials is provided by the back stress as shown in Figure 2b. AISI 420 exhibits a pronounced decrease in the back stress values comparable to that developed by EUROFER 97; the friction stress seems not to have an important role in any cases.

3.2. Microstructure Evolution

The microstructural stability of quenched and tempered ferritic/martensitic steels during subsequent annealing times up to 1100 h at 550°C has been reported.[11] Armas et al.[6] studied the cyclic behavior of these steels, showing that the microstructural stability could be destabilized under cyclic strain conditions. The apparent stability of the martensite structure changes under cyclic conditions being gradually replaced by the development of a cell structure. According to Marmy and Kruml[1] the evolution of the typical martensite lath structure to a cell structure in low-carbon steels was reported to be already established after few cycles during fatigue tests.

Figure 3a shows the as-received state of EUROFER 97, the structure is composed mainly of sub micrometric equiaxed subgrain being the dislocation density inside the subgrains very high. Figure 3d is the characteristic subgrain structure obtained for EUROFER 97 fatigued at RT using a plastic strain range of 0.2%. In this micrograph,

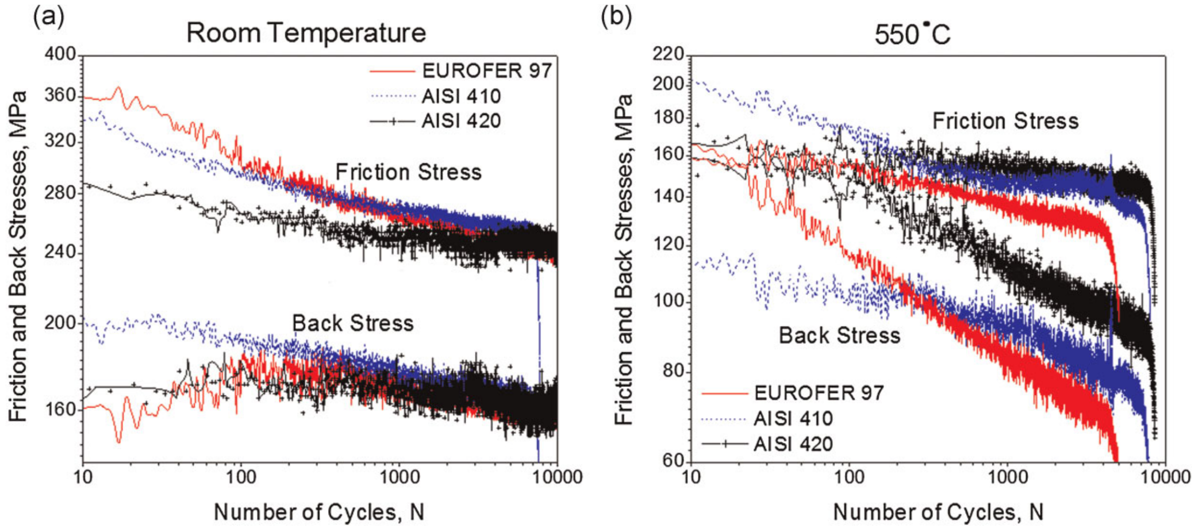

Figure 2. Friction and back stresses behavior, with 0.2% plastic strain range, observed a) at RT and b) at 550°C.

it is important to note that cycling at RT induces two visible microstructural evolutions: the decrease in the dislocation density and the annihilation of some subgrain boundaries.

In order to analyze the evolution of the dislocation structure during cycles, interrupted tests up to 10 and 500 cycles were carried out in EUROFER 97. Figure 3b shows the sample tested up to 10 cycles, the comparison between this micrograph and the as-received material does not present marked differences. On the contrary, the sample tested up to 500 cycles (Figure 3c) show a pronounced microstructural evolution. As can be seen in these micrographs, the dislocation density in the interior of the subgrains showed a significant decline.

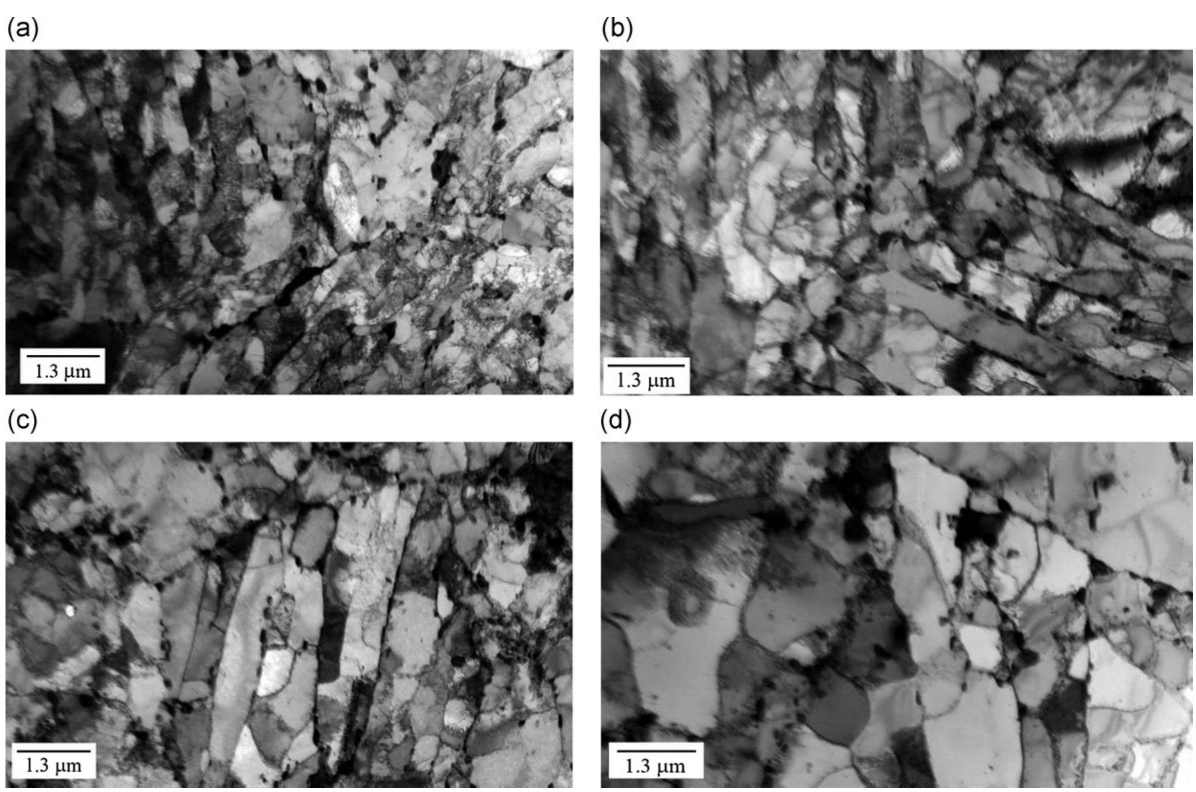

Figure 3. a) Tempered martensite structure of EUROFER 97 in as-received samples. Characteristic microstructure of samples fatigued using $\Delta\varepsilon_p = 0.2\%$, up to b) 10 cycles c) 500 cycles and d) 11 000 cycles, at RT.

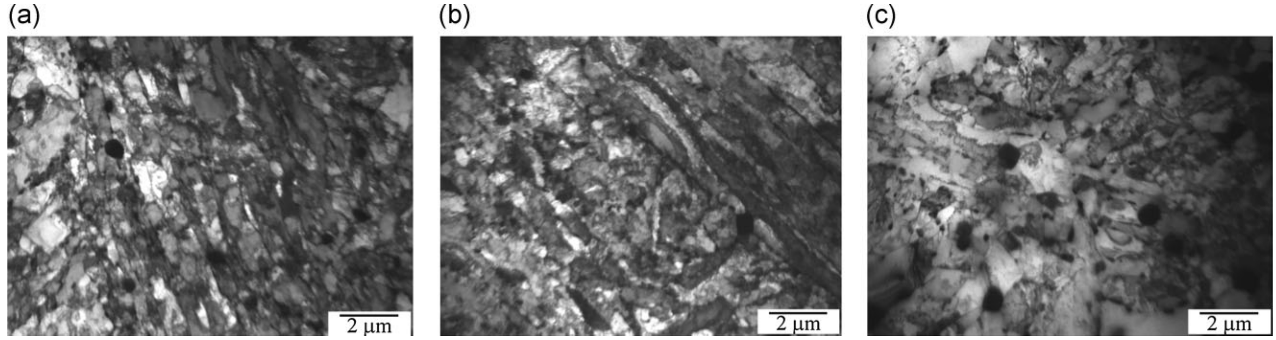

Figure 4. TEM observations of the microstructure in the as-received materials: a) EUROFER 97; b) AISI 410; c) AISI 420.

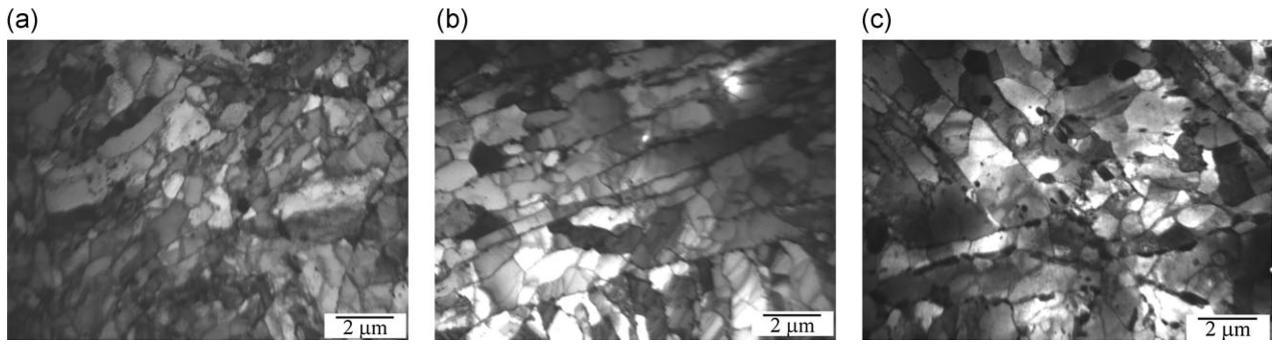

Figure 5. Characteristic microstructure of samples fatigued at RT, using $\Delta\varepsilon_p = 0.2\%$: a) EUROFER 97; b) AISI 410; c) AISI 420.

A comparison of the as-received microstructure obtained on the three different steels is showed in **Figure 4**. In any case, the original parallel martensitic laths are partitioned in sub-micrometrics equiaxed subgrains. In addition, a high density of dislocations produced during quenching of these steels remains after tempering. Comparing the three micrographs in Figure 4, there is a close resemblance of the subgrain sizes between EUROFER 97 and AISI 410 steels in the initial state. For AISI 420 slightly larger subgrains were observed, Figure 4c. The dislocation density in the interior of the subgrains for AISI 420 appears to be lower than the ones presented in EUROFER 97 and AISI 410.

After cycling at RT, **Figure 5**, a resemblance in microstructural features is observed for the different steels, including AISI 420, i.e., subgrain sizes and dislocation densities seems to be closer. At high temperature, for the three steels, the mean free dislocation density inside subgrains showed analogous evolution as the one observed at RT, **Figure 6**. Moreover, in addition to the annihilation of the free dislocations inside the subgrains, many of the subgrain boundaries have disappeared, leading to an increment on subgrain sizes.

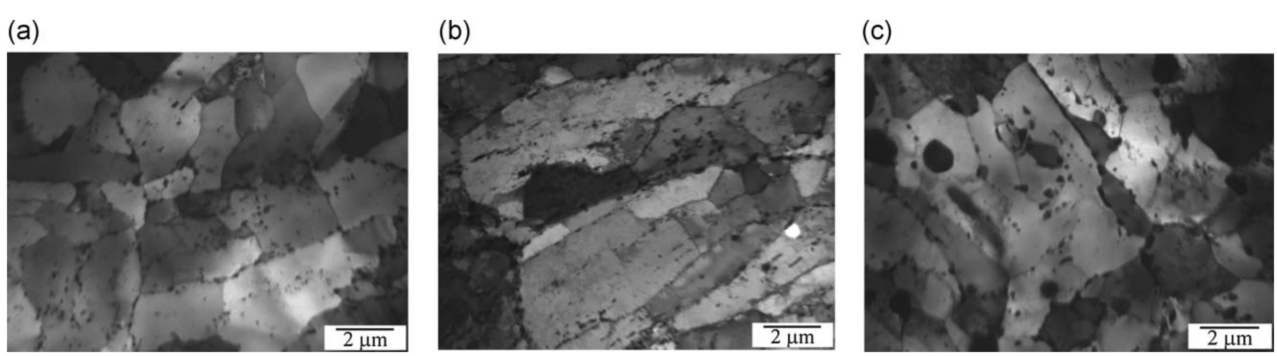

Figure 6. Characteristic microstructure of samples fatigued at 550°C, using $\Delta\varepsilon_p = 0.2\%$: a) EUROFER 97; b) AISI 410; c) AISI 420.

4. Discussion

At RT, Figure 2 shows that the influence of the stress components on the softening is different for the three steels, being the back stress the least influential. Indeed, the friction stress shows the highest decrease during cycling, which is more pronounced at the beginning of the fatigue life. Moreover, for AISI 420, the friction stress is the only contribution to the cycling softening, the variation of the back stress is negligible. As can be seen in the micrographs, the three steels present a low dislocation density inside the subgrains after cycling. Moreover, EUROFER 97 shows subgrains almost free of dislocations already at 500 cycles (Figure 3c). This fact may confirm that the friction stress is mainly associated with the change in the free dislocation density inside the subgrains and that the most important changes take place at the beginning of the fatigue life. On the contrary, for the three steels, the back stress shows a lower decrease, which can be attributed to the partial annihilation of the subgrain boundaries, i.e., the increment on subgrain sizes. Interestingly, for AISI 420, in which the initial subgrain size is larger than in the other two steels, the mean size appears not to suffer an appreciable variation during cycling. Therefore, the larger value of the subgrain size would influence in the variation of the back stress during cycling; on the level of the cyclic softening attained by the steel and lastly on the value of the flow stress at the beginning of the fatigue life.

At high temperature, cycling leads to a very pronounced decreasing behavior of both the back and the friction stress components. This behavior is consistent with the fact that, at 550°C, the mean dislocation density inside subgrains shows analogous evolution as that observed at RT. Nevertheless, as it was aforementioned, the most important contribution to the cyclic softening at high temperature is provided by the back stresses. As can be seen in Figure 6, at 550°C, in addition to the clean-up of dislocations located in the interior of the subgrains, the density of subgrain boundaries is substantially reduced and even can completely disappear. The detailed study of the dislocation structure of these three steels has shown that EUROFER 97 presents the fastest annihilation of the subgrain boundaries that leads to larger decreasing values of the back stress in comparison with AISI 410 and AISI 420.

5. Conclusions

Reduced activation ferritic/martensitic steels present softening under cyclic loadings. Low-cycle fatigue tests were performed in three different steels, the RAFM European steel EUROFER 97 and the commercials AISI 410 and AISI 420 at RT and at 550°C. The cyclic softening exhibited by these materials was studied through the back and friction stresses evolutions. Microstructural changes were studied using transmission electron microscopy.

1. The main results obtained are the following: Two main microstructural evolutions were observed during cycling for the different materials: the free dislocation density decreases and mean the subgrain size grows. In particular for EUROFER 97 the most significant microstructural changes take place at the beginning of the fatigue life.
2. At room temperature: The cyclic softening presented by AISI 420 is less pronounced than that exhibited by EUROFER 97 and AISI 410. In any case, the friction stress showed the most important contribution to the cyclic softening. This can be attributed to the annihilation of dislocations that were initially inside the subgrains. The decrease of the back stress for EUROFER 97 and AISI 410 was not as significant as the friction stress. Only few subgrain boundaries disappear after cycling. In AISI 420 no influence of the back stress was observed with cycling.
3. At 550°C: The three steels show pronounced softening being the least pronounced for AISI 420. At this temperature, the back stress plays an important role in the cycling softening of the three steels. Mean subgrain size presents a marked increment due to the disappearance of subgrain boundaries. The friction stress showed a similar decrease at RT; this fact is in agreement with TEM observations where the final dislocation density inside subgrains is close to that observed at 20°C.

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References

[1] P. Marmy, T. Kruml, *J. Nucl. Mater.* **2008**, *377*, 52.
[2] M. F. Giordana, I. Alvarez-Armas, A. Armas, *Acta Microsc.* **2010**, *19A*, 84.
[3] P. F. Giroux, F. Dalle, M. Sauzay, C. Caës, B. Fournier, T. Morgeneyer, A. F. Gourgues-Lorenzon, *Proc. Eng.* **2010**, *2*, 2141.
[4] B. Fournier, F. Dalle, M. Sauzay, J. Longour, M. Salvi, C. Caës, I. Tournié, P.-F. Giroux, *Mater. Sci. Eng. A* **2011**, *528*, 6934.
[5] T. Ishii, K. Fukaya, Y. Nishiyama, M. Suzuki, M. Eto, *J. Nucl. Mater.* **1998**, *258–263*, 1183.

[6] A. F. Armas, C. Petersen, R. Schmitt, M. Avalos, I. Alvarez, *J. Nucl. Mater.* **2004**, *329–333*, 252–2256.
[7] H. Cottrell, *Dislocations and Plastic Flow in Crystals*, Oxford University Press, London **1953**.
[8] A. Seeger, *Defects in Crystalline Solids*, The Phys Soc, London **1955**, p. 328.
[9] B. Fournier, M. Sauzay, C. Cäes, M. Noblecourt, M. Mottot, *Mater. Sci. Eng.* **2006**, *A437*, 183.
[10] M. F. Giordana, I. Alvarez-Armas, M. Sauzay, A. Armas, *Key Eng. Mater.* **2011**, *465*, 358.
[11] W. Jones, C. R. Hils, D. H. Polonis, *Metall. Trans.* **1991**, *22A*, 1049.