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The influence of the glycerin concentration on the porous structure of ferritic stainless steel obtained by anodization

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Abstract
Anodizing of ferritic stainless steel has been used for decoration purposes to obtain a barrier effect. The most commonly used electrolyte for this process is INCO (5M H_2SO_4 + 2,5M CrO_3). INCO electrolyte with glycerin addition induces the formation of ordered porous structures, because glycerin reduces the electrolyte conductivity. Ferritic stainless steel was anodized in electrolyte composed by 2M H_2SO_4 with glycerin addition in different concentrations, without chromium oxide addition. It was observed that the addition of 90 v/v% glycerin promoted a reduction in the electrolyte conductivity, which caused an increase in the anodizing potential. The glycerin addition to the electrolyte diminished the oxide dissolution in the electrolytic solution, promoting the formation of an oxide with an ordered porous structure.

Keywords: Anodizing; ferritic stainless steel; glycerin; porous structure.

Influencia de la concentración de glicerina en la estructura porosa obtenida por anodización de acero inoxidable ferrítico

Resumen
La anodización de acero inoxidable ferrítico ha sido utilizada con fines decorativos, para obtener una capa de barrera. El electrólito más comúnmente utilizado para este proceso es el INCO (5M H_2SO_4 + 2,5M CrO_3). Con adición de glicerina induce la formación de estructuras porosas ordenadas, pues la glicerina reduce la conductividad del electrólito. Se anodizaron muestras de acero inoxidable ferrítico, en electrólito de 2M H_2SO_4 con la adición de glicerina en diferentes concentraciones, sin adición de óxido de cromo. Se observa que la adición de glicerina en un 90 v/v % promovió la reducción de la conductividad del electrólito, lo que ocasionó un aumento del potencial de anodización. Los resultados muestran que la adición de glicerina al electrólito redujo la disolución del óxido en la solución electrolítica y promovió la formación de un óxido con una estructura porosa ordenada.

Palabras clave: Anodización, acero inoxidable ferrítico, glicerina, estructura porosa.

1. Introduction

The resistance of stainless steel to corrosion is high for a wide range of conditions [1], mainly environmental ones. As chromium is the main alloy element, the addition of nickel and molybdenum improve resistance to corrosion [2]. These materials are less corrosion susceptible and more resistant to attack than common steel, but they are not completely immune [3].

The presence of determined levels of chromium in a stainless steel promotes resistance to corrosion [2]. Chromium dissolved in the iron lattice forms a thin oxide film on the steel surface, referred to "passive layer," protecting the metal against corrosion [4].

Metal corrosion protection has been widely studied [5-7]. Anodizing is an electrochemical process that promotes the

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obtaining of a protective oxide on the metal surface. This process is carried out by the immersion of the metal in an electrolytic bath, in which the metal is the anode; cathode can be an inert metal, for instance, platinum. The Anodizing process enhances the obtaining of barrier and porous oxide layers [8].

Barrier oxides obtained by the anodizing process are able to hinder electron conduction, which enhances a resistive character and a relatively uniform thickness, in addition to being compact, dense and thin [9]. In order to bring about the formation of a barrier layer, the electrolyte must not attack the formed oxides [10].

On the other hand, the formation of porous oxides is favored with electrolytes that chemically attack the oxide. It takes place due to the dissolution of the barrier layer in the electrolyte, which generates attacked spots on the surface. That generation takes place inside, specifically in the smallest pores, in the center of the hexagonal cells. A single potential is developed in each attack point, which then becomes a source of current. In the oxide-electrolyte interface there are ions that supply oxygen to transform the attacked point to oxide. At the same time, the reduction of the thickness of the barrier layer which occurs due to the action of dissolving the electrolyte in the pore base tends to decrease. Then, there is a pore lengthening that facilitates the ions' pass and the heat outflow, aiding the oxide film dissolution [10,11].

Currently, the anodizing process is widely used in stainless steel for aesthetic purposes [12], so the works developed on anodizing such as surface treatment for stainless steel are very few [13].

The electrolyte INCO (5M H_2SO_4 + 2,5M CrO_3) is widely used because it is extremely conductive [14]. In any case, given its high toxicity, chromium has to be replaced, and this has provided the motivation for the research of alternative electrolytes. Studies showed that valve metal anodizing in electrolyte containing glycerin induces the formation of ordered porous structures, due to the fact that glycerin reduces the electrolyte conductivity [15].

An increasing interest in applications for glycerin has been motivated by the increase of biodiesel production, since glycerin is one of its co-products. Glycerin ensures high viscosity and low conductivity to the electrolyte. It promotes the formation of porous oxides on the metals during the anodizing process [16].

The aim of this study is to evaluate the influence of different glycerin concentrations on the 2M H_2SO_4 electrolyte, in the properties of the oxides formed on ferritic stainless steel during the anodizing process.

2. Experimental

2.1. Elaboration of the samples

AISI 430 ferritic stainless steel plates of 20 mm x 20 mm x 0.5 mm were supplied by Aços Riograndense, with the composition that is shown in Table 1.

Samples were sanded down with silicon carbide paper (200 – 4000) and polished using 1 μm diamond paste and neutral alcohol in the Pentec polishing machine Polipan-U.

Table 1.
Alloy elements percentage in the AISI 430.

Alloy element	%
Cr	16.11
Ni	0.2563
Mn	0.3467
Si	0.3587

Source: the authors.

Table 2.
Anodizing electrolytes.

Sample	Anodizing electrolyte (%v/v)
100H	100% 2M H_2SO_4
90H10G	90% 2M H_2SO_4 + 10 % glycerin
50H50G	50% 2M H_2SO_4 + 50 % glycerin
10H90G	10% 2M H_2SO_4 + 90 % glycerin

Source: the authors.

After the surface preparation, electric contact was mounted with a copper wire and two layers of acetone-based lacquer on the unpolished side and about 2 mm on each border on the polished side. After this process, the samples were anodized in the solutions specified in Table 2. Each sample remained with an exposed area of approximately 2.5 cm^2 during the anodizing process.

Tests were carried out at room temperature. A current density of 4 $mA.cm^{-2}$ during three minutes [17]. The sample of AISI 430 stainless steel was the anode and a platinum wire was the cathode. The equipment used possesses a 0 – 300 V potential source and a 0 – 500 mA current source. Figure 1 shows the assembly of the anodizing process.

To determine the electrolyte conductivity a microprocessed conductivimeter pH determination was made at 25 ºC. These equipments are found at the Central Analítica at Universidade Feevale.

Morphological analysis of the obtained oxides was undertaken by scanning electronic microscopy (SEM). With the SEM micrographs and the Image Tool 3.0 software, pore diameter was measured. Through the energy dispersive spectrometer (EDS) coupled to the SEM equipment, a chemical elements mapping of the samples was undertaken.

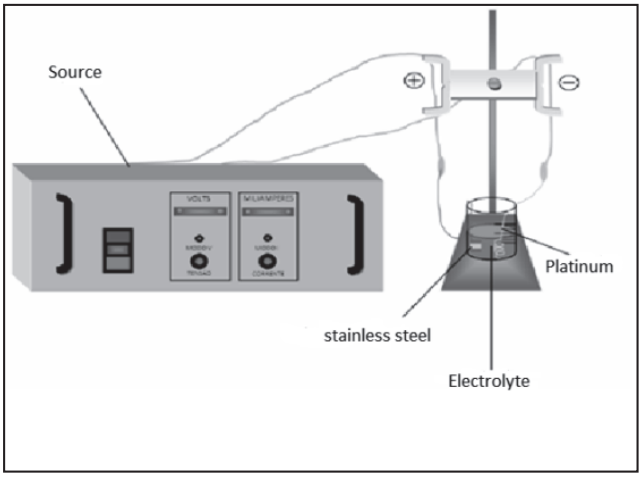

Figure 1. Anodizing process assembly.
Source: the authors.

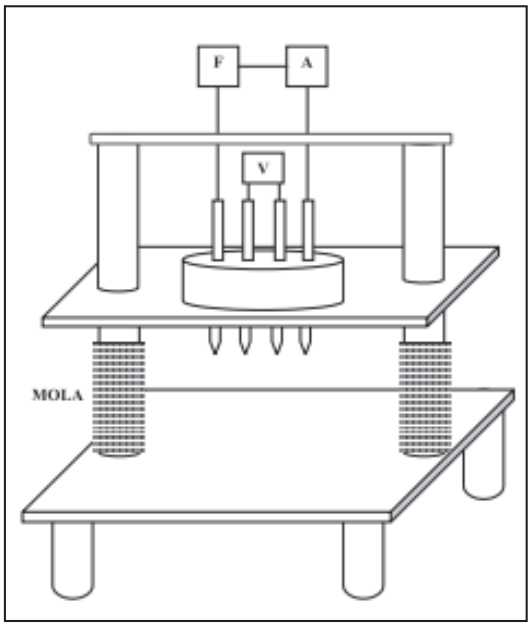

Figure 2. Four point probe method assembly to measure electrical conductivity with current source (F), amperometer (A) and voltmeter (V).
Source: the authors.

Surface structural characterization was carried out by X-ray diffraction (XRD) in a Bruker AXS D5000. The Kα beam was generated by a copper tube with 40 kV and 25 mA.

The four point probe method was used to determine the oxides electrical conductivity. A variable potential/current source 20 V/20 A, fabricated by Universidade Feevale was used, applying a voltage to close the circuit. Current was measured with a Minipa ET 2042C tester and for voltage an Agilent 34401 tester was used. The assembly can be seen in

Atomic force microscopy (AFM) was carried out in a SHIMADZU SPM 9500J3 in contact mode at LAPEC laboratory in the Universidade Federal do Rio Grande do Sul (UFRGS).

3. Results and discussion

3.1. Electrolyte pH and conductivity

Table 3 shows the pH and conductivity values corresponding to the different anodizing electrolytes. All the samples showed an acidic character; however, the increase in the glycerin percentage promoted a slight increase in the pH value. It was not possible to measure conductivity for the samples 100H, 90H10G and 50H50G due to the limitations of the equipment used. Sample 10H90G showed less conductivity compared to the other samples.

Table 3.
Anodizing electrolytes pH and conductivity.

Sample	pH	Conductivity (mS.cm^{-1})
100H	0.41	> 1412
90H10G	0.42	> 1412
50H50G	0.48	> 1412
10H90G	0.74	394 E-3

Source: the authors.

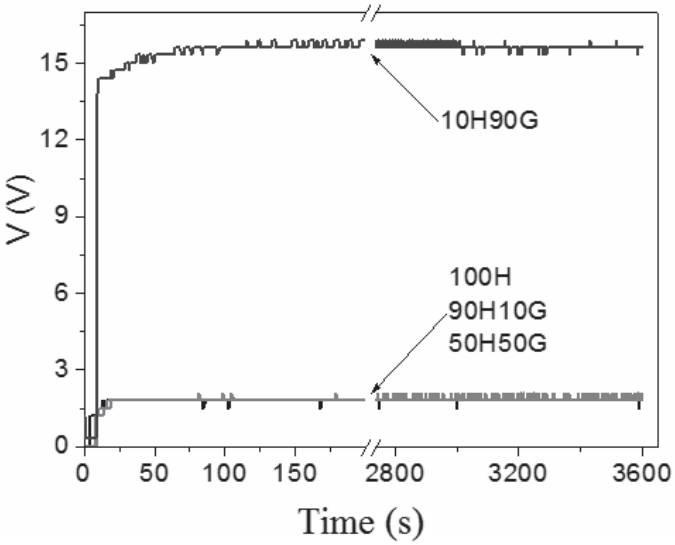

Figure 3. Potential transients V versus time.
Source: the authors.

3.2. Anodizing curves

Figure 3 shows the potential transients measured during anodizing. 10H90G had an anodizing potential of 1.5 V; those points out were about the formation of an oxide on the sample surface. Samples 100H, 90H10G and 50H50G had anodizing potentials close to 1.5 V for 1 hour and maintained the same behavior. As formerly stated, the conductivity of these samples are too high. This can be related to the intense oxide dissolution, hindering their accrual on the sample surface. It keeps the anodizing potential reduced for those samples.

3.3. Morphological analysis

In the micrographs, sample 100H (Figure 4a-b) shows an important presence of pores formed due to the intense oxide dissolution in the electrolyte.

Sample 90H10G (Figure 4c-d) has an intense porous surface, but with less oxide dissolution compared to the 100H sample. Micro-arc oxidation or dielectric breakdown studies carried out in anodizing of magnesium, in an electrolyte that contained silicate with glycerin addition, showed that there was an improvement in the coating, with less pores and microcracks. Glycerin addition to the electrolyte increases the micro-arc oxidation stability; it reduces the spark size. Consequently, smaller and more homogeneous pores are obtained [18].

In sample 50H50G (Figure 4e-f), a smaller number of pores with less depth compared to the former ones are observed. In sample 10H90G (Figure 4g-h), there was less pore formation. These results prove that with an increase in the amount of glycerin in the electrolyte reduces oxide dissolution, making the surface homogeneous with a reduced number of pores. According to the study [19], glycerin addition increases the electrolyte viscosity, that reduces the oxide dissolution and, with that, pore formation.

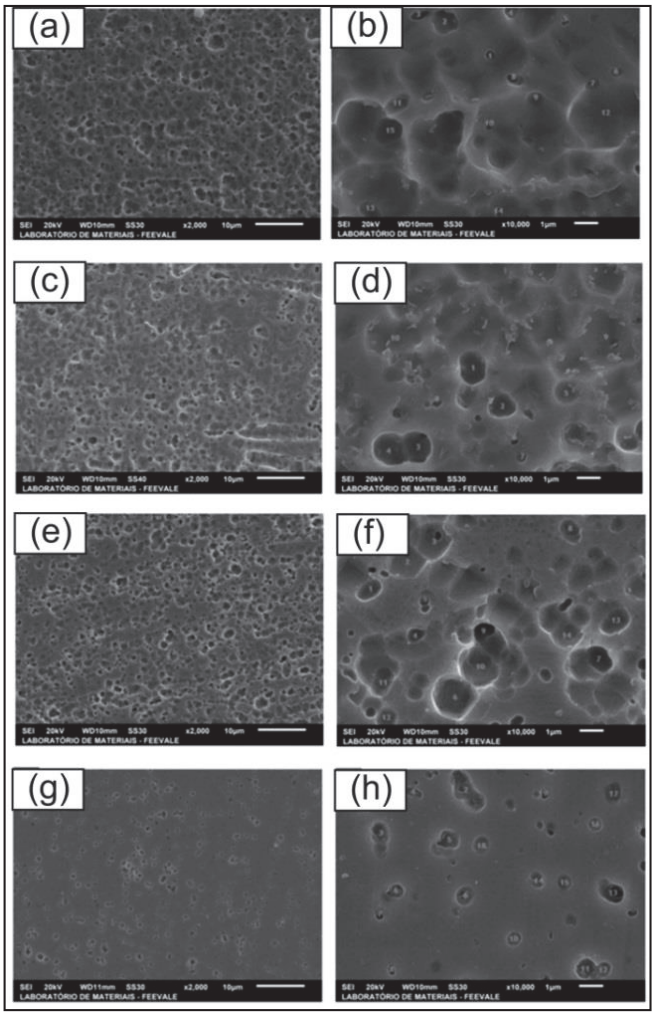

Figure 4. SEM micrographs for the samples 100H (a-b), 90H10G (c-d), 50H50G (e-f) e 10H90G (g-h), with magnifying of 2,000x e 10,000x respectively.
Source: the authors.

Authors [17] have reported behaviors like those observed in this work. Another work [20] observed that the polarization curve pointed out that the dissolution is accompanied by hydrogen evolution; meanwhile, oxygen reduction can accompany passive film growing. The film seems to be finely porous and reveals cracks along the stainless steel grain boundaries, due to the improved dissolution in these areas. Film growth can be obtained by dissolving cycles of stainless steel, capable of activating or transpassivation, and subsequent deposition of the material on the film.

The proposed iron passivation mechanism [21-23] indicates that iron dissolves as ferrous ion to form ferric hydroxide with water; finally, film formation takes place by ferric hydroxide dehydration. These processes can be simplified according to equations [1-3], although it is known that the anions in solution, specially the oxygenated species, participate in the passivation reaction, where M represents the metal, $M(OH)_3$ is the metallic hydroxide and $M_2O_3(H_2O)_3$, the passive film [22].

$$M \rightleftharpoons M^{2+} + 2\bar{e} \quad (1)$$

Table 4.
Pore diameter for the anodized samples.

Sample	Diameter (μm)
100H	0.84 ± 0.60
90H10G	1.28 ± 0.77
50H50G	1.20 ± 0.42
10H90G	0.72 ± 0.23

Source: the authors.

$$M^{2+} + 3H_2O \rightleftharpoons M(OH)_3 + 3H^+ + \bar{e} \quad (2)$$

$$2M(OH)_3 \rightleftharpoons M_2O_3(H_2O)_3 \quad (3)$$

As stated above, glycerin addition to the electrolyte provoked a reduction in the number of pores on the samples surface. However, samples 90H10G and 90H10G showed the highest pore diameter (Table 4), promoted by the glycerin addition, compared to 100H. Sample 10H90G had the least number of pores as well as the smallest pore diameter, when compared to the other samples.

Figure 5 shows the EDS images and the point at which elemental determination was undertaken. Table 5 shows the EDS results for the anodized samples. It is observed that all the samples, the oxygen, iron and chromium remains unaltered with the anodizing electrolyte. So, it is possible to foresee the oxide formation according to the amount of oxygen.

It was expected that for sample 100H the amount of oxygen would be the smallest. But, namely, the presence of chromium in stainless steel promotes the formation of a thin oxide on the surface of the metal. That is why it is thought that the high amounts of oxygen present in the samples come from the alloy natural oxide, instead of evidencing the formation of a specific oxide after the anodizing.

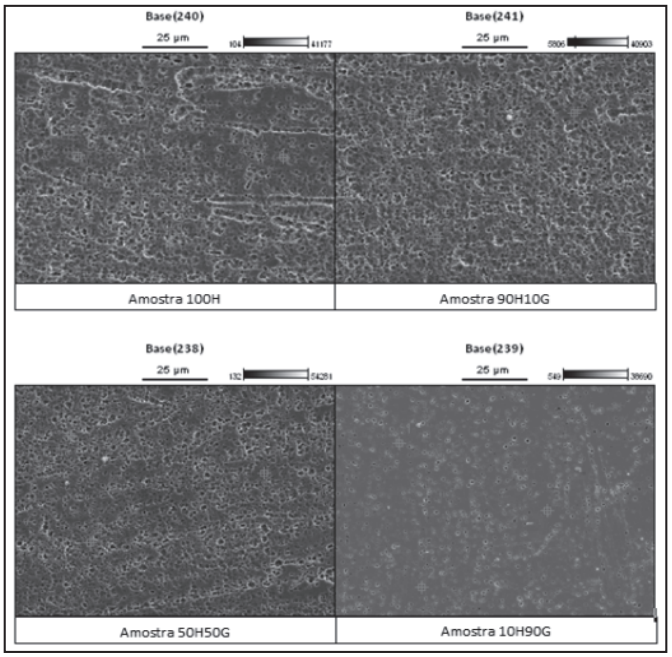

Figure 5. SEM-EDS micrographs of the samples.
Source: the authors.

Table 5.
Elemental compositions of the samples obtained by EDS.

Sample		Atomic %		
		O	Fe	Cr
100 H	Point 1	60.00	32.91	7.09
	Point 2	59.94	32.76	7.20
	Point 3	60.00	32.83	7.17
90H10G	Point 1	60.00	32.65	7.35
	Point 2	60.00	32.75	7.25
	Point 3	60.00	32.83	7.17
50H50G	Point 1	60.00	32.89	7.11
	Point 2	59.98	32.67	7.32
	Point 3	60.00	32.84	7.16
10H90G	Point 1	60.00	33.01	6.99
	Point 2	60.00	32.97	7.03
	Point 3	60.00	32.91	7.09

Source: the authors.

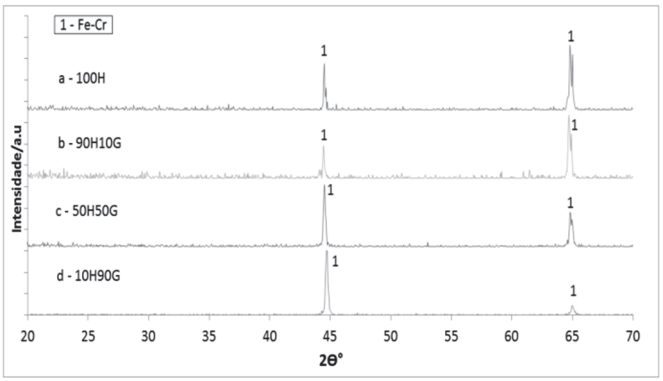

Figure 6. Diffractogram for the anodized samples.
Source: the authors.

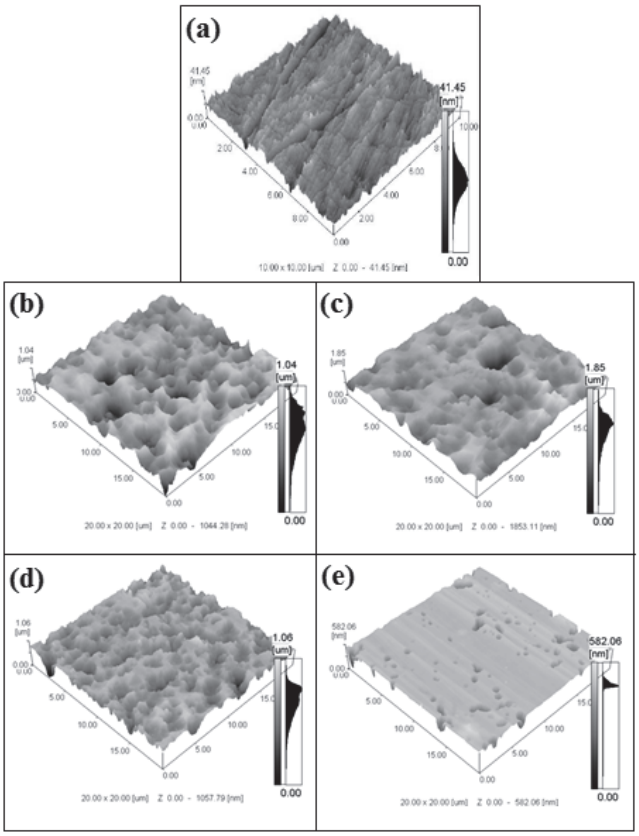

Figure 7. 3-D AFM images for the samples (a) non-anodized stainless steel*, (b) 100H, (c) 90H10G, (d) 50H50G and (e) 10H90G.
Source: the authors. *(Leite, 2011)

3.4. XRD analysis

Figure 6 shows the X-ray diffractograms for the samples anodized in different electrolytes.

It is possible to observe that the systems 100H, 90H10G, 50H50G and 10H90G were amorphous. Meanwhile, anodized samples with different electrolyte composition did not affect the substrate chemical composition. Only iron and chrome were detected because they are substrate components.

3.5. AFM

Figure 7 shows the 3-D atomic force microscopy images for the samples.

It can be observed that the samples had a rough surface, confirming what was observed in the SEM results.

Table 6 shows the average roughness (Ra) and the depth roughness (Rz) of the samples. Anodized samples present higher Ra and Rz values than the original stainless steel. The increase in the glycerin concentration in the electrolyte (10H90G) diminished the Ra and Rz values, which indicates a reduction of the attack to the oxide. On the other hand, Rz value is too high for all the electrolytes, indicating the presence of deeper pores on the anodized samples when compared to the non-anodized steel.

Table 6.
Measurements of Ra and Rz of samples

Sample	Ra (nm)	Rz (nm)
Stainless Steel *	3.593	20.092
100H	135.9	518.0
90H10G	178.8	918.5
50H50G	105.2	516.0
10H90G	24.6	278.1

Source: the authors.*(Leite, 2011)

3.6. Electrical conductivity

Table 7 shows the resistivity and conductivity values for the studied samples. It can be observed that all the conductivity values are found in an order of 105 (Ω m)-1. This is related to the decrease in the oxide dissolution process; however, it cannot be stated that there was the formation of an oxide layer, based only on these results.

Table 7.
Resistivity and conductivity values for the studied samples.

Samples	Resist. (Ω.m)	Condut. $(\Omega.m)^{-1}$
SS *	2.97 E-6	3.81 E+5
100H	3.58 E-6	2.79 E+5
90H10G	1.95 E-6	5.12 E+5
50H50G	5.10 E-6	1.96 E+5
10H90G	2.80 E-6	3.58 E+5

Source: the authors.* Values for non-anodized stainless steel (SS) (Leite, 2011).

4. Conclusions

The study conducted showed that the addition of glycerin to the 2M H_2SO_4 electrolyte diminished its electrical conductivity, which increases the potential transient during the anodizing process.

The decreased electrolyte conductivity promoted a decrease in the formed oxide dissolution, allowing its deposition on the metallic substrate, forming a porous oxide layer. Regarding the SEM images the formation of a porous oxide on the sample surface promoted by anodizing was verified.

The sample anodized in electrolyte with the highest glycerin concentration (10H90G) had the highest number of pores, but with a reduced diameter compared to the other samples.

In spite of the modification of the electrolyte composition with the addition of glycerin that made its conductivity vary, XRD evidenced that the chemical composition of the substrate remained the same.

Based on the aforementioned results, it can be concluded that ferritic stainless steel anodizing in 2M H_2SO_4 and glycerin promoted the formation of an oxide on the sample surface, but the latter one maintained its chemical structure. Due to the addition of glycerin, oxide dissolution in the electrolyte was minimized. This promotes the formation of pores on the oxide, which harms its protective performance.

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