COBEM 2023 27TH INTERNATIONAL CONGRESS OF MECHANICAL ENGINEERING

ABCM Brazilian Society of Mechanical Sciences and Engineering
27th ABCM International Congress of Mechanical Engineering
December 04-08, 2023, Florianópolis-SC, Brazil

COB-2023-2175
MICROSTRUCTURAL CHARACTERIZATION AND EVALUATION OF CORROSION RESISTANCE IN STAINLESS STEEL APPLIED IN THE PULP AND PAPER INDUSTRY

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Abstract. *The occurrences of failures in industrial equipment result in higher maintenance costs. One of the main failure mechanisms is due to corrosion, caused by the performance of equipment in aggressive environments that compromise the integrity of the system. This work focused on the analysis of stainless steel samples from industrial components in a pulp and paper industry, seeking to identify the failure mechanisms related to corrosion. The samples were submitted to metallographic tests, chemical analysis and electrochemical tests to evaluate their resistance to intergranular and pitting corrosion after a heat treatment of sensitization. The results indicated that the 316L steel samples had better corrosion resistance than the 304 steel samples. It was observed that the sensitization effect was detected through the difference in chromium content in the regions close to the grain boundaries, indicating the formation of precipitation of chromium carbides. This analysis revealed that using materials degraded by the industrial process can lead to different results from the literature, as the loss of their chemical properties directly affects corrosion resistance. Therefore, it is essential to consider the composition of materials used in corrosive environments to avoid failures and additional maintenance costs.*

Keywords: *Pulp and paper industry, Corrosion, Stainless steels, Metallography, Electrochemical tests.*

1. INTRODUCTION

Corrosion, in addition to directly generating losses, such as equipment failures during an operation, is also responsible for part of the indirect losses, such as unscheduled downtime, reduced system efficiency or making components more susceptible to other types of failures, such as fatigue, cracks and creep (Affonso, 2006; Telles, 2003). In the pulp

production process, after the wood chip formation stage, this material undergoes cooking processes in a chemical solution in digesters to form the pulp. The cellulose is then screened, bleached, dried and converted into various types of paper. During the process, corrosion is an obvious problem due to the aggressive working environments. Associated with factors that influence the progression of a corrosive process, such as in welding processes that form a heat-affected zone (HAZ), degradation becomes even more aggressive. Studies were carried out to evaluate the corrosion behavior and develop new alloys and conditions that resist the aggressive environment (Silva, 2013; D'Avila, 2016; Scherer, 2015; Gogola, 2014). Thus, it is necessary to use equipment made of materials that resist the aggressive products used, such as stainless steel (Castro, 2009; Klock, Andrade and Hernandez, 2013; Garner, 2017).

Stainless steels are widely used in industry due to their corrosion resistance, mechanical properties at high temperatures and toughness. The presence of chromium contents above 12% improves the corrosion resistance of steel, forming a layer of chromium oxide on the surface through the passivation process. Stainless steels can be classified as martensitic, ferritic, austenitic, duplex and precipitation-hardened, depending on their microstructure (Bhadeshia and Honeycombe, 2006; Silva and Mei, 2010). Austenitic stainless steels are widely used in the pulp and paper industry, especially in piping, pulping and bleaching processes, due to their good corrosion resistance and high toughness. The addition of molybdenum and nitrogen increases the corrosion resistance of these alloys in chloride environments. Stainless steel grades with low carbon content (with a maximum of 0.035%C) contribute to preventing intergranular corrosion. AISI 304, AISI 316 and AISI 317L steels are examples of austenitic stainless steels with different compositions to meet specific corrosion resistance requirements (Tuthill, 2017; Wensley, 2017). Corrosion characterization based on morphology is useful to understand the mechanism and apply appropriate protection measures. Corrosive processes can lead to uniform or localized corrosion, such as alveolar and pitting corrosion, or erosion-corrosion caused by fluid flow. Intergranular corrosion occurs when there is preferential dissolution of elements at the grain boundaries. This analysis is important to determine the best form of prevention and treatment against corrosion (Gentil, 2011; Nunes, 2007).

After being subjected to heat exposure, as in the case of a welding process, stainless steels can undergo a phenomenon known as sensitization, due to the thermal cycles involved, with temperatures between 500°C and 950°C in the HAZ. This welding heat depletes chromium from the microstructure in the HAZ due to the precipitation of chromium carbides at grain boundaries, making this zone susceptible to intergranular corrosion. To evaluate this resistance to intergranular corrosion, heat treatments are carried out by exposing samples to the sensitization temperature and conducting corrosion tests (Nunes, 2007; Gentil, 2011; Tuthill, 2017). Therefore, studying the corrosion process in critical conditions of the pulp and paper production system is of great importance, in search of the development and selection of new materials that meet the requirements of the aggressive environment in which they are found.

The objective of this study is to carry out a descriptive and comparative analysis between stainless steel samples taken from equipment in a paper and cellulose industry, evaluating the susceptibility to intergranular corrosion after thermal exposure and characteristics linked to pitting corrosion, using optical and electronic microscopy. scanning to check the phases present and their interaction with the types of corrosion studied after each test carried out. Corrosion tests were carried out using the electrochemical techniques of Double Cycle Potentiodynamic Electrochemical Reactivation (DL-EPR) and Anodic Potentiodynamic Polarization.

2. METHODOLOGY

2.1 Materials

In this study, four types of samples obtained from different stages of the production process of a pulp and paper industry were analyzed. Each sample was properly identified, as shown in Figure 1.

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

Figure 1. Samples received for analysis. a) B50; b) B54; c) B56; d) Upper Profile Plate – SUP.

Samples B50, B54 were taken from white water pumping systems originating from the pressing of the paper sheet, while B56 is white water originating from the dewatering of the paper sheet. The SUP sample was taken from a paper machine dryer exhaust.

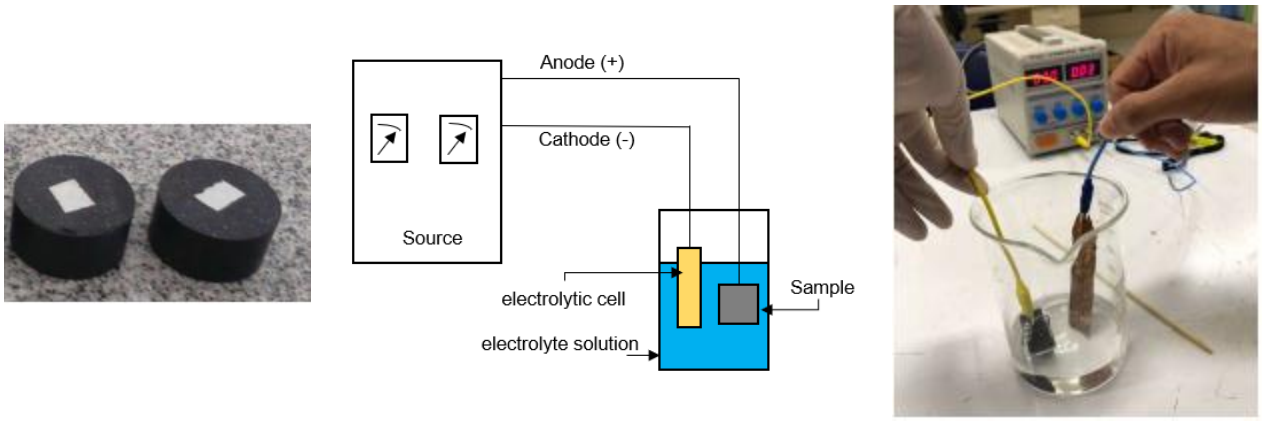
In order to assess how thermal exposure during the manufacturing process affects corrosion resistance, the samples were subjected to a sensitization treatment. The samples were heated at a temperature of 675°C for 1 hour and then quickly cooled in water, following ASTM G108-94 (2015).

2.2 Chemical and microstructural characterization

Chemical analysis was performed using Optical Emission Spectroscopy using an Oxford Instruments spectrometer, model Foundry-Master Pro. Using the results of the chemical analysis, it was possible to classify the sample material.

The procedures for preparing the specimens for metallography were carried out in accordance with the ASTM E3-11 (2017) standard. The samples were analyzed in their original state and after heat treatment. After cutting the samples, they were subjected to hot mounting using bakelite, as shown in Figure 2(a). Then, the specimens were sanded and submitted to the polishing process, using 1 µm and 0.3 µm alumina paste, with the aid of a polisher.

For stainless steels, chemical etching is carried out through the electrolytic etching process, using oxalic acid as a reagent while a voltage is applied from a source, as shown in Figure 2(b). For this purpose, an electrolytic cell composed of a copper plate with an approximate thickness of 2 mm was used, following the procedure described in Miranda (2018), Figure 2(c). The applied voltage was 3V, as presented in Lima Filho (2013).



(a) (b) (c)

Figure 2. a) Specimens after polishing; b) Illustrative scheme; c) Conducting the test.

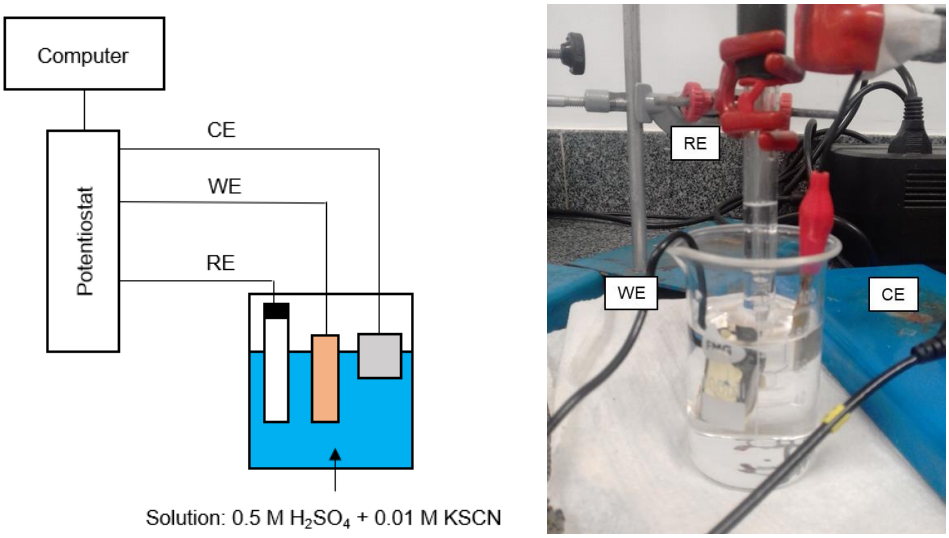
To visualize the microstructure of the samples after metallographic and corrosion tests, an Optical Microscope (OM) Olympus CX31 was used through the software Motic Images Plus 2.0. For a better verification of the intergranular corrosion present in the samples after the DL-EPR corrosion tests, the surfaces of the specimens were observed in the Scanning Electron Microscope (SEM), brand ZEISS model EVO MA10 operating at a voltage of 30 kV. With the aid of Energy Dispersion Spectroscopy (EDS), measurements were performed along the images obtained in the SEM to verify the chemical composition of the phases present in the microstructure of the samples. In this study, the EDS aims to demonstrate the effect of sensitization, identifying differences in the contents of chemical elements, such as chromium and carbon, in regions close to the grain boundaries, allowing to verify the presence of carbide precipitation.

2.3 Electrochemical tests

In this study, two types of corrosion tests were conducted: the Double Cycle Electrochemical Potentiodynamic Reactivation Technique (DL-EPR), with the objective of determining the susceptibility to intergranular corrosion between the samples, and the Anodic Potentiodynamic Polarization Technique, used to analyze the resistance to pitting corrosion. These tests were carried out using a digital potentiostat/galvanostat model PGSTAT302N from Metrohm Autolab, an electrochemical cell to position the electrodes and contain the solution, as well as a computer with Nova 2.1. To perform the corrosion tests, the samples were cut and cold-embedded using epoxy resin (resin to catalyst ratio of 2:1). Then they were connected to a metallic wire to create a circuit with the potentiostat. After the resin curing process, the samples were sanded according to the specifications of each test and had their area delimited to approximately 1 cm^2 using nail polish, in order to avoid crevice corrosion. Three tests were conducted for each material, using different specimens in each test.

In addition to the samples in their original state, the DL-EPR test was also conducted on heat-treated (HT) samples, following the guidelines of ASTM G108-94 (2015), subjecting them to a temperature of 675°C for 1 hour and subsequently quenching in water. Before immersion in the solution, the samples were cleaned with detergent and distilled water and air dried. The test solution consisted of a mixture of sulfuric acid (H2SO4) and potassium thiocyanate (KSCN) in the proportion of 0.5 M H2SO4 + 0.01 M KSCN, with 70 mL transferred to the electrolytic cell. The cell temperature was maintained at room temperature throughout the test, as shown in Figure 3. The potentiostat works coupled to three

electrodes that are immersed inside the electrochemical cell together with the electrolyte solution: the working electrode (studied sample, WE), the saturated calomel reference electrode (RE) and the counter electrode (CE) of platinum.

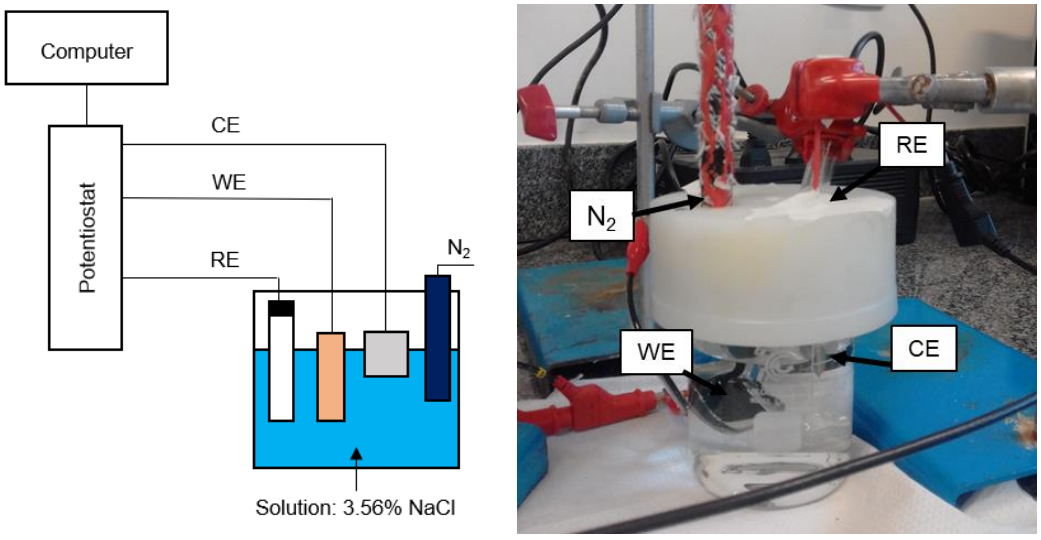


(a) (b)

Figure 3. a) Schematic drawing of a DL-EPR assay emphasizing the electrochemical cell; b) Wiring diagram of the electrochemical cell.

The sample was immersed in the solution for 30 minutes to stabilize the corrosion potential (OCP - Open Circuit Potential). Then, using the potentiostat, an anodic polarization was induced in the sample at a sweep rate of 1.67 mV/s (6 V/h), until reaching a potential 750 mV higher than the OCP. After reaching this value, a reverse bias was performed in the cathodic direction until returning to the OCP. From the curves obtained, the activation (ia) and reactivation (ir) current peaks of each sample were verified to analyze the degree of sensitization. According to Wolynec (2003), the measurement of the degree of sensitization (DS) can be obtained through the relationship between the two peak currents, ir/ia.

For the anodic potentiodynamic polarization test, only the samples in the original state were analyzed. The procedures followed the ASTM G61-86 (2018) standard. The test solution contained 3.56% sodium chloride (by weight), transferring about 100 mL to the electrochemical cell. During the test, the cell was kept at room temperature. After cleaning, the sample was transferred to the electrochemical cell, where it remained in the test solution, initially deaerated using nitrogen gas (N2) for 30 minutes to remove oxygen from the solution, and then maintained for another 30 minutes to determine the OCP. Figure 4 illustrates the schematic of the electrochemical cell.



(a) (b)

Figure 4. a) Schematic drawing of a potentiodynamic polarization test emphasizing the electrochemical cell; b) Wiring diagram of the electrochemical cell.

After measuring the OCP, a sweep was performed from this value in the anodic direction, with a rate of 0.167 mV/s (0.6 V/h), continuing until the current reached 5 mA. The data were then plotted on a graph with a logarithmic scale. Through this polarization curve, it was possible to identify the pitting potential after the passivation of the material.

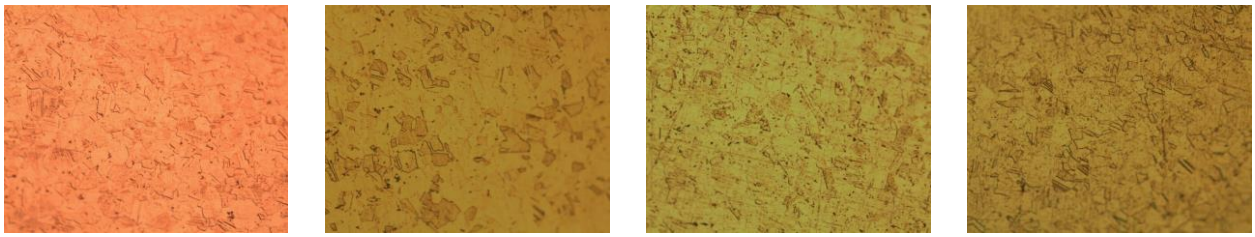
3. RESULTS AND DISCUSSION

The chemical analysis proves, according to Silva and Mei (2010), that the materials of the analyzed samples are austenitic stainless steels, due to their percentage of chromium, carbon and nickel.

Table 1. Main experimental results chemical analysis.

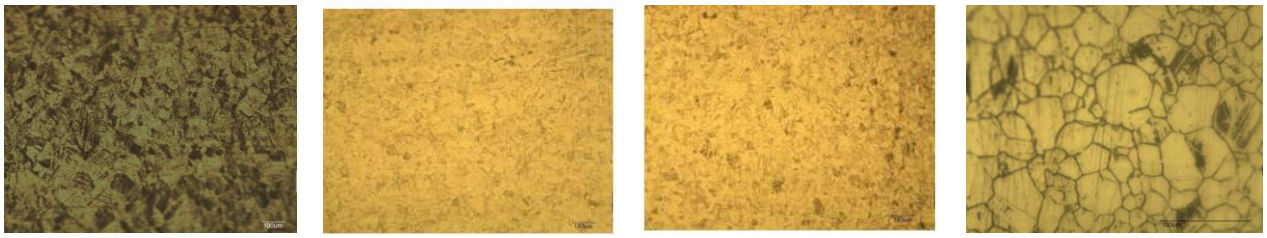
Sample	Chromium (%)	Carbon (%)	Nickel (%)	Molybdenum (%)	Classification
B50	18.5	0.0380	8.7	0.07	304 steel
B54	16.4	0.0265	11.3	1.74	316L steel
B56	16.4	0.0213	11.7	1.79	316L steel
SUP	18.0	0.0536	9.2	0.18	304 steel

The results of metallographic analyzes via MO for the original "as received" samples can be seen in Figure 5, and the heat-treated samples in Figure 6. In the SUP sample, the heat treatment had a direct impact on the kinetics of chromium carbide formation, promoting its precipitation and, as a consequence, intergranular attack occurred close to the grain boundaries, as also verified in Monfardini et al. (2021). According to Nunes (2007), the electrolytic attack in an oxalic acid solution can be used to assess the susceptibility to intergranular corrosion, which was also verified in previous studies, such as those by Viana (2014), Silva (2007) and Palácio (2008). Table 2 presents the main results obtained through the DL EPR test for the samples as received and heat treated.



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

Figure 5. Micrograph of samples "as received" via OM, etched with 10% oxalic acid. a) B50 (200x); b) B54 (200x); c) B56 (200x); d) SUP (200x).



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

Figure 6. Micrograph of samples with heat treatment via OM, etched with 10% oxalic acid. a) B50 (100x); b) B54 (100x); c) B56 (100x); d) SUP (400x).

Table 2. Analysis of the degree of sensitization by the DL-EPR test.

Sample	Medium OCP [V]	Standard deviation	DS Medium (Ir/Ia)	Standard deviation	Degree of sensitization
B50	-0.4546	0.0037	0	0	None
B50 HT	-0.4627	0.0023	0.1468	0.0340	Fully sensitized
B54	-0.4078	0.0035	0	0	None
B54 HT	-0.4167	0.0087	0	0	None
B56	-0.4018	0.0034	0	0	None
B56 HT	-0.4117	0.0069	0.0032	0.0055	None
SUP	0.4566	0.0048	0	0	None
SUP HT	-0.4581	0.0012	0.0398	0.0065	Partially sensitized

Analyzing the degree of sensitization found for austenitic stainless steels, Viana (2014), Bendini Junior (2016), Carneiro (2014) and Muri (2011) suggest that a stainless steel can be classified as non-sensitized, partially sensitized or fully sensitized. If DS < 0.01, the steel is considered not sensitive, if 0.01 ≤ DS ≤ 0.05, the steel is partially sensitive, and when DS > 0.05, the steel is considered to be fully sensitive. For the samples as received, by the degree of sensitization obtained, it is evaluated that there is no presence of regions that are susceptible to intergranular corrosion. As for heat-treated samples, only samples B50 and SUP suffered sensitization effects, classified as fully sensitized and partially sensitized, respectively.

The curves obtained in the DL-EPR test are shown in Figure 7, where it can be verified in the sensitized samples the existence of a reactivation current high enough so that it could be noticed. The result of the B50 sample was compatible with the answer presented by Palácio (2008) considering the same material and heat treatment.

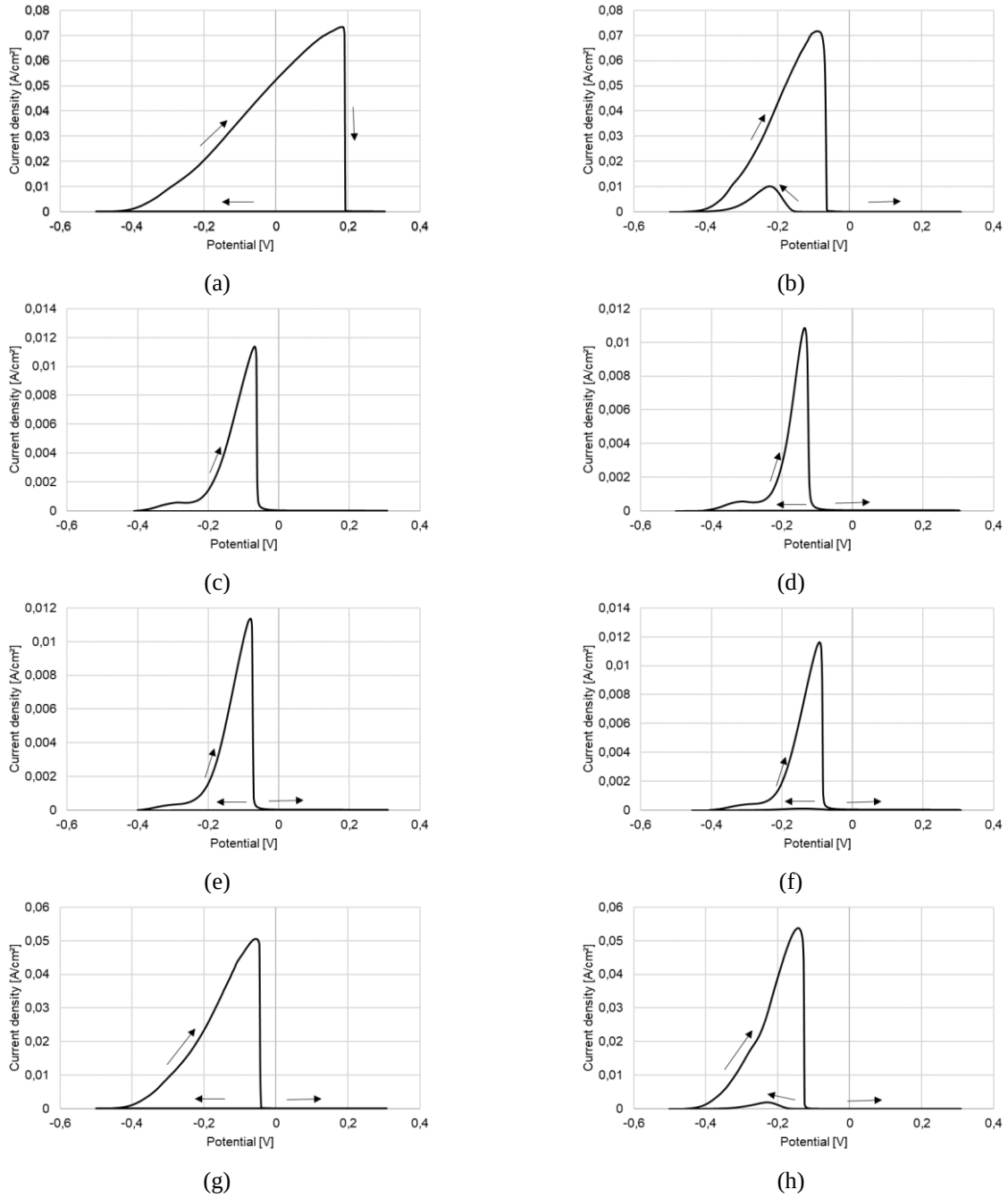
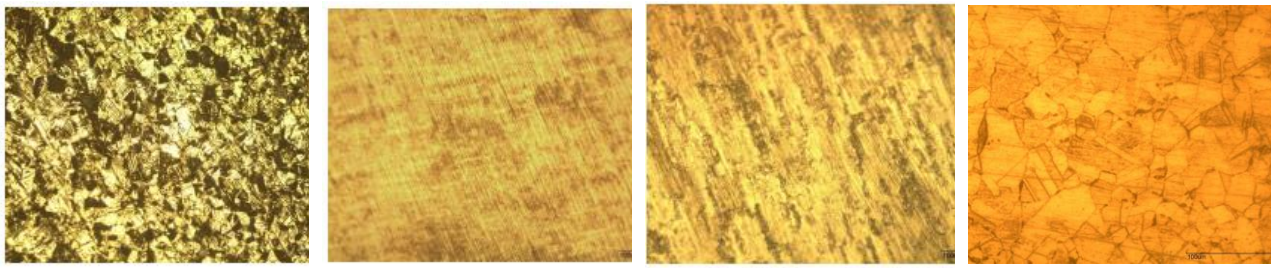


Figure 7. Sample DL-EPR assay curve. a) B50 as received; b) B50 with heat treatment; c) B54 as received; d) B54 with heat treatment; e) B56 as received; f) B56 with heat treatment; g) SUP as received; h) SUP with heat treatment

When analyzing the micrographs obtained, represented in Figure 8, it was not possible to identify the grain boundaries with the precipitated carbides for the B50 sample, even though it was shown to be sensitized in the DL-EPR test. However, when examining the surface of the sample in the SEM, Figure 9, intergranular corrosion was confirmed, showing the loss of material in regions close to the grain boundaries. For the SUP sample, it was possible to identify corrosion by MO and also by SEM. Using EDS, analysis of the chemical composition near the grain boundaries was carried out to confirm the formation of carbides. The analyzed points are shown in Figure 8, with the respective compositions of chromium. A significant increase in the chromium content near the grain boundaries were observed. For samples B54 and B56, the material did not show sensitization, the presence of molybdenum and lower carbon content are decisive in suppressing this effect.



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

Figure 8. Micrograph of samples with heat treatment via OM, after DL-EPR test. a) B50 (100x); b) B54 (100x); c) B56 (100x); d) SUP (100x).

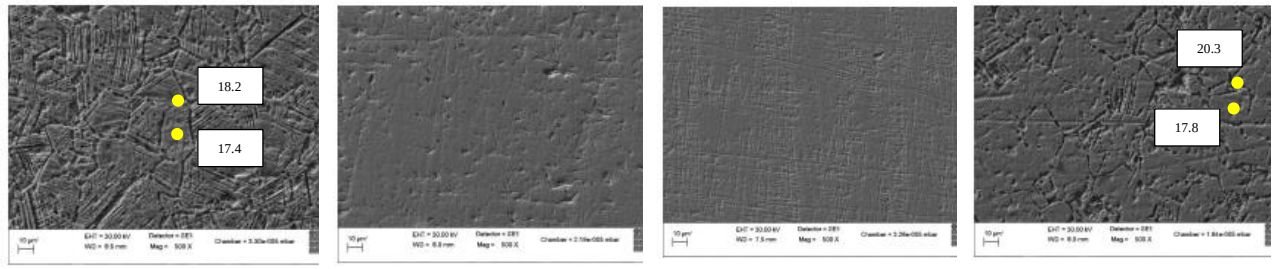


Figure 9. Micrograph of samples with heat treatment via MEV, after DL-EPR test. a) B50 (500x); b) B54 (500x); c) B56 (500x); d) SUP (500x).

Regarding the anodic potentiodynamic polarization test, Figure 10 illustrates the curves obtained for the studied samples. The pit potential (E_{pite}) is indicated by the red arrow, which was obtained by the tangent method, also performed by Garcia (2014).

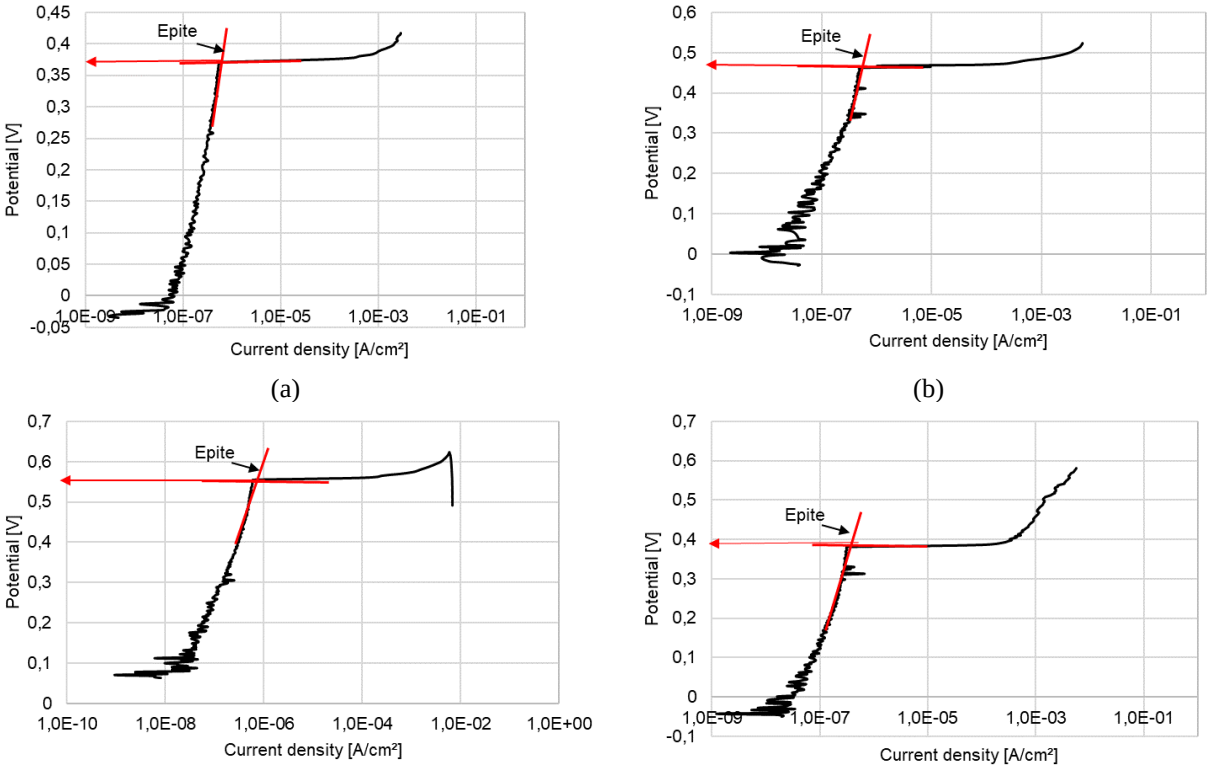


Figure 10. Anodic potentiodynamic polarization curves, samples such as received. a) B50; b) B54; c) B56; d) SUP.

Table 3 presents the main results obtained through the anodic potentiodynamic polarization test for the samples as received. Note that samples B54 and B56 showed higher pit potential values than samples SUP and B50. This happens because they are 316 L stainless steels, in which, according to Carbó (2008), the addition of molybdenum makes the material more resistant to pitting corrosion. Therefore, the behavior of the pitting potential is equivalent to the increase in the content of molybdenum and nickel present, showing the influence of these elements in the regeneration of the passive layer.

Table 3. Result of samples tested by anodic potentiodynamic polarization.

Sample	Medium OCP [V]	Standard deviation	Pitting Potential [V]	Standard deviation
B50	-0.0336	0.0041	0.3650	0.0031
B54	-0.0597	0.0364	0.4651	0.0086
B56	0.0424	0.0165	0.4591	0.0985
SUP	-0.1238	0.0539	0.3969	0.0137

Using the OM, it was possible to identify the pits formed for each sample, as shown in Figure 11. It can be seen that the form of attack by pitting corrosion is localized, where regions adjacent to the pit do not show any change in microstructure.

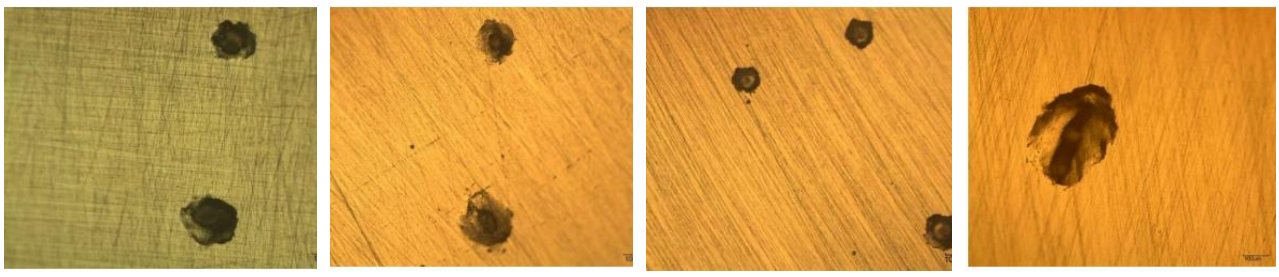


Figure 11. Micrograph of samples, as received, after assay of anodic potentiodynamic polarization, via OM. a) B50 (100x); b) B54 (100x); c) B56 (100x); d) SUP (100x).

4. CONCLUSION

The results of this work showed that the characterization of samples through chemical analysis agrees with metallographic tests. Only with metallography, it was possible to verify susceptibility to intergranular corrosion in the heat-treated SUP sample, evidenced by electrolytic attack with oxalic acid.

It was verified that samples B54 and B56, both type 316L, did not show sensitization with heat treatment, as verified by the DL-EPR test and by OM and SEM analyses, which did not show intergranular corrosion. The B50 and SUP samples (classified as 304 steel) were sensitized. However, even with similar chemical compositions, they had different results in the DL-EPR test. B50 was fully sensitized, while the SUP sample showed partial sensitization. Analyzes using EDS demonstrated the precipitation of chromium carbides through an increase in the chromium content at the grain boundary.

In relation to the anodic potentiodynamic polarization test, the pitting potentials were higher for samples B54 and B56, confirming the resistance to pitting corrosion presented by 316L steels. The molybdenum and nickel content present in these materials had a significant effect on increasing corrosion resistance. Therefore, in the two corrosion tests carried out, it was confirmed that alloy 316L presented a better response to resistance to intergranular corrosion and pitting than alloy 304.

The use of materials that are highly degraded by the industrial process can make it difficult to carry out corrosion tests and lead to results that are not compatible with the literature, such as the difference in sensitization presented by B50 and SUP, which in theory are made of the same material. However, the results obtained reflect the current state of the samples and show how the loss of their chemical properties directly influences corrosion resistance.

5. ACKNOWLEDGEMENTS

To the Federal Institute of Espírito Santo.

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