



MICROSTRUCTURAL BEHAVIOR OF 316L AUSTENITIC STAINLESS STEEL UNDER GROOVE PRESSING (GP)

Comportamiento microestructural del acero inoxidable austenítico 316L sometido a presión calibrada (GP)

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ABSTRACT

The microstructural behavior of 316L austenitic stainless steel sheets subjected to severe plastic deformation (SPD) by the constrained groove pressing (CGP) technique without mass flow limitation up to 4 passes was analyzed. Two tool steel dies were used: a corrugated die with geometry ($t = 2$ mm and angle of 45°) and a flat die generating a maximum equivalent deformation of $\epsilon \sim 4.64$. The microstructural behavior was analyzed using characterization techniques: Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Electron Backscatter Diffraction (EBSD). The results were analyzed from three different perspectives: 1. SEM was used to evaluate the presence of martensite induced by deformation. 2. The presence of martensite of the ϵ and α' types were confirmed by XRD, and 3. The effect of severe plastic deformation on the behavior of the austenitic grain in a steel with an SFE=28 mJ/m² was analyzed by EBSD. The results showed a significant reduction in the austenitic grain size in the presence of special $\Sigma 3$ -type boundaries. Regarding the deformation mechanisms, the presence of dislocation gliding and mechanical twinning was observed, with the transformation of austenite $\rightarrow$ twinning austenite $\rightarrow$ martensite (α').

Keywords: constrained groove pressing; martensite transformation; mechanical twinning; stainless steel; X-ray diffraction.

RESUMEN

El comportamiento microestructural de placas de acero inoxidable austenítico 316L sometido a deformación plástica severa (SPD) mediante la técnica de presión calibrada restringida con limitación en el flujo masico (GP) hasta 4 pases fue analizada. Para esto se utilizaron 2 matrices de acero para herramientas, una matriz corrugada con una geometría ($t = 2$ mm y ángulo de 45°) y otra matriz plana, lo cual generan una deformación máxima equivalente de $\epsilon \sim 4,64$. El comportamiento microestructural se analizó mediante técnicas de caracterización tales como: microscopía electrónica de barrido (SEM), difracción de rayos X

(XRD) y difracción de electrones retrodispersados (EBSD). Los resultados se analizaron desde tres puntos de vista diferentes: 1. Mediante SEM se evaluó la presencia de martensita inducida por deformación. 2. Mediante DRX se ratificó la presencia de martensita del tipo ϵ y α' y 3. Mediante EBSD se analizó el efecto de la deformación plástica severa sobre el comportamiento del grano austenítico en un acero con una SFE=28 mJ/m². Los resultados mostraron una reducción significativa del tamaño de grano austenítico con presencia de límites especiales del tipo $\Sigma 3$. En cuanto a los mecanismos de deformación, se observó la presencia el deslizamiento de las dislocaciones y maclado mecánico, con transformación de austenita $\rightarrow$ austenita maclada $\rightarrow$ martensita (α').

Palabras clave: acero inoxidable; difracción de rayos X; maclado mecánico; presión calibrada restringida; transformación martensítica

1. INTRODUCTION

The AISI 316L stainless steel is a material widely used in the petrochemical industry, marine industry or in industries located in saline environments, due to its good corrosion, chloride, and oxidation resistance. This material contains a high content of Cr and Ni, which range for Cr between 16% and 18%, and for Ni between 10% and 14%. This combination provides good corrosion resistance, ductility and weldability. However, it has low hardness and mechanical strength, characteristics that cannot be improved by heat treatment [1] [2]. This steel contains low carbon content and other alloying elements that prevent the transformation of phases during the heating or cooling processes, which implies that the improvement in its mechanical behavior is based only on grain refinement processes, according to the Hall Petch equation [3][4].

Severe plastic deformation (SPD) techniques are widely used methods to produce significant grain size refinement at the micro and nanometer scales [5]. Different SPD methods effectively modify the microstructure associated with defect generation, thereby improving the mechanical properties. Among the most common SPD methods include Constrained Groove Pressing (CGP) and Groove Pressing (GP), which consist of producing repetitive shearing of sheets between asymmetrical corrugation and flattening dies. Other techniques are High Pressure Torsion (HPT), Equal Channel Angular Pressing (ECAP), Twist Extrusion (TE), and Accumulative Roll Bonding (ARB) [6], [7]. These methods are performed using equipment and devices with special geometrical characteristics that do not allow free flow of the material during plastic deformation, resulting in the application of high hydrostatic pressure. High hydrostatic pressure combined with high shear stress results in a high density of defects (vacancies, dislocations) in the crystal lattice, especially dislocations that produce significant refinement of the grains in the material [8], [9]. Lei et al. [10] found that dynamic plastic deformation at high stresses facilitates deformation, leading to high tensile strength on the order of 1.40 GPa for 316L stainless steels. However, that process reduces ductility to the order of about 2%. In the search for a balance of properties, they also found that controlled annealing treatments reduced tensile strength from 1.4 to 1.0 GPa and improved ductility by 27%; this is due to the recrystallization of ultrafine grains that are located around the nanoscale grains.

On the other hand, Kim et al. [11] evaluated two types of treatments during the rolling at room temperature of a 316L austenitic stainless steel. In the first case, there was a thickness reduction of 20%, and in the second case of 80% with the addition of heat treatment at 760°C for 5 h, called R20 and R80+HT, respectively. They found that the RT80+HT deformation process allowed the formation of heterogeneous dual structures (austenite and phase (σ)) compared to homogeneous structures (austenite) in the R20 process. Regarding the mechanical properties, the ultimate tensile strength (UTS) and ductility of the material subjected to the R80+HT process were higher than those obtained for the R20 process. They found that the balance between tensile strength, ductility, and work hardening ability was due to a combined effort between martensitic transformation and heterogeneous hardening induced by the presence of a dual behavior in the material. A similar result was obtained by Singh et al. [14] who determined that the improvement in the mechanical behavior of 316 stainless steel after CGP was due to the combined presence of high dislocation density, presence of martensitic transformation, formation of a substructure, and the reduction of grain size. Aragón-Lozano et al. [12] also reported significant reduction in austenitic grain size and the presence of deformation-induced martensite in a 316L stainless steel deformed by CGP with up to 4 passes. They reported an improvement in the corrosion resistance in chloride-rich environments after four passes of ~45%. This behavior was associated with the presence of a passive Cr_2O_3 layer favored by the exposure time and level of plastic deformation applied. The aim of this paper is to analyze the microstructural behavior of 316L austenitic stainless steel deformed by the severe plastic deformation technique (Groove Pressing), quantifying the effect of plastic deformation on the continuous and discontinuous dynamic recrystallization processes with emphasis on the influence of the stacking failure energy on the hardening processes, either by mechanical twinning, deformation-induced martensitic transformation, or dislocation glide.

2. METHODS

AISI 316L austenitic stainless steel sheets of 20 mm x 96 mm x 2 mm were used in this study. The material composition was provided by the producer in the quality certificate and is summarized in Table 1.

Table 1. Chemical composition of AISI 316L austenitic stainless steel

Element	C	Cr	Ni	Mo	Mn	Si	N	S	P	Fe
w.t %	0.018	16.8	10.2	2.04	0.74	0.59	0.01	0.001	0.035	Bal.

The sheets were subjected to homogenization annealing for 60 minutes at 1000°C with water quenching. Subsequently, two CGP dies made of AISI A2 type tool steel with dimensions of 96 mm x 96 mm, one corrugated and one flat, were used. The geometry of the corrugated matrix consists of 2 mm teeth and 45° angles (see Figure 1). The material was subjected to the constrained groove pressing technique without mass flow restriction (GP) at room temperature up to a maximum equivalent strain of $\varepsilon \sim 4.64$ and molybdenum disulfide (MoS_2) was used as a lubricant.

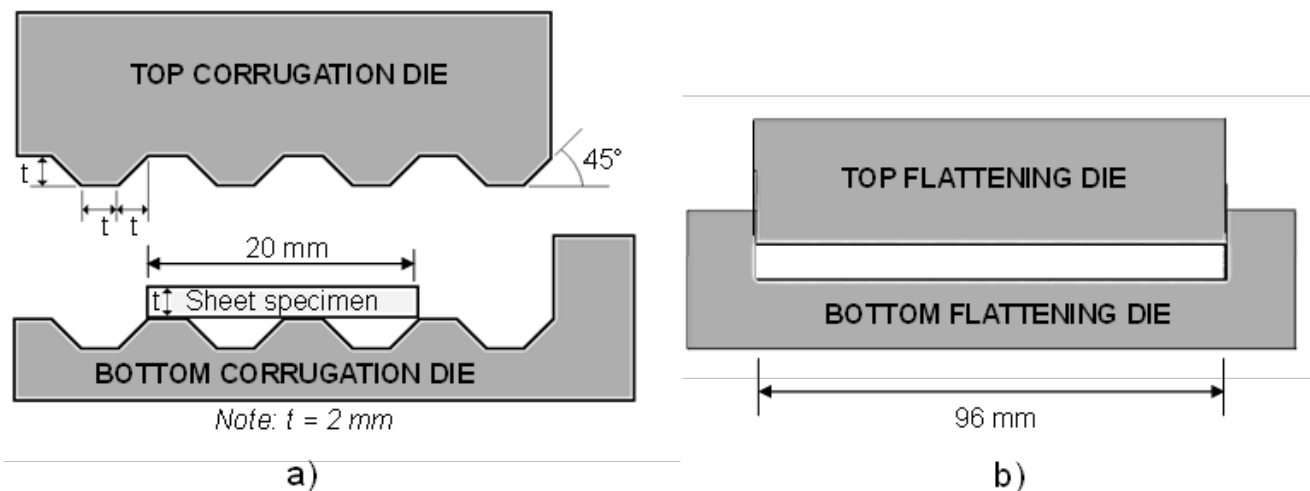


Figure 1. Schematic of the severe plastic deformation (GP) system used in this study.

The microstructures of the samples were characterized by electron backscattered diffraction (EBSD) on the transversal section of the samples, which were cut from the center of the CGP specimens and mechanically polished from 1500 grit sandpaper to $0.02 \mu\text{m}$ colloidal silica suspension following standard metallographic procedures. The EBSD measurements were performed using a JEOL JSM-7001F field Emission SEM operating at 20 kV. The Oxford Instruments HKL Channel 5 software package was used to process the obtained results. Different step sizes of $0.2 \mu\text{m}$ (annealed steel) and $0.1 \mu\text{m}$ (deformed steel) were used. Misorientations below 3° were not considered in the postprocessing data procedure. Additionally, to corroborate the presence of martensite induced by deformation, the following techniques were used: X-ray diffraction using a PANalytical Multi-Purpose diffractometer model X'Pert PRO MRD with copper radiation ($K\alpha=1.54187 \text{ \AA}$) and Scanning Electron Microscopy using a Hitachi SU3500 SEM.

3. RESULTS AND DISCUSSION

A. Microstructural Characterization

Figure 2 shows the microstructural behavior of the stainless steel before the CGP process (0 passes). Figure 2a shows the presence of a completely austenitic microstructure formed by grains of different sizes ($6.9 \pm 8.15 \mu\text{m}$), some with the presence of twins, which increases the heterogeneity of the microstructure. Additionally, a remanent lamination texture is noted with the presence of the δ phase ($\text{Fe}_{36}\text{Cr}_{12}\text{Mo}_{10}$) between and on the grains. Regarding the preferred grain orientation, Figure 2b shows a random behavior with a prevalence of grains oriented in the $\langle 111 \rangle$ direction, the product of the annealing heat treatment. The misorientation of grain boundaries shows a small presence of low-angle grain boundaries of the order of $\sim 8.5 \%$ and large amount of high-angle grain boundaries of the order of 91.5% . 25.9% of that are of the $\Sigma 3$ type ($60^\circ \langle 111 \rangle$).

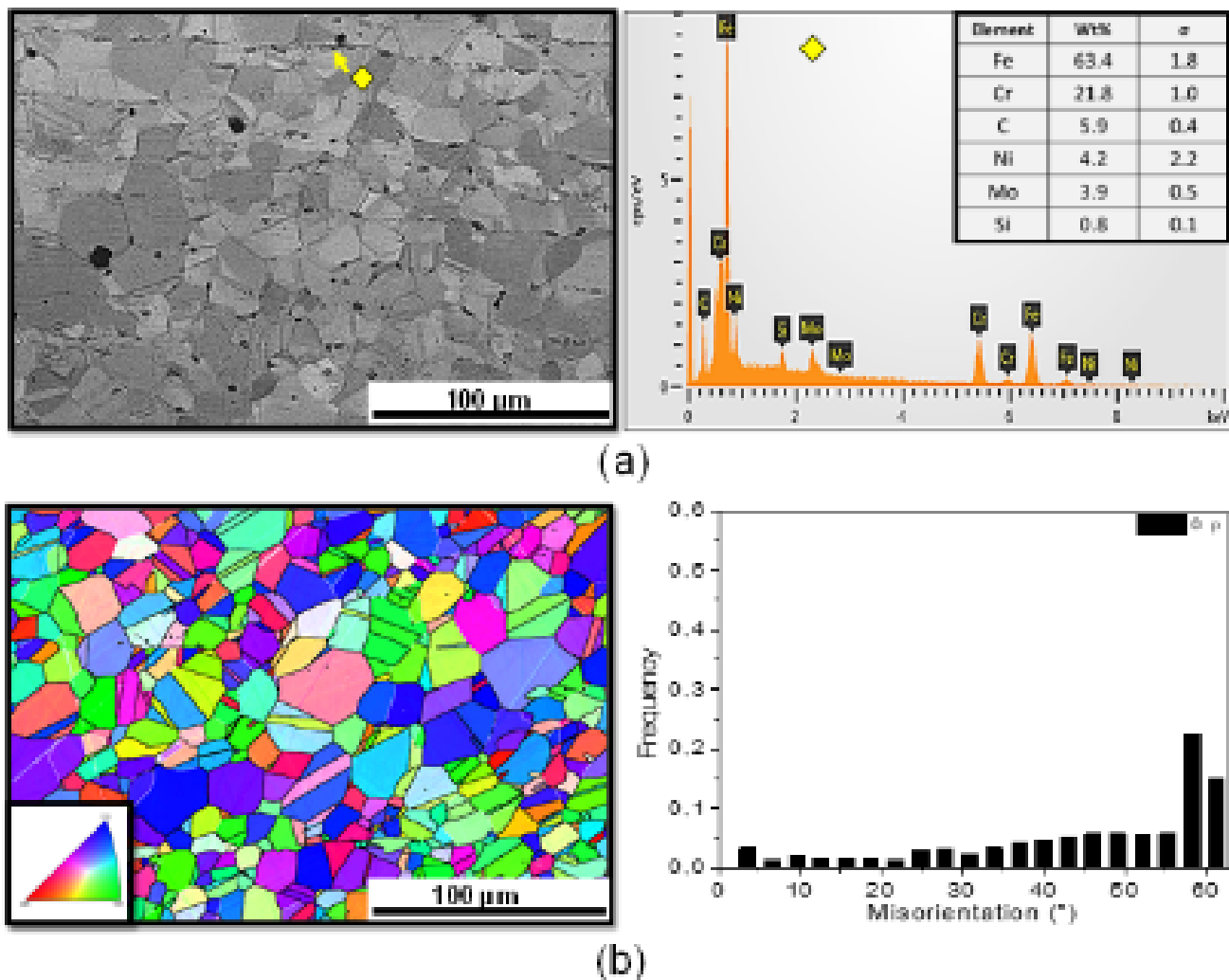


Figure 2. Microstructural behavior of 316L stainless steel in the annealed state (0 passes). (a) SEM-EDS and (b) Inverse pole figure via EBSD with subgrain boundaries ($3^\circ \leq \theta < 15^\circ$, white lines) and grain boundaries ($\theta \geq 15^\circ$, black lines).

Regarding the material deformed with up to four passes by CGP, the microstructural behavior is summarized from three different points of view, as shown in Figures 3 and 4. The first analysis was performed using SEM (Figure 3a), where the presence of deformation-induced martensite (DIM) on the surface of the material can be observed. According to Singh et al. [13], the presence of martensite is a function of the amount of cold work applied, the deformation temperature, and the chemical composition of the stainless steel. The second analysis was performed using XRD (see Figure 3b), which confirmed the presence of martensite and defined the type of martensite present in the material after the severe plastic deformation processes. The results show that after the first pass by CGP, the presence of martensite ϵ (100), ϵ (210), ϵ (101), and α' (101) was observed. After the second pass, peaks associated with martensite ϵ (100) and ϵ (210) were not observed because they transformed into martensite α' (101) and ϵ (101).

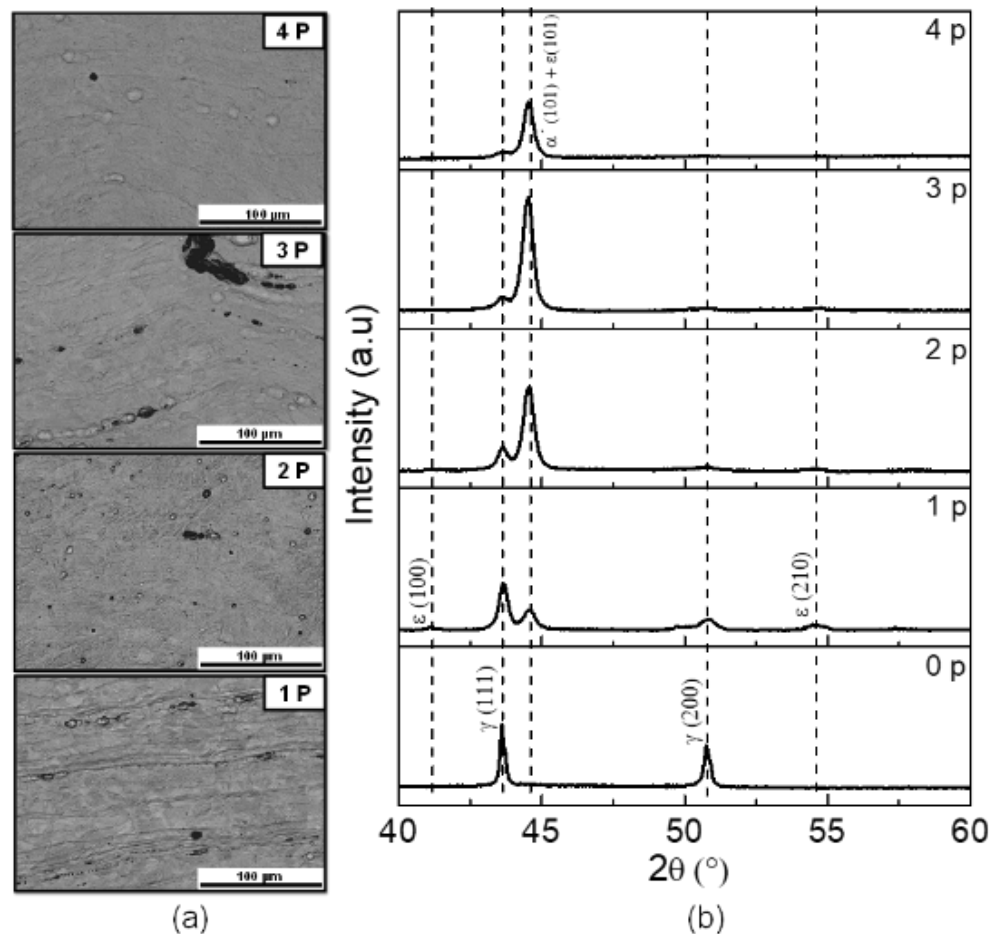


Figure 3. Microstructural behavior of 316L stainless steel processed by CGP. (a) SEM and (b) X-ray diffraction analysis.

Strain-induced martensitic transformation is mainly due to nucleation sites generated from defects, such as strain-induced twins [14], stacking faults, or ϵ -type martensite. [13], [15]. CGP-induced deformations facilitate the transformation from austenite to martensite improving the mechanical strength of the steel. In these cases, the austenite grain is refined to the order of ultrafine grains, which offer larger nucleation sites promoting the formation of DIM [13]. A very important topic in this type of material is the stacking failure energy (SFE), since it represents the energy generated per unit area by the interruption of the normal stacking sequence. The balance of forces between dislocations allows control of the mechanisms of martensite formation, among other aspects. Sato et al. [14] propose two routes based on the stacking failure energy (SFE) for the martensitic transformation of steels (See Figure 4). The first is the transformation of: austenite $\rightarrow$ martensite (ϵ) $\rightarrow$ martensite (α') (for SFE values $< 18 \text{ mJ/m}^2$); and the second is the transformation of: austenite $\rightarrow$ twinning austenite $\rightarrow$ martensite (α') (for SFE values $> 18 \text{ mJ/m}^2$).

The stacking failure energy (SFE) can be affected by factors such as chemical composition, temperature, and grain size. Some researchers have found correlations for the determination of the SFE in stainless steels using thermodynamic calculations, chemical composition, or by X-ray diffraction analysis [11], [16], [17]. The value calculated for the steel in this study was 28.2 mJ/m^2 , based on the chemical composition of the material, which is very similar to that used by Li et al [16]. The SFE value (28

mJ/m²) indicates a possible transformation of austenite to martensite α' from the mechanical twinning deformation mechanism and dislocation glide. Due to the intermediate value in the stacking failure energy, it is unlikely to find ϵ -type martensite (HCP) in this study [14] [15]. Nevertheless, as shown in Figure 3, after one pass through CGP in stainless steel, the presence of slip bands in the austenitic grains is noted, which some present twinning character and others induce martensite (α') nucleation generated by atomic stacking, stacking faults, accumulated dislocations or secondary twinning generated by increased deformation [18], [17].

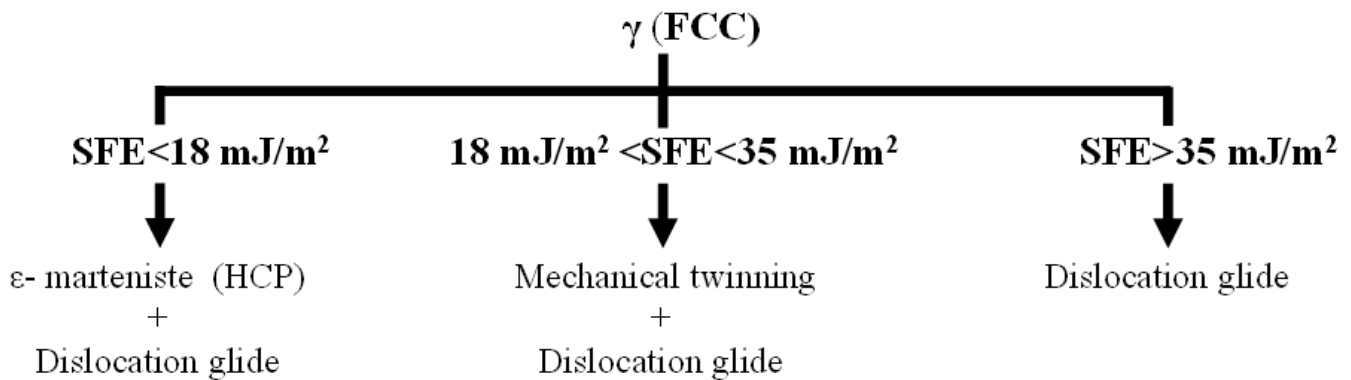


Figure 4. Deformation mechanisms controlled by stacking failure energy [15].

In order to clarify the presence of special grain boundaries induced by plastic deformation, a third EBSD analysis was performed. Figure 5 summarizes the changes generated during the plastic deformation process via CGP on the austenitic grain. It can be observed that the increase in the amount of deformation by CGP increases the fraction of low-angle grain boundaries ($3^\circ \leq \theta < 15^\circ$, white lines) and reduces the fraction of high-angle grain boundaries ($\theta \geq 15^\circ$, black lines), especially the twins ($\theta = 60^\circ$, blue lines) compared to the material without deformation. The twins deform towards the deformation direction, which is a typical behavior of a plastic deformation process. Subsequently, with the increase in deformation and due to the continuous and discontinuous dynamic recrystallization processes, the subgrain boundaries are transformed into high-angle grain boundaries (HAGB), achieving a recovery of the structure. The grain size obtained after the CGP process showed a variation from $3.28 \pm 4.16 \mu\text{m}$ in the first pass to $0.45 \pm 0.78 \mu\text{m}$ in the fourth pass, showing a high standard deviation, which is typical in this type of process due to the different energy levels in the grains resulting from the presence of defects such as vacancies, dislocations and grain boundaries that promote recrystallization processes.

As for the special grain boundaries, only the presence of coincident sites lattice (CSL) or primary twins ($60^\circ < 111^\circ$) of the $\Sigma 3$ type was analyzed because they are in higher proportion. A significant reduction in this type of grain boundaries is noted with increasing deformation compared to the undeformed material. The undeformed material presented 25.9% of $\Sigma 3$ passing after deformation by CGP to values of 0.9% for the first pass, 1.7% for the second pass, 3.1% for the third pass, and 2.5% for the fourth pass. Sing et al. [13] reported that deformation up to 2 passes by CGP in 316L stainless steel generated an increase in the number of low-angle grain boundaries influenced by the amount of plastic deformation applied. These grain boundaries are associated with the formation of subgrains, resulting from an increase in dislocations, which improves the strength of stainless steel.

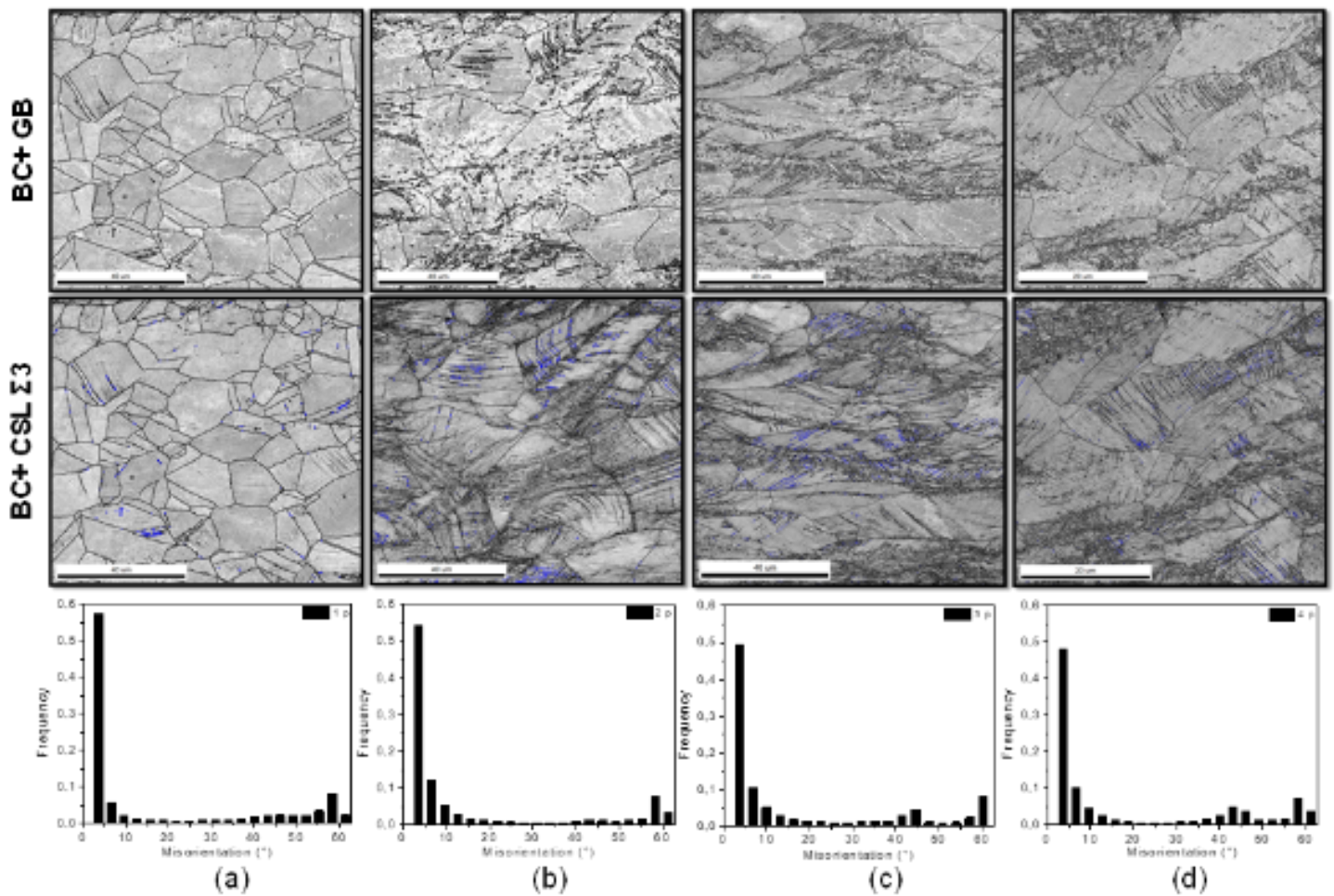


Figure 5. Contrast band with grain boundaries misorientation and special grain boundaries type $\Sigma 3$ ($60^\circ < 111 \rangle$) (twins-blue lines) via EBSD. (a) 1 pass, (b) 2 passes, (c) 3 passes, and (d) 4 passes.

4. CONCLUSIONS

Based on the microstructural behavior of an AISI 316L stainless steel subjected to severe plastic deformation by the groove pressing technique, the following conclusions were generated:

- It was observed that the undeformed 316L stainless steel presents an austenitic twinned grain with 25.9% of special grain boundaries of type $\Sigma 3$ ($60^\circ < 111 \rangle$) and randomly oriented with some prevalence to the $\langle 111 \rangle$ direction and with average grain size of $6.9 \pm 8.15 \mu\text{m}$ with presence of χ phase ($\text{Fe}_{36}\text{Cr}_{12}\text{Mo}_{10}$) in the hot rolling direction.
- It was observed that 316L stainless steel when subjected to severe plastic deformation processes presented several microstructural changes, which were evaluated from two different points of view: Surface changes in the microstructure, where the formation of deformation-induced martensite (martensite (α')) was observed in two different routes. The first transformation occurs by the transformation of austenite (γ) $\rightarrow$ martensite (ϵ) + mechanical twins $\rightarrow$ martensite (α'), and the second transformation occurs by the intersection of twins and sliding of dislocations, which is influenced by its intermediate stacking fault energy SFE (28 mJ/m^2). 2. Changes generated on

the austenitic grains, in this case, the sequential generation of low-angle grain boundaries were observed, which were reduced with the increase of plastic deformation passing to high-angle grain boundaries through continuous and discontinuous dynamic recrystallization processes. Additionally, the presence of special $\Sigma 3$ -type grain boundaries was observed, which were eliminated during plastic deformation passing from 25.9% (OP) to 0.9% after the first pass and finished at 2.5% after the fourth pass.

AUTHORS' CONTRIBUTION

Oscar Fabián Higuera-Cobos: Conceptualization, research, methodology, visualization, writing, review, and editing.

María Mercedes Cely-Bautista: Conceptualization, research, methodology, visualization, writing original draft, review, and editing.

Cristian Antonio Pedraza-Yepes: Software, validation, writing, review and editing.

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