Evaluation of the Corrosion Resistance of AISI 316l Steel Subjected to Severe Deformation Using the Groove Pressing Technique

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Abstract

In this investigation, samples of AISI 316L steel were subjected to severe plastic deformation by the groove pressing (GP) technique using 2 dies A2 type tool steel dies with dimensions of 96 mm X 96 mm, a corrugated die with 2 mm teeth and 45° angle and a flat die. Each pass through the GP die includes 2 states of corrugated and 2 states of straightening with a 180° rotation between each of them. This configuration provides the material with an equivalent theoretical deformation per pass of ε~1.16. The material was deformed by 4 passes per GP to an equivalent deformation of ε~4.64. Prior to the deformation, the specimens were subjected to an annealing heat treatment for 1 hour at 1000 °C with water cooling, in order to eliminate the lamination texture. The annealed and deformed material was

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characterized chemically and microstructurally by X-ray fluorescence and scanning electron microscopy, respectively. In order to evaluate the corrosion behavior of the material, linear polarization resistance and analysis by Tafel plot were used in a 0.6 M NaCl solution for 0 and 24 hours. The results show an atypical behavior regarding the corrosion resistance of AISI 316L steel. An increase in corrosion resistance of 45% of the material was observed after 4 passes per GP compared to annealed material (0 passes).

Keywords*:* austenitic stainless steel; corrosion rate; groove pressing; linear polarization resistance; severe plastic deformation; Tafel plot.

Evaluación de la resistencia a la corrosión del acero AISI 316l sometido a deformaciones severas mediante la técnica presión calibrada

Resumen

En esta investigación, muestras de acero AISI 316L fueron sometidas a deformación plástica severa por la técnica presión calibrada (GP) mediante el uso de 2 matrices de acero de herramientas tipo A2 con dimensiones de 96 mm X 96 mm, una matriz corrugada con dientes de 2 mm y ángulo de 45° y una matriz plana. Cada pase por la matriz GP incluye 2 estados de corrugado y 2 estados de enderezado con una rotación de 180° entre cada uno de ellos. Esta configuración provee al material una deformación teórica equivalente por pase de ε~1.16. Al material fue deformado por 4 pases por GP hasta una deformación equivalente de ε~4.64. Previo a la deformación, las probetas fueron sometidas a un tratamiento térmico de recocido durante una 1 hora a 1000 °C con enfriamiento en agua, con el fin de eliminar la textura de laminación. El material en estado de recocido y deformado se caracterizó química y microestructuralmente mediante fluorescencia de rayos X y microscopía electrónica de barrido, respectivamente. Con el fin de evaluar el comportamiento a la corrosión del material, se utilizó la resistencia a la polarización lineal y el análisis mediante las curvas de Tafel en una solución de 0.6 M de NaCl por un tiempo de 0 y 24 horas. Los resultados muestran un comportamiento atípico en cuanto a la resistencia a la corrosión del acero AISI 316L. Se observó un aumento en la resistencia a la corrosión del 45% del material después de 4 pases por GP en

comparación con el material recocido (0 pases).

Palabras clave: acero inoxidable austenítico; curvas de Tafel; deformación plástica severa; presión calibrada; resistencia a la polarización lineal; velocidad de corrosión.

Avaliação da resistência à corrosão do aço AISI 316l submetido a deformações severas mediante a técnica pressão calibrada

Resumo

Nesta pesquisa, amostras de aço AISI 316L foram submetidas a deformação plástica severa pela técnica pressão calibrada (GP) mediante o uso de 2 matrizes de aço de ferramentas tipo A2 com dimensões de 96 mm X 96 mm, uma matriz corrugada com dentes de 2 mm e ângulo de 45° e uma matriz plana. Cada passe pela matriz GP inclui 2 estados de corrugado e 2 estados de endireitado com uma rotação de 180° entre cada um deles. Esta configuração provê ao material uma deformação teórica equivalente por passe de ε~1.16. O material foi deformado por 4 passes por GP até uma deformação equivalente de ε~4.64. Prévio à deformação, as provetas foram submetidas a um tratamento térmico de recozimento durante uma 1 hora a 1000 °C com esfriamento em água, com o fim de eliminar a textura de laminação. O material em estado de recozimento e deformado caracterizou-se química e microestruturalmente mediante fluorescência de raios X e microscopia eletrônica de varredura, respectivamente. Com o fim de avaliar o comportamento à corrosão do material, utilizou-se a resistência à polarização linear e a análise mediante as curvas de Tafel em uma solução de 0.6 M de NaCl por um tempo de 0 e 24 horas. Os resultados mostram um comportamento atípico em quanto à resistência à corrosão do aço AISI 316L. Observou-se um aumento na resistência à corrosão de 45% do material depois de 4 passes por GP em comparação com o material recozido (0 passes).

Palavras chave: aço inoxidável austenítico; curvas de Tafel; deformação plástica severa; pressão calibrada; resistência à polarização linear; velocidade de corrosão.

I. INTRODUCTION

Austenitic stainless steels (ASS) have wide applications in fields that include the chemical, nuclear, petrochemical, maritime and metal implant technology due to their privileged properties, such as their excellent corrosion and oxidation resistance, good malleability, weldability and biocompatibility. However, austenitic stainless steel applications are limited by their relatively low elastic limit. Because these materials are not heat treatable, their mechanical properties can only be improved through cold work hardening. Over the years, many efforts have been made to obtain a good combination of high strength and excellent plasticity. Grain refinement using severe plastic deformation techniques is the most promising approach to improve the elastic limit without sacrificing plasticity considering that when processing this material by cold work, its grain refinement is accompanied by the transformation of martensite. Hence the importance of studying the effect of plastic deformation on corrosion resistance and the mechanical behavior of these materials [1-3].

The techniques of Constrained Groove Pressing (CGP) and Groove Pressing (GP) are severe plastic deformation (SPD) techniques in which ultra-fine grain size sheets can be obtained by applying high repetitive shear deformations. This is obtained by deforming sheets between grooved and flat asymmetric matrices (Figure 1), in turn.

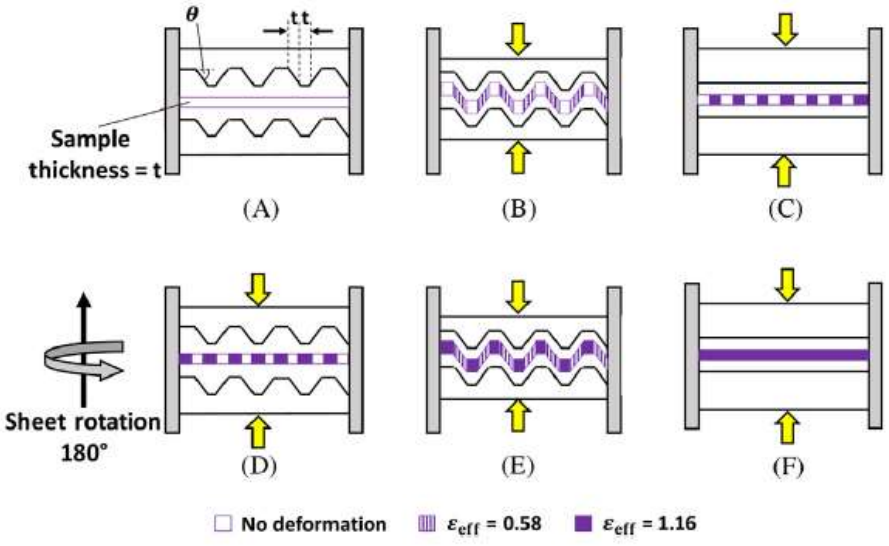

Fig. 1. Constrained Groove Pressing (CGP) [5].

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Due to its potential for large-scale industrial production, this process is prospective for application in the aeronautical, aerospace, transportation and many other engineering fields [3]. In CGP, the sheet is restricted within the matrix. On the other hand, when processing through the Groove Pressing (GP) method, there is no restriction, and the sample flows freely along the longitudinal and transverse directions [4].

Until now, there are very few studies in the literature that analyze the effect of severe plastic deformation on the corrosion resistance of 316L stainless steels with ultra-fine grain. Therefore, in this work we study the effect of grain refinement and deformation-induced martensitic transformation using the calibrated pressure technique on the corrosion resistance of AISI 316L stainless steel in saline solution [1].

II. METHODOLOGY

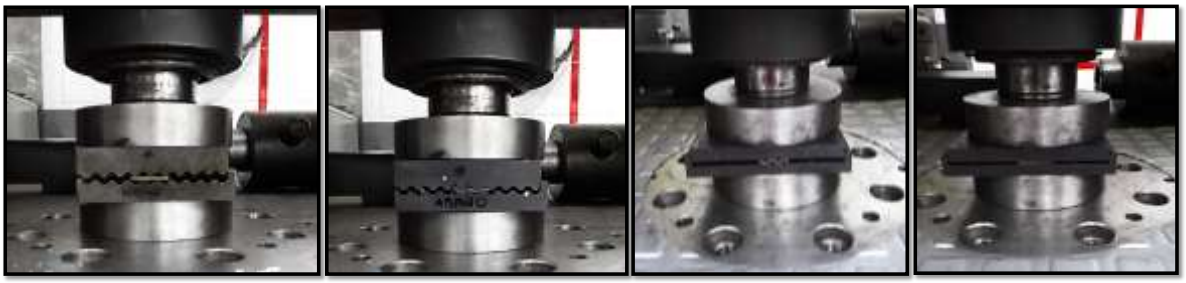
In this study, a cold rolled 316L stainless steel with 2B finish and dimensions of 1524 mm x 3048 mm and 2 mm thick was used. The chemical composition of the material was supplied by the manufacturer in the quality certificate and was corroborated by the X-ray fluorescence technique (Table 1) [6,7].

Table 1. Comparison of the chemical composition supplied by the production company (YCINOX.LTD), the ASTM A240 and the results by XRF [7].

Ítem	Manufacturer information	UNS Designation (máx.)	XRF	
			Result	Error (±)
Name	316L	S31603	316	
Carbon, **C**	0.018	0.030	Does not detect	
Silicon, **Si**	0.590	0.750	0.339	0.153
Manganese, **Mn**	0.740	2.000	0.853	0.080
Phosphorus, **P**	0.035	0.045	0.000	0.093
Sulfur, **S**	0.001	0.030	0.000	0.118
Nickel, **Ni**	10.220	10.00 - 14.00	10.280	0.270
Chromium, **Cr**	16.790	16.00 - 18.00	16.696	0.109
Molybdenum, **Mo**	2.040	2.00 - 3.00	2.062	0.052
Nitrogen, **N**	0.010	0.100	Not detected	
Iron, **Fe**	*69.556	Balance	69.119	0.866

For the development of the GP tests, the 20 mm x 96 mm x 2 mm plates of AISI 316L austenitic stainless steel were initially subjected to homogenization annealing

at 1000 ° C for 60 minutes at a heating rate of 30 ° C / min with a subsequent cooling in water. This process was performed in order to eliminate the texture of the laminate [7, 14]. Subsequently, the material was deformed at room temperature by the GP technique at a deformation rate of 2 mm / min (Figure 2) to an equivalent maximum deformation of $\varepsilon\sim$ 4.64 [4] and molybdenum disulfide was used as a lubricant. (MoS2).



(a) (b) (c) (d)

Fig. 2. Specimen under GP process [7, 14].

The scanning electron microscopy technique was used for microstructural analysis. The samples were cut from the center of the GP specimens and mechanically polished to 0.3 µm alumina solution. The used equipment was a scanning electron microscope HITACHI SUM3500 at a voltage of 20 kV [6]. For the corrosive analysis, linear polarization resistance (RPL) techniques and analysis using Tafel curves were used. The equipment used was the Potentiostat Galvanostat PG - TEKCORR 4.2 USB which has a National Instruments NI USB 6009 data acquisition card [8]. As a reference electrode a saturated Calomel electrode (SCE) was used, as a counter electrode a graphite electrode was used and as a working electrode the material under study, with an exposure area of 1cm^2. The performed assembly is summarized in Figure 3.

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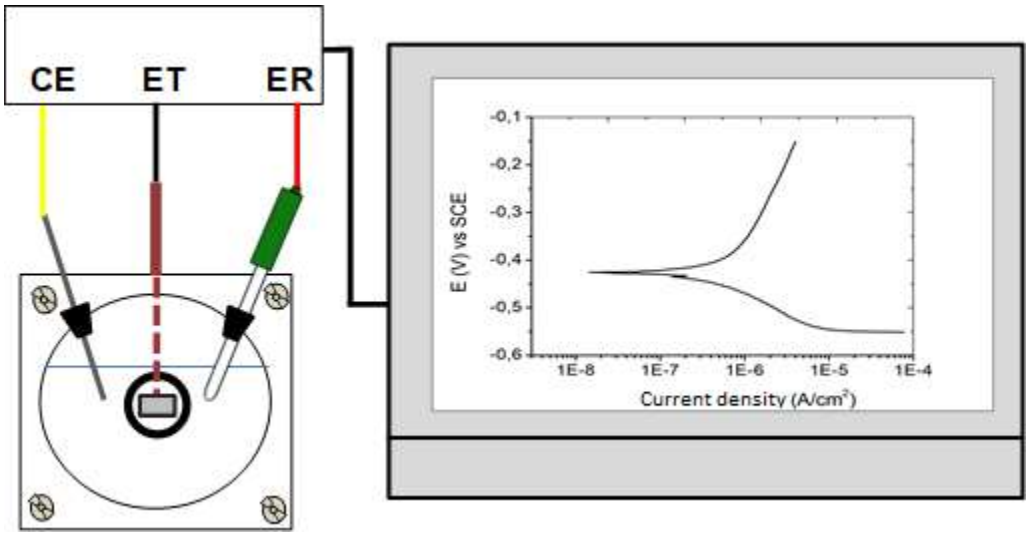

Fig. 3. Scheme of electrochemical tests.

As an electrolyte, a solution of sodium chloride with a concentration of 0.6 M was used. First, the linear polarization curves were performed and after 10 seconds, the linear polarization resistance, with the parameters described below. For the Tafel curves, the voltage data was recorded every 0.5 seconds for a time of 120 seconds (2 min) in order to achieve the slopes of the anodic and cathodic curves. And for resistance the linear polarization was performed according to ASTM G59-14 [15] where the voltages are carried from -20mV to + 20mV with respect to Ecorr. The scanning speed was 10mV / s recording the current value per second. Quantitative values of corrosion parameters, such as anodic and cathodic slopes (βa, βc), Tafel constant (β), corrosion current density (Icorr), corrosion potential (Ecorr) penetration corrosion rate (Vcorr) were determined from the aforementioned electrochemical techniques. Where β and Vcorr were obtained indirectly from the polarization graphs applying the criteria of ASTM G102-15 [9].

III. Results and Analysis

Hereunder, the effect of the degree of severe plastic deformation obtained by the GP technique on the corrosion behavior of AISI 316L stainless steel in 0.6M saline solution will be analyzed.

A. Microstructural Analysis

Figure 4 shows the microstructural variations obtained for the different degrees of deformation obtained 0, 1, 2, 3 and 4 passes per GP. As can be seen there is a reduction in grain size after the first GP pass (dmedium = 11µm) compared to the material in annealing state (dmedio = 22µm) a similar behavior was reported by Satheesh and Raghu [13], during processing by Constrained Groove Pressing of pure aluminum. The average diameter of the alloy deformed by 2, 3 and 4 passes could not be quantified due to the presence of the deformation-induced martensite that prevented its visualization. In addition, it was evident after the first pass the presence of mechanical twinning. The presence of martensite in this material changed the magnetic behavior of the material [1,7,11,14].

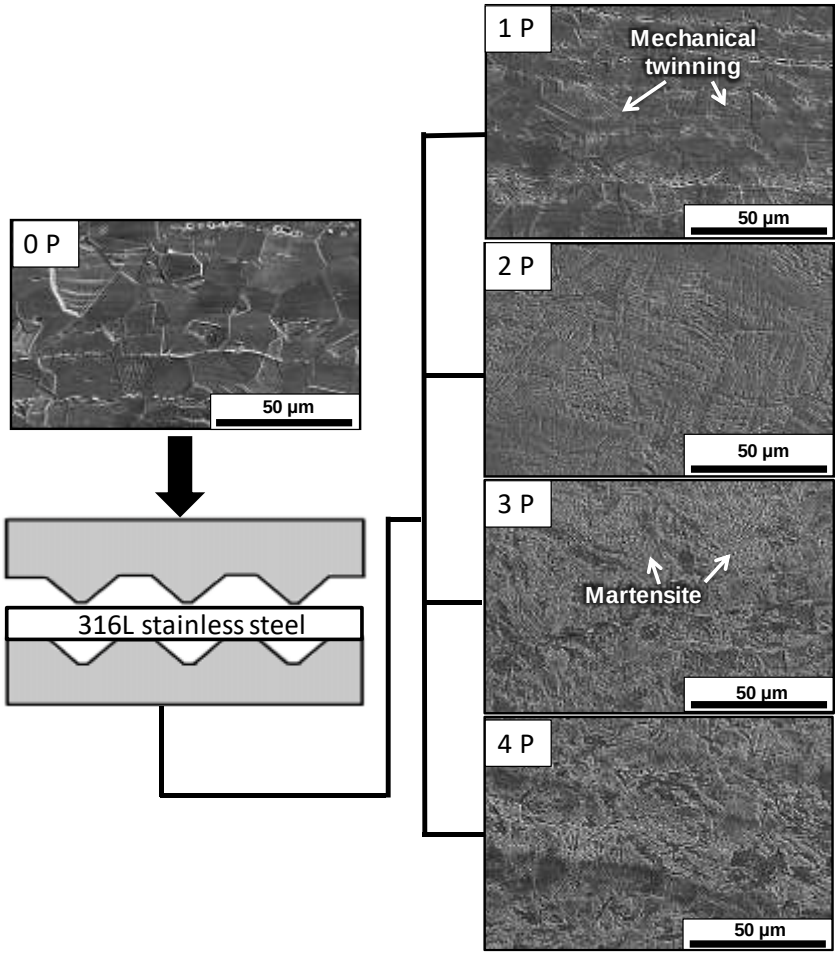

Fig. 4. Evolution of the microstructure of AISI 316L steel deformed by GP [7, 14].

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B. Electrochemical analysis

1) Tafel Curves. The results of Tafel curves in 3.5% saline medium NaCl for a dive time of 0 and 24 hours for 0, 1, 2, 3, 4 passes are shown in Figure 5.

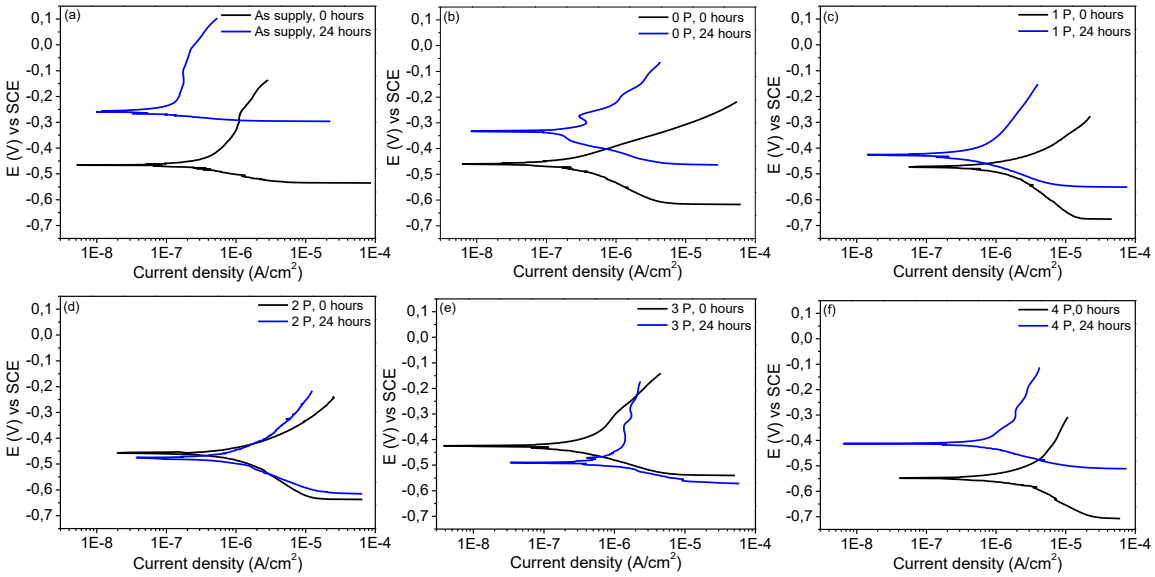

Fig. 5. Tafel curves, (a) As supply, (b) 0 pass, (c) 1 pass, (d) 2 pass, (e) 3 pass, (f) 4 pass.

As can be seen, in all the curves obtained in the reduction zone there is no linear tendency of the current density values to form a vertical line parallel to the axis of the potential (current corrosive limit) [12]. In addition, in Figure 5 it can be seen that for each of the specimens there is a tendency for improvement in corrosion resistance, that is to say that the potential of Tafel curves at 24 hours for the as supply condition, 0, 1 and 4 passes remain above the curves at 0 hours, these being more positive, therefore, it is corroborated that the corrosion resistance for the 24-hour specimens is greater, this can be explained by the presence of a protective film of uniform chromium oxide (Cr_2O_3), this process is known as passivation, it can also be observed that for 3 passes, the change in current density in the anodic curve with respect to the potential is smaller compared to the other specimens, which is an indication that it is more resistant to corrosion. Finally, for 2 passes it was possible to observe graphically that there was no significant change in the potentials, nor in the slopes at 0 and 24 hours, so that a clear answer cannot be obtained as to

whether the corrosion resistance increased or decreased. Due to this behavior, it was necessary to perform the pertinent calculations that allowed to determine the corrosion rates, in order to perform a more detailed analysis of its corrosive behavior [8-13]. Through the assistance of the PG 01X18E1CE6 software of the equipment, the anodic and cathodic slopes ("βa" anodic slope, "βc" cathodic slope) were determined, which are summarized in Tables 2 and 3 in order to calculate the slope of Tafel "B", according to ASTM-G102-15 [9] and thus determine the corrosion rate [10-12].

2) Linear polarization resistance (Rp). Table 4 summarizes the values of Rp where "M" is the slope and R^2 the correlation factor, checking the linear response between the current and the voltage with respect to the Ecorr posed by the Butler-Volmer equation for small polarizations (- 20mV at +20 mV vs Ecorr) [12].

Table 2. Values of the Tafel curves for 0 hours.

0 hour specimen	βa(mV/dec)	βc (mV/dec)	Ecorr (mV)	B
As supply	34.95	-33.56	-464.8	7.44
0 passes	74.88	-74.68	-460	16.26
1 pass	30.8	-30.94	-472.32	6.71
2 passes	52.36	-52.15	-458.03	11.36
3 passes	24.44	-24.55	-425.85	5.32
4 passes	60.53	-60.22	-547.45	13.12

Table 3. Values of the Tafel curves for 24 hours.

24 hour specimen	βa(mV/dec)	βc(mV/dec)	Ecorr (mV)	B
As supply	19.78	-19.29	-255.420	4.246
0 passes	84.26	-84.21	-332.080	18.312
1 pass	48.54	-48.75	-426.000	10.575
2 passes	38.25	-38.75	-475.650	8.369
3 passes	45.56	-45.46	-488.800	9.893
4 passes	44.84	-44.54	-410.500	9.715

Table 4. Results of linear polarization resistance, kΩ.

Specimen	Exposure time			
	0 hours		24 hours	
	M	R^2	M	R^2
0 passes	32.765	0.9498	33.502	0.9659
1 pass	15.892	0.9769	26.87	0.924
2 passes	19.000	0.9536	20.212	0.9833
3 passes	67.918	0.9472	40.836	0.9596
4 passes	11.901	0.9788	23.258	0.9382

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The Rp values obtained were between 11.091 and 67.918 KΩ for 0 hours and for 24 hours' values between 20.212 and 40.836 KΩ were obtained. Figure 6a shows the tendency of the linear polarization resistance with respect to the different degrees of deformation with 0 and 24 hours of exposure. Taking into account the values described in Table 4, it can be seen that there is a tendency to decrease the resistance to polarization except for the 3-pass specimen, which obtained the highest value of 67.918 kΩ and 40.836 kΩ for a time 0 and 24 h exposure, respectively. The lowest value recorded was for deformed specimens for four passes, with a value of 11.901 KΩ with the same exposure time (0 hours).

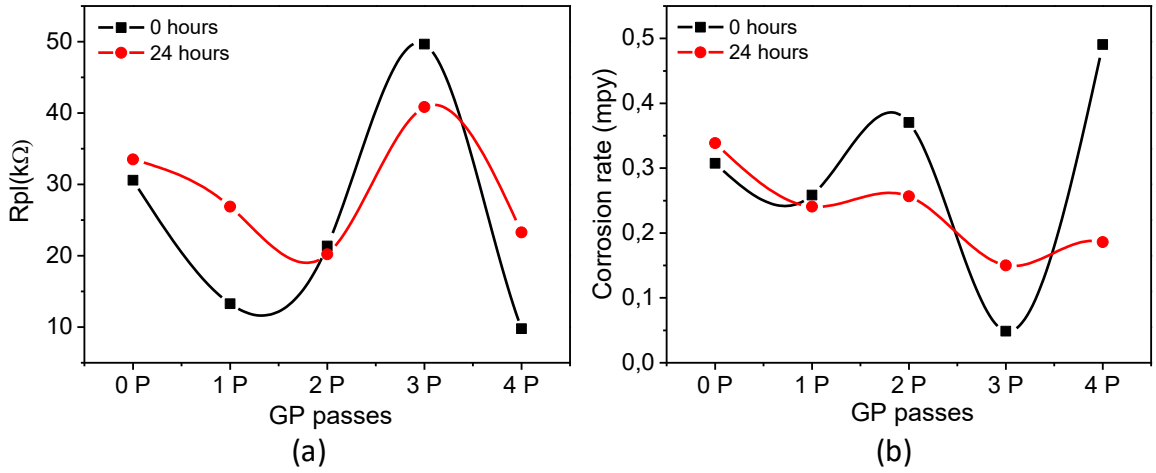

Fig. 6. Effect of the degree of severe plastic deformation by GP on (a) Linear polarization resistance and (b) Corrosion rate for each exposure time.

According to the provisions for the calculation of the corrosion rate in ASTM-G102-15 [9], the equivalent weight of the material was first determined based on the chemical composition of the steel provided by the manufacturer and the densities of corrosion current (i_{corr}) that were calculated from the polarization resistance values and the Tafel constants in order to determine the corrosion rate in mils per year (mpy). The% error between theoretical and experimental values did not exceed 0.5%. Figure 6b shows the behavior of the corrosion rate in mils per year for different degrees of deformation, with respect to the exposure time in which the test was

performed, as can be detailed for the 0-hour specimens, a clear trend is not observed. On the other hand, for the specimens evaluated at 24 hours, an improvement in the corrosion resistance of the material with respect to the number of passes by GP is observed. This behavior is very similar to the one reported by H. Miyamoto and M. Yuasa, [2], they found a marked improvement in Fe-Cr alloys, where passivation becomes more stable due to the formation of ultra-fine grains induced by SPD. Thermodynamically, it has been described that the effect of residual stresses on the metal favors the increase in the tendency to corrosion, however, the energy produced by cold work, measured by differential scanning calorimetry is usually less than 30 J / g, therefore, is much smaller than is necessary to produce a change in the free energy of the system [3,10].

IV. Conclusions

The study was conducted with the purpose of obtaining a better understanding of the effect of severe plastic deformation (SPD) on the corrosion behavior of AISI 316L austenitic stainless steel after being subjected to the calibrated pressure technique. The effect of plastic deformation on this material allows a reduction in the size of austenitic grain and favors the processes of mechanical grinding and martensitic transformation induced by deformation, which increases the mechanical resistance of the material, affecting its electrochemical behavior. In that order of ideas, this paper presents the following conclusions:

With the implementation of the technique of severe plastic deformation type calibrated pressure (GP), a significant reduction of the grain size in the material was obtained, moving from grains with an average diameter of 22.1 μm in an annealing state to mean diameters of 11,0 μm after the first GP pass, the deformation-induced martensitic transformation was also evidenced, which was checked by changing its magnetic properties.

Regarding the corrosive behavior, a greater resistance to corrosion was observed in general for the specimens evaluated after 24 hours, compared to those of 0 hours, this is due to the presence of a passive layer in the material (Cr_2O_3), which becomes more stable at longer exposure times. The results showed an atypical behavior in

terms of corrosion resistance, obtaining improvements in the corrosive behavior of the material as the grain size decreased with the increase of plastic deformation in the specimens from 0 to 4 passes. An increase in corrosion resistance of 45% was observed.

AUTHOR'S CONTRIBUTION

The contribution in the work by the authors was made as follows: Walter Aragón, Luis Fernández and José Luis Tristancho evaluated the corrosive behavior of 316L steel subjected to severe plastic deformation by GP; Oscar F. Higuera and Cristian Pedraza developed the plastic deformation tests by GP and the thermal cycles used in the study

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