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Dynamic strain ageing evidences during low cycle fatigue deformation in ferritic–martensitic stainless steels

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Abstract

The influence of dynamic strain ageing (DSA) on the strain cyclic behaviour of ferrite–martensite stainless steels was investigated at temperatures ranging from room temperature to 823 K. For fully annealed AISI 420 initial hardening followed by a saturation stage was observed at each test temperature. This steel was found to be susceptible to DSA as evidenced by the temperature independent stress saturation observed between 523 and 723 K. Normalized and tempered MANET II and F82H mod. softens during cyclic loading at all temperatures. In this steel DSA manifestations were observed on plotting the peak tensile stress difference between hysteresis loops obtained at different strain rates. Strongly abnormal behaviour with higher peak tensile stresses corresponding to slower strain rates was observed in the temperature range between 500 and 700 K. It is proposed that DSA mechanisms caused by the drag of solution carbon atoms is responsible for this unusual behaviour.

1. Introduction

High-chromium ferritic–martensitic steels containing 8–14 wt% chromium have received extensive use at elevated temperatures, because of their economical combination of good mechanical and corrosion properties. They are currently being considered as candidate alloys in first wall applications for fusion reactor systems. The pulsed mode operation of fusion devices leads to cyclic loading at high temperatures of the structural materials during operation. This fatigue process may be sufficiently severe to cause damage to the material.

Among all the studies performed to evaluate cyclic hardening mechanisms in stainless steels, only a few investigations were made in connection with the presence of impurity atoms such as carbon and nitrogen in interstitial positions. This mechanism, known as dynamic strain ageing (DSA), occurs when the rate of straining is such that the interstitial atoms can diffuse and pin the mobile dislocations.

Although the effects of strain ageing on cycling of steels are already well known [1], they were scarcely studied on stainless steels. Several authors [2–5] have reported the strong influence of DSA on the cyclic behaviour of austenitic stainless steels. As a result, they all have found a marked cyclic hardening and an inverse dependence of the maximum tensile stress amplitude on both temperature and strain rate in the DSA domain. Armas et al. [6] have recently analysed the cyclic behaviour of the AISI 430F ferritic stainless steel in the normalised condition and have observed a pronounced cyclic hardening stage of extended duration when cycled between 673 and 773 K.

However, several works [7,8] in the literature about low cyclic fatigue of martensitic stainless steels at RT and higher temperatures have revealed a cyclic softening tendency only. These steels were always tested in normalised and tempered condition. The cyclic softening behaviour is considered to be the result of the rearrangements of dislocations previously introduced by the quenching. Serrations and changes in the decreasing rate of yield stress and tensile strength in the temperature range from 523 to 723 K, observed in these steels during monotonic tensile deformation [9], are typical manifestations of DSA. Although this temperature range

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belongs to the temperature region of interest for the thermonuclear fusion reactor design, there are scarce information available on the effects of DSA on cycling deformation and accompanying substructure.

In order to obtain basic information about the DSA influence on fatigue behaviour of martensitic stainless steels, low cycle fatigue tests were carried out under isothermal conditions on type AISI 420, the German MANET II and the Japanese Reduced Activation Ferritic Steel F82H mod. The original purpose was to analyse the cyclic behaviour of AISI 420 with a ferrite-pearlite structure in order to prove if a cyclic hardening similar to that observed in AISI 430F is also present. Since it was not possible to obtain this structure in MANET II and F82H mod., they were studied in normalised and tempered condition. During tempering at 1023 K, the fully martensitic structure of these steels transforms into a ferrite matrix with a high dislocation density and carbide precipitates. In this way, all the steels under study are composed of ferrite with differences in dislocation density and carbide population. The carbon concentration in the ferritic matrix will be different according to the carbon percentage of the steels.

2. Experimental details

Fully reversed total axial strain controlled low cycle fatigue (LCF) tests which were conducted in LCF testing machines equipped with radiant heating facility, INSTRON 1362 in case of AISI 420 and MANET II and MTS 810 in case of F82H mod.

Tests were carried out on three types of 9–12% Cr steels:

1. A commercial type AISI 420 martensitic steel.
2. MANET II, as the European Reference Heat of ferritic–martensitic steels.
3. F82H mod. a Japanese Reduced Activation Ferritic–martensitic steel.

The chemical compositions of the materials are given in Table 1. Cylindrical fatigue specimens were machined with a uniform gauge section of 5 mm diameter and 10.5 mm length, in case of AISI 420 and MANET II and 8.8 mm diameter and 21 mm length in the case of F82H mod.

According to the purpose of this paper, it is important to carry out tests with samples containing the lowest possible dislocation density to avoid eventual "hiding" effects. To obtain this condition, the steel AISI 420 was fully vacuum heat treated for 1 h at 1173 K with slow furnace cooling. The structure of a ferrite matrix with pearlite nodules of this annealed steel is shown in Fig. 1.

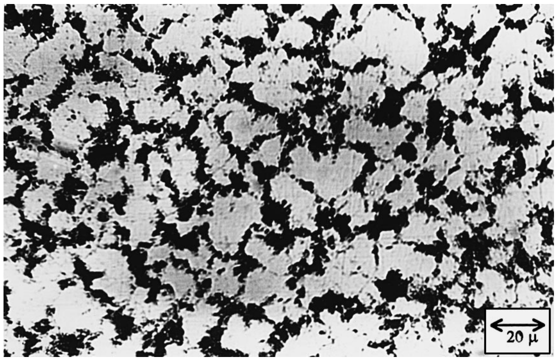

Fig. 1. Microstructure of fully annealed AISI 420.

Annealing conditions for MANET II and F82H mod. (values for F82H mod. in brackets) were: Austenitization 1348 K/30 min (1313 K/38 min), air cooled and tempering 1023 K/2 h (1023 K/1 h), air cooled. A fully martensitic and δ-ferrite-free structure transforms during tempering into the so-called "tempered martensite" structure. This consists of elongated subgrains with walls containing a high dislocation density and mostly $M_{23}C_6$-carbides lying along prior austenite grain boundaries and tempered lath boundaries.

An optical micrograph shows the microstructure of tempered steel in Fig. 2. According to Finkler and Schirra [10], even under a very low cooling rate, a free martensite matrix with a low density of dislocations would be impossible to obtain in this steel.

3. Results and discussion

The LCF cyclic stress response of type AISI 420 stainless steels in the range 300–823 K is shown in Fig. 3. The set of curves was obtained using the same total strain range of $\Delta\varepsilon_t = 0.01$ and strain rate of 2×10^{-3} s^{-1}.

All curves show a short initial hardening stage followed by a saturation period independent of test tem-

Table 1
Chemical analysis of materials

Steel	Element wt% (Fe bal.)											
	C	Si	Mn	S	P	Cr	Ni	W	Mo	V	Ta	Nb
AISI 420	0.27	0.36	0.35	0.02	0.03	12.3	0.13	–	0.07	0.04	–	0.03
MANET II	0.11	0.18	0.85	0.004	0.005	10.3	0.65	–	0.58	0.19	–	0.14
F82H mod.	0.09	–	0.156	–	–	7.68	0.021	2.2	0.003	0.162	0.03	0.01

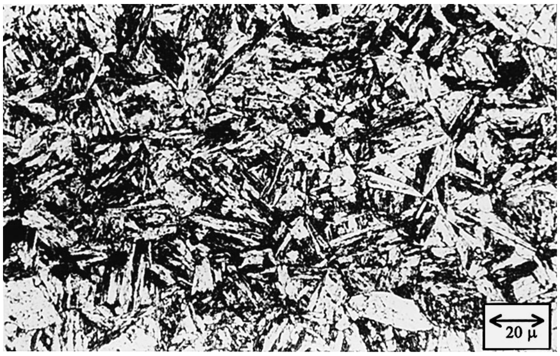

Fig. 2. Microstructure of tempered MANET II.

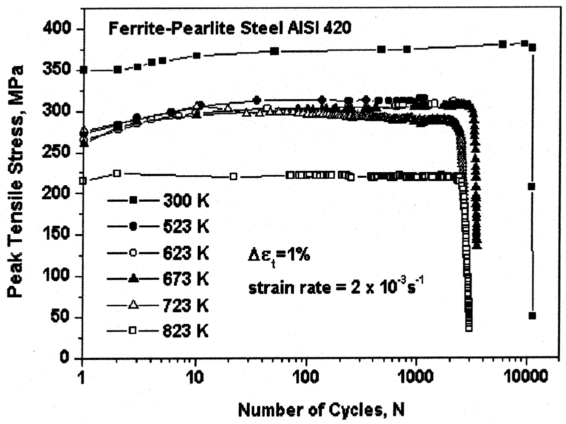

Fig. 3. Cyclic stress behaviour of AISI 420.

perature. This saturation stage remains up to failure, except in some cases where the stress slightly decreases. The predominant feature of the figure is the small variation among saturation stress values of each curve in the temperature range between 523 and 723 K.

The fact that the saturation stress is independent of temperature, between 523 and 723 K, suggests that DSA influences the cyclic behaviour of the steel. Dislocation-solute atoms interactions would become temperature independent in this temperature range and for the specified mechanical conditions. However, no pronounced cyclic hardening was observed in this steel. On the other hand, pronounced hardening was reported previously [6] at 723 and 773 K in a normalised AISI 430F ferritic steel. In this ferritic steel the normalizing treatment produces a carbon supersaturated solid solution in the matrix. The structure of fully annealed AISI 420 steel also consists of a ferritic matrix with a significant amount of carbides that had precipitated during furnace cooling. In this way, the carbon concentration in the matrix is smaller than in the normalised AISI 430F. Taken into account the above, it is adequate to assume that only solid solution strengthening would be the most important cyclic hardening mechanism in AISI 420 cycled in the DSA region. However, as a consequence of the lower carbon concentration, it would not be strong enough to produce an enhanced dislocation multiplication during cycles to cause a pronounced cyclic hardening.

In order to identify the DSA influence on the cyclic behaviour of normalised and tempered MANET II, the peak tensile stress dependence on the strain rate was examined at different temperatures. Tests with total strain range of 0.006 were performed in the following way on sample 1: They were started at a temperature of 423 K and after 200 cycles increased stepwise to 521, 577, 623, 673, 723, 773 until 823 K. At each temperature, the strain rate was changed first from strain rate 1 of 1.5×10^{-3} s^{-1} to strain rate 2 of 1.5×10^{-4} s^{-1} and then to strain rate 3 of 1.5×10^{-5} s^{-1}. Finally, the test was finished by cycling at 623 K with the initial strain rate 1 of 1.5×10^{-3} s^{-1} up to failure.

For comparison the tempered sample 2 was totally cycled at 623 K with the initial strain rate. Fig. 4 shows the mechanical behaviour of samples 1 and 2 of tempered MANET II. Both samples soften independently of test temperature. Sample 1 was tested at 623 K during two steps, firstly, after three lower temperature steps and secondly after four higher temperature steps. Sample 2 coincides with the first step of sample 1 cycled at 623 K. It can be suggested that the structure developed in sample 1 during the three lower temperature steps would be similar to that developed in sample 2 at this state. On the other hand, the second cyclic step at 623 K of sample 1 after four higher temperature steps presents a similar cyclic softening rate as sample 2. But peak tensile stresses of sample 1 are smaller than those of sample 2. These two effects could be rationalised on considering that tempered MANET II softens by fatigue loading due to a rearrangement of the transformation induced dislocation substructure into a lower dislocation density

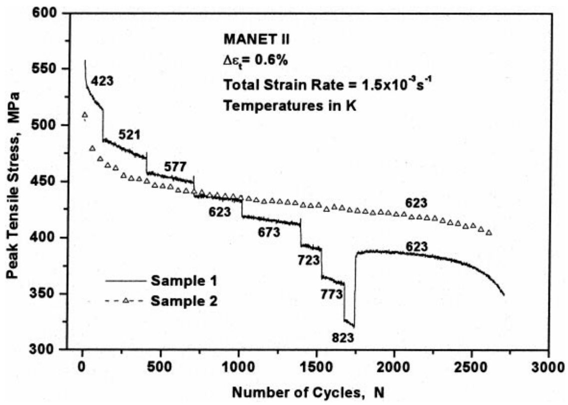

Fig. 4. Cyclic stress behaviour of MANET II.

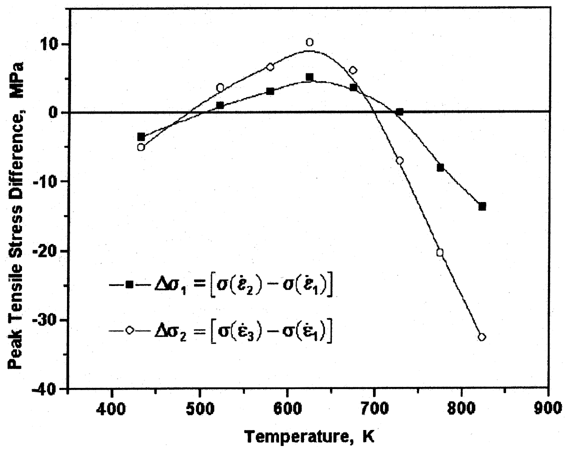

Fig. 5. Peak tensile stress differences vs. temperature measured on MANET II. $\Delta\sigma_1$: 1.5×10^{-4} s^{-1} – 1.5×10^{-3} s^{-1}; $\Delta\sigma_2$: 1.5×10^{-5} s^{-1} – 1.5×10^{-3} s^{-1}.

and lower internal stress. Alvarez-Armas et al. [11] proposed for the accelerated softening observed during thermal fatigue of MANET I (other variant of the German steel DIN 1.4914), that a synergetic process between high temperature and mechanical cycling improves the recovery process promoting the development of an equiaxed subgrain structure. It is proposed that the resulting structure formed in sample 1 after the three lower temperature steps is a consequence only of the cyclic process without an appreciable temperature influence. On the other hand, the structure formed after the four higher temperature steps would be influenced by both, temperature and mechanical cycling, becoming softer due to a lower dislocation density as a product of recovery.

According to the experimental procedure previously described, strain rate changes were carried out. Defining $\Delta\sigma_1$ as the difference between peak tensile stress of hysteresis loops with strain rate 1 of 1.5×10^{-3} s^{-1} and strain rate 2 of 1.5×10^{-4} s^{-1} and $\Delta\sigma_2$ as the corresponding to strain rate 1 of 1.5×10^{-3} s^{-1} and strain rate 2 of 1.5×10^{-5} s^{-1}. Fig. 5 shows both peak tensile stress differences as a function of temperature. Negative values in the curves are related to a "normal" behaviour with lower peak tensile stress for the slower strain rates. Between 500 and 700 K an anomalous behaviour region is observed with higher peak tensile stress corresponding to the slower strain rates. Both curves present a maximum at approximately 623 K. Positive values in these curves clearly indicate that higher stresses are required to reach the imposed strain amplitude for slower average dislocation velocities. It is proposed that this velocity dependent strengthening effect could be attributed to DSA. In the temperature range from 500 to 700 K solute atoms acquire enough mobility to diffuse to the dislocation cores producing a drag effect on the mobile dislocation. This drag effect will be more pronounced for the slowest dislocation velocities.

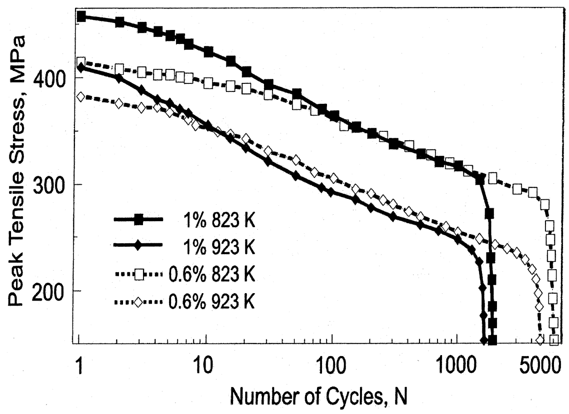

Fig. 6. Cyclic stress behaviour of F82H mod.

LCF tests of reduced activation ferritic steel F82H mod. have been performed at 823 and 923 K with two different total strains and a strain rate of 3×10^{-3} s^{-1}. Fig. 6 shows that softening occurs since the beginning of the tests independent of temperature and total strain amplitude.

The noticeable variation between the initial softening stage of curves with the same strain amplitude for both test temperatures suggests that no DSA influences of cyclic behaviour of this steel could be detected. The absence of strain ageing in this steel could be attributed to the higher temperature range of the tests.

4. Conclusions

In order to evaluate the influence of DSA during fatigue, low cycle fatigue tests were performed on three ferritic-martensitic stainless steels in the temperature region between room temperature and 823 K. The main results obtained are the following:

1. For full annealed type AISI 420 steels, a temperature independent saturated peak tensile stress was observed between 523 and 723 K.
2. Temperature and strain rate change tests were carried out on normalised and tempered MANET II. Cyclic softening was observed between 423 and 823 K.
3. Anomalous mechanical behaviour characterised by higher peak tensile stresses of the hysteresis loops corresponding to lower strain rates was observed in the temperature range between 500 and 700 K on MANET II. This effect could be attributed to DSA.
4. During LCF-tests of the RAF steel F82H mod. no DSA influences of cyclic behaviour could be detected at temperatures above 823 K.

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References

[1] L.F. Coffin, Jr., J. Basic Engrg. Trans. ASME 87D (1965) 351.
[2] K. Tsuzaki, T. Hori, T. Maki, Y. Tamura, Mater. Sci. Eng. 61 (1983) 247.
[3] K.B.S. Rao, M. Valsan, R. Sandhya, S.L. Mannan, P. Rodriguez, High Temp. Mater. Proc. 7 (1986) 171.
[4] R. Zauter, F. Petry, H.J. Christ, H. Mughrabi, ASTM STP 1186 (1993) 70.
[5] A.F. Armas, O.R. Bettin, Y. Alvarez-Armas, G.H. Rubiolo, J. Nucl. Mater. 155–157 (1988) 646.
[6] A.F. Armas, I. Alvarez-Armas, M. Avalos, C. Petersen, R. Schmitt, in: C. Varandas, F. Serra (Eds.), Fusion Technology 1996, Elsevier, Amsterdam, 1997, p. 1359.
[7] K. Kanazawa, K. Yamaguchi, K. Kobayashi, Mater. Sci. Eng. 40 (1979) 97.
[8] S. Kim, J.R. Weertman, Metall. Trans. A 19 (1988) 999.
[9] P. Marmy, Y. Ruan, M. Victoria, PSI-Bericht 37, Paul Scherrer Institut, Wuerenlingen, Switzerland, 1989.
[10] H. Finkler, M. Schirra, Steel Res. 67 (1996) 328.
[11] I. Alvarez-Armas, A.F. Armas, C. Petersen, Fatigue Fract. Engrg. Mater. Struct. 17 (1994) 671.