



# Experimental characterization of short crack nucleation and growth during cycling in lean duplex stainless steels



R. Strubbia<sup>a,\*</sup>, S. Hereñú<sup>a</sup>, A. Giertler<sup>b</sup>, I. Alvarez-Armas<sup>a</sup>, U. Krupp<sup>b</sup>

<sup>a</sup> Instituto de Física Rosario – CONICET, Universidad Nacional de Rosario, Argentina

<sup>b</sup> Faculty of Engineering and Computer Science, University of Applied Sciences Osnabrück, Germany

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## ABSTRACT

Considering that many applications of Lean Duplex Stainless Steels (LDSSs) involve cyclic loading, the aim of this paper is to study short crack initiation and growth during low (LCF) and high cycle fatigue (HCF) in AL 2003 (UNS S32003). Electron Backscattered Diffraction (EBSD) analysis of plastically active grains allows to determinate the slip systems and their associated Schmid factor (SF). Additionally, the dislocation structure developed during cycling is observed by transmission electron microscopy (TEM). Whereas in HCF cracks nucleate at grain boundaries, during LCF cracks nucleate along intrusion/extrusions in ferritic grains and as they reach austenitic grains grow along active slip systems or by double slip system. Moreover, phase boundaries and grain boundaries act as effective barrier against crack propagation.

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## 1. Introduction

Duplex stainless steels (DSS) are two-phase alloys with approximately equal proportions of ferrite ( $\alpha$ ) and austenite phases ( $\gamma$ ). The duplex microstructure combines properties of both phases that may even be enhanced in the mixture; the ferrite provides high strength and resistance to stress corrosion cracking, while the austenite contributes to good ductility and general corrosion resistance [1,2]. Recently, many companies have tried to reduce the cost of duplex stainless steels replacing more expensive elements, like Ni, by other lower cost elements, giving rise to a low-alloyed grade of DSS named Lean DSS. To maintain the typical microstructural balance of DSS, the reduction in Ni content is compensated by an increasing amount of Mn and N elements.

LDSSs are being used in many industrial applications subject to cyclic loading [3]. Fatigue damage generally initiates at plastic strain concentration sites. These are the result of different stages during fatigue, which starts with the evolution of dislocations structure and the consequent localization of the strain and the final nucleation and propagation of cracks [4]. Particularly, short crack behavior is influenced not only by loading conditions but also by microstructural features including chemical composition, volume fraction of phases, phase distribution, grain size and heat treatment [5]. Some researchers [6–9] have studied short crack nucleation in standard DSS during fatigue. However, in low-alloyed

duplex stainless steels there is scarce information correlating the dislocation structure, the evolution of the surface damage and short crack nucleation on low-alloyed duplex stainless steels [10]. Therefore, the aim of this work is to study microstructural short cracks nucleation and propagation during LCF as well as short cracks nucleation HCF in a LDSS and the correlation with the dislocation structure developed in both fatigue regimes.

## 2. Material and experimental procedure

### 2.1. Material

The investigated material was the AL 2003 (UNS S32003) LDSS. This steel was received in longitudinally welded stainless steel pipe. The manufacturing process of the pipes includes a hot rolled stage and a subsequent welding of the tube. A thermal treatment at 1050 °C followed by a rapid water quench was finally carried out to the tube. The chemical composition of the present LDSS in weight percent is: C:0.021; Cr:22; Ni:3.8; Mo:1.8; Mn:1.73; Si:0.22; P:0.024; N:0.18; Fe: balance. The yield stress of the LDSS AL 2003 is 579 MPa. From slabs taken parallel to the axis of the pipe, flat specimens for low and high cycle fatigue were prepared. Fig. 1a shows the geometry of the specimen used for fatigue tests. An optical micrograph from the as-received LDSS is observed in Fig. 1b. The microstructure morphology consists mainly of a lamellar structure of bright austenite islands embedded in a grey etched ferrite matrix. This steel presents approximately the same volume fraction of austenite and ferrite. The EBSD technique was used for

\* Corresponding author.

E-mail address: [strubbia@ifir-conicet.gov.ar](mailto:strubbia@ifir-conicet.gov.ar) (R. Strubbia).

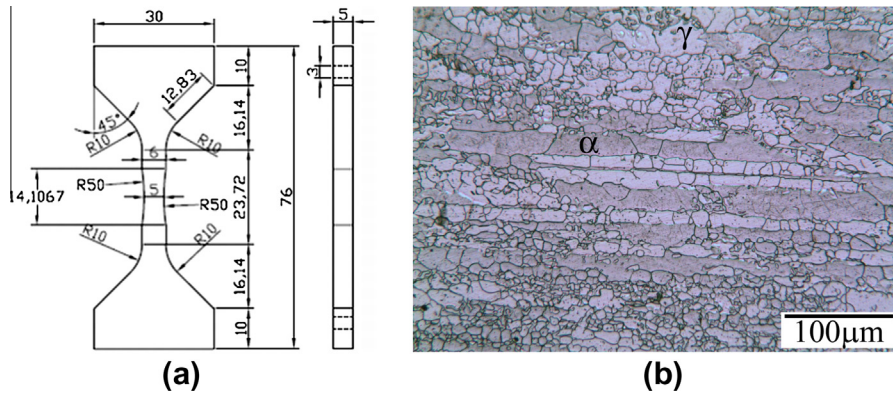


Fig. 1. (a) Specimen geometry and (b) microstructure of AL 2003.

grain size determination. The average grain size and their standard deviations are  $7 \pm 4 \mu\text{m}$  for the ferritic phase and  $4 \pm 2 \mu\text{m}$  for the austenite. Vickers hardness was measured in both phases; the values obtained were  $310 \pm 10$  for ferrite and  $320 \pm 10$  for austenite.

## 2.2. Specimen preparation

Specimens for mechanical tests were initially ground and polished with sequentially finer grits paper and finally were electrolytically polished; using A2 supplied by Struers as electrolyte. This surface preparation allows the observation of the structure during tests and the acquisition of good quality electron back-scattered diffraction patterns (EBSD).

## 2.3. Mechanical tests

In order to observe the damage evolution during LCF tests, a central sector of the specimens was selected. Surface damage observations of this zone were performed by in situ microscopy before and during the LCF test using an optical system composed of a CCD camera JAI mod. CM-140MCL with a  $50\times$  objective, focal length of 13 mm, depth of field of  $\pm 1 \mu\text{m}$  and a  $12\times$  ultra zoom device mounted on the fatigue test machine. LCF tests were conducted at room temperature under fully reversed plastic strain control, at a constant total strain rate  $\dot{\epsilon} = 2 \times 10^{-3} \text{ s}^{-1}$ , with a plastic strain range  $\Delta\epsilon_p = 0.2\%$ . The LCF test with these conditions was repeated five times so as to discern the occurrence of the initial slip lines with the surface relief in each phase and to calculate their corresponding standard deviations.

Besides, push–pull HCF tests were carried out in servo-hydraulic testing system at room temperature under stress control with stress amplitude of  $\sigma = 425 \text{ MPa}$ , stress ratio  $R = -1$  and frequency  $f = 10 \text{ Hz}$ . In situ observation of specimens during HCF was not possible because the optical system mounted on the HCF testing machine does not allow discerning the small grains of the AL 2003. This optical system is composed of a long-distance QUESTAR optical microscope coupled to a digital camera.

In order to compare the strain rates in LCF and HCF tests, the total strain rate of HCF tests was estimated. This quantity was approximated as the ratio between twice the total strain range measured at midlife to fracture ( $N_f/2$ ) and the frequency, resulting that  $\dot{\epsilon}$  is of the order of  $1 \times 10^{-1} \text{ s}^{-1}$ .

## 2.4. Slip systems determination

After LCF and HCF tests a Scanning Electron Microscope (SEM) equipped with EBSD detector was used. EBSD measurements provide the Euler angles ( $\varphi_1, \Phi, \varphi_2$ ) that characterize the

three-dimensional orientation of the grains. These data enable to determine the slip systems, their SF and their angles relative to the tensile axis. On the other hand, the angles between the surface slip markings and the loading axis were measured. The comparison of these angles with the calculated ones permits to identify the activate slip systems and their corresponding SFs.

## 2.5. TEM observation

In order to analyze the dislocation structure after fatigue, thin foils were prepared from discs cut parallel to the tensile axis of the specimen using a double-jet electropolisher. The dislocation structures of the as-received material and of the specimens cycled up to failure were observed by TEM operating at 100 kV.

## 3. Results and discussion

Fig. 2 shows the dislocation structure in the as-received condition in both phases of AL 2003. It is important to remark that after the thermomechanical manufacturing process, considerable dislocation density remains in the bulk.

The optical in situ observations during LCF reveals that the first deformation lines appear in the ferrite phase after  $100 \pm 50$  cycles (Fig. 3a) while in the austenitic phase they appear after  $300 \pm 100$  cycles (Fig. 3b). As cycling proceeds the surface damage evolution is mainly concentrated in the ferrite (Fig. 3c). According to a previous study in this steel [10], some of the slip lines in ferrite evolve to extrusions at the surface where short cracks finally nucleate. The preceding fatigue damage evolution is corroborated in the present work. Moreover, the surface damage observation has also enabled to note that the slip markings have different morphology in each phase. While in the austenite phase the slip bands are straight, in ferritic grains slip lines are wavy. This wavier feature of the ferrite phase is attributed to the high capacity of cross-slip developed by b.c.c structures that allow the activation of several slip systems.

An initiated crack within the ferritic phase can present two alternative crack growing mechanism as it reaches austenitic neighbouring grains (Fig. 4): grows along a favourable slip plane with high SF (stage I), or alternating between two slip systems (stage II). This last growing mechanism has already been reported by others authors [11,12]. Additionally, these authors propose a useful method to calculate the plane of the crack in stage II. Applying this procedure to the present steel, it is found that some cracks grow by double slip on lattice planes of  $\{110\}$  type (Fig. 4). It is important to remark that independently of the growing mechanism (stage I or II), cracks grow approximately perpendicular to the specimen axis.

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