EBSD STUDIES ON THE INFLUENCE OF TEXTURE ON THE SURFACE DAMAGE MECHANISMS DEVELOPED BY AGED DUPLEX STAINLESS STEELS CYCLICALLY LOADED

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Abstract. Interrupted low cycle fatigue tests up to 20% of fatigue life have been performed on electropolished specimens of an aged superduplex stainless steel. Crystallographic orientation results obtained by EBSD analysis have allowed to determine the individual orientation of each individual grain as well as the slip systems plastically active. EBSD results demonstrate the requirement for the accomplishment of several conditions for a slip plane/system to become active. These conditions are different for austenite and ferrite.

Resumen. Se han llevado a cabo ensayos de fatiga oligocíclica, hasta un 20% de vida a fatiga, en probetas electropulidas de un acero inoxidable superduplex envejecido térmicamente. Los resultados de orientación cristalográfica obtenidos mediante análisis por EBSD han permitido determinar la orientación individual de cada uno de los granos, así como los sistemas plásticamente activos. Los resultados obtenidos demuestran que varias condiciones relacionadas con la orientación del grano son requeridas para que un sistema de deslizamiento sea activo. Estas condiciones son diferentes para la austenita y la ferrita.

1. INTRODUCTION

It is well-known that all forms of surface damage caused by cyclic loading are strongly influenced by the microstructure, i.e. grain size, grain orientation, grain boundary geometry, precipitates and other second phase particles.

The effect of grain boundaries on the fatigue resistance of polycrystals has been studied for many years because in some metals and alloys crack nucleation takes place at these sites as a consequence of elastic and plastic incompatibilities [1]. It is known that grain boundaries (GBs) have a preponderant role in small crack propagation by acting as microstructural barriers to small crack propagation [2-6]. This fact has promoted an increasing interest for the knowledge of the role developed by GBs on fatigue damage initiation and propagation. Since crack initiation and growth are strongly influenced by the orientation of adjacent grains, it results very important to know and understand how this factor determines fatigue damage formation.

Recently, with the development of the EBSD technique, bicrystals have primarily been used to clarify GB effects on the transmission of the plastic activity from one grain to another during monotonic deformation of polycrystalline materials. Studies have been mostly centered in aluminum bicrystals, copper bicrystals [7] and Fe-3Si alloy bicrystals [8]. Also, a few studies deal with the influence of texture on the cyclic damage, e.g. the interaction of PSBs generated during cyclic loading with large- and low- angle GBs in copper bi- and multi-crystals [9-10].

In thermally aged duplex stainless steels (DSSs), slip bands represent the main crack initiation site, either in austenite or ferrite depending on the applied strain amplitude [11]. Moreover, grain boundaries have been described to actively act as microstructural barriers to small crack propagation. Thus, grain boundary constraints probably play the most important role in the crack initiation and propagation process of the DSSs under consideration and slip incompatibilities at the interphase contribute to an increasing stress value at these sites. In this sense, the capability for crossing grain or phase boundaries, which is strongly dependent on the misorientation between neighbouring grains, would be determinant in the fatigue process.

The scope of this paper is to investigate by means of the EBSD technique, the parameters that influence the fatigue damage process in each of the constitutive phases (ferrite and austenite) of the superduplex stainless steel (SDSS) EN 1.4410 in aged condition (200 hours at 475 ºC, conditions that induce the α' formation within ferrite leading to a strong embrittlement of the material [12]). Orientation studies in ferritic and austenitic grains have been performed in order to investigate the slip activity developed within each individual phase.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

The material used in this investigation is a superduplex stainless steel EN 1.4410 (commercial designation SAF 2507). It was supplied in the form of cylindrical bars of 20 mm in diameter, by Sandvik AB, Sweden. Table 1 gives its chemical composition in weight percent. The microstructure of the superduplex consisted of 56% austenitic grains on a ferritic matrix, and the shape of the grains is highly elongated in the rolling direction. This direction corresponds with the loading axis in all the specimens tested.

Table 1. Chemical composition in weight percent and volume fractions of EN 1.4410.

	Cr	Ni	Mo	N	α/γ (%)
EN 1.4410	25.0	7.0	3.79	0.26	46/54

Cylindrical specimens were machined from the bars, but for EBSD studies, a perfectly well-polished flat area is required. Thus, previous to the machining of this plane, a study of the texture of the material parallel and perpendicular to the rolling direction was performed. The results demonstrated that the SDSS used in this investigation did not present a preferred radial texture. Therefore, three planes with 2.5 mm in width and 8 mm in length were mechanized at each 120º around the specimen's circumference, as it is shown in Fig. 1, and following the rolling direction to ensure the equilibrium of the stresses in the gauge section. In order to obtain a good quality in the EBSD patterns, the specimens and the flat areas were first mechanically and then electrolytically polished with 10% perchloric acid and 90% acetic acid solution at 45V during 10 – 15 seconds. Then specimens were rinsed and cleaned with ethanol and acetone in an ultrasounds bath.

Prior to the fatigue tests, texture measurements were performed on a scanning electron microscope JEOL 6100 equipped with an EBSD system. Orientation imaging microscopy (OIM) commercialized by TSL was used for EBSD measurements. The specimen was mounted on a support which ensures an angle of 70º required for EBSD technology and automatic scans were performed in beam control mode with OIM software. For each specimen a minimum of eight scans (100 x 100 μm^2 each scan) were performed on the electropolished flat area. The scan spatial resolution selected was 2 steps/μm.

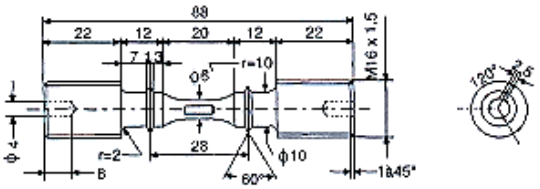


Figure 1. Geometry of the specimens used for EBSD studies (units in mm).

Fatigue tests were performed using an electromechanical Instron machine (model 1362). Two axial extensometers directly attached to the specimen were used to measure and control strain. All tests were performed under plastic strain control, R = -1 and 0.3 Hz. Only one plastic strain amplitude was considered, $\Delta\varepsilon_{pl}/2 = 2 \times 10^{-3}$, value that corresponds to an austenitic/ferritic-like behaviour of the material, i.e. both constitutive phases participate in plastic strain accommodation [13]. Tests were stopped at 20% of fatigue life.

Following, the selected areas previously studied by EBSD were analyzed by SEM, with the purpose to match the fatigue damage with the texture measurements (Fig. 2). It is worth to point out that investigations have demonstrated that cyclic loading do not produce changes of the misorientation of adjacent grains [14].

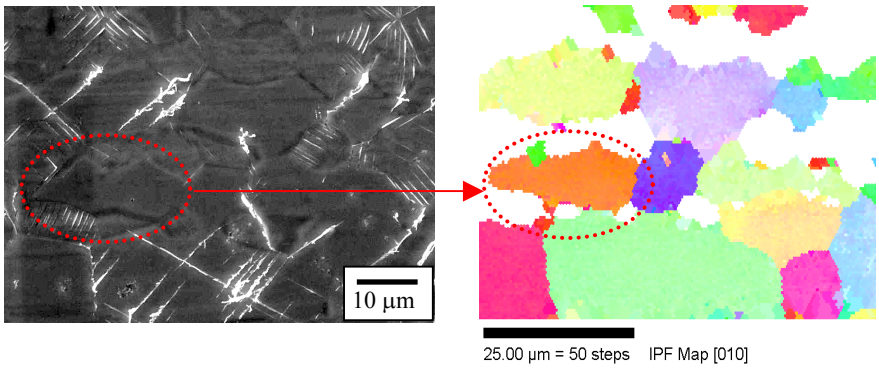


Figure 2. SEM photograph of surface damage on an aged specimen tested at 20% N_f and its corresponding EBSD mapping for ferrite.

3. RESULTS AND DISCUSSION

SEM observations showed (Fig. 2) that contrary to the homogenously damaged austenite with high density of PSBs, aged ferrite presented grains with low density of pronounced extrusions, and others practically without any visible signs of surface damage. This difference in plastic activity within the constitutive phases of the aged DSSs can be attributed to the ageing treatment. For DSSs, thermal ageings at temperatures below 500 ºC lead to the formation of the α' phase within ferrite via a mechanism of spinodal decomposition or via a mechanism of nucleation and growth. Independently of the mechanisms, the formation of these areas riches and depleted in Cr within ferritic grains leads to significant changes in the intrinsic mechanical properties of ferrite, and also of the dual-phase alloy. Thus, an increase in σ_{YS}, σ_{UTS} and hardness is observed. Therefore, in aged DSSs, the yield stress of the constitutive phases are markedly different, i.e. the softer austenitic phase is the first to participate in plastic strain accommodation, whereas at this plastic strain amplitude, the role of the aged ferrite is scarce. Moreover, a change in the glide and fracture mechanisms within ferrite takes place, i.e. ferrite starts to develop a more planar dislocation glide due to the demodulation of the composition during dislocation motion and also microcleavage of ferrite is observed [15].

In f.c.c. metals the observed surface traces result from dislocation movement in four {111} slip planes each with three possible <110> slip directions. Therefore, for austenite 12 possible glide conditions were considered. However, for b.c.c. structures the dislocation glide takes place always following the <111> direction. Experimental results have revealed that the slip planes correspond primarily to the type {110}, {112} and {123}, although sometimes planes with higher Miller index are activated [16]. However, only {110}<111>, {112}<111> and {123}<111>, i.e. 42 slip systems, were taken into account for ferrite.

The values for the Schmid factor (μ) were calculated for each active slip system for both constitutive phases of the aged SDSS from the experimentally determined G orientation matrices which were obtained from the Euler angles measured and calculated by the EBSD system. The value of μ for the primary slip system plastically active for austenite was found to be higher than 0.4. Secondary slip systems presented values of μ higher than 0.35 and, moreover, it was proved that the no-activation of a secondary slip system was not due to the inexistence of slip systems with μ higher than 0.35. Finally, it was stated that slip systems with a $\mu < 0.35$ were never observed to become plastically active for this plastic strain amplitude and this percentage of fatigue life. Therefore, the value of μ is a parameter very decisive for the development of the plastic activity of an austenitic slip system. However, the activation of a slip system also depends on other factors concerning to the texture which will be studied with detail further.

The determination of the slip system plastically active for ferrite was more complex as a consequence of the 42 slip systems that can participate in plastic strain accommodation. In most of the α grains with surface damage analyzed, two or more slip systems presented the same traces, i.e. the same angle, with respect to the loading or the surface axis. Therefore, all these slip systems were considered. Most of them presented values of μ higher than 0.3; however slip systems with $\mu < 0.3$ were also stated to develop plastic activity whereas other slip systems with higher μ value did not become active. Table 2 shows the distribution of the Schmid factor for each plastically active slip system. From this table it can be stated that for {110} planes, μ takes values mainly higher than 0.4, whereas for the other slip planes, i.e. {112} and {123}, although $\mu > 0.4$ is predominant, planes with lower values can easily become plastically active. Therefore, the Schmid factor is not a determinant factor to induce the activation of a slip system within ferritic grains and other parameters must be considered to explain whether or not a ferritic grain results plastically strained.

Finally, the values for all the slip systems of undamaged α grains were calculated. Results confirmed the existence of slip systems with $\mu > 0.4$ and from a statistic point of view, no significant differences between the Schmid factors of plastically deformed grains and those grains in which no plastic deformation could be observed. This point suggests that the plastic deformation occurring near grain boundaries is mainly a consequence of the grain boundary constraints. Therefore, the development of slip activity within α grains depends on the applied stress, grain size, as well as the internal stresses concentrated at the grain boundary which at the same time are strongly dependent on the plastic activity developed at the neighbouring grains and their relative orientation.

Table 2. Schmid factor distribution for each set of slip planes plastically active in ferrite.

	$\mu > 0.4$	$0.4>\mu>0.3$	$0.3>\mu>0.2$	$0.2>\mu>0.1$	$\mu < 0.1$
{110}	82.6 %	17.4 %	-	-	-
{112}	53.8 %	7.8 %	20.5 %	-	17.9 %
{123}	58.7 %	12.0 %	9.3 %	9.3 %	10.7 %

The values of ϕ (angle of the normal to the slip plane with respect to the loading direction) and λ (angle of the normal to the slip plane with respect to the surface)

were also calculated for each active slip system of austenite and ferrite. From Fig. 3 it can be stated that for austenite, the higher frequency of ϕ was obtained for values in the range 40 - 60º, however a more uniform distribution was obtained for λ (Fig. 4) but values lower than 30º were not encountered.

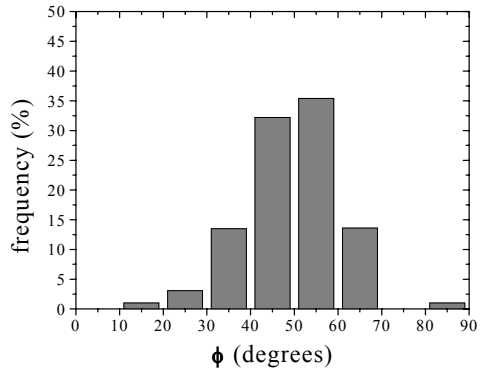


Figure 3. Distribution of ϕ for the austenitic phase of the aged EN 1.4410.

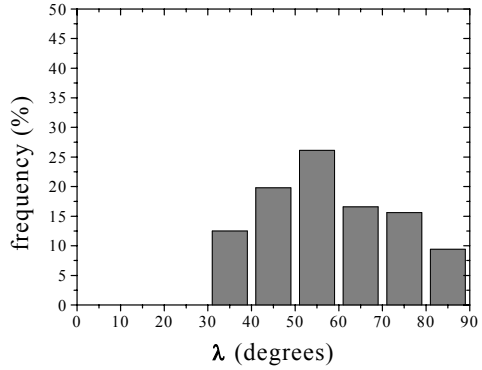


Figure 4. Distribution of λ for the austenitic phase of the aged EN 1.4410.

Fig. 5 shows the distribution for ϕ obtained for each set of slip planes considered for ferrite. For all the possible slips systems considered, the maximum frequency for ϕ was located at values within the range 30 - 60º, whereas for λ it was not possible to define a range of angles for which the slip activity is predominant as a more uniform distribution was obtained (Fig. 6). Nevertheless, it can be stated that a higher tendency for $\lambda > 60º$ was found. It is worth to note that, as it occurred with the austenitic phase, those values of $\lambda < 30º$ presented very low to no plastic activity.

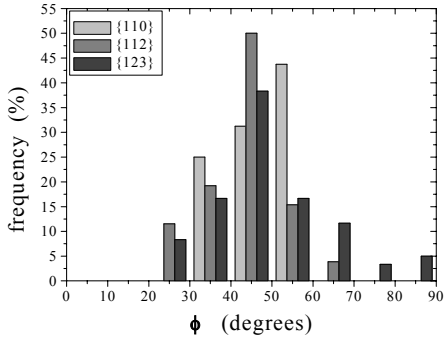


Figure 5. Distribution of ϕ for the ferritic phase of the aged EN 1.4410

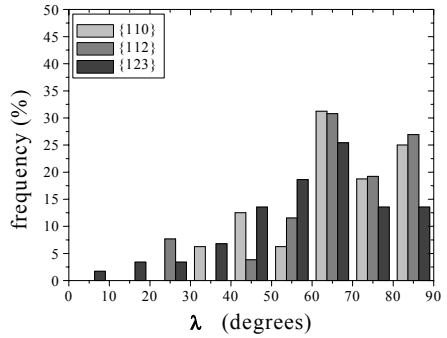


Figure 6. Distribution of λ for the ferritic phase of the aged EN 1.4410.

Afterwards, the parameters **a** and **b,** which were defined as the angle of the slip direction with respect to the loading axis and the surface, respectively, were studied for each active slip system. The values of **a** obtained for austenite (Fig. 7) showed a higher frequency within the range 30 - 50º meanwhile for **b**, a high scatter was found (Fig. 8). Also, it is worthwhile to note that the slip activity is concentrated in those slip systems with **b** > 30º.

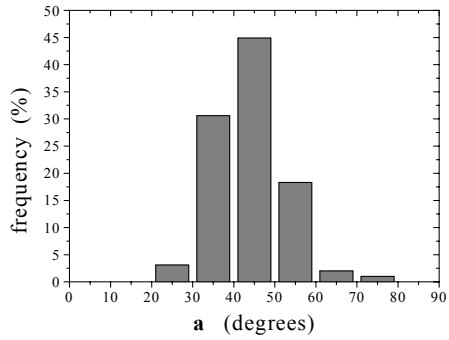


Figure 7. Distribution of **a** for the austenitic phase of the aged EN 1.4410.

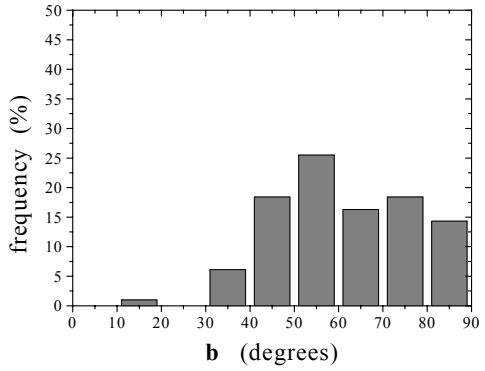


Figure 8. Distribution of **b** for the austenitic phase of the aged EN 1.4410.

When analyzing the values of **a** obtained for the slip planes {110},{112} and {123} of aged ferrite, the same tendency as for austenite can be observed (Fig. 9). Thus, for values of **a** within the range 30 - 60º a maximum frequency was attained (Fig. 7). Referring to the parameter **b**, again a big scatter on the distribution of angles was reached (Fig. 10), except for the values corresponding to the {112} planes, where a maximum frequency was attained for angles within the range 70 – 80º. Note that, for all the slip systems considered, only those with **b** > 30º presented plastic activity.

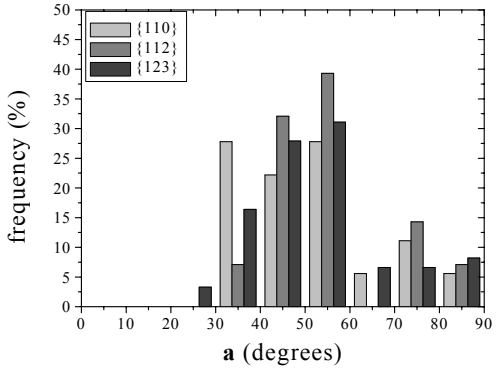


Figure 9. Distribution of **a** for the ferritic phase of the aged EN 1.4410.

Also, it has been proved that those grains without any detectable slip activity at the surface presented values of **ϕ** and **a** within the range of maxima frequencies encountered. This fact in conjunction with the existence of slip planes with high Schmid factor values of the primary slip system point out that the plastic inactivity of the aged ferritic grains can not be related to the absence of slip systems favourably oriented. Therefore, it could be thought that as austenite is the major responsible for plastic strain accommodation at this plastic strain amplitude, some ferritic grains could be only elastically strained which would explain the absence of slip markings on the surface. Therefore, the development of plastic activity would be related to the grain size (usually small grains are easily strained) as well as the plastic activity developed by their neighbouring grains, i.e. the stress constraints at the grain or interphase boundaries.

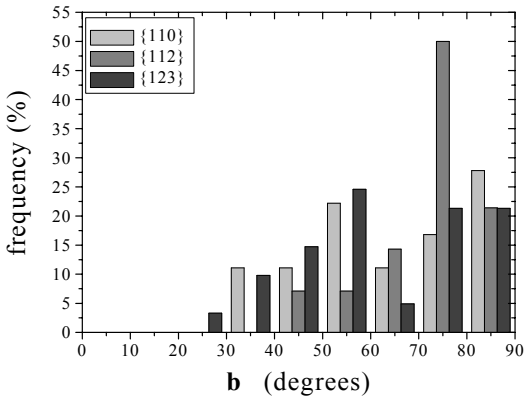


Figure 10. Distribution of **b** for the ferritic phase of the aged EN 1.4410.

Finally, the angle of the traces with respect to the loading axis were analysed. From Fig. 11 and 12, it can be stated that whereas for austenite no direct correlation between the angle of the trace and the sense of the applied stresses could be discerned (Fig. 11), for ferrite, higher frequencies were obtained for those traces with an inclination of 40 - 60º (Fig. 12). Studies on the fatigue behaviour of aged DSSs have demonstrated that at this strain amplitude, ferrite participates in the plastic strain accommodation and its inherent brittleness leads to a microcleavage fracture mechanism. From SEM analysis, it has been stated that these planes are oriented at 40 - 60º with respect to the loading axis, values coincident with the active planes determined by EBSD. But, these plastically active planes do not correspond to the typically cleavage planes described in the literature for ferrite, i.e. {001} planes [17]. However, it is worth to point out that the b.c.c. ferrite may experiment deformation twinning which can cause cleavage in age-hardened DSSs [18-19]. Since the habit plane of twins in ferrite corresponds to the {112} planes [17]), the high plastic activity, demonstrated by EBSD, of these set of planes, may point out that microcleavage of aged ferrite occurs along the twin habit planes.

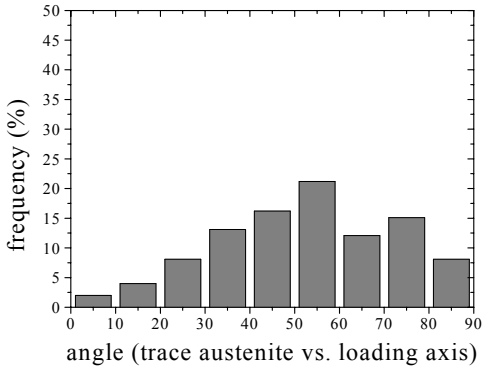


Figure 11. Distribution of the angle of the traces with respect to the loading axis for austenite of the aged EN 1.4410.

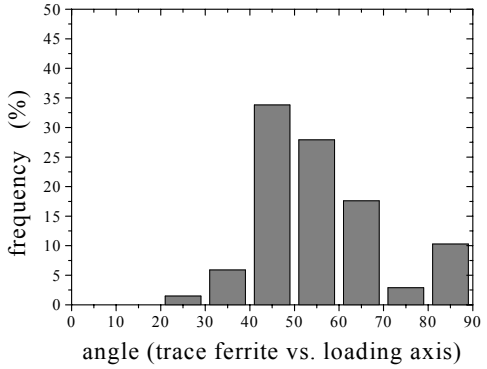


Figure 12. Distribution the angle of the traces with respect to the loading axis for ferrite of the aged EN 1.4410.

4. CONCLUSIONS

The plastic activity of austenite depends on its intrinsic glide and orientation characteristics, i.e. Schmid factor, glide direction, etc. whereas for ferrite the plastic activity is strongly influenced by the plastic activity developed within the neighbouring grains as well as their relative misorientation.

The absence of slip traces within a α grain cannot be explained by a low Schmid factor or by a negligible component of the slip vector. Therefore, such grains are only elastically strained, and the absence of plastic activity could be attributed to their grain size as well as the plastic activity developed in their neighbouring grains.

Deformation twinning may cause cleavage within ferrite of the aged SDSS, which is expected to occur on the {112} twin habit planes.

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