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Effect of thermal aging on the microstructure and mechanical properties of stainless steel UNS S31803

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ABSTRACT

Duplex stainless steel UNS S31803 exhibits high mechanical strength with high corrosion properties, due to its microstructure composed of ferrite and austenite phases, in equal proportion. When the UNS S31803 steel is submitted to high temperatures, some precipitations can occur, such as nitrites, carbides, and third phases (e.g. sigma phase -σ, and alpha prime - α'). These phases are deleterious in relation to the mechanical properties and corrosion resistance, and their effects are analyzed regarding the properties of the steel. In order to evaluate the precipitation of this deleterious phase, isothermal treatment was done at 500°C for 144 hours (α' phase) and at 850ºC for 80 minutes (σ phase). The results were obtained through the microstructural analysis and tensile tests. The presence of sigma phase was verified in the grain boundary, an increase in the mechanical resistance with a loss of toughness. There was as well as an increase in mechanical resistance with the precipitation of α', with less loss of ductility than that observed in the experiments involving the presence of sigma phase.

Keywords: Duplex stainless steel; Embrittlement; Sigma phase; Mechanical properties.

1. INTRODUCTION

Stainless steels, in addition to having good mechanical properties, are very resistant to corrosion. Chromium is the main element responsible for corrosion resistance. This material may still have high mechanical properties due to the presence of nickel, nitrogen, and other elements. The stainless steels can be formed by the austenitic, ferritic, martensitic, precipitation hardened and duplex (austenitic and ferritic) microstructures.

Duplex stainless steels are steels resistant to uniform and pitting corrosion, so they are widely used in the chemical and petrochemical industry. They exhibit high resistance to stress corrosion, but are not immune to cracking. The best mechanical properties and resistance to corrosion are obtained with a microstructure composed of equal parts of ferrite and austenite and are free of third phases [1-3]. The austenitic phase is formed by solid state transformation from the ferritic phase. In these steels, the equilibrium of the chemical composition and the heat treatment affects the composition of the phases. The distribution of phases, morphology, and texture of both phases are important factors that can influence the properties of this material [4]. Duplex stainless steels have high levels of alloying elements such as Cr, Ni, and Mo with the presence of N. These elements must be suitably balanced in order to obtain similar volumetric fractions of both phases and give both ferrite and austenite mechanical strength and corrosion [5]. In addition to the ferritic - austenitic phases found in duplex stainless steels, third phases may precipitate in these steels. At high temperatures 600-900ºC, during hot forming or welding operation, precipitation of the σ phase can occur, among others. According to FONSECA *et al.* [6], during the duplex stainless steels welding, special care must be taken with the heat input in order to maintain the balance of the austenite and ferrite phases and prevent the precipitation of deleterious phases, especially the sigma phase. In the heating range, 350-550°C, spinodal decomposition of the ferrite phase can also occur in small precipitates of α' (rich in Cr) and α (rich in Fe) [1].

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The α' phase is composed essentially of Fe and Cr. This phase presents BCC (body-centered cubic) structure. The α' phase is small (20 to 200Å) and has a high resistance to growth, even for long exposure times [7]. The presence of the α' phase increases the hardness and induces high brittleness in the steel. The precipitation of the α' phase occurs due to a miscibility gap in the Fe-Cr system. In this gap the ferrite can decompose in α' (rich in Cr) and α (rich in Fe) [8]. The highest incidence of the α' phase occurs at 475°C [9].

The sigma phase (σ) is an intermetallic Fe-Cr-Mo phase. This phase forms at elevated temperatures (600-900°C). The presence of the σ phase has greater influence on ferrite compared to austenite [10]. The σ phase can occur in processes involving high temperatures, such as casting, rolling, welding, forging, and heat treatments. It is difficult to prevent precipitation of the σ phase when the Cr content is above a certain level (above 20 wt%) in stainless steels with the presence of Mo [11].

The focus of this work is the influence of the precipitation of the σ and α' phases on the mechanical properties and microstructure of duplex stainless steel UNS S31803.

2. EXPERIMENTAL PROCEDURES

The duplex stainless steel UNSS31803 used in this research was supplied by APERAM South America, Timóteo City, Minas Gerais State, Brazil in the form of cold-rolled sheets with dimensions of 1.5 x 150 x 300 mm^3 and was annealed at 1060°C. Table 1 shows the chemical composition of the steel as received; the composition was obtained using a CS-400 carbon analyzer and an ARL4460 optical emission spectrometer (APERAM South America, Timóteo City, Minas Gerais State, Brazil).

Table 1: Chemical composition of the UNS S31803 duplex stainless steel (wt%).

C	Cr	Ni	Mo	N	Si	Mn	V	Co	Sn	Ti	Nb	Cu	W
0.02	22.38	5.35	3.04	0.15	0.31	1.82	0.06	0.02	0.01	0.04	0.04	0.20	0.01

The isothermal aging treatments were performed in two stages. The first one consisted of a treatment at 500°C (144 hours) and the second at 850°C (80 minutes). For both aging treatments, the oven was preheated at the treatment temperature and cooling was carried out in water at 20°C.

For microstructural analysis, a VEGA3 Tescan scanning electron microscope (SEM) was used. Samples taken from the sheets were embedded in Bakelite with the thick longitudinal section exposed in accordance with ASTM Standard (E3-11) [12]. The chemical attack to reveal the microstructure was performed with the Behara reagent (1g $K_2S_2O_5$ in 15 mL HCl diluted in 85 mL of distilled water, with an exposure time of 20 s) [13, 14].

The tensile tests were performed according to ASTM Standard (E8/E8M-16a) [15]. The test was performed at a speed of 2 mm/min using an EMIC tensile test machine, model DL20000.

3. RESULTS AND DISCUSSION

3.1 Microstructural characterization

Figure 1 shows SEM images for the sample as received (without isothermal treatment) and aged at 500ºC and 850ºC.

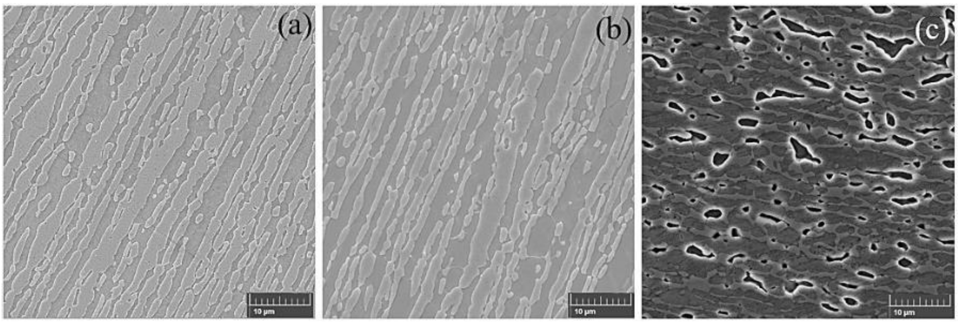

Figure 1: Stainless steel UNS S31803 duplex microstructures: (a) as received; (b) aged at 500ºC; (c) aged at 850ºC. SEM.

According to Figure 1(a,b), the steels did not present any microstructure variation, such as phase distribution and phase morphology as a result of the aging treatment at 500ºC. Although α' precipitation most likely occurred, given its small size (20 to 200 Ǻ), its identification cannot be revealed by SEM.

Figure 1(c) presents the microstructure of duplex stainless steel aged at 850ºC. It can be observed that there was a great precipitation of the σ phase dispersed in the matrix, preferably in the adjacencies of the grain boundaries (Figure 2). The microstructure (Figure 1-c) is composed of ferrite matrix (light gray) with austenite island (dark) and σ precipitates.

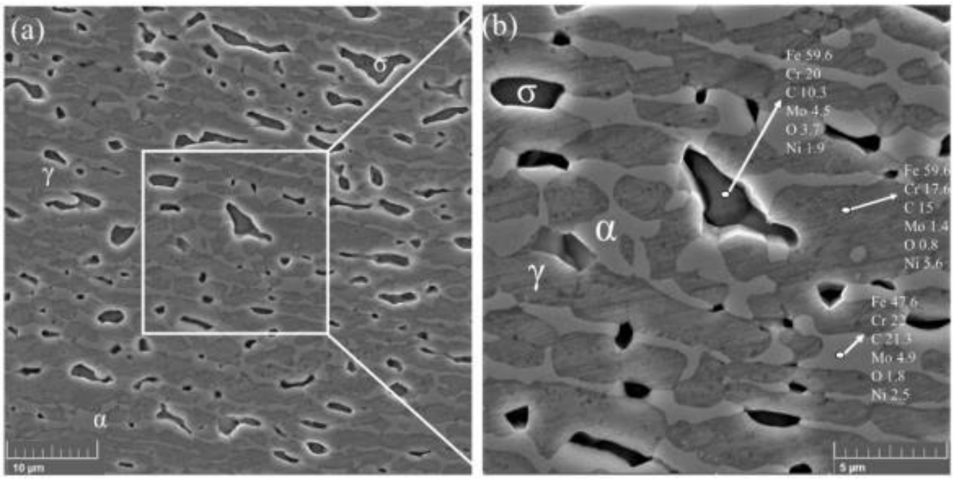

Figure 2: Stainless steel UNS S31803 duplex microstructures aged at 850ºC. SEM.

Figure 2 (a-b) shows the existence of a phase very rich in Cr and Mo, allowing the conclusion to be the σ phase. The precipitation of the sigma phase occurs preferentially in ferrite near the ferrite / austenite grain contours. According to DEL ABRA-ARZOLA *et al.* [16], this occurs due to precipitation kinetics from the ferrite where Cr and Mo elements are more stable. With the formation of the σ phase, diffusion of Cr and Mo occurs to the intermetallic phase enrichment. Therefore, ferrite looses its stabilizing elements and becomes secondary austenite (γ2) [17].

Figure 3 shows phase fractions obtained in the steel UNS S31803: as received (cold rolled and annealed at 1060ºC), aged at 500ºC and 850ºC. The quantitative analysis was performed according to ASTM E562-11 [18]. As can be observed, the steel as received exhibited approximately equal fractions of α and γ phases. In the steel aged at 850ºC, there was a decrease in the α phase due to the σ phase precipitation. In the steel aged at 500ºC, no significant change in the α and γ phase fractions was observed.

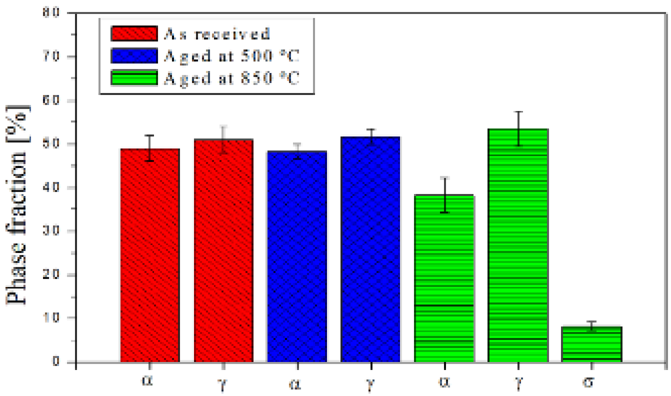

Figure 3: UNS S31803 Phase fraction.

3.2. Tensile results

Tensile results for the three samples in each aging temperature of treatment and as received are presented in Figure 4. Figure 5 shows ultimate tensile strength results with their respective standard deviations.

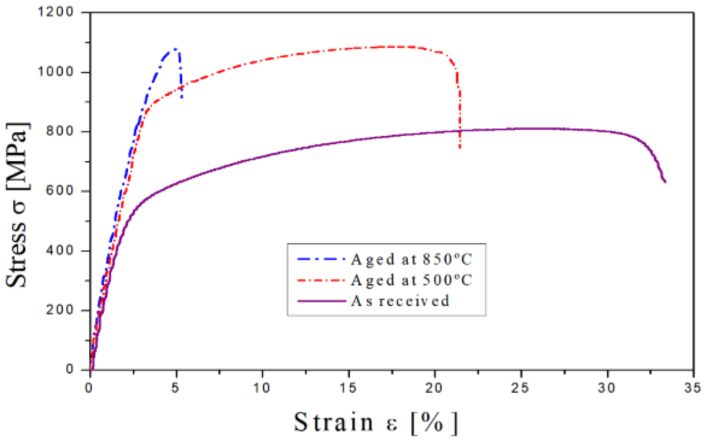

Figure 4: Tensile stress–strain curves for duplex stainless steel UNS S31803.

The aging treatment increased the ultimate tensile strength although it decreased the ductility (Figure 4). This reduction was even more significant for treatment at 850°C, which was in the range of 5%. This is one of the parameters for the loss of ductility.

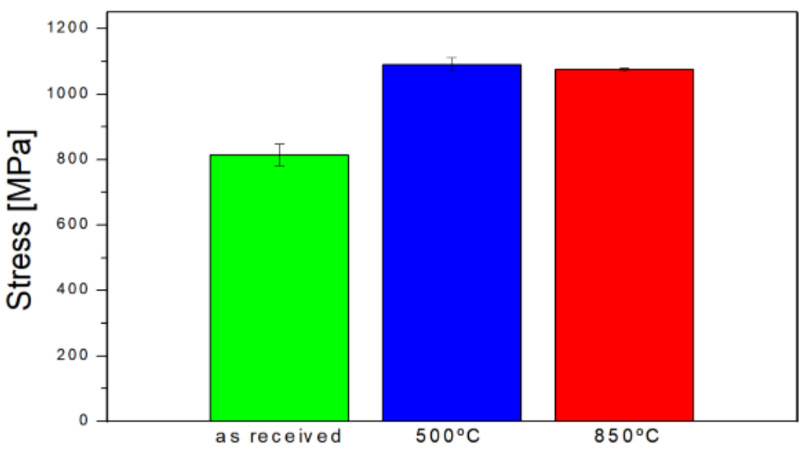

Figure 5: Ultimate tensile strength for each treatment and as received.

The analysis of the results presented by the graph of stress-strain (Figure 4) and by the bar graph (Figure 5) showed a pronounced increase in tensile strength values after the isothermal treatments. Similar results were obtained by SHAMANTH and RAVISHANKAR [19] in their study on the reversal of α' phase precipitation that causes embrittlement. During 100 hours of treatment, the material presented an increase in the resistance limit and a loss in ductility. This increase in mechanical strength was a consequence of the precipitation of the α' phase, that interfered with the movement of the dislocations, causing the loss in ductility [19]. The reduction in deformation occurred mainly in the first 100 hours of aging. From the experimental tensile results obtained, it was concluded that the tension increased from 814 MPa to 1091 MPa, although the ductility, or deformation, decreased with the aging time from 33% to 22%. In an investigation performed by FARGAS *et al.* [20], in which they studied the effects of heat treatments at high temperatures on the mechanical properties of the duplex stainless steel UNS S31803, it was concluded that there was an increase of approximately 30% in the resistance limit of the material, and a reduction in the deformation. They attributed this increase to the high percentage of σ phase precipitation. The results of the current study obtained experimentally for samples treated at 850ºC presented the same behavior as that reported by these researchers.

From the stress-strain curve, it was possible to calculate the material toughness. Toughness is the amount of energy absorbed per unit of volume until fracture, that is, the amount of energy that the material can withstand, without causing its rupture. One way to evaluate the toughness is to consider the total area under the stress-strain curve. Therefore, to determine the toughness modulus (U_T), there is an approximate method for materials with ductile behavior proposed by Equation (1) [21].

$$U_T = \frac{\sigma_y + \sigma_{uts}}{2} \varepsilon_f \qquad (1)$$

(σ_y – yield strength; σ_{uts} – ultimate tensile strength; ε_f – strain).

For materials with brittle behavior, the toughness is calculated according to equation (2).

$$U_T = \frac{2}{3} \sigma_{uts} \, \varepsilon_f \qquad (2)$$

The toughness unit (U_T) is Nmm/mm³. The toughness is a parameter that includes both the mechanical strength as well as the ductility of the material [21]. The results are presented in a bar graph (Figure 6) with respective standard deviations.

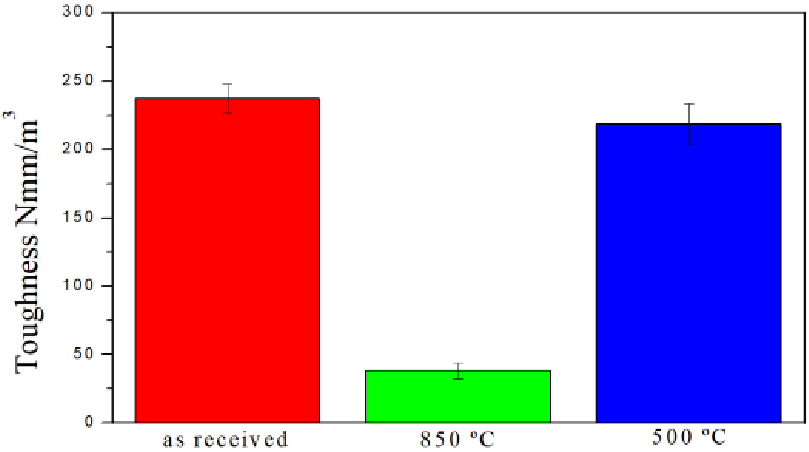

Figure 6: Toughness of the UNS S31803 stainless steel duplex.

Figure 6 presents the toughness mean value for both aging temperatures, as well as for material as received. The loss in the toughness of the materials with isothermal aging treatments can be observed in this figure. A study by TAVARES *et al.* [1] confirms this reduction in the toughness of the stainless steel UNS S31803 after aging treatment at 800ºC. It was concluded that the increase in treatment time and the increase in the percentage of σ phase precipitation decrease the toughness considerably.

A study by ÖRNEK *et al.* [22] reported that the deformation of samples treated at 475ºC showed a significant decrease after 20 hours, indicating clear signs of embrittlement. The same behavior was observed in this work, according to Figure 6. The spinodal decomposition and precipitation of third phases in the ferrite phase appear to be mainly responsible for the embrittlement of the material. The reason is that ferrite is the matrix phase in the duplex microstructure and, therefore, the main load carrying element [22].

3.3 Microphraphy

The fractures resulting from the tensile tests were analyzed. Figure 7 shows the fracture of the duplex stainless steel UNS S31803 untreated (as received).

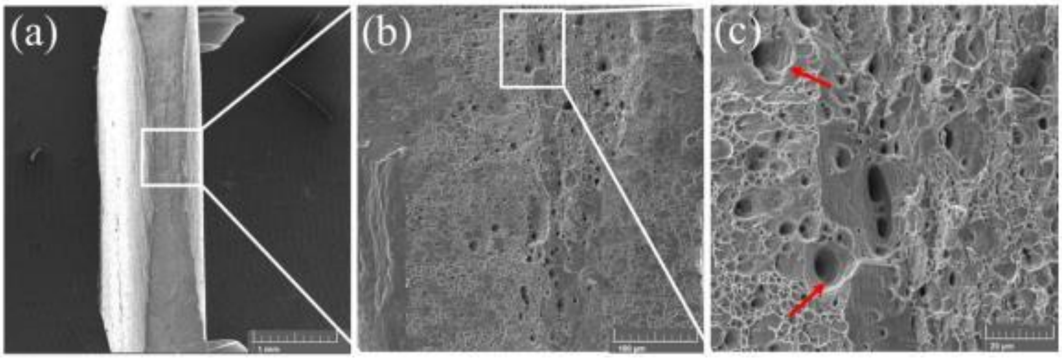

Figure 7: Tensile microfractography of UNS S31803 steel as received. SEM.

The fracture presents the aspect of ductile fracture, with the presence of micro voids. These micro voids are nucleated in regions of localized deformation, known as necking (Figure 7-a). As the deformation, or necking, increases, these micro voids coalesce and eventually form a continuous fracture surface. This type of fracture presents several dimples, indicated by the red arrows (Figure 7-c). Depending on the microstructure and plasticity of the material, the dimples can be deep and conical [23]. Figure 8 presents fractures of the sample aged at 500°C after the tensile test was performed.

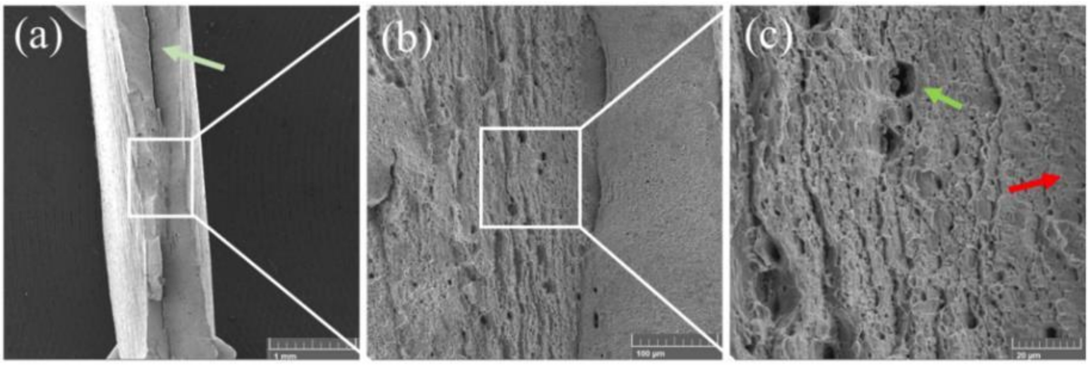

Figure 8: Tensile microfractography of UNS S31803 steel aged at 500ºC. SEM.

Figure 8a shows signs of embrittlement with more extensive cracks. Areas of fracture by cleavage are indicated by the red arrow and the presence of micro voids by the green arrow (Figure 8c). ÖRNEK *et al*. [22] studied the embrittlement caused by the α' phase in 2205 duplex stainless steel. They observed that the fracture shows cracks with deep and fibrous appearance as presented in Figure 8a. The dimples and heterogeneous fractured regions were observed, indicating the loss of strain homogeneity and local brittleness (Figure 8c) [22].

When comparing the fracture with the tensile tests results (Figure 4), it is possible to confirm the decrease in the deformation, with the necking less pronounced (Figure 8a). There was also a decrease in the number of micro voids (Figure 8c), compared to the material as received (Figure 7). The reduction in micro voids and slight necking confirm the embrittlement generated by the presence of the α' phase [22].

Figure 9 presents SEM-generated images of the fractures in the samples aged at 850ºC for 80 minutes.

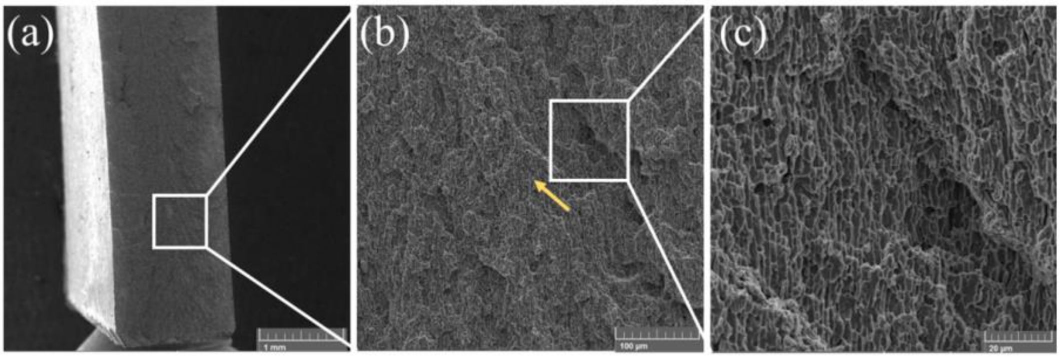

Figure 9: Tensile microfractography of UNS S31803 steel aged at 850ºC. SEM.

Figure 9 presents the fracture of the specimen aged at 850°C. It shows the fragile characteristic. Figure 9c indicates the presence of cleavage. The tensile specimens were broken without plastic deformation or necking zone. The presence of sigma phase led to a fragile behavior. FARGAS *et al.* [20] demonstrated that besides the amount of intermetallic phases, their morphology is of great importance. The σ phase particles contributed to the nucleation of cracks.

4. CONCLUSIONS

- UNS S31803 steel aged at 500ºC and 850ºC presented higher mechanical resistance compared to steel as received (annealed at 1060ºC);
- The toughness of the steel aged (500ºC and 850ºC) was decreased compared to steel as received (annealed at 1060ºC);
- There was reduction of the ferrite fraction with the precipitation of σ phase in the steel aged at 850ºC;
- The steel as received (annealed at 1060ºC) presented fracture completely ductile, with pronounced necking;
- The steel aged at 850°C showed fragile fracture;
- Steel aged at 500ºC presented a region with ductile fracture (presence of dimples) and fragile fracture (presence of cleavage).

5. ACKNOWLEDGEMENTS

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6. REFERENCES

[1] TAVARES, S.S.M., PARDAL, J.M., GUERREIRO, J.L., *et al.* "Magnetic detection of sigma phase in duplex stainless steel UNS S31803", DOI: 10.1016/j.jmmm.2010.02.055*Journal of Magnetism and Magnetic Materials*. v. 322, pp. 29-33. 2010.

[2] CAVALCANTI, D.A., MUTERLLE, P.V., REINKE, G. "Effect of Sigma Phase Precipitated at 850 °C on Corrosion Behaviour of UNS S82441 Duplex Stainless Steel", DOI: 10.1590/S1517-707620190003.0775*Matéria (Rio J.)*. v. 24, n. 3. 2019.

[3] AGUIAR, I.V., ESCOBAR, D.P., SANTOS, D.B., *et al.* "Microstructure characterization of a duplex stainless steel weld by electron backscattering diffraction and orientation imaging microscopy techniques", DOI: 10.1590/S1517-707620150001.0022*Matéria (Rio J.)*. v. 20, n. 1. 2019.

[4] EGHLIMI, A., SHAMANIAN, M., ESKANDARIAN, M., *et al.* "Evaluation of microstructure and texture across the welded interface of super duplex stainless steel and high strength low alloy steel*",* DOI: 10.1016/j.surfcoat.2014.12.060. *Surface and Coatings Technology*. v. 264, pp. 150-162. 2015.

[5] TOTTEN, G.E. Steel heat and treatment handbook. Oregon: Editora Taylor and Francis Group. 2006.

[6] FONSECA, C.S., SILVA, S.**N.**, GOMES, H.G., *et al.* "Evaluation of the sigma phase precipitation susceptibility in duplex stainless steel SAF2205 welding", DOI: 10.1590/S1517-707620190003.0786. *Matéria (Rio J.)*. v. 24, n. 3. 2019.

[7] ALVAREZ-ARMAS, I., DEGALLAIX-MOREUIL, S. Duplex stainless steels. John Wiley & Sons, 2013.
[8] HÄTTESTRAND, M., LARSSON P., CHAI, G., *et al.* "Study of decomposition of ferrite in a duplex stainless steel cold worked and aged at 450–500ºC", DOI: 10.1016/j.msea.2008.09.021. *Materials science and engineering A*. v. 499, pp. 489-492. 2009.
[9] WENG, K.L., CHEN, H.R., YANG, J.R. "The low-temperature aging embrittlement in a 2205 duplex stainless steel", DOI: 10.1016/j.msea.2003.12.051. *Materials science and engineering A*. v. 379, pp. 119-132. 2004.
[10] DEGTYAREVA, V.F., DUBROVINSKY, L., KURNOSOV A. "Structural stability of the sigma phase Fe-Cr under pressure up to 77 GPa", DOI: 10.1088/0953-8984/21/7/075706. *Journal of physics: Condensed Matter*. v. 21, pp. 1- 4. 2009.
[11] HSIEH, C., WU, W. "Overview of intermetallic sigma phase precipitation in stainless steels", DOI:10.5402/2012/732471. *International scholarly research network*. v. 2012, pp. 1-16. 2012.
[12] ASTM E3- 11. Standard guide for preparation of metallographic specimens. 2017.
[13] DE LACERDA J.C., CÂNDIDO, L.C., GODEFROID, L.B. "Effect of volume fraction of phases and precipitates on the mechanical behavior of UNS S31803 duplex stainless steel", DOI: 10.1016/j.ijfatigue.2014.12.015. *International journal of fatigue*. v. 74, pp. 81-87, 2015.
[14] NOMANI, J., PRAMANIK, A., HILDITCH, T., *et al.* "Investigation on the behavior of austenite and ferrite phases at stagnation region in the turning of duplex stainless steel alloys", DOI: 10.1007/s11661-016-3472-0. *Metallurgical and materials transactions A*. v. 47, pp. 3165-3177. 2016.
[15] ASTM E8/E8M – A16a. Standard test methods for tension testing of metallic materials. 2016.
[16] DEL ABRA-ARZOLA, J.L., GARCÍA, M.A.R., CRUZ, V.L.H., *et al.* "Study of the effect of sigma phase precipitation on the sliding wear and corrosion behaviour of duplex stainless steel AISI 2205", DOI: 10.1016/j.wear.2017.12.019. *Wear*. v. 400-401, pp. 43-51. 2018.
[17] POHL, M., STORZ, O., GLOGOWSKI, T. "Effect of intermetallic precipitations on the properties of duplex stainless steel", DOI: 10.1016/j.matchar.2006.03.015. *Materials Characterization*. v. 58, pp. 65-71. 2007.
[18] ASTM E562-11. Determining volume fraction by systematic manual point count. 2011.
[19] SHAMANTH, V., RAVISHANKAR, K.S. "Dissolution of alpha-prime precipitates in thermally embrittled S2205-duplex steels during reversion-heat treatment", DOI: 10.1016/j.rinp.2015.10.004. *Results in physics*. v. 5, pp. 297-303. 2015.
[20] FARGAS, G., ANGLADA, M., MATEO, A. "Effect of the annealing temperature on the mechanical properties, formability and corrosion resistance of hot-rolled duplex stainless steel", DOI: 10.1016/j.jmatprotec.2008.04.026. *Journal of materials processing technology*. v. 209, pp. 1770 -1782. 2009.
[21] GARCIA, A., SPIM, J.Á., SANTOS, C.A. Ensaios dos Materiais, LTC, 2012.
[22] ÖRNEK, C., BURKE, M.G., HASHIMOTO, T., *et al.* "748 K (475ºC) embrittlement of duplex stainless steel: effect on microstructure and fracture behavior", DOI: 10.1007/s11661-016-3944-2. *Metallurgical and materials transactions A*. v. 48, pp. 1653-1665. 2017.
[23] ASM International handbook committee. ASM Handbook: Fractography. USA; 1987.

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