Sigma phase detection in duplex stainless steel by induced magnetic field

E M Silva¹, W M L Fialho¹, H F B M Silva², J P Leite², J P Leite³ and A V G Oliveira⁴

¹Instituto Federal de Educação Ciência e Tecnologia da Paraíba, Av. Primeiro de Maio, 720 - Jaguariaíva, João Pessoa - PB, 58015-435, Brazil. Email: edgard@ifpb.edu.br, wmlfialho@gmail.com
²Universidade Federal da Paraíba Jardim Universitário, s/n - Castelo Branco, João Pessoa - PB, 58051-900, Brazil. Email: josinaldo@ct.ufpb.edu.br, hicarofelipe@gmail.com
³Universidade Federal de Campina Grande, R. Aprígio Veloso, 882 - 58429-900, Brazil. Email: joao.leite100@yahoo.com.br;
⁴Instituto Federal de Educação Ciência e Tecnologia do Ceará, R. José Quintino, s/n - Prado, Cedro - CE, 63400-000. Email: venturagol14@gmail.com

Abstract. Duplex stainless steels when subjected to temperatures above 600 °C have its tenacity decreased by the formation of sigma phase. This phase has high hardness and is rich in chromium and reduces the matrix of this element. In this study, field line density measurements, obtained in the reversibility region of magnetic domains, and application of artificial neural networks are used to monitor the formation of this undesirable phase. Samples of a stainless steel SAF 2205 were subjected to aging at temperatures of 800 °C and 900 °C, in order to obtain different amounts of sigma phase. The amount of this phase was obtained by image processing and the density of field lines through a Hall Effect sensor. Charpy impact tests were performed. The field lines densities were used for training of an artificial neural network and correlated with the presence of sigma phase and embrittlement of the material. The results showed that the method was able to correlate the parameters studied with the presence of the sigma phase and toughness of the material studied in both temperatures.

1. Introduction

Duplex stainless steels possess desirable properties of both the face-centered cubic (austenitic) and body-centered cubic (ferritic) phases within their microstructures. The ferrite and austenite phases are present in roughly equal volume fractions. They have excellent strength and toughness, improved corrosion resistance (especially to localized corrosion) and exceptional resistance to halide stress corrosion cracking. However, when these steels are heated above 600 °C there is the formation of non-magnetic embrittlement σ phase. This has hardness around 900 Hv and is rich in chromium. An amount of 3% of this is able to compromise the material toughness [1-7].

The most important metallurgical process in duplex stainless steels is the eutectoid decomposition of the ferrite (δ) to sigma (σ) phase and secondary austenite (γ) through the reaction (δ - σ + γ 2) which happens due to thermal effects (Tavares, 2010).[3].
The ferrite phase is ferromagnetic in contrast to the paramagnetic austenite and sigma phases. Therefore, the magnetic methods seem to be adequate for testing duplex stainless steels, since they are sensitive to the amount and structure of the ferromagnetic ferrite phase. The intensive use of ferromagnetic materials within the engineering applications makes the loss of magnetic flux to be one of main techniques widely used to determine the presence of discontinuities [8, 9]. The disturbance of the magnetic flux near the discontinuities enables the contactless sensing of the vertical component of the magnetic field. Hall sensors have been used for detecting size and position of cracks in materials. These sensors are also sensitive detection of surface flaws in metallic materials, especially under ac excitation (Bi, 1998) [10].

Cavalcante, et al., 2008 [11], used Hall voltage measurement for training an artificial neural network, in order to make microstructures identification. The two techniques combined proved to be promising. The results were based on application of an external field, no saturation of the samples and therefore no measurement of the remaining field.

Some previous works have also proved the applicability of magnetic measurements on the detection of spinodal decomposition of the ferritic phase and the presence of σ in duplex stainless steels [12, 13]. The magnetic susceptibility was found to decrease during prolonged thermal aging due to precipitation of both phases. The non-magnetic σ phase reduce the permeability of the materials and so the magnetic susceptibility.

In this study, field line density measurements, obtained in the reversibility region of magnetic domains and application of artificial neural networks are used to monitor the formation of this undesirable σ phase. A duplex stainless steel 2205 with different amount of σ phase were used.

2. Experimental procedures
Samples of a SAF 2205 steel were subjected to treatments at temperatures of 800 °C and 900 °C for times of 0.25, 1 and 2 hours. Its surfaces were attacked using the Behara and 10% KOH reagents and analyzed through an optical microscopy (NIKON FX 35XD OM). The amount of sigma phase was determined by analysis of surfaces etched with the KOH reagent subjected to image processing by segmenting images and quantification by an image processing program developed in the laboratory. Forty images were captured and the volume fraction was determined with a 5% confidence interval. The microstructures were also analyzed by scanning electron microscopy (SEM) for microstructural characterization. Charpy impact test was performed on the samples subjected to the same treatments. A specimen of the as received material was also studied.

The measurements of induced fields in the material due to the interaction between an external field and the one were carried out as the configuration of the Fig. 1.

![Figure 1. Experimental equipment of test: (1) Power supply; (2) Solenoid; (3) Hall sensor; (4) Sample; (5) Data acquisition board; (6) Computer; (7) Bench test; (8) Potentiometer.](image-url)
It has a solenoid which is responsible to generate the external magnetic field. The magnetic flux density is determined by a Hall Effect sensor (SS495A model).

The neural network applied was a feed forward backpropagation type with feedback from output to input, with tansig activation function, supervised learning by error correction, standard mode, with the entrance with two neurons formed by induced fields and voltage values applied to the solenoid. Twenty neurons were used in the intermediate layers and an output with the absorbed energy values. The objective was to determine the impact energy of the samples analyzed from the applied voltages and field induced in the working region of the sensor.

3. Results and discussions
Optical and electron micrographs of the SAF 2205 steel for treatment condition of 800 °C and 2 hours are shown in Fig. 2 and 3. Figure 2 shows an image of the material surface attacked with KOH and with Behara in figure 3.

![Figure 2. Micrograph of SAF 2205 steel treated at 800 °C for 2 hours obtained by MO (Attack: KOH).](image)

The precipitation of σ phase and the eutectoid reaction δ > σ + γ' can be noted in both figures. Tavares, et al., 2010[3], studying the same steel found that σ phase can precipitate in the (δ / γ) grain boundary and also from the ferrite δ. The transformation in the interfaces (δ / δ) and (δ / γ) were observed in Fig. 3. Chen [1] and Tavares [3] observed that this phase precipitates is the only one revealed by electrolytic corrosion with KOH. In the regions of discontinuous precipitation the σ and γ' phases are formed side by side and as the σ grain boundary is attacked the γ' seems to revealed.

![Figure 3. Micrograph of SAF 2205 steel treated at 800 °C for 2 hours obtained by SEM (Attack: Behara).](image)

By analyzing treated at 900 °C for 2 hours in figure 4 it appears the same precipitation but in larger quantity due to the higher kinetic.
The embrittlement of duplex stainless steel in the temperature range from 600 °C to 1000 °C is due to the presence of sigma phase. This has a hardness about 1000 HV and a percentage of 4 % is sufficient to reduce the toughness around 90 % [14]. Figures 5 shows the change of the impact energy, induced magnetic field and the amount of σ phase, for a voltage of 10 V applied on the solenoid. It was plotted in figure 5 the values obtained at 800 °C and 900 °C temperature. From the analysis of Fig. 5 it can be seen that the absorbed energy and induced magnetic field values decreased with increasing of the amount of σ phase. Lo et al (2007)[12, 13] observed similar behavior studying the effect of formation of sigma phase by magnetic susceptibility measurements.

Figure 5 shows that there is a critical value of the induced field to the embrittlement of the material. A value of 690 Gauss was found to a 10 voltage applied in the solenoid. In order to correlate the different voltage values with the absorbed energy, a neural network was constructed, so any solenoid voltage could be used for future monitoring application.

Figure 5 Change in absorbed energy and induced field due to the amount of sigma phase for a 10 V voltage.

Figure 6 shows the simulated results with the applied voltage, showing that there is a good correlation between them. However, some differences in simulated values were observed, but this can be improved by increasing the amount of data acquisition.
4. Conclusions
A methodology formed by the use of induced magnetic field and neural network was applied to follow the σ phase transformation in an SAF 2205 duplex stainless steel, leading to the following conclusions:

The use of magnetic flux densities in the reversible region of magnetic domains is able to detect variations in the duplex stainless steel microstructure.

There is a critic induced field able to define the low impact energy of the material. A value of 690 gauss was obtain is the one to a voltage of 10V.

The magnetic field combined with a neural network can be used to follow the embrittlement of the studied stainless steel.

Acknowledgements
The authors thanks to the financial support of IFPB and CNPq.

References
[1] Chen T H., Weng K L and Yang J R. 2002 The effect of high-temperature exposure on the microstructural stability and toughness property in a 2205 duplex stainless steel Mater. Sci. Eng. A 338, p. 259.
[2] Jiang Z L, Chen X Y, Huang H and Liu X Y 2003 Grain refinement of Cr25Ni5Mo1.5 duplex stainless steel by heat treatment Mater. Sci. Eng. A 363, p. 263.
[3] Tavares S S M, Pardal J M , Guerreiro J L, Gomes A M and Da Silva, M R 2010 Magnetic detection of sigma phase in duplex stainless steel UNS S31803 J. Magn. Magn. Mater. 322, p. 129.
[4] Mohapatra J N, Kamada Y, Murakami T, Echigoya J, Kikuchi H and Kobayashi S 2013 Magnetic hysteresis loop technique as a tool for the evaluation of σ phase embrittlement in Fe–Cr alloys J. Magn. Magn. Mater 327, p. 71–75.
[5] Normando P G, Moura E P , Souza J A, Tavares S S M and Padovese L R 2010 Ultrasound, eddy current and magnetic Barkhausen noise as tools for sigma phase detection on a UNS S31803 duplex stainless steel Mater. Sci. Eng. A 527 p. 2886–2891.
[6] Camerini C, Sacramento R, Areiza M C, Rocha A, Santos R, Rebello J M and Pereira G 2015 Eddy current techniques for super duplex stainless steel characterization J. Magn. Magn.
Mater. 388 p. 96–100.

[7] Nunes T M, De Albuquerque V H C, Papa J P, Silva C C, Normando P G, Moura E P and Tavares J M R S Automatic microstructural characterization and classification using artificial intelligence techniques on ultrasound signals Expert Syst. Appl. 40 p. 3096–3105.

[8] Enokizono M, Tokada T, Tuchida Y and Hachiki N 1999 Finite element analysis of moving magnetic flux type sensor developed for nondestructive testing IEEE. Trans. Magn. 35 p. 1853.

[9] de Macedo Silva E, Leite J P, de França Neto F A, Leite J P, Fialho W M L, de Albuquerque V H C and Tavares J M R S 2016 Evaluation of the Magnetic Permeability for the Microstructural Characterization of a Duplex Stainless Steel J. Test. Eval. 44 p. 20130313.

[10] Bi Y and JILES DC 1998 Dependence of magnetic properties on crack size in steels IEEE, Trans. Magn. 34 p. 2021.

[11] Cavalcante A F, Albuquerque V H C, Silva E M, Santos N C and Silva A A 2008 Um estudo da aplicação de um sensor Hall e uma rede neural artificial para reconhecimento dos diferentes tipos de tratamento térmico pelo qual um aço SAE 1045 foi submetido Iberian Latin American Congresso on Computacion Methods in Engineering – XXIX CILANCE. Maceio, Alagoas, Brazil.

[12] Lo K H, Lai J K L, Shek C H and Li D J 2007 Effects of pre-treatment on the magnetic susceptibility and ageing behaviour of duplex stainless steels Mater. Sci. Eng. A 452 p. 78.

[13] Lo K H, Lai J K L, Shek C H and Li D J, 2007 Magnetic and transformation behaviour of duplex stainless steels under non-isothermal conditions and temperature-fluctuation monitoring Mater. Sci. Eng. A 452 p. 149.

[14] Glosh S K and Mandal S 2008 High temperature ageing behaviour of a duplex stainless steel Materials Characterization 59 p.1776.