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DETECTION OF FERRITE PHASE TRANSFORMATION BY INDUCED MAGNETIC FIELD ON A DUPLEX STAINLESS STEEL

Edgard M. Silva
Ana L. S. S. Andrade
Francildo O. Silva
Michelline M. R. Silva
Instituto Federla da Paraíba, Av. Primeiro de Maio, 720, João Pessoa, Paraíba, Brasil
edgard@ifpb.edu.br, aluciassouza@gmail.com, francildo_oliveira@hotmail.com , michelline.nery@uol.com.br

Josinaldo P. Leite
João Pereira Leite
Mickael M. R. Silva
Universidade Federal da Paraíba, Cidade Universitário Castelo Branco, João Pessoa, Paraíba, Brasil
josinaldo@ct.ufpb.br, joao.leite100@yahoo.com.br, mickaelmrodrigues@gmail.com

Abstract. *The production of ferrite transformation in duplex stainless steel phase is responsible for the embrittlement of duplex stainless steels. A small amount of sigma phase, for example, is enough to produces unfavorable mechanical and corrosion properties due its brittle nature. In this work, a new affordable approach to detect and follow-up the kinetic of the ferrite phase transformation, based on the measurement of an induced magnetic field generated due to the interaction between an external magnetic field and the microstructure under study is evaluated. To validate the proposed approach, induced magnetic field values are compared to the values of the Charpy impact energy, optical microscopy analysis and X-ray diffraction. The results obtained with the 2205 duplex stainless steel show that there is a relationship between the impact energy, and induced magnetic field, and thus the successfully follow-up of the material embrittlement is possible.*

Keywords: *ferrite phase, induced magnetic field, duplex stainless steel.*

1. INTRODUCTION

Duplex stainless steel have outstanding properties, such as good mechanical, corrosion and fatigue resistances, good toughness and fair impact resistance. These reasons have led to its use in high reliability engineering applications, where failures can cause environmental damage, human and money losses. Due to the above mentioned, duplex stainless steel has been used and studied in areas such as naval, nuclear, oils and gas, and in components such as reactor, pipes, pressures vessels and pumps. However when these steels are submitted to thermal cycling at temperatures above 300 ^{o}C, it appears brittle phases such as chi (χ) and chromium nitrate (Cr_2N), σ and α`. These phases appear due to ferrite decomposition (SILVA 2009, TAVARES 2010, SILVA 2016, FREITAS 2011).

The austenite and ferrite phases form the duplex stainless steels, but when heat treated, only de ferrite phase suffers decomposition. The ferrite phase is ferromagnetic, while the austenite phase and the embrittlement sigma phases are paramagnetic. Two phases are responsible for the embrittlement of the duplex stainless steel, the sigma phase and the alpha line phase called α` that is a ferrite phase rich in chromium. The sigma phase is formed above 550 ^{o}C and the alpha line phase (α') below this temperature. Therefore, the ferrite decomposition leads to changes in the magnetic properties of the material and so magnetic-based inspection methods seem to be adequate for testing these steels (MOHAPATRA 2013, LO 2007, LO 2010). It is also known that the magnetic properties like coercivity and remanence are affected by microstructural modifications, and that the remanence measurement of the magnetic hysteresis loop is able to follow the formation of the sigma phase even when in low volume fraction (MOHAPATRA 2011 and 2013).

The presence of paramagnetic phases in heat treated duplex stainless steel changes the material magnetic permeability and therefore also the magnetic susceptibility. Hence, magnetic susceptibility based tests have been applied to material samples with different amounts of these phases, and decreased susceptibility has been found when the thermal aging times are high due to the formation of the sigma phase. These tests have been used not only to detect the sigma phase but also to identify the alpha line phase (α') and the same trend has been observed (LO 2007, 2010). Additionally, the magnetic permeability assessed based on the interaction between a DC magnetic field and the microstructure was used in (SILVA 2016) to follow the ferrite decomposition into alpha line phase (α') in a duplex stainless steel. The authors showed that although alpha line phase had nanostructure, the presence of this phase could be successfully detected.

In this work, it is proposed a new test for determining the ferrite phase. It is based on the determination of an induced magnetic field which appears due to interaction between an external field and the material. The magnetic filed is generated by direct current and in a region of reversibility of magnetic domains..

2. EXPERIMENTAL PROCEDURE

Samples of a SAF 2205 duplex stainless steel were thermally aged in an electric resistance furnace at 800 ºC for times of 0.25, 1 and 2 hours, and at 900 ºC for 2 hours in order to obtain different amounts of ferrite phase that were quantified through optical microscopy (FX 35XD NIKON Optic Microscopy, Germany). In these temperatures the ferrite phase decomposes to form the sigma one. The difference between the amount of ferrite in the as received material and the sigma phase of the treated samples correspond to percentage of ferrite in each condition. The samples were prepared by mechanical polishing and electrolytic etch in a 10% KOH solution, being the voltage and time applied equal to 3 V and 15 s, respectively. The electrolytic etching with 10 % KOH solution reveals mainly sigma phase. The amount (% volumetric fraction) of sigma phase presented in each sample was determined using a computational tool, which is based on techniques of image processing using the threshold filter. Forty images were acquired from each material sample and the volume fraction was determined adopting a confidence interval of 95%. All samples had dimension equal to 10x10x5 mm^3. The microstructure of the as received condition was attacked by the Behara solution. The Charpy impact test was performed on the samples subjected to the same thermal treatments, and the microstructures of the fracture surfaces were also analyzed trough scanning electron microscopy (SUPERSCAN SSX-550 SEM, Shimadzu Corporation, Japan). To study the formation of sigma phase, X-ray diffraction test was performed using an X-ray Diffractometer from Shimadzu Corporation (Japan), model XRD-6000 vertical type, with Cu κ-α radiation. The scanning angle adopted varied from 0 to 120^o with steps of 0.02º.

For the application of the induced magnetic fields and acquisition of their values, the experimental setup shown in Figure 1 was used. In this setup, a solenoid is responsible for generating the external magnetic fields. The generated magnetic flux density is determined by a Hall Effect sensor (SS495A model, Honeywell S&C, USA.). An external magnetic field of 211.5 A/m was applied in the experiments. In order to assure statistical significance, fifty measurements of five hundred signals each were acquired from each material sample under study.

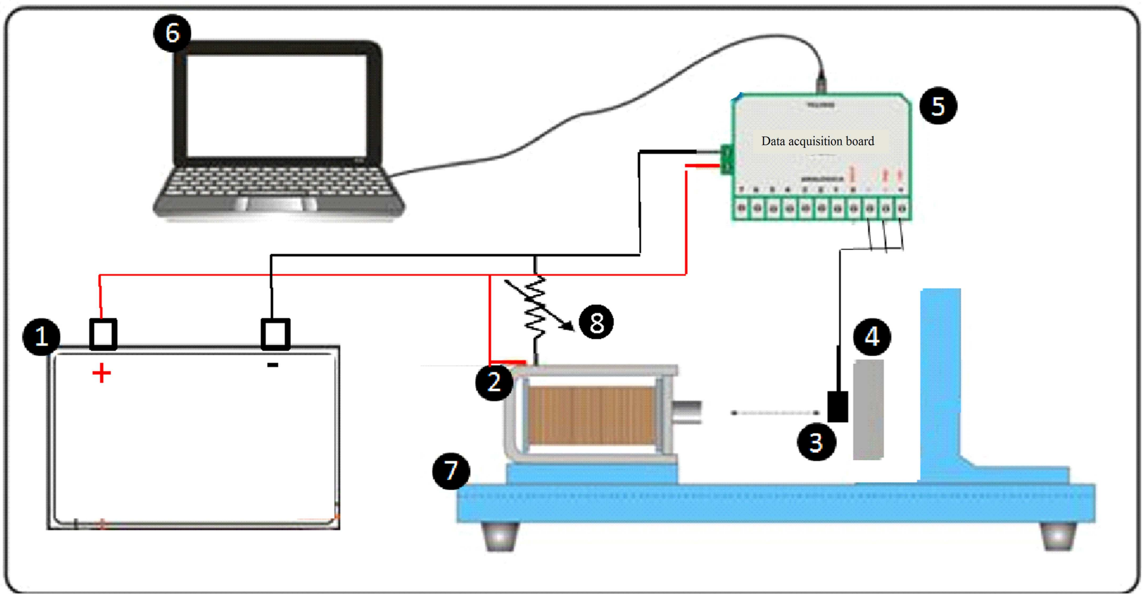
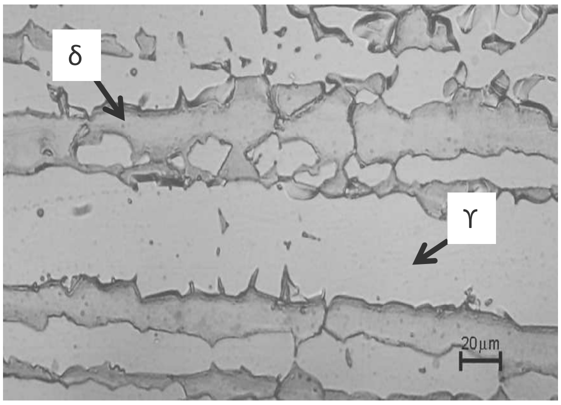


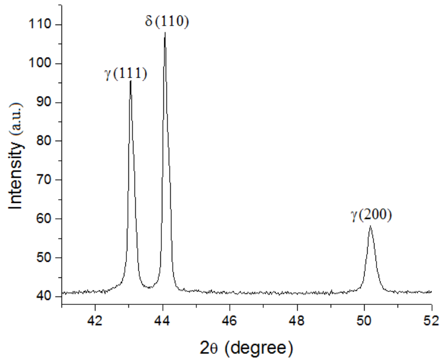
Figure 1. Setup experimental: (1) Power supply, (2) Solenoid, (3) Hall sensor, (4) Sample, (5) Data acquisition board, (6) Computer, (7) Bench test and (8) Potentiometer.

3. RESULTS AND DISCUSSION

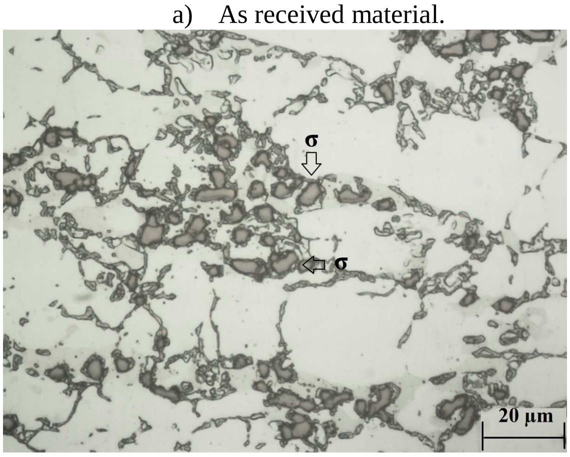
Figure 2 shows the microstructure of the as received material and at 900^{o}C for 2 hours, as well as, their X-ray diffractions. It can be note from Figures 2a and 2b the presence of ferrite (δ) and austenite phase (γ). The ferrite phase decomposes to form the sigma when the duplex stainless steel is heated above 600 ^{o}C (TAVARES 2010). The microstructure of sample aged at 900^{o}C (Figure 2c) reveal the presence of the sigma phase that is the production of the ferrite decomposition. The specimen was attached with KOH reagent which revels sigma phase. The amount of ferrite was obtained by the difference of ferrite of the as received material and the values of sigma. The X-ray diffraction of Figure 2c confirm of the discussed phases.



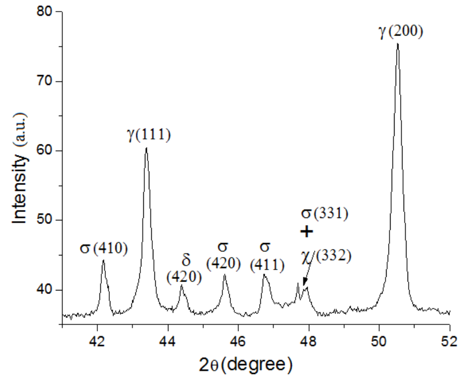
a) As received material.



b) X-ray of the as received material.



c) Sample aged at 900^{o}C for 2 hours.



d) X-ray diffraction of the sample aged at 900^{o}C for 2 hours.

Figure 2. Microstructure of the as received material (Figure 2a) and at 900^{o}C for 2 hours (Figure 2c), as well as, their X-ray diffractions. Figure 2b X-ray of diffraction of the as received material and Figure 2d of sample aged at 900^{o} for 2 hours.

Figure 3 shows the varying induced magnetic field and the Charpy energy as a function of the amount of ferrite phase. It can be seen a clear relationship between the measurements, indicating that this technique is able to identify the embrittlement of the steel by comparing the induced magnetic field and the amount of ferrite. The decomposition of the ferrite phase in duplex stainless steels at 800 ^{o}C causes the formation of the paramagnetic sigma phase. Since ferrite is ferromagnetic, then if the sample has less ferrite the magnetic permeability of the material drops (SILVA 2016). Small contents of the sigma phase promote considerable decrease of toughness; for example, 3% of content causes a reduction of 78% in the absorption of the impact energy. The correlation between this phase and the impact energy has been reported (FARGAS 2009, MARTINS 20080. As already stated, the sigma phase presents high hardness (around 900 HV) (MOURA 2008, ZOU 2010). The reduction of ferrite phase from 47% to 44% is enough to produces a reduction of 40% in the absorbed impact energy leading to the embrittlement of the material. This is due to the formation of 3 % of sigma phase. The technic discussed can be a useful tool to monitor structures in service, since it can detect the presence of the sigma phase even when in low amounts. Other techniques like the ones based on eddy currents and ultrasound are not so precise for short aging times of around 0.25 h at temperatures of 800 and 900 ^{o}C (NORMANDO 2010,[8, 26].

Based on the experimental findings, the measurement of induced magnetic fields can be used to detect, in service, material embrittlement due to the sigma phase. For example, welded joints can be tested by performing analysis before the welding, or in regions distance from the welded regions, and after the welding; and, if the reduction in the induced magnetic field corresponds to a content of 4 % of sigma phase, then it should be interpreted as this result is a strong indication that the structure under analysis needs to be repaired.

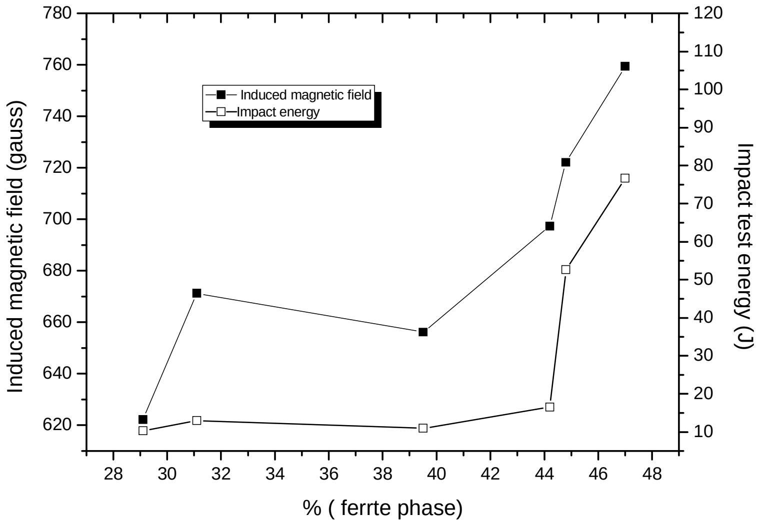


Figure 3.Induced magnetic field and the Charpy energy as a function of the amount of ferrite phase.

4. CONCLUSIONS

The present work introduced a new technique for following the formation fo ferrite phase in a duplex stainless steel, reaching the following conclusions:

The ferromagnetic phase is decomposed in sigma paramagmentic phase in duplex stainless steels. This phenomenon lower the magnetic permeability fo the material. The measuments of the induced magnetic field are affected by this phase transformation. This technique is albe to detect the quantity of ferrite phase as well as monitoring the embrittlenet of the material.

5. ACKNOWLEDGEMENTS

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