INFLUENCE OF HEAT TREATMENT TIME ON MAGNETIC NOISE DETERMINATION FOR SIGMA PHASE DETECTION IN DUPLEX STAINLESS STEEL SAMPLES OF DIFFERENT THICKNESSES

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ABSTRACT
This work is the result of a study on the influence of heat treatment time on duplex stainless steel (AID) samples when subjected to a magnetic interaction generated from the application of sine waves applied to AID samples in natura/without treatment (CR) and heat-treated under specific conditions. The study correlates the analysis of magnetic noises, which were generated in the magnetic reversibility region, to the ability to detect the deleterious sigma phase in the material and predicts the best conditions for wave application, such as frequencies and amplitudes to be considered to identify the presence of the deleterious phase in the material.

Keywords: Duplex Stainless Steel (AID). Sigma phase. Frequency. Sine waves. Magnetic permeability. Magnetic Noise.

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INTRODUCTION

The study of magnetic noises was first perceived in an essay carried out by Heinrich Barkhausen in the mid-twentieth century. In his study, Barkhausen realized that by magnetizing a piece of ferromagnetic material surrounded by a coil with its terminals connected to an acoustic amplification apparatus, this system was able to detect audible sounds through a loudspeaker (TAVARES *et al*., 2019) (NETO, 2021). The noises heard were originated by microscopic impacts that occurred inside the material due to the displacement of the walls of the magnetic domains that constitute the material that generated electrical voltage peaks after a variable magnetic field was applied (DIWAKAR *et al*., 2022).

The concept of magnetic permeability is related to the ease with which a magnetic flux can pass through a material or even the measure of the amount of magnetization that a material can undergo or the ease with which a magnetic field can be induced in that same material. Magnetic permeability is represented by the Greek letter μ. It measures the ability of a material to offer greater or lesser resistance to magnetic induction. Ferromagnetic materials have μr (relative magnetic permeability) values greater than 1, indicating that they facilitate the passage of magnetic flux. On the other hand, diamagnetic and paramagnetic materials have μr values equal to or less than 1, which means that they offer greater resistance to magnetic flux (LIMA, 2021);(NASCIMENTO JUNIOR, 2011); (LEITE, 2014).

Regarding duplex stainless steels (AID), it is known that they are formed from the junction of the ferrite and austenite phases, which emerged in the early twentieth century and only from the 1970s onwards began to be studied and produced on a commercial scale (FRANCIS; BYRNE, 2021). This steel alloy has in its composition amounts of chromium, in approximately equal portions both in the ferritic phase and in the austenitic phase (HÄTTESTRAND et al, 2009). This composition gives this type of steel a miscellany, in relation to some specific properties, such as: the high strength of ferrite associated with the ductility and toughness of austenite, which gives the material a high resistance to corrosion, an improvement in terms of yield strength and improved resistance to stress corrosion, in relation to stainless steels composed only by austenitic phase (XU et al, 2019) (FRANCIS; BYRNE, 2021). Due to its mechanical properties, AID is used in a range of applications, such as: oil and gas and chemical process industries, in the naval and pulp and paper sectors, pollution control, mineral processing, and civil and structural engineering (XU et al, 2019). Despite the numerous advantages, duplex stainless steels have characteristics such as those mentioned above when working with this material considering a temperature range between -50 and 250 °C (HÄTTESTRAND et al, 2009). AID is led to embrittlement when the material is exposed to temperatures above 250 °C to close to 550 °C, where a reaction caused by spinodal decomposition occurs, since the alpha-line phase (α') is precipitated, and this phase is responsible for compromising the mechanical properties of AID, leading to a decrease in the corrosion resistance of this type of material (FONTES et al, 2009). When there is a temperature increase between approximately 600 °C and 1000 °C, precipitation of several secondary phases such as sigma (σ), chi (χ), secondary austenite (γ2), nitrides ($Cr_{2}N$), carbides ($M_{23}C_{6}$) and carbonitrides (FERNANDES et al, 2021).

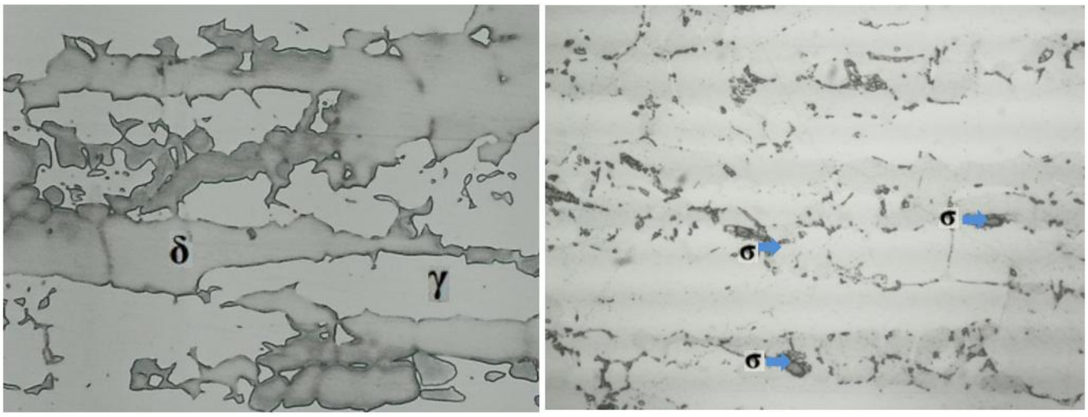
This research aimed to show the characteristics, such as the frequency and amplitude of the signal generated by sine waves and consequently the best working condition for the application of this type of wave that is conducive to the detection of the deleterious sigma phase (σ) inside the AID based on the analysis of magnetic noise in the region of reversibility of the magnetic domains, at first, taking Barkhausen's magnetic noise studies as a reference.

MATERIAL AND METHODS

The study was carried out using samples of a duplex stainless steel (SAF 2205). Specimens with the following dimensions:24 mm in diameter; 2mm thick; 8 mm thick. Regarding the treatment time: Samples in natura or without heat treatment as received (CR); samples thermally aged in a JUNG Model 912 induction furnace at 850ºC for 15 minutes (t15) and for 60 minutes (t60). In order to obtain the deleterious sigma phase in the IDA in a shorter time interval, the sample was heat-treated at 850ºC for a minimum of 15 minutes, and this time, according to the specific literature, Normando et al.,(2010), was sufficient time for the emergence of 4% sigma phase in the AID. Samples cooled in the open air and at local room temperature then sanded, polished and chemically attacked on their surface with 10% potassium hydroxide in such a way as to accentuate through oxidation the region where the sigma phase is located for its microscopic visualization. For the microscopic analysis, a NIKON FX 35XD microscope with a maximum magnification of 400x was used.

Figure 1 shows the micrograph (with 400X magnification) of how the AID SAF 2205 is presented: (a)AID (in natura) / without heat treatment showing the ferrite(δ) and austenite(γ) phases; (b) Heat-treated AID at 850ºC showing the presence of the deleterious phase sigma(σ) (RODRIGUES, 2022).

Figure 1: Micrograph (400X magnification) of the microstructure of a SAF 2205 Duplex Stainless Steel (AID). (a) Micrograph of the sample of an AID in natura (without heat treatment)/CR. (b) Micrograph of the sample of an IDA heat-treated at 850°C.

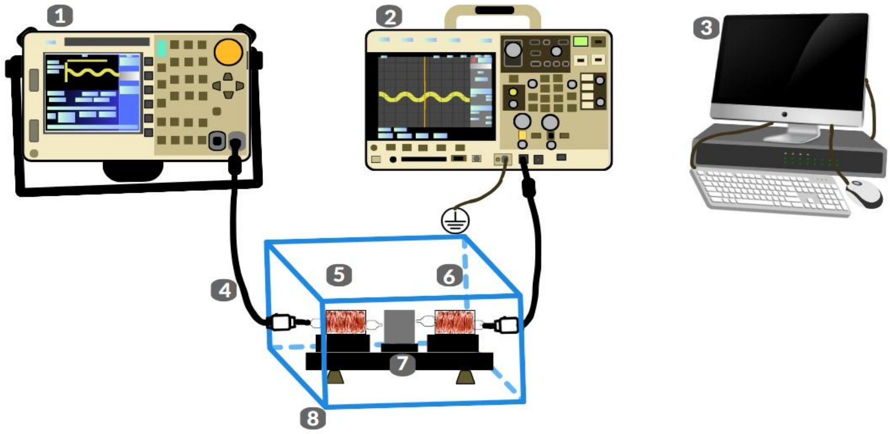


(a) (b)

Image Source: RODRIGUES,2022

Figure 2 represents the test bench for the detection of magnetic noise generated in the magnetic reversibility region. The assembled experimental apparatus does not generate a magnetic field capable of reaching the range of magnetic irreversibility. It is composed of two coils (BG – wave generation coil and BC – wave capture coil). The BG is connected at its input to a signal generator while its output makes contact with the material sample. The capture coil (BC) is in contact at its input with the material sample and at its output it is connected through a cable to an oscilloscope, which in turn was connected to a computer for the acquisition of the rms values of the signal after the wave passes through the sample of the material under study. Sine waves were applied at different frequencies between 0.1 and 50Hz with a signal amplitude of 1V (or $2V_{pp}$) to capture the noises and make observations regarding how effectively this system allows regulating the frequency and amplitude capable of detecting the presence of the sigma phase in the material. The waves specified in the wave generator by regulating the frequency and amplitude voltage of the signal in V_{pp} values were applied both in the 2mm and in the 8mm samples, in natura (without heat treatment, as received - CR) and in those heat-treated at 850ºC for 15 minutes and 60 minutes. In the test, the samples were placed between the coils and isolated from the external environment by a metal housing in the style of a Faraday cage. Then, sine waves with an amplitude of 2V pp were applied at the frequencies of: (0.1/0.2/0.4/0.6/0.8/1/2/3/4/5/10/15/20/25/50)Hz and the rms of the noise signals were captured using the oscilloscope and taking the data through an output peripheral (pen drive) to be statistically treated on the desktop computer through a specific program called Barkhausen Analysis that was developed at the Laboratory of Simulation and Behavior of Materials of the IFPB (GSCMat).

Figure 2: Illustration of the type of bench for measuring magnetic noise.
1. Signal generator. 2. Oscilloscope. 3. Desktop. 4.Armored cable. 5. Generating coil (BG). 6. (BC)Pickup Coil. 7. AID sample.



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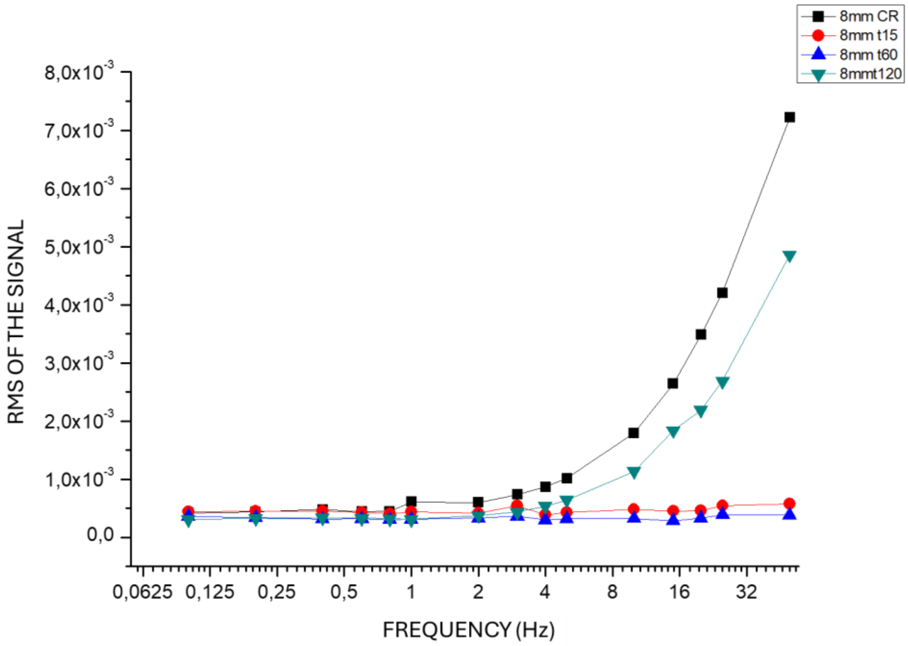
RESULTS

For all the IDA samples under study, after the application of a variable magnetic field, rms of magnetic noise signals were captured that behaved according to the configuration expressed in Figure 3. When analyzing the behaviors of the captured magnetic noise signals, the rms values of the signal remain very close both for the heat-treated samples (t15, t60 and t120) and for the untreated samples (CR) up to the frequency of 5Hz for the two thicknesses (2mm and 8mm) of the samples studied. With the increase in frequency from 5 Hz, it is observed that there is a significant difference between the rms values of the noise in the thermally treated samples, which are lower than the rms values of the signal noise in relation to those obtained for the in natura (CR) samples. The behavior of the captured signal, as can be seen in the graph of Figures 3 and 4, occurs regardless of the thickness of the samples up to the frequency application range of 5 Hz. Above this frequency value, what can be extracted is that for the samples of lower thickness, the rms values of the noise, although they are increasing as a function of the increase in frequency, maintain a certain proximity when considering the samples of lesser thickness. From the application of sine waves, with frequencies above 5Hz, it is observed that there is a discrepancy or distance in the rms values of the noises in relation to the conditions of the material in natura (CR) for the samples with greater thickness.

Figure 3: Graph of the RMS values of noise as a function of frequency. Sample condition: heat treated for 15 minutes(t15), 60 minutes(t60), 120 minutes(t120) at 850ºC. In natura/without heat treatment(CR). Thicknesses: 2mm and 8mm.



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Figures 4 and 5 show that there is a difference between the amplitudes of the rms values of noise from the frequency of 5Hz and that the relative difference between these rms of magnetic noise is more noticeable in the thicker samples when comparing CR and heat-treated samples. For the samples of lower thickness, studied in the same frequency range, the rms of the noises show that they are very close to each other, with a low range of amplitude of value that makes it impossible to distinguish between the noises in the samples with and without heat treatment, not being possible this differentiation and not allowing the system to identify the presence of the deleterious phase in the material at the lower thickness of the sample.

Figure 4: Graph of the RMS values of noise as a function of frequency. Sample condition: heat-treated for 15 minutes(t15) at 850ºC. In natura/without heat treatment(CR). Thicknesses: 2mm and 8mm

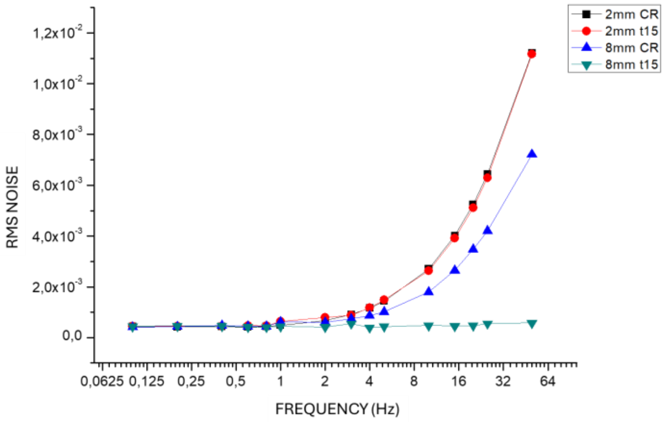

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Figure 5: Graph of the RMS values of noise as a function of frequency. Sample condition: heat treated for 60 minutes (t60) at 850ºC. In natura/without heat treatment (CR). Thicknesses: 2mm and 8mm

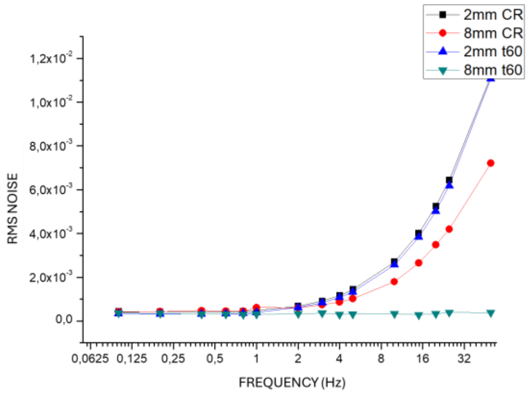

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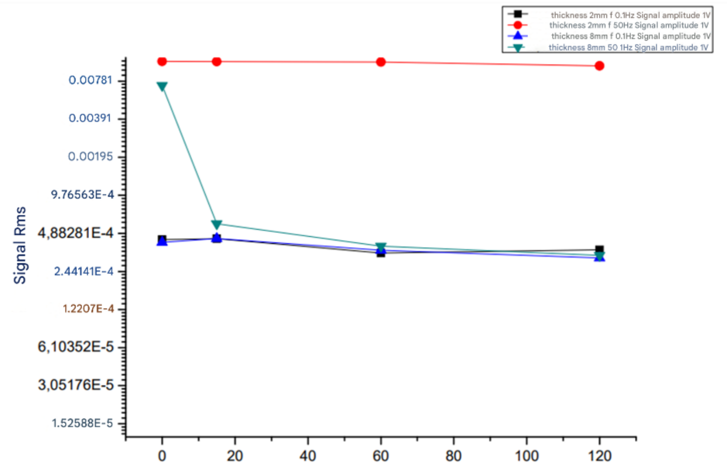
Figure 6 shows how the rms of the noise signal behaves as a function of the heat treatment time (0 min(CR); 15 minutes(t15); 60 minutes(T60); 120 minutes (t120) at the extremes of the frequency range of the study, i.e., considering the lowest and highest value of the applied frequency (0.1Hz and 50Hz) in the samples of 2mm and 8mm thickness. It is observed that as the sample was heat-treated at different treatment times (t15, t60 and t120) and for the different frequencies applied, those that were heat-treated showed practically constant rms values of the noise signal and values close to each other for both the 2mm and the 8mm samples. at the lowest frequency. On the other hand, at the higher frequency applied for the generation of the wave, the rms values of the noise are presented with higher values in both thicknesses.

Figure 6: Graph of the RMS values of noise as a function of the heat treatment time at 850ºC. 2mm samples at frequencies 0.1Hz and 50Hz with 1V amplitude. 8mm samples at frequencies 0.1Hz and 50Hz with 1V amplitude.



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DISCUSSION

Figure 6 shows that for the lowest thickness and at the highest frequency, the rms values of the noise remained higher than the values obtained at the highest frequency for the thicker samples. This difference in the rms values measured in the samples of different thicknesses (2mm and 8mm) and in the higher frequency of the study made it possible to see a significant difference between the rms values of the noise. This difference in rms values leads to the identification that there is an influence of the presence of the deleterious phase on the acquisition of noise signals and that this difference between these rms values of noise is more useful in the study of identifying the presence of the deleterious phase in the material when dealing with the sample with the greatest thickness at the frequency of 50Hz. It is important to note that with the increase in the time of heat treatment until the Limit of the total formation of the sigma phase, the noises decrease regardless of the thickness of the material, although in this study it was impossible to observe this in a more precise way with the samples of the lowest thickness due to the small range of difference between the amplitudes of the RMS values of the noise being confused because they are very close at the lowest frequency. According to Huallpa (2016), corroborates in his work, he observed that the decrease in noise signals due to the kinetics of sigma phase formation over the time of heat treatment, because the ferromagnetic ferrite phase present in the in natura (CR) material tends to decrease as there is an increase in temperature and that the time of subjection to heat treatment can lead to a decrease in the ferromagnetism of the material when the transformation occurs of ferrite in the sigma phase (σ), which in addition to being harmful to the properties of the material, when formed inside the steel increases paramagnetism, reducing ferromagnetism and consequently this phenomenon can be observed through the reduction of the RMS of the captured noises.

CONCLUSION

In this study, different frequencies were applied to generate a sine wave in order to detect the presence of the deleterious phase to the material. The noises generated refer to the collision between the walls of the magnetic domains inside the microstructure of the material and may be able to identify the existence of the sigma phase inside. In view of this study, the heat treatment conditions to which the AID samples were subjected were considered and to what extent these treatment times, as well as the dimensions of the samples, interfered in the rms values of the generated signal. After the study it was observed that at frequencies higher than 5Hz and using an amplitude of 2Vpp it is possible to identify the presence of the sigma phase present inside the sample material and that the difference in the thickness of the sample did not affect the detection of the deleterious phase while the increase in temperature leads the steel to decrease its ferromagnetism becoming more paramagnetic directly impacting the decrease of the rms values of the noises, In other words, the RMS was little impacted by these dimensional factors. Regarding the time of exposure to heat treatment, it was observed that with the increase in the heat treatment time (t15, t60 or t120) in the formation stage of the deleterious sigma phase, the rms values of the noise decreased in the fall and in the thickness of the study sample. And that waves generated at frequencies higher than 5Hz stimulate the generation of noise and that at the frequency of the top of the studied range with the amplitude of 2Vpp, 50Hz, it is shown as the best characterization region for the application of sine wave for the detection of the sigma phase in the AID.

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