

## Evaluation of voltametric profiles for quantitative estimation of sigma phase ( $\sigma$ ) content in duplex stainless steel UNS S31803: an ohmic and layers porous resistance models (LPRM) analysis

## Avaliação de perfis voltamétricos para estimativa quantitativa de Fase Sigma ( $\sigma$ ) em aço inoxidável duplex UNS S31803: análise de modelos Ohmico e de resistência de camada porosa (LPRM)

DOI:10.34117/bjdv9n2-060

Recebimento dos originais: 09/01/2023

Aceitação para publicação: 10/02/2023

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### ABSTRACT

The voltammetric profile parameters of potential and peak current intensity, for different scan rates were obtained for heat treated duplex stainless-steel samples. The Ohmic and Layer Porous Resistance models were considered for quantitative estimation of deleterious phase surface content (sigma phase). A 4 mol L<sup>-1</sup> KOH selective solution and a specially designed electrolytic cell were used. The observed linear slope for  $E_p$  vs  $v^{1/2}$  and  $I_p$  vs  $v^{1/2}$  linearity profiles are in accordance with the considered LPR Model. The parameter profile of peak potential ( $E_p$ ), with the square root of the scan rate (1 mV s<sup>-1</sup>, 2 mV s<sup>-1</sup>, 5 mV s<sup>-1</sup>, 7 mV s<sup>-1</sup> and 10 mV s<sup>-1</sup>), suggests the growth of a film rich in Fe and Cr content that could be correlated to the sigma phase level with the dependence of  $E_p$  on

scan rate been considered a better parameter, than peak current, for detect small levels of sigma phase for a specific scan rate. The non-dependence of  $E_p$  from surface area, make it a more feasible parameter for a lower sigma phase level quantitative analysis.

**Keywords:** intermetallic phases, oxide growth, linear voltammetry.

## RESUMO

Os parâmetros de perfil voltamétrico de potencial e intensidade de pico de corrente, para diferentes taxas de varredura foram obtidos para amostras de aço inoxidável duplex tratados com calor. Os modelos de Resistência de Camada Porosa (LPR) e Ôhmico foram considerados para estimativa quantitativa de conteúdo de superfície de fase deletéria (fase sigma). Uma solução seletiva de  $4 \text{ mol L}^{-1}$  KOH e uma célula eletrolítica especialmente projetada foram usadas. Os perfis de linearidade observados para  $E_p$  vs  $v^{1/2}$  e  $I_p$  vs  $v^{1/2}$  estão de acordo com o modelo LPR considerado. O perfil do parâmetro de potencial de pico ( $E_p$ ), com a raiz quadrada da taxa de varredura ( $1 \text{ mV s}^{-1}$ ,  $2 \text{ mV s}^{-1}$ ,  $5 \text{ mV s}^{-1}$ ,  $7 \text{ mV s}^{-1}$  e  $10 \text{ mV s}^{-1}$ ), sugere o crescimento de um filme rico em conteúdo Fe e Cr que poderia ser correlacionado ao nível de fase sigma com a dependência de  $E_p$  na taxa de varredura de potenciais foi considerado um parâmetro melhor, do que a corrente de pico, para detectar pequenos níveis de fase sigma para uma taxa de varredura específica. Além disso, a não dependência de  $E_p$  da superfície, torna-se um parâmetro mais viável para uma análise quantitativa de nível de fase sigma mais baixa.

**Palavras-chave:** fases intermetálicas, crescimento de óxido, voltametria linear.

## 1 INTRODUCTION

With a balance of austenite ( $\gamma$ ) and ferrite ( $\delta$ ) phases, duplex stainless steels are in demand in a wide range of the industrial sector, where high mechanical properties and corrosion resistance are required (DONIK et al., 2009; SWENS and KOLSTER, 1991).

The good corrosion resistance of these materials is provided by the formation of a continuous and thin oxide film, mainly of  $\text{Cr}_2\text{O}_3$ , on the surface (DONIK et al., 2009; LUIZ et al., 2020; ÖRNEK et al., 2019). In this regard, the existence of a passive film for superduplex steel type UNS S32750 is considered by (ÖRNEK et al., 2019) generally in the form of a double oxide/hydroxide layer, one as a barrier layer rich in Cr and a superior external one in Fe.

A study about stainless steel UNS S31803 passivation, concluded that an increase in pH to more alkaline values at the metal/solution interface has a marked influence on the electrochemical behavior of this passive film, that favors reactions with more reactive oxides according to (SÁNCHEZ et al., 2012).

(ABDO et al., 2021), analyzed the electrochemical corrosion behavior of a UNS S31803 laser welded metal, in an environment with different pH values, and also observed different bilayer structures for the formed film, changes with the degree of acidity and alkalinity, formed by Fe and Cr oxides in the same structure.

By other hand, the exposure of duplex stainless steels to high temperatures, in the range of 700 – 900 °C, promotes the precipitation of unwanted phases (deleterious / intermetallic phases) such as sigma ( $\sigma$ ) and Chi ( $\chi$ ), non-magnetic Fe-Cr bonds, which has the effect of reduced the mechanical properties of the material, this affirmed by (POHL et al., 2007).

Related to the precipitation of the  $\sigma$  phase, (MAGNABOSCO, 2009) reports three mechanisms have been described, which involve the decrease of Cr in the surrounding metallic matrix of this secondary phase: nucleation and growth from ferrite, eutectoid decomposition of ferrite, with formation of secondary austenite ( $\gamma^2$ ), and growth from austenite. This author by The Time – Temperature - Precipitation (TTP) diagram, for analysis of the  $\sigma$  phase in the UNS S31803 interval of 700-900°C, shows that, for 850°C, there is a higher precipitation kinetics of the  $\sigma$  phase with preferential nucleation in  $\delta/\gamma$  interface and growth strongly linked to the consumption of  $\delta$ .

(HASKEL et al., 2015) analyzed the presence of deleterious phases in UNS S31803 steel by the linear scan voltammetry (LSV) technique and it was observed that with increases of the precipitate contents, the voltammograms showed changes for the current density peaks,  $J_p$ , allowing the determination of 0.9% of  $\sigma$  phase, a value below of the critical range which is between 1 and 2%.

Electrolytic solutions like KOH, NaOH presents high sensibility for reaction with Cr in the  $\sigma$  and  $\chi$  phases that is oxidized to higher states ( $\text{Cr}^{+3}$  to  $\text{Cr}^{+6}$ ). This selective behaviour is an advantage to be used for metallographic and potentiodynamic tests (HASKEL et al., 2019; DE SAMPAIO et al., 2020). From the results obtained by (HASKEL et al., 2019), electrochemical cells with micro electrode concepts with seems to be better for the analysis, providing current densities in the order of microamperes and an outstand sensitivity result for small levels of  $\sigma$  and  $\chi$  deleterious phases quantification, this has also been proved by (DE SAMPAIO et al., 2020).

Considering the mentioned parameters and the LSV technique applied for film growth analysis on metallic surfaces, it is worth mentioning the Ohmic

(D'ALKAINE and CORDEIRO, 1984; D'ALKAINE et al., 1993; D'ALKAINE et al., 2004) and Porous Layer Resistance models (CALANDRA et al., 1974; BRUNETTI et al., 1999).

To describe the voltametric growth of a continuous oxide film on some metallic surface, D'Alkaine and Cordeiro in 1984 (D'ALKAINE et al., 1993) started from the hypothesis that the behavior of this film was similar to that of a gel, with an Ohmic relation between current intensity – overpotential, ( $I/\eta$ ), regardless the electric field magnitude.

This Ohmic model described as linear relation between the peak current density  $J_p$  and scan rate square root, in terms of the film ionic resistivity and its specific volume, is expressed by the equation 1:

$$J_p = \frac{1}{(\rho \cdot C)^{1/2}} v^{1/2} \quad (1)$$

where  $\rho$  is the film ionic resistivity and  $C$  its volume per unit electric charge.

The layer pores resistance model (LPRM) describes the growth of a solid, isolating, low-conductivity film, with initially random nucleation and propagation over the electrode surface. This film behaves as a physical barrier to flow and current and has a constant thickness with the presence of small pores in the layer formed. This kind of film formed is subject also to an ohmic resistance control but in a layer-pore system model (CALANDRA et al., 1974; BRUNETTI et al., 1999).

For the description of the anodic growth process of this film, an insoluble compound is formed under voltametric conditions on the electrode surface (CALANDRA et al., 1974).

According to (CALANDRA et al., 1974; BRUNETTI et al., 1999), voltammograms correspondent to systems where the LPRM model could be applied, showed linear dependence peak current intensity,  $I_p$  and peak potential,  $E_p$  with the square root of the scan rate,  $v$ , to consider the degree of coverage in the peak condition,  $\theta_p$ , independent of scan rate as

$$I_p = \left\{ \frac{3 \cdot Q_0 \cdot \theta_p}{b \cdot [1 - \theta_p]} \right\}^{1/2} (1 - \theta_p) v^{1/2} \quad (2)$$

$$E_p = \left\{ \frac{3 \cdot Q_0 \cdot \theta_p}{b \cdot (1 - \theta_p)} \right\}^{1/2} [R_0(1 - \theta_p) + b\theta_p^{1/2}] v^{1/2} \quad (3)$$

Although the film thickness is a function that changes temporally along with the is the degree of coverage of the electrode,  $\theta$ , at the peak voltametric condition, is considered to be constant in accordance with (BRUNETTI et al., 1999).

Considering the alkalinity of the medium and the constituent elements of the electrode surface, it is important to consider the work done by (PAPASSIOPI et al., 2014) indicating that for Cr(III) salt solutions an alkaline medium promotes the precipitation of  $\text{Cr}(\text{OH})_3(\text{H}_2\text{O})_3$  (metastable) crystalline networks that become amorphous by dehydration, and for reduction of Cr(VI) to Cr(III) in the presence of Fe, the precipitate is a mixed phase  $\text{Fe}_{(1-x)}\text{Cr}_x(\text{OH})_3$ , compounds that can be suggested as precipitates covering the electrode surface of area  $A$ .

In this presented paper, it was considered that reaction has a higher kinetic for the  $\sigma$  phase than that for the base material to determine, with high sensibility, low amounts of  $\sigma$  phase. Thus, a linear dependence related the increase of  $J_p$ , due to the  $\sigma$  phase level, for example, with the square root of the potential sweep rate, could suggest the ohmic model to describe the anodic film growth over a specific phase on the surface of UNS S31803 steel. Also, the experimental voltammograms correspondent to the UNS S31803, submitted to heat treatment, could consider the LRPM model as an alternative for analysis to its  $\sigma$  content, through the evaluation of  $I_p - v^{1/2}$  or  $E_p - v^{1/2}$  response.

## 2 MATERIALS AND METHODS

Duplex stainless-steel plates of UNS S31803 with dimensions 10 mm x 08 mm x 05 mm and solubilized at 1100°C were used. The solubilized sample is denoted by  $S_1$  and was taken as reference material (absent of intermetallic precipitates), while  $S_2$  is the sample subjected to a heat treatment of 870°C for 60 min for the precipitation of the intermetallic  $\sigma$  phase and was cooled in water at room temperature of 25°C. To quantify the of  $\sigma$ -phase precipitated amount in the samples, a metallographic analysis was carried out with the samples embedded in Bakelite, sanded with 120, 220, 400, 600, and 1200 mesh and polished in felt with alumina solution at a granulometry of 0.3 micron. The microstructure of the samples surfaces was revealed by electrochemical surface attack with a 10% KOH solution and application of 2.5 V of DC through a graphite cathode and

platinum electrode for anodic contact with the work electrode (samples) for 40 s. The metallographic images were obtained and processed on an OLYMPUS optical microscope, MODEL BX-51M, with OIM ANALYSIS 5.0 software and 20x magnification, indicates 2.8%  $\sigma$ -phase content for the 60 min treated S<sub>2</sub> sample.

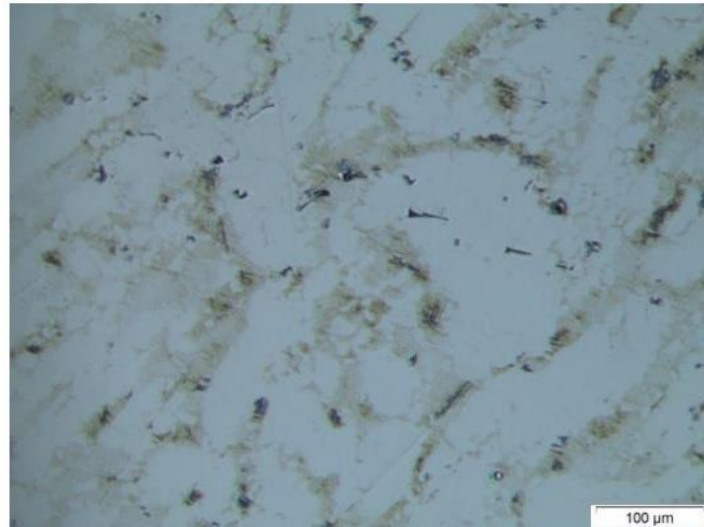
In order to carry out the tests by linear voltammetry, an especially designed electrochemical cell with 5 ml of electrolyte solution was used. The samples (UNS S31803) were the work electrode, platinum wire the counter and a Saturated Calomel Electrode as the reference electrode. A PalmSens 2 electrochemical interface device complements the experimental assembly. The electrochemical cell used were made with Teflon with a hole of 0,7854 mm<sup>2</sup>, for the contact with the work electrode plate. For the selective attack of the  $\sigma$  phase, an electrochemical solution of 4 mol L<sup>-1</sup> KOH was used. The applied scan rates were 1 mV s<sup>-1</sup>, 2 mV s<sup>-1</sup>, 5 mV s<sup>-1</sup>, 7 mV s<sup>-1</sup> and 10 mV s<sup>-1</sup>, tests performed in duplicate.

### 3 RESULTS AND DISCUSSION

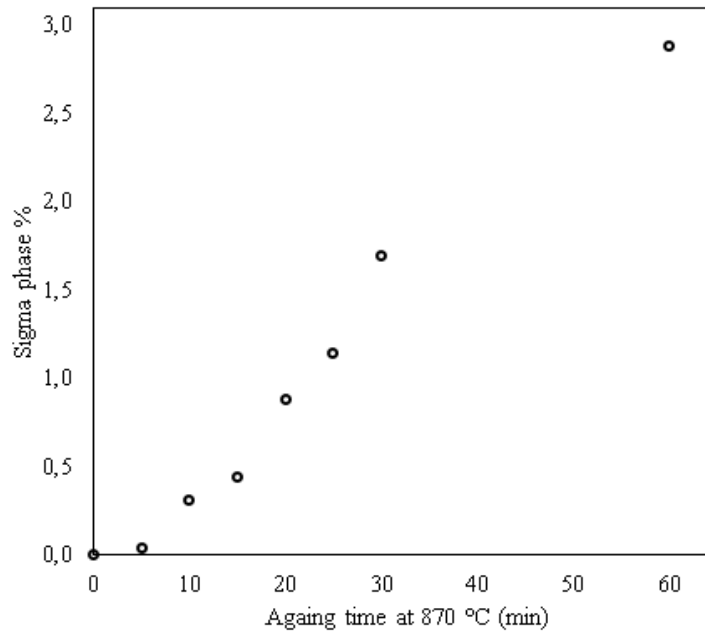
The Figure 1 corresponds to the metallographic test of S<sub>2</sub> sample of the UNS S31803, presents the profile of the different phases on the surface, obtained with electrolytic attack in 10% KOH solution, 2.5 V for 40 s, after isothermal treatment at 870 °C for 60 min. The intermetallic precipitates are revealed in the darkest shade while the ferrite phase has a yellowish-brownish tendency and the austenite has a light silvery color.

The determination of the percentage in area of the amount of  $\sigma$  precipitates for this heat treatment resulted in 2.8% and the profile of the content of this phase with the time of heat treatment is presented (Graphic 1), which indicates, starting at 10 min of heat treatment, a rapid increase in the amount of precipitated phase in the samples.

Figure 1. Micrograph of UNS S31803 sample corresponding to metallographic test after isothermal treatment at 870°C for 60 min.

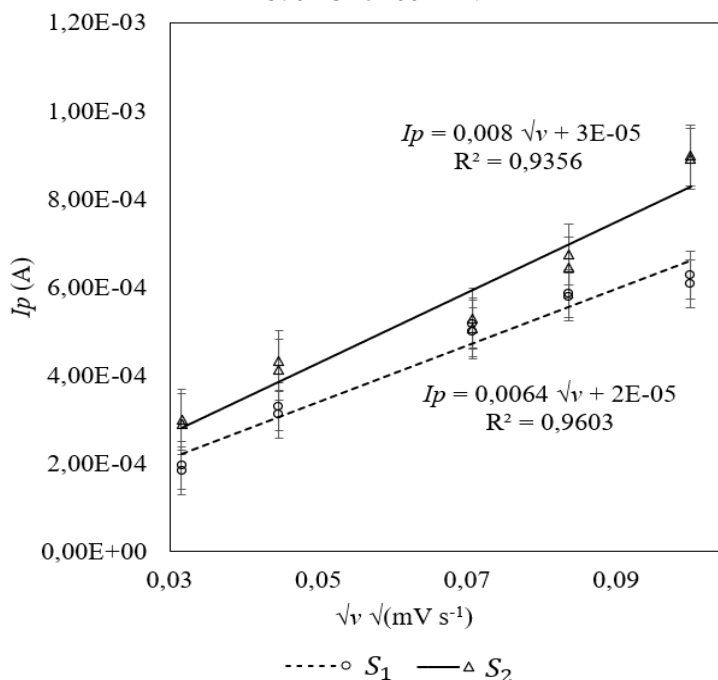


Graphic 1. Percentage of phase  $\sigma$  per unit area as a function of isothermal treatment time.



The experimental voltametric results for samples S1 and S2 resulted in an almost linear and parallel relation of parameters  $I_p$  and  $v^{1/2}$ , as indicated by the curves (Graphic 2). The results demonstrated an increase of  $2.77E-04 \pm 0.3(A)$  in the  $I_p$  value for sample S2, in relation to S1 for  $10 \text{ mV s}^{-1}$ .

Graphic 2. Comparison of  $I_p$  vs  $v^{1/2}$  profiles for samples  $S_1$  and  $S_2$  of UNS S31803 steel correspondent to voltammetry obtained from tests carried out with  $4 \text{ mol L}^{-1}$  KOH solution and  $1 \text{ mV s}^{-1}$ ,  $2 \text{ mV s}^{-1}$ ,  $5 \text{ mV s}^{-1}$ ,  $7 \text{ mV s}^{-1}$  and  $10 \text{ mV s}^{-1}$  scan rates.  $S_2$  subjected to heat treatment at  $870 \text{ }^\circ\text{C}$  for 60 min.



The KOH concentration used for this analysis confirm the suitable condition (pH 13.97), as already described by (HASKEL et al., 2015; HASKEL et al., 2019; DE SAMPAIO et al., 2020), for a selective interaction with the intermetallic phase.

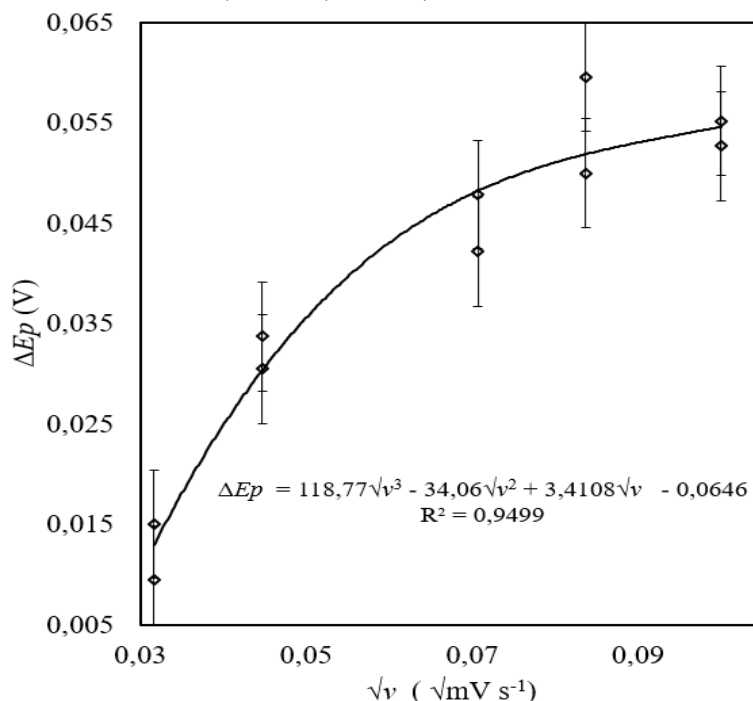
The deviation from linearity in  $I_p$  for  $S_1$  sample, for higher  $v$ , could be associated with film aging process, accord to (D'ALKAINE et al., 1993), follow an increase in its  $\rho$  with, accord to equation (1), a consequence decreases in  $J_p$ . Another alternative consideration is depletion of  $\text{Cr}(\text{OH})_3$  concentration, over the  $\sigma$  phase, due to mass transport at higher scan rate, as proposed by fundamental electrochemistry for totally irreversible systems.

For the  $S_2$  sample, the variation in linearity can be attributed to the random distribution of  $\sigma$  phase at the different points of analysis. These effects related to variation in  $I_p$  vs  $v^{1/2}$  linearity profiles could impart a better technique sensitivity for higher scan rates were aging process and mass transport phenomena could become significative in relation to the film formation reactions over the  $\sigma$  phase. A result that corroborates with this phenomenon is observed by (Graphic 3), in which the effect of peak potentials variation for  $S_2$ , in relation to  $S_1$ , with the sweep rate



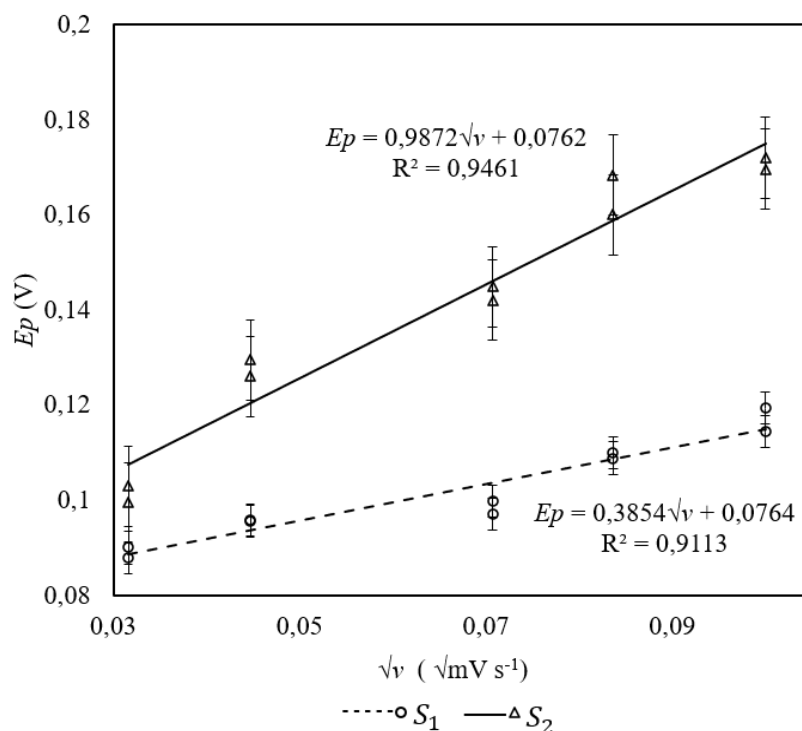
shows a maximum, corresponding to  $10 \text{ mV s}^{-1}$  ( $0.1 \text{ (V s}^{-1})^{1/2}$ ). After this maximum, the curve evolution indicates limited scan rates to be avoided.

Graphic 3. Variation of  $E_p$  vs  $v^{1/2}$  with  $4 \text{ mol L}^{-1}$  of KOH for sample  $S_2$  heat treated at  $870^\circ\text{C}$  for 60 min with  $1 \text{ mV s}^{-1}$ ,  $2 \text{ mV s}^{-1}$ ,  $5 \text{ mV s}^{-1}$ ,  $7 \text{ mV s}^{-1}$  and  $10 \text{ mV s}^{-1}$  scan rates.



The  $E_p$  vs  $v^{1/2}$  linear profiles for  $S_1$  and  $S_2$  samples of (Graphic 4), indicate a good fit for the LPR Model. It could be obtained an increase in the  $E_p$  value for  $S_2$  of about  $0.05 \pm 0,048 \text{ V}$  in relation to  $S_1$  for the rate as high as  $10 \text{ mV s}^{-1}$ . This result is better than that when considered  $I_p$ . The  $E_p$  vs  $v^{1/2}$  slop for  $S_1$  sample is about  $20 \text{ mV}$  for each tenfold increase in scan rate corresponding to the condition for totally irreversible system  $30/\alpha n \text{ mV}$  at  $25^\circ\text{C}$  (for  $n=3$  ( $\text{Cr}^{+3}$ ) and  $\alpha=0.5$ , number of electrons and transport coefficient, respectively) (BARD and FAULKNER, 2022). The higher  $E_p$  vs  $v^{1/2}$  of  $S_2$  slop indicates surface different reactions due to  $\sigma$  phase passivating process, probably, influenced by  $\text{FeOOH}$  precipitates as proposed for the passive film by (Yao et al., 2019). Another important result could be considered in regard to the non-dependence of  $E_p$  from electrode area allowed a much feasible analysis of  $\sigma$  phase levels.

Graphic 4.  $E_p$  vs  $v^{1/2}$  profiles for samples  $S_1$  and  $S_2$  of UNS S31803 steel. Heat treatment at 870°C for 60 min. Voltammetry performed with 4 mol L<sup>-1</sup> KOH solution and 1 mV s<sup>-1</sup>, 2 mV s<sup>-1</sup>, 5 mV s<sup>-1</sup>, 7 mV s<sup>-1</sup> and 10 mV s<sup>-1</sup> scan rates.



The linear slope for  $E_p$  vs  $v^{1/2}$  for  $S_1$  could be associated to a complete film formation process, accord to LPRM model. Considered the aging process, as presented for Ohmic model, an increase in the scan rate, could induces passivation films with more positive  $E_p$ . For  $S_2$  sample, the more positive  $E_p$  and greater slope for the  $E_p$  vs  $v^{1/2}$  curve may represent a faster reactivity of the intermetallic precipitate, due to its higher composition in Cr, at in the aging step.

The  $\text{Cr}(\text{OH})_3$ ,  $\text{FeOOH}$  precipitates present in the passive film according to (YAO et al., 2019), could experience an increase in the formation rate due to the strongly alkaline medium. This consideration is supported in what was explained by (LEE and HERING, 2005), about the dissolution kinetics of  $\text{Cr}(\text{OH})_3(\text{s})$ , which was thermodynamically favored in an oxidant environment, on considering the compounds present in the film formed on the surface of a UNS S31803 steel under basic pH conditions (SÁNCHEZ et al., 2012).

Therefore, on dynamics of film growth, its precipitates of hydroxides, components of the passive layer, could have a marked tendency to transform into oxides at an aging step. Estimate the  $\sigma$ -phase content, present in UNS S31803 steel, could then be performed with greater reliability through  $E_p$  analysis, when considering Fe-Cr oxides filled at the

pores of the formed layer and scan rates up to the maximum observed at (Graphic 3). According to the LPRM model, the higher increase in the intensity of  $E_p$  could be a better parameter to analyze intermetallic alloy amount levels.

#### 4 CONCLUSION

Voltametric parameters of peak potential and current intensity were evaluated for different scan rates of tests performed for UNS S31803 duplex stainless-steel samples, in a selective electrolyte of 4 mol L<sup>-1</sup> KOH solution, after heat treated. The LSV technique was applied and analysis predicted by the Ohmic and Porous Layer Resistance models were performed. It was observed important effect of aging process from the  $\sigma$  phase passivation and that this process is faster for greater the scan rate.

The parameter profile of  $E_p$  with the  $v^{1/2}$ , suggests the growth of a film rich in Fe and Cr content that could be correlated to the  $\sigma$  phase level and that dependence of  $E_p$  on the scan rate could be considered a better parameter, than peak current, for detect small levels of  $\sigma$  phase for a specific scan rate. The non-dependence of  $E_p$  from electrode area allowed a much more feasible analysis of  $\sigma$  phase levels.

#### ACKNOWLEDGEMENTS

The present study was partly funded by the Coordination for the Improvement of Higher Education Personnel – Brazil (CAPES) – Finance Code 001.

The authors declare that there is no conflict of interest regarding this work. This manuscript has not been published elsewhere and is not being considered by another journal.

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