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MONITORING THE 475 °C EMBRITTLEMENT BY BARKHAUSEN NOISE MEASUREMENTS IN A DUPLEX STAINLESS STEEL

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Abstract. Duplex stainless steel (DSS) have been largely used as a structural material in various engineering sectors, such as pulp and paper industries and oil and gas industries. The microstructure of DSS is formed by balanced proportions of austenite and ferrite. This combination provides high levels of strength and corrosion resistance. However, this material suffers from the so-called 475 °C embrittlement when exposed to temperatures between 280-500 °C. Previous studies point out that this embrittlement happens through spinodal decomposition, a spontaneous phase transformation phenomenon. To avoid accidents in DSS structures in service, nondestructive testing are needed to detect the embrittled phase and prior intervention on the equipments. Magnetic Barkhausen Noise (MBN) has been used for parameter survey in tests of this type. When ferromagnetic material is exposed to a alternating magnetic field, MBN is emitted due to the interaction between the magnetic domain walls and microscopic anchor points. In this work, electromagnetic testing based on MBN detection is used for monitoring the embrittlement. The data obtained was compared to Charpy impact testing and Rockwell C hardness measurements. The results showed that there is a direct correlation between the two destructive tests and the RMS values obtained by MBN, indicating that the 475 °C embrittlement can be detected using this technique.

Keywords: Duplex stainless steel, 475 °C embrittlement, Nondestructive testing, Magnetic Barkhausen noise.

1. INTRODUCTION

The physical properties of the materials are directly influenced by their microstructures. According to Tavares *et al.* (2010), new phases can be formed when commercial steels are exposed to thermal cycles, due to extreme operation conditions or welding processes. A undesirable phase in the material can cause hardening, compromising the mechanical properties of a structure. Monitoring the formation of these phases is from great interest, as it helps finding the moment for prior maintenance, consequently avoiding accidents.

The microstructure of duplex stainless steels (DSSs) are formed by 50 % of ferrite and 50 % of austenite. The stainless is achieved by high amounts of Cr in both phases (Hättestrand *et al.*, 2009) along with Mo and N additions, which improves especially pitting corrosion resistance. A beneficial effect on mechanical properties is also noted, achieving superior performance when compared to austenitic stainless steels (Xu *et al.*, 2019).

DSSs has a variety of applications, in petrochemical, food, pulp and paper industries, oil and gas sectors, power plants and more (Silva *et al.*, 2014). However, their operations are limited below 250 °C, as it becomes brittle above this temperature (Hättestrand *et al.*, 2009). When DSSs are exposed to thermal cycles at 250-500 °C range, it is known that the ferritic phase decomposes into a iron-rich phase (α) and chromium-rich precipitates (α'). This reaction is known by 475 °C embrittlement, for being faster under such temperature (Tavares *et al.*, 2010).

Phase separation can occur by two mechanisms: (a) spinodal decomposition and (b) nucleation and growth. According to Sahu *et al.* (2009), spinodal decomposition is the formation of two phases of the same crystal lattice, due to the miscibility gap in the alloy system. This phenomenon is most often observed (Silva *et al.*, 2021) as the main cause for the 475 °C embrittlement of DSS alloys, since α and α' are both bcc phases (Hsieh *et al.*, 2016) and the miscibility gap is present at 280-500 °C temperature range of the Fe-Cr diagram (Sahu *et al.*, 2009).

At the initial periods of spinodal decomposition, α' is formed coherent with the matrix. Later the coherency is lost with the growth of this phase (de Albuquerque *et al.*, 2010). Thus, α and α' lowers dislocation mobility, increasing the hardness of the material. Their presence leads to a significant loss in both toughness and corrosion resistance (Xu *et al.*, 2019).

Duplex stainless steels are suitable for aggressive environments. Phase separation becomes a major concern, as it can affect performance and safety of industrial parts. Although nano-scaled α' precipitates can only be detected by atom

probe field ion microscope (Sahu *et al.*, 2009), indirect, nondestructive testing (NDT) can be used as a safer and easier monitoring alternative. Depending on the NDT applied, disassembling is not necessary as it is in microscopy techniques. There are plenty of methods for NDT, such as ultrasound, neutron diffraction, x-ray diffraction, penetrant testing and magnetic Barkhausen noise (MBN) (Franco Grijalba and Padovese, 2018; Lin *et al.*, 2023).

This last one, the Barkhausen Magnetic Noise, is an electromagnetic phenomenon that occurs in ferromagnetic materials when an external magnetic field is applied to the material, which, due to its characteristics, becomes magnetized, causing movements in the domain walls (Qiu *et al.*, 2021). Thus, the kinetics of magnetic domains is related to the formation of this characteristic signal (Fagan *et al.*, 2023). With the use of an inductor positioned near the magnetized piece, it is possible to detect and collect noise generated by this movement (He *et al.*, 2018). This specific signal is traditionally collected in the literature at a bandwidth frequency between approximately 1 and 100 kHz (Stupakov *et al.*, 2020). It is called Barkhausen Magnetic Noise, in honor of Heinrich Barkhausen, a German physicist who made significant contributions to the field by discovering and studying the phenomenon in the early 20th century (Tavares *et al.*, 2019). This noise exhibits variations throughout the material due to numerous conditions, such as grain boundaries, discontinuities, phase changes, impurities, and residual stresses (Limon Leyva *et al.*, 2018). Subsequently, other factors have been identified as influential in the behavior of MBN, which are: carbon content, surface condition, and the material's organizational structures. This enables this technique to be a highly useful tool for detecting internal defects or discontinuities that may compromise the structural integrity of the material (Deng *et al.*, 2018; Neyra Astudillo *et al.*, 2022). Additionally, the use of MBN also provides the possibility to quantify magnetic anisotropy through data collection from the analyzed material (Silva *et al.*, 2016).

In the present work, the spinodal decomposition mechanism in a duplex stainless steel is monitored by analysis of magnetic Barkhausen noise, using a technique in which the receiver coil is positioned on the opposite surface of the emitter coil, in order to analyze a larger volume of material. Sine waves with a frequency of 10 Hz are applied to samples treated at 475 °C with different thermal aging times and the results correlated with measurements of absorbed energy by impact.

2. METHODOLOGY

The investigated steel was a piece of the commercial duplex stainless steel SAF 2205. Seven specimens of 30x20x8 millimeters were extracted by electrical discharge machining. One specimen was separated to be tested as-received, and the other six were exposed to thermal aging at 475 °C, according to the following periods: 1, 4, 8, 12, 39 and 100 hours. The chemical composition of investigated specimens is shown in Tab. 1.

Table 1. Chemical composition of investigated specimens.
 Source: Materials Behavior Simulation Research Group - GSCMat IFPB.

C	Mn	P	S	Si	Cr	Ni	Co
0.055	1.333	0.019	0.002	0.458	21.823	6.085	0.130
Cu	Mo	N	Nb	Al	Sn	Ce	Fe
0.191	3.609	0.180	0.028	0.013	0.012	0.020	66.496

The experimental setup (Fig. 1) is composed by a waveform generator (1), the analyzed sample (3), a pair of coils (2,4), Faraday cage (5), wiring connections (6), an oscilloscope (7) and a computer (8). The wires used are shielded, to alleviate electromagnetic interferences of the environment alongside the Faraday cage. The coils used are identical and are 19.5 mm long, with 6,000 turns of enameled copper and AISI 1020 steel core.

As shown in Fig. 1, a sample is placed in the space between the coils to be magnetized. The waveform generator sends a sine wave with 3 V of amplitude and 10 Hz of frequency to the emitter coil, which transforms it in an alternating magnetic field. The signal pass through the sample, interacting with its magnetic domains. When it reaches the receiver coil, it is reestablished as an alternating electrical signal. The resulting noisy signal is sent and displayed into an oscilloscope. For each sample, 100 measurements from the signal were made, with an acquisition time of 1 second, 10,000 points. The measurements are saved through USB connection and analyzed in a specific computer program which allows to manipulate the measurements. The data were transposed into the frequency domain through Fourier transform and 2000 Hz high-pass filter was applied. The root mean square of the results were calculated after applying the filters and used as the analysis parameter for magnetic Barkhausen noise.

Three specimens for Charpy impact test were made for each treatment condition as well as for the initial condition. The specimen as received had its microstructure characterized by optical microscopy and etched with beraha reagent.

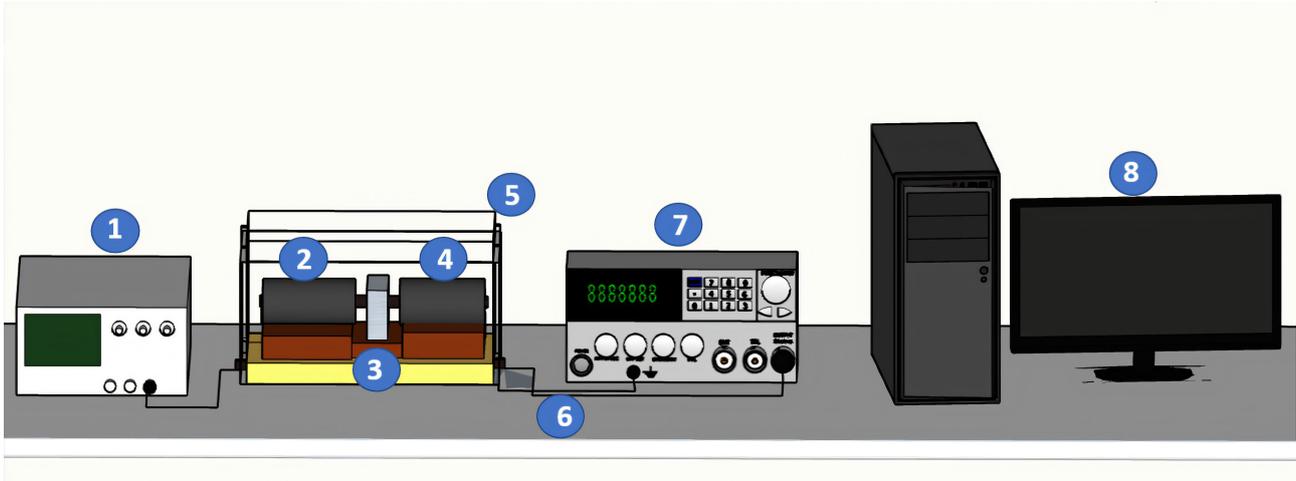


Figure 1. Experimental setup.

3. RESULTS AND DISCUSSION

Figure 2 shows the material as-received. Islands of austenite are observed in a ferrite matrix. The decomposition mechanism is spontaneous and, for SAF 2205 steel, has its highest kinetic temperature at 475 °C. The precipitates formed are nano-scaled and cannot be observed by optical microscopy, only by electron microscopy or by indirect measurements such as ultrasonic measurements, electrical resistivity or magnetic permeability measurements (de Albuquerque *et al.*, 2010).

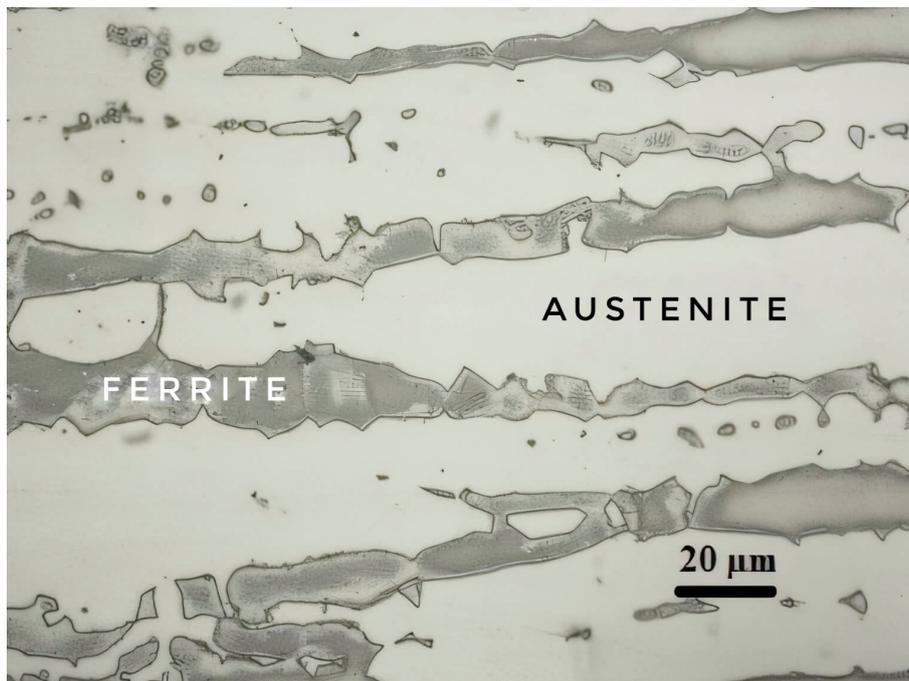


Figure 2. Optical microscopy of the SAF 2205 duplex stainless steel as received.

Figure 3 shows the variation of RMS and absorbed energy by impact measurements for samples treated at 475 °C as function of thermal aging time. It is observed a drop in the values of absorbed energy by impact, from 88 J for the as-received condition to 21 J in samples aged for 100 hours. This 76 % drop is due to the formation of finely dispersed precipitates formed by spinodal decomposition. These hinder the sliding of planes and thus reduces the toughness of the material.

The RMS values increase in samples aged up to 4 h, followed by decrease until the last 100 h aged sample. The regions of increase correspond to decomposition of ferritic phase due to spinodal decomposition. For times up to 4 h the precipitates are in the initial stage of formation and are not effective to block the movement of the magnetic domains. Thus, MBN increases when the magnetic domains detach from the anchor points. However, for samples aged above 12 h,

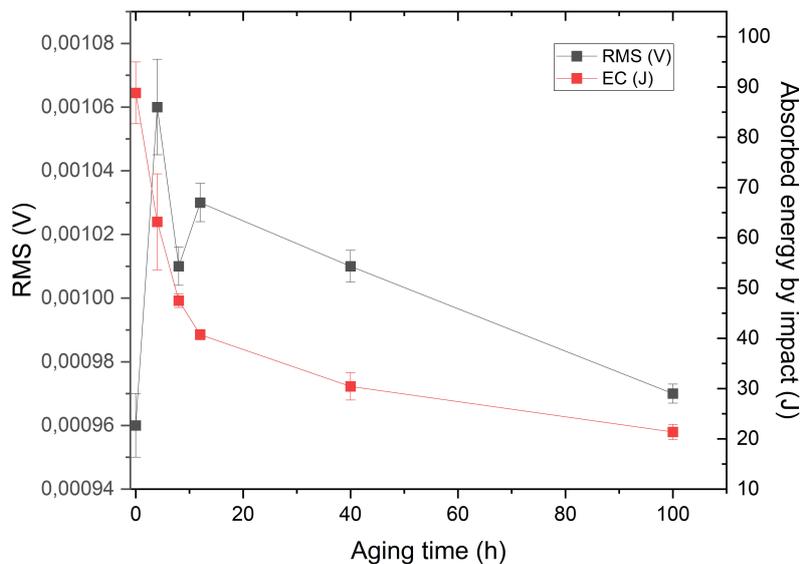


Figure 3. Variations of RMS and absorbed energy by impact measurements as function of thermal aging time at 475 °C.

there is a reduction of the RMS values, when the precipitates start to act as anchor points of the magnetic domains. The RMS values starts to follow the behavior of the measurements of absorbed energy by impact. The period of reduction of the RMS values around 4 hours can be associated with coherence loss between the ferrite and the α' phase formed, which initially is coherent with the matrix. When α' loses this coherence, it starts to own a sharply defined grain contour, leading to the hindering of the magnetic domains walls (de Albuquerque *et al.*, 2010; Silva *et al.*, 2014, 2016).

Figure 4 shows the variation of RMS and Rockwell C hardness as function of thermal aging time for the studied DSS. It is noted that the hardness values have a rapid increase in samples aged up to 12 hours, followed by a stabilization tendency. The formation of the finely dispersed precipitates within the ferritic phase hinders the sliding of the planes and increases the hardness of the material. The behavior of the hardness measurements is inverse of the absorbed energy by impact and indicates that the highest hardening rate occurs in the region where the new phase formed still has coherence with the matrix and tends to stabilize with the consolidation of the formed precipitate.

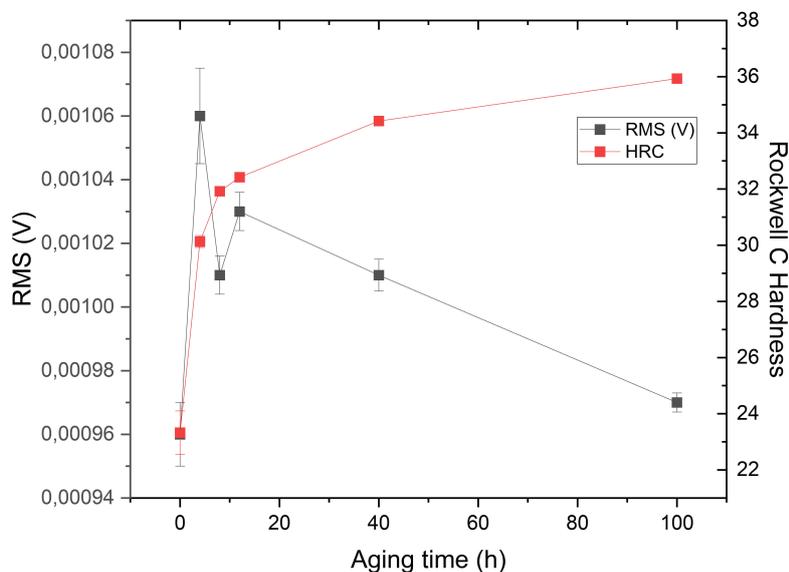


Figure 4. Variations of RMS and Rockwell C hardness as function of thermal aging time at 475 °C.

The results show that RMS measurements are capable of monitoring the precipitates formed by spinodal decomposition and points out that electromagnetic test by MBN is the effective nondestructive alternative for monitoring stainless steels.

4. CONCLUSIONS

Different stages of embrittlement by thermal aging of DSS SAF 2205 were discussed. Through this study, it was found that monitoring the α' phase formed by spinodal decomposition through a nondestructive test based on magnetic Barkhausen noise measurements is possible.

A further study will be conducted in samples aged at 425 °C. At this temperature, the formation of the precipitates are not quite as fast. If MBN measurements are indeed effective at other range of temperature, the same effects in RMS values may be observed, but at lower intensity compared the present results at 475 °C.

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