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Fatigue mechanisms in an austenitic steel under cyclic loading: Experiments and atomistic simulations

E.A. Soppa*, C. Kohler, E. Roos

Materialprüfungsanstalt (MPA) Universität Stuttgart, Pfaffenwaldring 32, 70569 Stuttgart, Germany

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ABSTRACT

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1. Introduction

Strong and ductile steels with excellent corrosion resistance are of particular interest for various applications. Deformation induced martensitic transformation of fcc austenite into bcc α' martensite offers a unique possibility to enhance significantly the yield stress and the tensile strength of the material at a remaining good toughness [30]. This is the reason for the great research interest in metastable austenitic steels [1,7,8,20,28,29,37]. Nevertheless, despite an intensive research work, reliable models of martensitic transformation suitable for practical assessment of the specimen/component lifetime are missing up to now [32]. The interaction between microstructure and nucleation of martensite and its impact on crack initiation and crack growth is a key factor in this respect. Idrissi et al. [10] investigated, by transmission electron microscopy, a relationship between the stacking fault character and plasticity mode active during loading: ε-martensite transformation or mechanical twin formation in Fe-Mn-Al-Si austenitic steels. Raabe [25] is exploring the influence of carbon content on martensitic transformation (TRIP) or twinning (TWIP) in austenitic steels using ab initio methods. Two variants of deformation induced martensite are mentioned in the literature: ε -martensite (hcp) and α' -martensite (bcc) for which different opinions concerning their nucleation sites in the microstructure

E-mail addresses: ewa.soppa@mpa.uni-stuttgart.de (E.A. Soppa), christopher.kohler@mpa.uni-stuttgart.de (C. Kohler), eberhard.roos@mpa.uni-stuttgart.de (E. Roos).

Experimental investigations on the austenitic stainless steel X6CrNiNb18-10 (AISI – 347) and concomitant atomistic simulations of a FeNi nanocrystalline model system have been performed in order to understand the basic mechanisms of fatigue damage under cyclic loading. Using electron backscatter diffraction (EBSD) the influence of deformation induced martensitic transformation and NbC size distribution on the fatigue crack formation has been demonstrated. The martensite nucleates prevalently at grain boundaries, triple points and at the specimen free surface and forms small ($\sim 1 \, \mu m$ sized) differently oriented grains. The atomistic simulations show the role of regions of a high density of stacking faults for the martensitic transformation.

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exist. According to Nishiyama [21] ε -martensite forms in the areas with a beneficial but irregular distribution of stacking faults. The formation of α -martensite is, according to Poulon et al. [24], connected with dislocation pileups on the active slip planes. Peralta et al. [22] found martensite nucleation sites at crossing points of shear bands. A certain amount of accumulated plastic strain [13,32] and a threshold value of plastic strain $\Delta \varepsilon_{\rm pl}/2=0.3\%$ [1,8] must be reached in order to trigger martensite formation. A modified version of the Olson-Cohen model presented by Lehnhoff and Findley [15] shows the dependence of transformed martensite on the local strain amplitude in the microstructure and its impact on mechanical behaviour of the material and its fatigue resistance [8]. It is known that the growth of microstructurally "short" cracks can last until 90% of the specimen's total lifetime in the HCF regime. The proper description of short cracks requires the consideration of the anisotropic material behaviour on the microscopic scale [12,19,22,26]. According to [17] cracks initiate in an austenitic steel AISI 316L under LCF-loading in persistent shear bands. Chauvot and Sester [5] examined the crack growth in X6CrNiNb18-10 at room temperature by replica. Unlike the authors of this paper they did not find the deformation induced martensitic transformation in any stage of the specimen lifetime. Roth et al. [27] performed in situ and ex situ cyclic deformation experiments in the HCF-regime on a metastable austenitic steel in combination with electron backscatter diffraction. They found that the majority of cracks initiate at twin boundaries in the absence of a martensitic phase. At the tip of a propagating short crack martensitic transformation first takes place. The instability of retained austenite in the plastic zone at the crack tip leads to the

^{*} Corresponding author.

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Table 1 Chemical composition of the X6CrNiNb18-10 steel (in weight %) [6].

С	Si	Mn	Р	S	Cr	Ni	Nb	Ta
0.043 Balance	0.410 e Fe	1.900	0.019	0.002	17.150	10.300	0.660	0.008

formation of martensite [9]. Stolarz et al. [36] observed in a high purity Fe-17Cr-13Ni alloy loaded with a strain amplitude of +0.4% that cracks nucleate in transformed α' -regions irrespective of the grain size and martensite content. All authors share the opinion that the growth rate of short cracks decreases rapidly due to their interaction with microstructural barriers (e.g. [14]). This process is connected with crack closure and a reduction of the propagation rate. The crack closure is enforced by a volume expansion of about 2% during the martensite formation [27,41].

2. Material and experimental methods

2.1. Material

Austenitic stainless steel X6CrNiNb18-10 with the chemical composition as presented in Table 1 was used in this study. This material delivered by DMV STAINLESS Deutschland GmbH was manufactured by melting metallurgy and casting with subsequent plastic forming and solution heat treatment at 1050 °C for 10 min and was water-quenched to room temperature [6].

2.2. Low cycle fatigue test and electron backscatter diffraction

Smooth cylindrical specimens [33] were uniaxially loaded in air at room temperature with different strain amplitudes between 0.25% and 1.5%, R = -1 with a strain rate of 0.1% s⁻¹ until initiation of macroscopic crack. For an interrupted LCF test combined with the electron backscatter diffraction (EBSD) technique, a strain amplitude of 1.5%, R = -1 was chosen. This relatively great loading amplitude should ensure a moderate number of loading cycles until break. The total length of the specimen of 60 mm allowed a non-destructive analysis in the scanning electron microscope after a defined number of cycles and a new loading after breaks. For the observation of the microstructure alteration and crack development at different stages of the specimen lifetime, two narrow flat bands ($3 \text{ mm} \times 20 \text{ mm}$), symmetrically placed on the specimen lateral surface, were grounded and electrolytically polished before loading.

EBSD was used to study the distribution of grain orientations on the specimen surface and to identify deformation induced α' -martensite in the austenitic matrix. The phase identification – austenite or α' -martensite – was based on the comparison of the measured diffraction patterns with the data stored in the ICCD database [11] for the assumed phase. Five overlapping measuring fields of $350 \times 350 \,\mu\text{m}^2$ localized on the flat polished area on the specimen surface were chosen for the EBSD measurements. The resolution of the standard scans was 1 µm; additional scans of interesting details were recorded with higher resolution.

3. Experimental results

3.1. Microstructure

Ni forms with Fe a solid solution, extends the γ -Fe field and stabilizes (partially) the austenitic phase until room temperature or even below it. Cr enhances the corrosion resistance, provided

Fig. 1. Microstructure of the X6CrNiNb18-10 after heat treatment and before mechanical loading.

that it is not exhausted to chromium carbides. The addition of 0.66% of Nb (Table 1) bound the entire C to niobium carbides (NbC) and thus stabilized the chemical resistance of the steel. The resulting microstructure after heat treatment (homogenization) consisted of austenitic grains with several twins and finely distributed NbC located along the sub-grain boundaries or lattice imperfections and forming structures similar to ropes of pearls in the grains (Fig. 1). Two classes of NbC size distribution, with average values of 33 nm and 410 nm, were found by using replica.

In addition to fine NbC, isolated large NbC particles (\sim 30 μ m) were found. They are primary carbides formed during casting and not dissolved during the short homogenization time of 10 min only.

3.2. Combination of interrupted LCF tests with EBSD measurements

Changes in the microstructure and fatigue crack formation in the specimen under cyclic loading were observed by a combination of interrupted LCF tests and EBSD analysis in SEM. The first EBSD scans were recorded on the undeformed specimen in the previously marked areas and served as a reference for further measurements. The subsequent scans were taken after 10, 50, 100, 130, 180, 230 and 300 cycles. The crystallographic orientation of grains and twins in the reference state was randomly distributed. In this state no α' -martensite was found. A small amount (less than 1%) of the bcc-phase was detected as delta-ferrite and was also partially caused by etching pits after electrolytic polishing. These small and flat etching pits had no influence on the crack formation. On the contrary, they were very helpful as identification marks making the mapping of the grain boundary contours to the SEM images easy and reliable [33].

3.2.1. Work hardening behaviour of the material under cyclic loading

Deformation and hardening behaviour of X6CrNiNb18-10 under cyclic loading depends on the strain amplitude (Fig. 2(a)). At the smaller amplitude of 0.25% a minor softening occurs, caused probably by arrangement of dislocations into energetically beneficial patterns. For the medium sized amplitudes until 0.75% the ultimate stress reaches the saturation stadium from the very beginning. For the strain amplitudes greater than 0.75% a pronounced work hardening caused by martensitic transformation was observed. The higher the loading amplitude the greater the volume fraction of α' -martensite in the microstructure. The curves of minimum and maximum stresses recorded during the LCF-tests performed with a strain amplitude between 0.25%-1.5% and separately for the interrupted LCF-tests (strain amplitude of 1.5%, R = -1) are shown in Fig. 2(a, b).

The development of the volume fraction of α' -martensite as a function of loading cycles is presented in Fig. 2(c). The volume

50 µm

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