

INFLUENCE OF THE PRESENCE OF THE BRITTLE PHASES AND OF THE TEMPERATURE ON THE FATIGUE CRACK PROPAGATION PROPERTIES OF THE DUPLEX STAINLESS STEEL UNS S31803.

Jorge Teófilo de Barros Lopes, teofilo@ufpa.br

Department of Mechanical Engineer – Federal University of Pará.
Rua Augusto Corrêa, 1, Bairro do Guamá, 66055-260, Belém – Pará – Brazil

Itamar Ferreira, itamar@fem.unicamp.br

Department of Materials Engineer – State University of Campinas
Caixa Postal 6122, CEP. 13083-970, Campinas – São Paulo – Brazil

Tácito Brandão Pinto, tacitobp@brfree.com.br

Department of Materials Engineer – State University of Campinas
Caixa Postal 6122, CEP. 13083-970, Campinas – São Paulo – Brazil

Abstract. Duplex stainless steels have exceptional combination of mechanical properties and good corrosion resistance under critical conditions of work, due to your special microstructure; but, they are less appropriate that the austenitics for applications above 523K and below 223K, due your fragility in these temperatures. This work purposes to study the behavior of the steel UNS S31803, concerning to the fatigue crack propagation rate and fracture mechanisms when submitted the low temperatures and microstructural variations. Fatigue crack propagation tests were conducted at three temperatures (297K, 253K and 223K) and under five material conditions (annealed and four microstructures with increasing fraction of precipitate phases obtained from isothermal heat treatments for 1123K in the times of 5, 15, 30 and 100 minutes). The form of analysis and discussion was the comparison of the $da/dN-\Delta K$ curves obtained in the studied conditions. The study of fracture mechanisms was made by means of SEM in the fracture surface of the specimens. The results showed that, at the temperature of 297K, when the fraction of precipitated phases increase, the fatigue crack propagation rate also increase and the fracture mechanisms tend to acquire brittle characteristics. In the negative temperatures, were observed a few decrease in the propagation rate for the annealed material and with the test carried at the temperature of 223K. The fracture mechanism alveolar with trend for appearance cleavage was observed with increasing the fraction of brittle phases. The aspect of the fracture surfaces did not present considerable modifications with the decrease of the test temperature.

Keywords: Fatigue, duplex stainless steel, heat treatments.

1. INTRODUCTION

The duplex stainless steels are of the materials series alloys with microstructures formed normally by two phases, which are ferrite and austenite, with volumetric fractions approximately the same. These alloys show good combination of mechanical properties and corrosion resistance when they are submitted to critical working conditions, this fact does that these materials an available alternative to conventional austenitic stainless steels. Other advantage of duplex stainless steel in relation to austenitic stainless steels is due it has few nickel, been thus, more materials cheap in relation others materials to several applications (karlsson, 1985), because the decreasing of nickel content favors a reduction on like cycle cost of these steel when it compares to austenitic steels. The good mechanical properties of these steel are due duplex structure: the ferrite promotes the mechanical properties, whereas the austenite assures the material toughness. The accurate amount of each phase on microstructure can be getting by amount of the main alloys compounds (Cr and Ni). The concentration of these elements is adjusted using the Fe-Ni-Cr ternary diagram, to obtain a microstructure of the same amounts of ferrite and austenite. Beyond of these, other elements as N, Mo, Cu, Si, Mg and W can be added into alloy to control characteristics of corrosion resistance.

For these reasons, the duplex stainless steel can be used in several applications, mainly on chemical and petroleum industries, pulp and paper industries, energy generation industry, nuclear reactors, in pollution controller machines and food and biomedical industry.

However, in relation of these steel versatility, proportioned by interesting combination of high mechanical properties and corrosion resistance, associated a low cost, it are less appropriate that the austenitic to application above of 523K, due the phases precipitation that make the ferrite brittle, and below 223K, because the ferrite absorbs few energy on fracture in low temperature.

The ferrite is more sensible to phase transformation that the austenite, in virtue of larger diffusion coefficient associated to crystal structure (CCC) and to high concentration of Cr and Mo dissolved (Dupouiron & Audouard, 1996; Zucato *et al.*, 2002). As outgrowth of ferrite instability, the secondary phases can be formed on temperature range 573K to 1273K, during isothermal annealing or incorrect heat treatment. On this temperature range, the precipitates are been observed in addition to ferrite and austenite, as follow: σ (Sigma), Cr_2N (chromium nitride), γ_2 (secondary austenite), χ (Chi), R, nitride π , ϵ (Cu), M_7C_3 e $M_{23}C_6$ (Josefsson *et al.*, 1991 Zucato *et al.*, 2002). Others precipitate as Laves phases (η) and G and carbides as M_6C too have been related, however in few scale. The σ phase is important, because is a hard

and brittle phase; the formation of it promotes the material toughness loss, as well as it influences the behavior in relation the corrosion; moreover, it shows the kinetics more quick on temperature interval reached during the welding or thermal mechanical process (Padilha, 1994).

This way, the heat treatment, as well as the duplex stainless steel welding, needs some special cares and preview knowledge of precipitation process of brittle phases, since the toughness loss is promoted by presence of these phases, can results in problems when it uses of the part and component treated or welded.

The good weldability is other advantage of actual duplex stainless steel, compared to usual austenitic steel; however, due to what it was exposed previously, the loss of corrosion resistance and the embrittlement of these materials will be able to be disclosed in case that is not followed the adequate procedures of welding (Karlsson et, 1995).

In relation of working temperature, several researches have been developed with objective of analyzing the influence of this variable on mechanical behavior of duplex stainless steels, under the influence of the test temperature and of the presence of precipitated brittle phases. Pinto (2001) developed a research about UNS S31803 duplex stainless, with purpose to study the mechanical behavior and the fracture micromechanisms of this material, by the test temperature influence and brittle phase presence. The mechanical behavior of material was available by mechanical properties analyzing the mechanical strength, ductility, toughness, fracture toughness and hardness; these properties were quantified for extracted parameters of tension test, hardness, impact and fracture toughness, with objective to evaluate what the parameters/tests/properties better detect the effect of temperature and metallurgical embrittlement on material mechanical behavior. Using specimens with increasing volumetric fractions of precipitates, obtained by heat treatment controlled, and testing on temperature of 297K, 253K, 223K e 183K, he concluded that these variables provide a considerable influence on mechanical behavior and micromechanisms of material fracture. However, nothing was done in relation of UNS S31803 duplex stainless steel behavior to fatigue crack propagation, as well as about the fracture micromechanisms related with this process, when the material is submitted on these conditions, that motivated the development of this study.

The mechanical fatigue is, actually, one of the biggest concerns of the professionals who act in the areas of projects, construction and maintenance of machines, equipments and structures. When the technology development found new components and equipment (for example, in the automobile and aeronautical industries), submitted continually to dynamic efforts and vibrations, the fatigue behavior it became 90% of motives of failure of metals components. This happen after a time of work, been usual in structural components of terrestrial vehicle or airship, turbine blade, spring, crane arm, elevation rope and others components or machines, as too, in artificial inserts (biomedical) and materials submitted constantly the repetitive efforts of the tension, compression, bend, vibration and thermal expansion/contraction, beyond others. Depending of fabrication process, some failures happen catastrophic way, causing material damages and victims, depending on the type of equipment and/or the place where this is being used.

As the duplex stainless steel is a great application industry material, know it fatigue behavior is very important, because these alloys are submitted to welding and heat treatment process, as well as can be using at places where the temperature varies, and for this, modify the mechanical properties of this material.

Considering these observations, this paper shows the SAF 2205 duplex stainless steel, UNS S31803, on neuter ambient, in relation the fatigue crack propagation rate and fracture mechanisms, when the material is submitted to different temperatures of test and variation on microstructure by brittle phases precipitated.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

The material used to this research was 2205 duplex stainless steel, UNS S31803 specification, made by Sandvik Steel Industry, as shape of rolled sheet with length 1500mm, width 500 mm and thickness 15 mm. In accordance with the product specification, it was submitted on solubilizing treatment to 1333K, followed of water quenching. The Tab. 1 and Tab. 2, respectively, show the chemical composition and mechanical properties of this material, informed in the product certified, as well as the normalized values by *American Society for Testing and Material* (ASTM A 790 790M/2003) and the found ones in others papers.

The tables show too the values got by composition analyze and mechanical test realized by Pinto (2001) on specimen taken from the same sheet used to this research, and thus, compares the characteristics of product with normalized values and others supplied on for literature.

Table 1. Chemical composition of studied Duplex Stainless Steel (%wt)

| Description | Fe | C | Mn | Si | Cr | Ni | P | S | Mo | Nb | N |
|-------------|------|---------------|--------------|--------------|---------------|-------------|---------------|---------------|-------------|------------|-------------------|
| (1) | bal. | 0.030 máx. | 2.00 máx. | 1.00 máx. | 21.0- 23.0 | 4.5- 6.5 | 0.030 máx. | 0.020 máx. | 2.5- 3.5 | - | 0.08-0.20 |
| (2) | - | 0.0224 | 1.71 | 0.27 | 22.43 | 5.55 | < 0.020 | 0.0007 | 3.36 | < 0.010 | 0.1694- 0.1711 |
| Certified | - | 0.017 | 1.56 | 0.40 | 22.59 | 5.32 | 0.021 | 0.0005 | 2.85 | 0.013 | - |

(1) Washko & Aggen (1990) e ASTM A 790/A 790M (2003); (2) Pinto (2001).

Table 2. Mechanical Properties of studied Duplex Stainless Steel.

| Description | S _{y0,2} (MPa) | S _{y1,0} (MPa) | S _u (MPa) | El _t (%) |
|-----------------------------|-------------------------|-------------------------|----------------------|---------------------|
| (1) | ≥ 450 | - | ≥ 620 | ≥ 25 |
| (2) | ≥ 450 | - | 680-880 | ≥ 25 |
| (3) (Cross direction) | 541 | 607 | 764 | 33.6 |
| Certified (Cross direction) | 614 | - | 772 | 36 |

(1) Washko & Aggen (1990) e ASTM A790 (1995); (2) Nilsson (1992); (3) Pinto (2001).

It was verified by literature, that the Sigma Phases (σ) and Chi (χ) formation on SAF 2205 duplex stainless steel occurs more quickly on temperature range 1123K to 1173K.

It was also verified that the conditions of heat treatment that promote the phase precipitation on this material, in different and increasing amounts to get this research, were fix by material aging on temperature of 1123K, in times that varied between 5 and 100 minutes. With base in those studies, four times of heat treatment were selected to produce microstructures with different precipitated phase's percentage, plus to specified by manufacturer, resulting five microstructural conditions, which are described on Tab. 3, together the symbology used. Therefore, the selection of these microstructural conditions promoted variations of embrittling phases fractions, since zero until larger percentages, because small amount of precipitated phases causes sensible modification on material mechanical behavior, in according with consulted references.

Table 3. Heat treatments to it obtain microstructural condition on material used to this research, and symbology adopted.

| Condition | Temperature | Total Time | Heating | Level |
|-----------|---|-------------|--------------|---------------|
| C1 | 1123K | 25 minutes | ± 20 minutes | ± 5 minutes |
| C2 | 1123K | 40 minutes | ± 20 minutes | ± 20 minutes |
| C3 | 1123K | 55 minutes | ± 20 minutes | ± 35 minutes |
| C4 | 1123K | 120 minutes | ± 20 minutes | ± 100 minutes |
| C0 | Solubilized Material at 1333K, indicated by manufacturer. | | | |

To the treatment, it used a resistance furnace EDG, model FC-1, with controller EDGCON 5P. To the cooling in water, it used a container with 100 liters of water. It controlled the water temperature without varies on the treatments. Due to quenching of specimens, others phases didn't precipitate during the cooling. All treated specimens showed overmetal, and after of heat treatment to the precipitation of embrittling phases, were machined by eletroerosion on final dimension, to promote surfaces without oxides and flat, in according with ASTM E 647 (ASTM, 2000) standard.

The microstructural analysis were realized with the objective to detect and amounting the precipitated phases, it observing the material three planes (L, T e S). To stipulate the planes and directions was observed the ASTM E 1823 (1996) criteria, and this way, standardize the research, to fracture planes and microstructural analyze. The most of analyzes was done on L plane, with T direction on horizontal line and S direction on vertical line, represented by LT and LS respectively.

To microstructural analyze in several conditions, were used the Optical Microscopy Technique (OM) and Scanning Electron Microscopy (SEM). Was used an optical microscope Neophot 32 to take the pictures and Quantimet 500 connected to microscope to amounting the phases. This analysis was done it marking the phases by color tonality difference.

The fracture surface analysis of specimens were done by Scanning Electron Microscopy (SEM), JEOL, JXA 840A model, observing the fracture, and the picture was obtained by detection of secondary electron on NORAN acquisition system, System Six model. Was observed the two specimens fracture to each microstructural condition and test temperature. It did, when was possible, a point analysis by energy dispersive spectrometry (EDS) to determine the precipitate composition, inclusion or surface specify fracture. Were done fatigue crack propagation test in three different temperatures to take a larger test temperature range and getting some modification on behavior material to fatigue crack propagation. The test temperatures were defined in 297K, 253K e 223K. The two first temperatures were defined, room temperature (297K), as standard, and the 223K, because is indicated on references with inferior limit to using the material. The 253K temperature was defined because is approximately the same between the two first, and too because is a usual test temperature, mainly in relation to welding process.

The specimens to fatigue crack propagation test were done in according with ASTM E 647 (ASTM, 2000) standard, whereas was used the compact tension (CT) with straight thru notch. The specimens were cut from sheet by

electroerosion, done the part of notch with final dimension, while other dimensions were submitted to overmetal to surfacing after the heat treatment. Was established the L fracture plane to taken the specimens, which, after cut, were heat treatment to each microstructure condition, being followed, too do by electroerosion, and other part of notch and the docking holes on machine of test. Finally, were milled on width (B) to final dimension, being the surfacing on lateral by grinding. The Fig. 1 shows the final shape of specimens used on this paper.

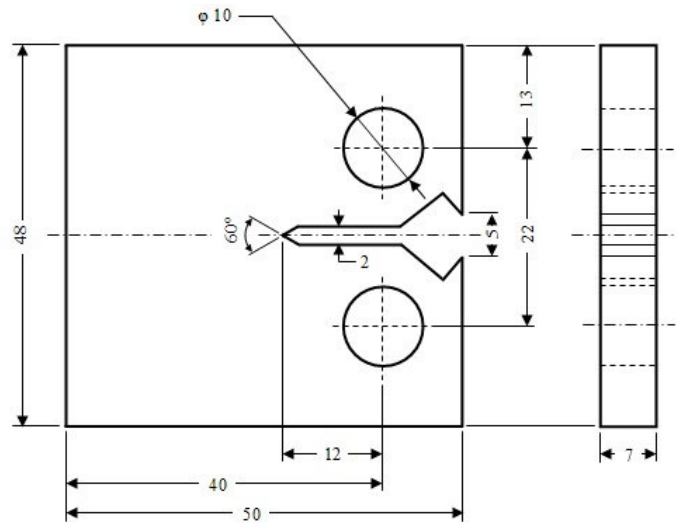


Figure1. Dimensions (millimeters) and fatigue crack geometry of specimens (ASTM, 2000).

The fatigue crack propagation tests were realized on servohydraulic machine MTS, 810 model, with Test Star II controller; MTS load cell, 661.21B-03 model, with capacity of 10.000N; MTS extensometer (“clip-on-gauge”), 632.02C-20 model, with initial length of 5 mm, opening of 3 mm, and working temperature between 173K and 423K.

Firstly, were nucleated the fatigue precrack, it using the test with K_{max} control, 0.1 load ratio and frequency of 20 Hz. After that, were realized the tests, it using the load amplitude controller, and using the same values of load ratio and frequency.

As the obtaining of the fatigue crack propagation curves from a rate 10^{-10} m/cycle, correspondent to ΔK_{th} (ASTM, 2000), would imply in a longer time test, but as this research is compares results of several curves of fatigue crack propagation rate on material, influenced by microstructural conditions and temperature, it was decided to work with ΔK levels into region II of propagation curve, correspondent to Paris’s law, and on larger extension of this region to executable time test, because the larger amount of specimens to testing and high nitrogen volume needs when the realization of tests on negatives temperatures. This way, were realized tests on several microstructural conditions, it varying the final max stress intensity factor ($K_{max\ final}$) to precrack and, indirectly, the max load (P_{max}) to test, using finally, the 15 MPa.m^{1/2} value as minimum limit to precrack $K_{max\ final}$, as establish ASTM E 647 (ASTM, 2000) standard as conditions to validation the tests. To some situations, high values $K_{max\ final}$ of precrack were using because not nucleate with value used. The cracks nucleated were done room temperature.

On the end of the tests, the equipment simultaneously showed in a table the number of cycle to crack growth, the values of K_{min} , K_{max} , P_{min} , P_{max} and the extensometer minimum and maximum opening (COD). From the data obtained, the equipment software controller did the calculus of curves points of fatigue crack propagation, $da/dN-\Delta K$, when of the selecting of fit method, being to this research the *seven points polynomial*; the values obtained were put in order table, whereas plotted the $da/dN-\Delta K$ curve it using the a graphic software.

To tests in low temperatures it used a MTS climatic chamber, 651.12C model with MTS 409.80 controller, that uses liquid nitrogen to cooling and electric resistance to heating, which a temperature range between 144K and 588K. During the tests, the thermalcouple of chamber controller was put on specimens. One thermalcouple, Salvterm 1200 K model, was fixed near to region of specimen center, to compares to temperature into interior of chamber with the specimen temperature.

After the put the specimens on chamber and positioned the extensometer and thermalcouple, the chamber was closed and was need to wait a time to stabilized of temperature shown by the chamber controller window, being this time five minutes to test at 223K and three minutes to test at 253K. After this stabilization, was waiting more five minutes to guarantee that all specimens it reached a wanted temperature, being thus initiated the test, in according with the conditions established by ASTM E 647 (ASTM, 2000) standard. About the variations observed on volumetric fractions of precipitated, the yield stress ($S_{y0,2}$) used on equation that test validity was determined by Pinto (2001). The elasticity module (E) was considered the same to all microstructural conditions and temperatures, about small alterations that can promotes on parameters values, being used to it the value of 196 GPa, as shows the Pinto (2001) studies.

3. RESULTS AND DISCUSSIONS

The Tab. 4 shows the values to volumetric fraction (VF) of intermetallic precipitated (σ and χ) on microstructural conditions analyzed on this paper. The Fig. 2 shows, as example, the microstructural condition aged C4 obtained by electrolytic etching with KOH and observation to optical microscope, where the austenite is disclosed on a white color, the ferrite in multicolor and the precipitated phases Sigma (σ) and Chi (χ) on black color.

Table 4. Media volumetric fraction of precipitated phases on several conditions (%).

| Condition | C0 | C1 | C2 | C3 | C4 |
|------------------------|----|-----|-----|-----|-----|
| FV ($\sigma + \chi$) | 0 | 0.7 | 1.7 | 2.8 | 8.6 |

The Fig. 3(a) shows the region II of the curve of fatigue crack propagation obtained for the UNS S31803 steel on microstructural condition solubilized (C0 condition) at room temperature. The results of fatigue crack propagation test to the five microstructural conditions analyzed in this paper obtained at room temperature and with ΔK initial values approximately the same, are compared on graphic of Fig. 3(b).

The parameters values m and C of Paris-Erdogan relation estimated to curve on C0 condition are same at 2.63 and 1.78×10^{-11} , respectively; this is coherent with studies developed by Iacoviello *et al.* (1999) that used the 22Cr5Ni duplex stainless steel showing others microstructural conditions when it compares to the solubilized conditions.

In all curves shown on Fig. 3(b), it can be observed that to small values of ΔK the fatigue crack propagation rates on the material that show embrittling phases are below of obtained solubilized material. From value determined ΔK to each condition, that decreasing with the precipitated phases FV, the crack growth rates to aged material will be the same, in the cases C1 and C2, or superiors, in the cases of C3 and C4, to condition material C0. It verified too, that the degree of growth of these rates shows a elevation with the increases FV of brittle phases, and that these different increases to larger ΔK ; this obeys the same tendency of results of Marrow and King (1994) and IACOVIELLO *et al.* (1999) obtains to duplex stainless steel.

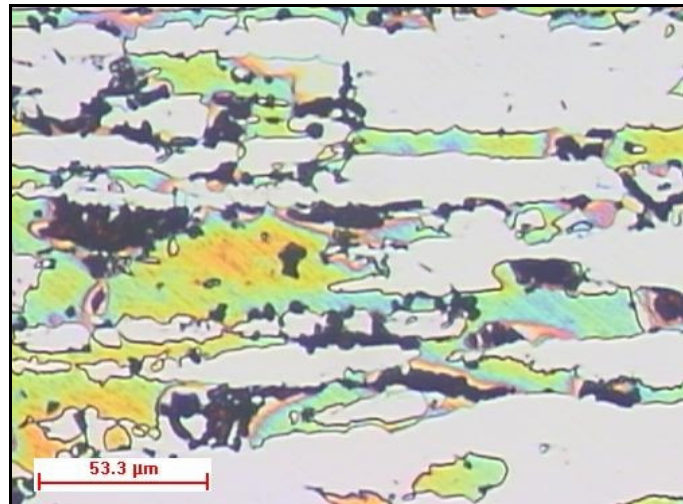


Figure 2. Condition C4 of material with the plane L observation and electrolytic etching, with KOH. MO.

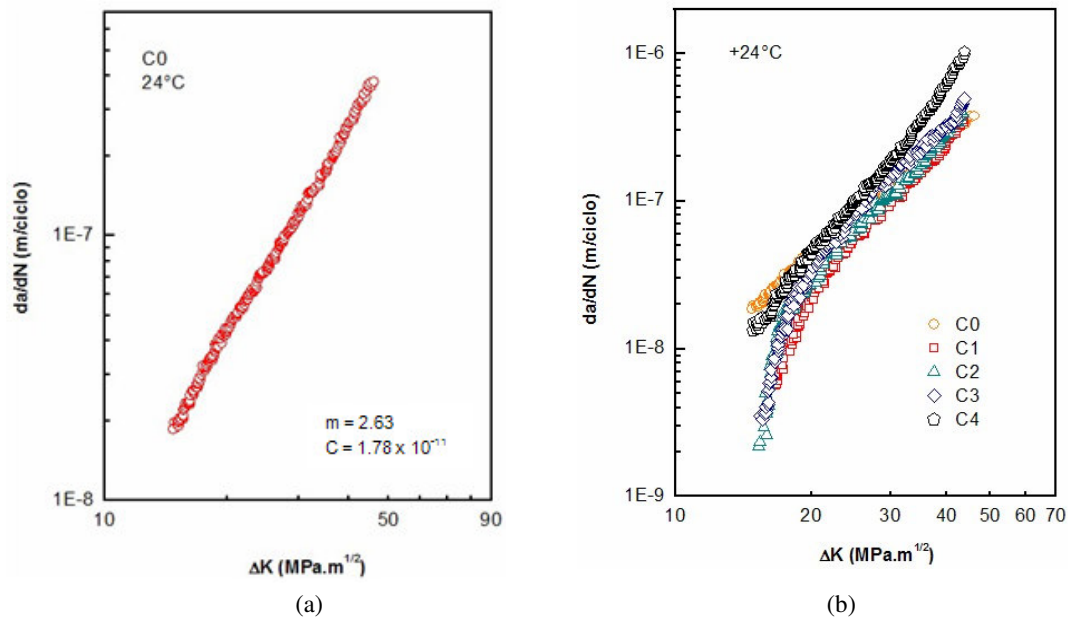


Figure 3. Fatigue crack propagation Curve Region II to UNS S31803 steel on condition C0 and room temperature (a). Fatigue crack propagation obtained to several microstructural conditions and room temperature (b).

The Fig. 3(b) shows, too, that little amount of embrittling phase are sufficient to promote variation in fatigue crack propagation rates on material.

Other observation important of the Fig. 3(b) is the tendency of reduction of ΔK_{max} in the fracture with the elevation of the volumetric fraction of intermetallic phases, which corresponds to reduction of the fracture toughness of the material, and that is according with the specified tests realized by Pinto (2001) to the same steel.

The influence of heat treatment conditions on region II of fatigue crack propagation curve is more evident when it analyzes the m and C parameters of Paris–Erdogan relation to the others treatment conditions studied, which are show on Tab. 5. By variation of theses parameters, it verifying that crack propagation rates were sensible to increases of FV of precipitated phases on material; to condition C4, where it has larger amount of these phases, the parameter value m was significantly greater when it compared with solubilized material. The inclination of curve $da/dN-\Delta K$ (parameter m value) to condition C4 is approximately 4.62, while that of the material on solubilized material is approximately 2.63; this fact shows difference considerable between material behavior to extreme conditions; near to curve validation limits, the crack propagation rate to material on condition C4 is three times greater that condition C0, to values near of ΔK to both conditions.

Table 5. m and C values parameters to several conditions of heat treatment and test realized to room temperature.

| Condition | m | C |
|-----------|------|------------------------|
| C0 | 2.63 | 1.78×10^{-11} |
| C1 | 3.71 | 2.69×10^{-12} |
| C2 | 3.75 | 2.40×10^{-12} |
| C3 | 4.03 | 1.35×10^{-12} |
| C4 | 4.64 | 3.16×10^{-13} |

Obs.: C values in m/cycle.

The variation on fatigue crack propagation behavior, proportioned by increases on brittle phases precipitated volume, mainly Sigma and Chi, as shown on Fig. 3(b), occurs because the microstructural characteristics submitted to material by formation of brittle phases that embrittling all material, decreasing it toughness and, consequently, promotes the high of variation observed on crack propagation rate on these conditions, relatively to process on solubilized condition. When the embrittling phase volumetric fraction increases, most regions of material embrittling and, consequently, larger will be the crack propagation curve inclination. In other way, the presence of brittle phases on grain boundary where usual it form, brittles these regions, permitting that crack propagates between it, doing that constitute, partially, the material fracture surface, that will be more rough in some areas. This way, when greater is phases brittle FV on material, greater will be the

roughness on fracture surface. The consequence of this fact is a increasing of the phenomenon influence of roughness-induced crack closure.

It infers, so, that the influence of brittle phases presence observed on behavior of fatigue crack propagation on SAF2205 duplex stainless steel, is due of competition of two factors. For other way, the roughness-induced crack closure promotes to start of process; however, when the ΔK value increases, the influence of cracks closure decreases, starting to prevail the brittleness of material that increases when phase precipitated volumetric increases. The balance of this commitment is the high level of crack propagation rate shown on Fig. 3(b).

The Fig. 4(a) e the Fig. 4(b) show the test temperature influence on fatigue crack propagation rate to solubilized material and on condition C4, respectively.

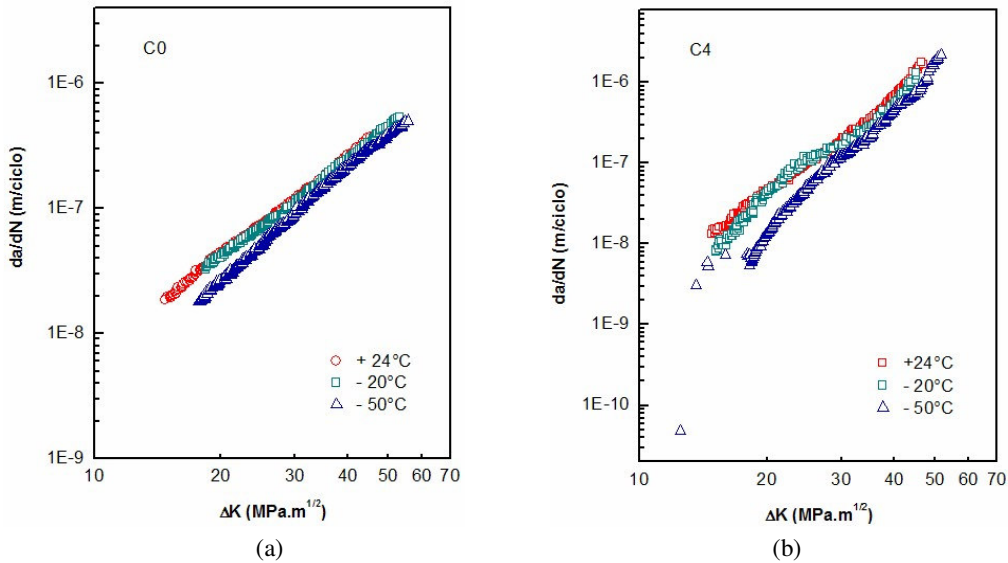


Figure 4. Fatigue crack propagation curves to UNS S31803 steel, in these three temperatures: to the solubilized material (a); on the condition C4 (b).

It has been observed, in all situations, that temperature of 253K practically doesn't modify the crack propagation rate, despite the increase of the volumetric fraction of embrittling phases. On temperatures of 223K, however, it verifies that this influence decreases the crack propagation rate, as too increases it variation with ΔK ; this fact is according, respectively, with others studies of Marrow and King (1994), about the fatigue crack propagation mechanisms on duplex stainless steel Zeron 100, and of El-Shabasy and Lewandovsky (2003), about the effects of several parameters on fatigue crack propagation on pearlitic eutectoid steel. So, the decreasing of temperature promotes the increase of the material resistance to crack propagation; however the variation of crack growth rates too will be high, tending to be the same, in all microstructural conditions, with the obtained to room temperature, when the ΔK increases.

The increases of crack propagation resistance observed on tests realized to temperature of 223K occurs because the growth crack is determined by plastic cyclic deformation on crack tip; as the plastic deformation resistance it increases with the low temperatures, the crack propagation resistance too increases. To other way, on steel, other factor influenced by temperature is related to ductile–brittle transition on behavior of material deformation plastic. This transition, in low temperatures, promotes increasing considerable of crack growth rate; this is characterized by elevation of m parameter on Paris-Erdogan equation. Therefore, the influence of low temperature on fatigue crack propagation behavior of 2205 duplex stainless steel, is due the modifying of two factors: the resistance increase of the plastic deformation on crack tip, that makes difficult the process, and ductile-brittle transition of this material that occurs near to temperature of 223K, that tends to makes high the variation of crack propagation rate.

To material on solubilized condition C0 and tested on temperature of 293K, was observed that the micromechanism more evident on crack propagation is a formation of ductile striations, as much in austenite as in ferrite, and that these striations are more evident to observe when ΔK values is increasing. The identifying of fracture surface of ferrite and austenite phases was done it using dispersive energy spectrometry analysis (EDS), it basing on Cr/Ni ratio (Makhlouf *et al.*, 2003), that is greater to ferrite, as can be observed on Tab. 6. The EDS analysis too shows that the austenite phase corresponds to area that has thin striations, confirming so, the studies of Iacoviello *et al.* (1999) and Makhouf *et al.* (2003). This difference on spacing between striations of two phases can be explain by austenite is low mechanical strength when it compared to ferrite.

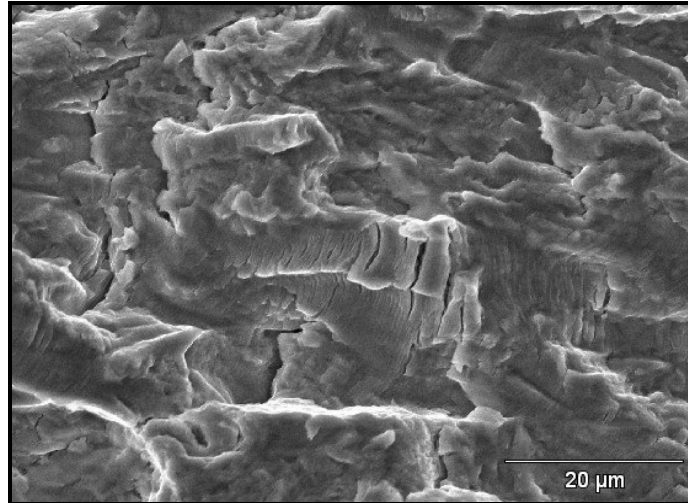


Figure 5. Fractured section of fatigue crack propagation specimens on condition C0, temperature of 297K and position $\Delta K=44.1 \text{ MPa.m}^{1/2}$.

Table 6. Ferrite FV and austenite obtained by AED in several surface points of one specimens on condition C0 and temperature 297K.

| Phase | Elements amounted (% wt) * | | | | | |
|-----------|----------------------------|--------------|-------------|-------------|-------------|-------------|
| | Fe | Cr | Ni | Mn | Si | Mo |
| Ferrite | 66.37 ± 0.53 | 24.70 ± 0.27 | 4.06 ± 0.35 | 1.42 ± 0.16 | 0.55 ± 0.05 | 2.89 ± 0.17 |
| Austenite | 67.88 ± 0.52 | 22.41 ± 0.26 | 6.27 ± 0.32 | 1.46 ± 0.16 | 0.37 ± 0.06 | 1.83 ± 0.16 |

Obs: * Media ± standard deviation.

The fractographic analysis of specimens tested to temperature of 297K and another four microstructural conditions shows a great influence of microstructural modifying, by brittle phases presence, on micromechanisms of fatigue crack propagation, consequently making brittle areas (cleavage), on ferrite grain fracture surface, as can be observed on Fig. 6.

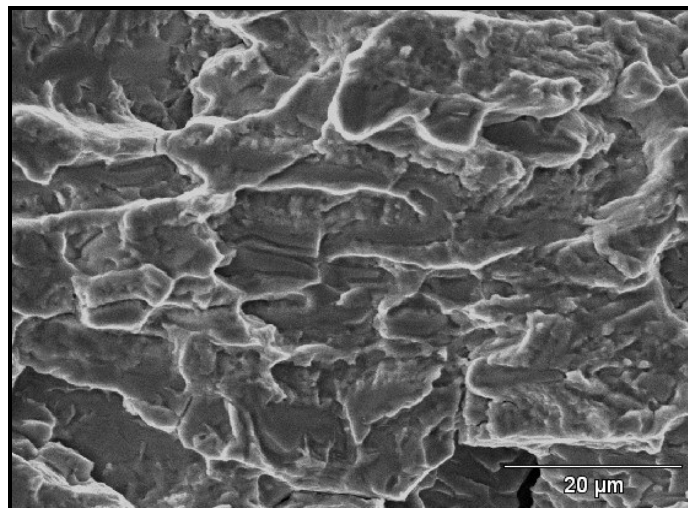


Figure 6. Fractured section of fatigue crack propagation specimens on condition C4, temperature of 297K and position $\Delta K = 48.6 \text{ MPa.m}^{1/2}$.

The fractographic analysis of tested specimens on negative temperatures, it compares to specimens tested to room temperature, shows that, independent of material microstructural condition, the fracture surface aspect don't show considerable modifications. Therefore, the negative temperatures on levels in that were used on this research, although promotes important modifications on fatigue crack propagation behavior on SAF2205 duplex stainless steel, influences little the fracture micromechanisms process.

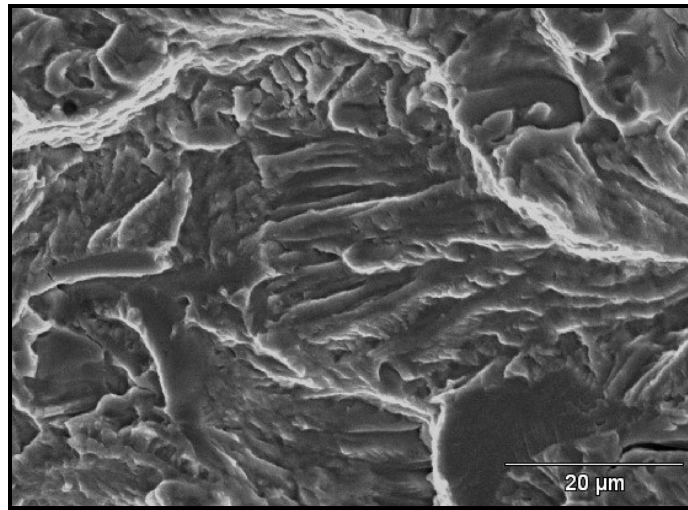


Figure 7. Fractured section of a specimen on condition C4, temperature of 223K and $\Delta K=31.7 \text{ MPa}\cdot\text{m}^{1/2}$.

4. CONCLUSIONS

The heat treatment realized on material in the temperature of 1123K by complete times of 25 to 120 minutes, followed of water cooling, promotes the precipitation of the Sigma and Chi phases, with reduction of ferrite fraction and increasing austenite fraction. The Sigma and Chi volumetric precipitated fractions varied of 0.7% to 8.6%, being the Sigma phase predominant;

The embrittling phases on material, on level of volumetric fractions get on this research, promoted the initial increasing of fatigue crack propagation resistance in all conditions analyzed, relatively to microstructural solubilized condition C0. It opposing to this tendency, the increasing amount of these phases makes reduced the fatigue crack propagation resistance, as well as accelerates the crack propagation; in the extreme condition – treated at 1123K to 100 minutes (C4) – the propagation rates surpassed to material on condition C0 to ΔK low values; so, the initial increasing on fatigue crack propagation resistance on aged material compared to solubilized material can be attributed to roughness-induced crack closure due the intergranular propagation predominant, proportionate by brittle phases precipitated on grain boundary;

Little increasing on volumetric fraction of phases precipitated were sufficient to promotes discrete modifications on fatigue crack propagation rates;

The fracture toughness of the material, measure by means of the parameter K_{max} , decreased with the elevation of FV of precipitated brittle phases.

The influence of test temperature reduction on fatigue crack propagation of 2205 duplex stainless steel occurs more clearly to 223K, due the alteration of two factors: the increasing of plastic deformation resistance on crack tip that increased the resistance to propagation, and the ductile-brittle transition that occurs in this type of steel near to temperature of 223K, that too increased the variation of the crack propagation rates. These factors acted together, and the crack growth rates decreased in relation to room temperature obtained, but tended to make it same by elevation of the ΔK value during the process;

The fracture micromechanism more evident on crack propagation on solubilized condition (C0) was the formation of ductile striations, as much austenite as ferrite. In other extreme condition with relation to precipitated phase fraction's, treated at 1123K to 100 minutes, the aspect of fracture surfaces showed a great influence of microstructural modification that these phases makes, on fracture micromechanisms, having as consequence the formation of brittle areas, probably, associated with the cleavage of ferrite;

Independently of material microstructural condition, the fracture surface appearance of specimens didn't show considerable modification when the temperature reduction of test on levels that were used on this research, although promote important modification on fatigue crack propagation behavior.

5. REFERENCES

- American Society for Testing and Materials, 1995, "A 790/A 790M, Standard Specification for Seamless and Welded Ferritic/Austenitic Stainless Steel Pipe", Philadelphia, 4p.
- _____. *E-647*. Standard test method for measurement of fatigue crack growth rates. Philadelphia, 2000. 42p.
- _____. *E-1823*. Standard terminology relating to fatigue and fracture testing. Philadelphia, 1996 (Reapproved 2002). 21p.
- Dupoiron, F.; Audouard, J. P. Duplex stainless steels: a high mechanical properties stainless steels family. *Scandinavian Journal of metallurgy*, v.25, p.95-102, 1996.
- El-Shabasy, A.B.; Lewandowski, J.L. Effects of load ratio, R, and test temperature on fatigue crack growth of fully pearlitic eutectoid steel (fatigue crack growth of pearlitic steel). *International Journal of Fatigue*, v.26, n.3, p.305-309, 2004.
- Iacoviello, F.; Boniardi, M.; La Vecchia, G.M. Fatigue crack propagation in austeno-ferritic duplex stainless steel 22 Cr 5 Ni. *International Journal of Fatigue*, v.21, p.957-963, 1999.
- Josefsson, B.; Nilsson, J. O.; Wilson, A. Phase transformations in duplex steels and the relation between continuous cooling and isothermal heat treatment. In: *Duplex Stainless Steels*, p.67-78, 1991.
- Karlsson, L.; Ryen, L.; Pak, S. Precipitation of intermetallic phases in 22% Cr duplex stainless weld metals. *Welding Journal*, p.28s-40s, january 1995.
- Makhlouf, K. et al. Corrosion fatigue crack propagation of a duplex stainless steel X6 Cr Ni Mo Cu 25-6 in air and artificial sea water. *International Journal of Fatigue*, v.25, p.167-179, 2003.
- Padilha, A.F.; Guedes, L.C. *Aços inoxidáveis austeníticos: microestruturas e propriedades*. São Paulo: Hemus Editora Ltda., 1994. Cap. 5: Fases formadas durante o envelhecimento ou serviço, p.77-106.
- Pinto, T.B. *Comportamento mecânico de um aço inoxidável duplex tipo 2205 sob influência da temperatura e da precipitação de fases frágeis*. Campinas: Faculdade de Engenharia Mecânica, Universidade Estadual de Campinas, 2001. 180p. Tese (Doutorado).
- Washko, S.D. & Aggen, G., 1990, "Wrought Stainless Steels", Metals handbook. 10.ed., American Society for Metals, Vol.1, Ohio, pp.841-907.
- Zucato, I. et al. Microstructural characterization and the effect of phase transformations on toughness of the UNS S31803 duplex stainless steel aged treated at 850°C. *Materials Research*, v.5, n.3, São Carlos, September 2002.

6. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.