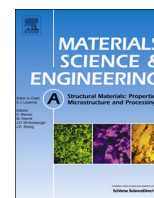




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# Deformation mechanisms induced under high cycle fatigue tests in a metastable austenitic stainless steel

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## ABSTRACT

Advanced techniques were used to study the deformation mechanisms induced by fatigue tests in a metastable austenitic stainless steel AISI 301LN. Observations by Atomic Force Microscopy were carried out to study the evolution of a pre-existing martensite platelet at increasing number of cycles. The sub-superficial deformation mechanisms of the austenitic grains were studied considering the cross-section microstructure obtained by Focused Ion Beam and analysed by Scanning Electron Microscopy and Transmission Electron Microscopy. The results revealed no deformation surrounding the pre-existing martensitic platelet during fatigue tests, only the growth on height was observed. Martensite formation was associated with shear bands on austenite, mainly in the {111} plane, and with the activation of the other intersecting austenite {111}⟨110⟩ slip system. Furthermore, transmission electron microscopy results showed that the nucleation of  $\varepsilon$ -martensite follows a two stages phase transformation ( $\gamma_{fcc} \rightarrow \varepsilon_{hcp} \rightarrow \alpha'_{bcc}$ ).

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## 1. Introduction

Automobile manufacturers are continuously seeking to decrease vehicle weight and polluting emissions, while improving crash performance by energy absorption of the different structural elements [1]. In this sense, recent developments of steel manufacturers have led to the commercialization of multiphase TRIP (transformation induced plasticity) and TWIP (twinning induced plasticity) steels, in which phase transformations and twin formation, respectively, represent the reinforcing mechanisms. These steels are particularly complex since the microstructure evolves with plastic deformation [2].

Metastable austenitic stainless steels can be considered as TRIP steels because plastic deformation, either during forming or under service loads, can lead to a strain-induced transformation from austenite to martensite [3]. These steels are candidate materials for body-in-white construction because they combine excellent formability, crash-absorbing capability and low life-cycle cost, together with good corrosion resistance [4]. However, they have a relatively low yield strength (230 up to 350 MPa) in the annealed state [1,5,6]. These values can be improved up to 1500 MPa by cold rolling. This process induces not only the formation of martensite but also the strain hardening of austenite, leading to high-strength stainless steels grades, although their ductility and formability are then reduced.

The amount of induced martensite depends on processing parameters, such as stress rate, temperature and rate of deformation [7], as well as composition [6,8,9]. Furthermore, plastic deformation of austenite creates the proper defect structure which acts as nucleation site for martensite formation [5]. The dislocation arrangements in the deformed austenite are strongly dependent upon the alloy chemistry, stress, strain, stress triaxiality, strain rate, initial micro-textures, slip systems, temperature of deformation and the extent of deformation-induced phase transformations [10]. The deformation process can induce the formation of two types of martensite in austenitic stainless steels:  $\varepsilon$  and  $\alpha'$ . The  $\varepsilon$ -martensite is hcp while  $\alpha'$  is bcc [11]. The typical transformation sequence can be summarized as  $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ , where the  $\gamma \rightarrow \varepsilon$  transformation has been proposed for austenitic stainless steels deformed under tension as well as by rolling, by a variety of authors [12,13]. On the other hand, the direct transformation of austenite into  $\alpha'$ -martensite,  $\gamma \rightarrow \alpha'$ , through dislocation reactions has also been found to be possible by Nolze [14]. Kundu and Bhadeshia [15] have shown that it is not necessary to consider the two-stage sequential transformation of austenite, first into  $\varepsilon$  and then into  $\alpha'$ , in order to calculate the transformation texture. It is well known that martensite transformation produces an increase in volume which results in the build-up of residual stress [16].

For a reliable application of metastable steels in automotive parts, their fatigue behaviour must be established. Extensive studies on the fatigue response of metastable stainless steels performed in the 1970s [17–19] reported different behaviours depending on testing conditions. In particular, the effect of

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induced martensite on the fatigue properties is still controversial. The formation of martensite during deformation is known to be harmful for low cycle fatigue (LCF) regime, while a small amount of martensite can be beneficial for high cycle fatigue (HCF) and very high cycle fatigue (VHFC) regimes. Recently, Müller-Bollenhagen et al. [20] concluded that automotive stainless steel structures undergoing cyclic loads beyond  $2 \times 10^6$  cycles should not exceed a deformation-induced martensite content of 27 vol%.

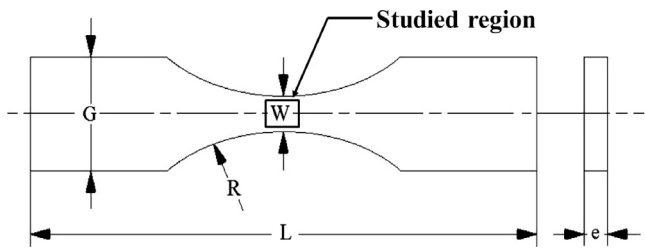
In this context, the aim of the present paper is to study the interaction between martensite and austenite deformation mechanisms involved during fatigue tensile tests. Although previous studies carried on metastable steels have proposed specific mechanisms there is a lack of microstructural observations. In fact, main current works are based on simulation and/or numerical analysis [21–23]. Therefore, advanced techniques such as Atomic Force Microscopy (AFM), Focused Ion Beam (FIB), Scanning Transmission Electron Microscopy (STEM) and Transmission Electron Microscopy (TEM) have been used in this investigation in order to not only support them but also analyse in detail the evolution of the microstructure in the austenitic grains through a HCF test.

**Table 1**  
Chemical composition of the studied stainless steel AISI 301LN (wt%).

	C	Cr	Ni	Mn	Si	Mo	N
AISI 301LN	0.02	17.48	7.03	1.23	0.45	0.12	0.12

**Table 2**  
Mechanical properties for the commercial AISI 301LN.

Ultimate tensile stress, $\sigma_{uts}$ (MPa)	Yield stress, $\sigma_{ys}$ (MPa)	HV <sub>10</sub>
922	670	264



**Fig. 1.** Schema of the fatigue specimen indicating the studied region.

## 2. Experimental process

### 2.1. Sample preparation

The studied material was a commercial AISI 301LN stainless steel, equivalent to EN 1.4318, supplied by Outokumpu (Finland) as sheets with a thickness of 2 mm, with an average grain size of  $40 \mu\text{m}$  and an initial percentage of martensite of  $8 \pm 1\%$ . The chemical composition and mechanical properties are shown in Tables 1 and 2, respectively. Fatigue samples were machined according to ASTM E 466-96 standard [24]. Fatigue specimens were machined by laser cutting. Before testing, they were polished with silicon carbide and then diamond suspension of 30, 6 and  $3 \mu\text{m}$ . Finally, a neutral suspension of 20 nm alumina particles was used in order to remove possible work hardening introduced during surface preparation.

### 2.2. Fatigue tests

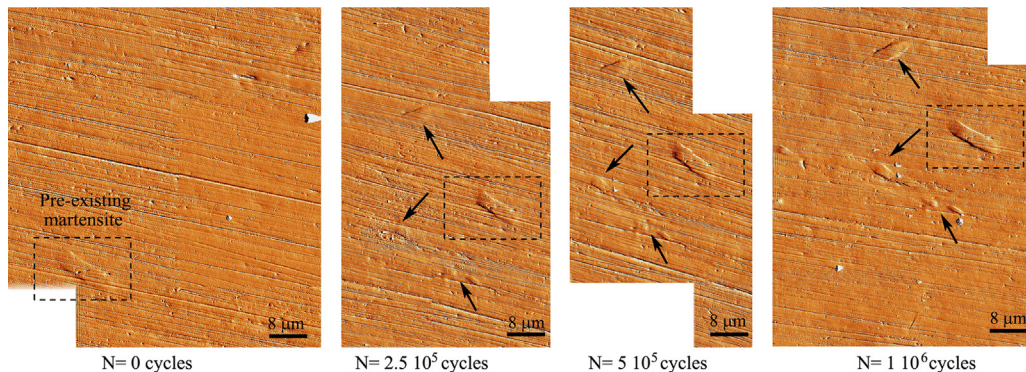
Specimens were subjected to fatigue tests at a stress ratio ( $R$ ) of 0.1 in a resonant testing machine Rumul Mikroton. Tests were conducted under load control at frequencies around 150 Hz. The testing procedure started with a  $\sigma_{max} = 450 \text{ MPa}$  corresponding to almost 50% of the ultimate tensile strength. The test was stopped at  $2.5 \times 10^5$  cycles and also after  $5 \times 10^5$ ,  $7.5 \times 10^5$  and  $10^6$  cycles. For each stage, AFM images were taken in the same region, located in a zone of maximum stress field concentration (Fig. 1), in order to observe the evolution of a pre-existing martensite platelet. The observed area was  $80 \mu\text{m}^2$  in size, approximately.

### 2.3. Surface observation

Surface observations were performed by AFM, working in tapping mode. A Dimension 3100 Microscope (Bruker) was used to carry out the different measurements. All the images and cross-section profiles were processed with the WSxM software [25] in order to check the roughness evolution along the fatigue life of a pre-existing martensite.

### 2.4. Sub-surface characterization

The damage induced under the pre-existing martensite during the fatigue tests was characterized using a dual beam FIB/SEM (Zeiss Neon 40). A thin platinum layer was deposited on the sample prior to FIB machining in order to minimize ion-beam damage. A  $\text{Ga}^+$  ion source was used to mill the surface at a voltage of 30 kV. The final polishing of the cross-sections was performed at 200 pA.



**Fig. 2.** AFM images (error signal mode) of the maximum stress field region in the initial state and after  $2.5 \times 10^5$ ,  $5 \times 10^5$  and  $1 \times 10^6$  cycles. The dash square exhibits the pre-existing martensite. Black arrows identify the new martensite platelets. It can be seen that the pre-existing martensite remains quite constant in shape and it only grows in height.

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