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Use of Magnetic Barkhausen Noise (MBN) to Follow Up the Formation of Sigma Phase in Saf2205 (UNS S31803) Duplex Stainless Steel

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Duplex stainless steels have a structure normally composed of austenite and ferrite in approximately equal proportions. In order to attain control of its fabrication processes and performance, it is important to understand its microstructural evolution, due to the formation of intermetallic phases such as sigma (σ) and chi (χ), which may cause a severe deterioration of mechanical properties. In the present study, the evolution of sigma phase during heat treatments at temperatures in which intermetallic phases can be formed (800ºC-900°C) was studied using magnetic analyses on a SAF2205 (DIN 1.4462/UNS S31803) steel. A significant reduction of the intensity of Magnetic Barkhausen Noise (MBN) was observed with the increase of heat treatment time, indicating a decrease in the quantity of ferromagnetic phases. For 24-hour-long treatments, the Barkhausen Noise signal is almost completely enclosed by the background noise, indicating the existence of a very small volume fraction of ferrite. If proper calibration samples are to be produced, this technique may be a viable method for non-destructive evaluation of field components working under thermal conditions that may cause the formation of intermetallic phases.

Keywords: *Magnetic Barkhausen noise (MBN); Duplex stainless steels; Sigma phase; Non-destructive testing; Feritscope*

1. Introduction

Duplex Stainless Steels (DSS) usually combines corrosion resistance with interesting mechanical properties. Their microstructure is normally composed of approximately equal fractions of austenite and ferrite. However, depending of the thermal cycles to which the steel is submitted, brittle undesirable phases may occur. Welding and hot forming operations are likely to cause harmful microstructural alterations, such as the formation of sigma (σ) and chi (χ) phases, or chromium nitrides (CrN and Cr_2N). In particular, sigma phase, which is paramagnetic, may be studied using magnetic measurements whenever it forms by a decomposition reaction of the ferromagnetic ferrite, causing a decrease in the amount of ferromagnetic constituents.

Since σ is the intermetallic phase more prominently formed during field applications of duplex stainless steel, its formation has been the object of several studies throughout the years, mostly focused on morphology and formation kinetics[1-8], as well as its effects on mechanical properties[9-11] and corrosion resistance[12-17]. Processes that include high temperature thermal cycling, such as welding, are particularly critical regarding the appearance of σ, due to the temperature range to which the heat affected zone is exposed in a poorly controlled manner. Thus, a method to evaluate the presence of sigma after such operations, even if qualitatively, is of great technological importance, especially if it can be used in field conditions and non-destructively.

Magnetic Barkhausen Noise (MBN) is based on the detection of a signal generated in ferromagnetic materials submitted to an external magnetic field. The signal is originated by the movement of magnetic domain walls, caused by the action of the external oscillating magnetic field. The signal detection occurs by induction of electric currents in a sensor coil.

The domain walls do not immediately follow changes in the magnetic field, as they encounter a resistance due to dissipative barriers. Thus, when a group of domain walls finally moves, it occurs abruptly and irreversibly. The internal energy of the systems rises, causing the emission of sudden peaks or magnetic field pulses. These field variations cause inducted voltage pulses in a sensor coil placed on the surface of the sample. These voltage pulses are referred to as Magnetic Barkhausen Noise, in honor of Heinrich Barkhausen, who discovered this phenomenon in 1919.

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Second phase particles, grain boundaries, dislocations and stress fields are effective barriers for the movement of the domain walls. For this reason, MBN is sensitive to microstructure, plastic and elastic strains in ferromagnetic materials[18-21]. It is also heavily influenced by defects such as porosity, inclusions, dislocations, pinning of punctual imperfections and stresses present[22,23]. Crystallographic texture may also have an influence on the intensity of the MBN signal, as it is widely known that certain orientations are more susceptible to magnetization than others.

Since sigma phase is not ferromagnetic, and formed by consumption of ferrite, its likely presence should be easily detected by magnetic measurements (it should be noticed that, without destructive testing, the decrease in magnetic signal cannot be uniquely attributed to sigma formation, since other non-ferromagnetic phases may form simultaneously, such as chi and nitrides)

The use of non-destructive methods to evaluate the formation of sigma phase has been reported before by several authors. Normando et al.[24] used different non-destructive techniques to evaluate the formation of sigma after heat treatments conducted at 800 and 900°C. The authors reported that the combination of different non-destructive techniques coupled with data processing procedures allowed for the identification of signals coming from samples with different amounts of sigma. Ginsztler et al.[25], compared potentiostatic etching and magnetic methods, and reported that Barkhausen noise measurements are accurate tools to evaluate the amount of ferromagnetic phase in superferritic stainless steels. Dobránszky et al.[26] also showed the efficiency of Barkhausen noise as a method to evaluate the decrease in ferrite fraction in a superduplex stainless steel.

2. Experimental Procedures

2.1 Material

The steel used in the present study is a duplex stainless steel type DIN 1.4462 (UNS S31803). The chemical composition of the steel is presented in table 1. It was provided in rolled, rectangular section bars, with a microstructure composed of austenite fibers aligned along the rolling direction, approximately equiaxial in a transverse section, embedded in a ferrite matrix. The microstructure of the as received material is shown in figure 1. The volume fraction of ferrite was measured by quantitative metallography. The measured values are given in table 2. The sample was submitted to Electron Backscatter Diffraction analysis for evaluation of texture in the as-received condition. The orientation map for the as-received material is shown in figure 2. The ferrite phase has a weak {001} <uvw> texture. No texture was detected in the austenite phase.

Table 1. Chemical composition of the steel used in this study.

C	Si	Mn	P	S	Co	Cr
0.016	0.66	0.62	0.009	0.0016	0.03	22.60
Mo	Ni	V	Cu	Al	B	N
3.06	4.73	0.02	0.07	0.013	0.0031	0.20

2.2 Heat treatments and metallographic characterization

A phase map as a function of temperature was calculated using ThermoCalc® software and the TCFE database, in order to choose the temperatures at which the heat treatments for formation of intermetallic phases would be done. Calculations were done taking into account the following elements: Fe, Cr, Ni, Mo, Cu, N, C, Si and Mn; and phases: Liquid, ferrite, austenite, Cr_2N, σ and $M_{23}C_6$. The calculated phase maps are presented in figure 3. Based on the calculated phase maps and on previous studies on sigma phase formation kinetics (e.g., references 3 and 4), the treatment temperatures of 800°C and 900°C were determined to be the most adequate ones. Although the phase map shows no formation of sigma at 900°C, it must be kept in mind that the map refers to equilibrium conditions, which are not attained in real conditions. Extensive formation of sigma phase was reported in a previous study by one of the authors[27] using this very same material. The treatments were conducted for 1h, 8h and 24h, followed by water quench. All heat treatments were conducted under vacuum, to prevent oxidation and nitrogen depletion near the surface. The samples used were 30x30x3 mm pieces, with the rolling direction parallel the 30x30 mm face, as shown in figure 4.

All samples (as-received and heat treated) were sectioned and polished down to 1 μm diamond using standard metallographic procedures. Etching was done using the tint etch described by Beraha and Shpigler[28] composed of 70 mL of distilled water, 30 mL of fuming hydrochloric acid and 1-2 g of potassium metabissulfite ($K_2S_2O_5$) for each 100 mL of solution. As a result, ferrite is brown or blue (depending on orientation and thickness of the deposited film), austenite is lightly etched (slightly yellow), and sigma remains unetched, allowing for unequivocal identification due to its bright white aspect. The volume percent of the sigma phase of the samples were analysed using "Image J" image analysis software.

2.3. Barkhausen noise measurements

The acquisition of the Barkhausen signal was done using excitation by a sinusoidal magnetic wave of 10 Hz, and a magnetic field of ± $1.2x10^4$ A/m. A pickup coil, placed perpendicular to the sample surface. The acquired signal was amplified and band pass filtered (1-150 kHz). The sampling frequency was 400 kHz. A schematic of the experimental setup for acquisition of the Barkhausen signal is given in figure 5[29,30].

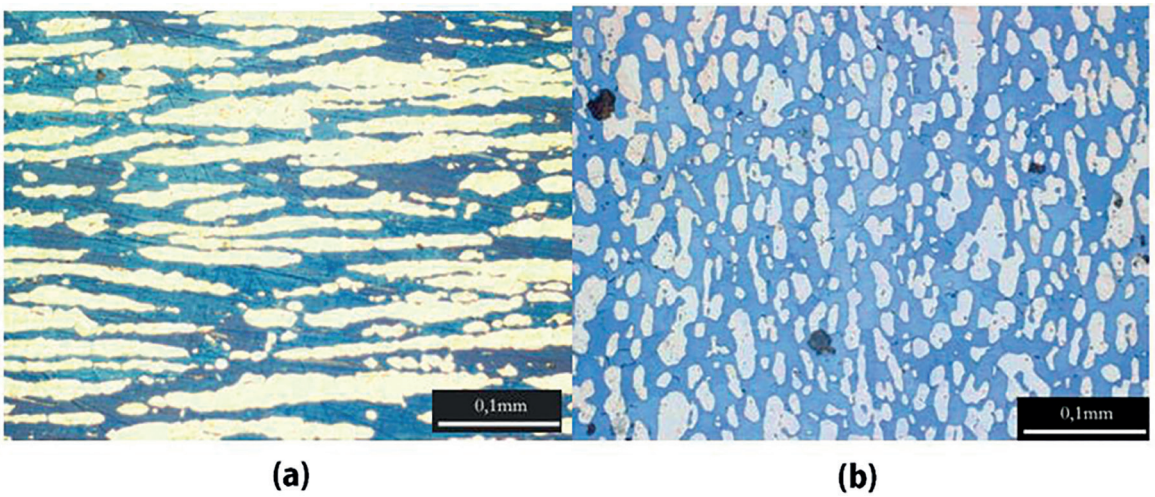

Figure 1. As-received condition of the material used in this study; a) parallel to rolling direction; b) transverse direction.

Table 2. Volume fractions of ferrite measured by quantitative metallography.

Direction	Ferrite volume fraction
Rolling Direction	53.1%
Normal Direction	58.8%

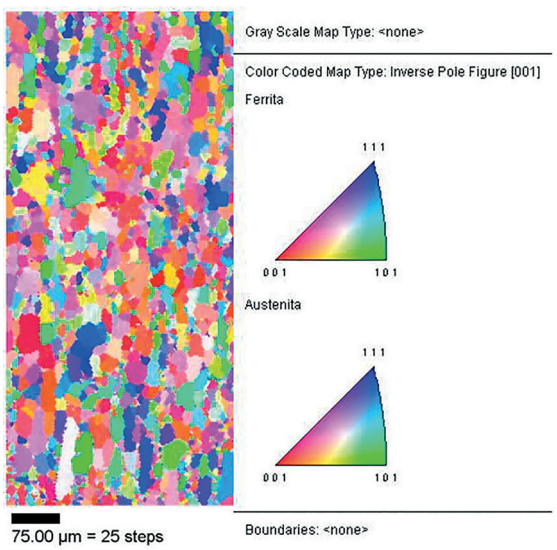

Figure 2. Orientation map of the as received material.

The RMS (Root Mean Square) voltage of the Barkhausen signal is defined by equation 1 as:

$$RMS = \sqrt{\frac{\sum^{i} V_i^2}{n}} \quad eq.1$$

In which V_i is the voltage of each signal individual peak and n is the total number of evented detected in a single measurement. The RMS voltage was then averaged over ten measurements[30]. By defining a bottom voltage level, the noise not belonging to the Barkhausen signal was eliminated from the MBN measurements. This threshold is determined by taking a time window with the background noise only and, then, calculating the RMS of this noise. Only those MBN voltages having amplitude higher than this threshold are considered for analysis.

2.4 Feritscope Measurements

In this work, a Feritscope MP30 (Fischer) was used to determinate the volume fraction of α-Fe (ferrite). The Feritscope is a commercially available device that has been developed for the non-destructive measurements of the ferrite content in austenitic and duplex steels in the range of 0.1 to 80% α-Fe.

3. Results and Discussion

Microstructures of the samples submitted to heat treatment are shown in figures 6 and 7. It can be clearly seen in all micrographs that the formation of sigma phase takes place by consumption of ferrite. As the reaction progresses, the fraction of ferromagnetic phase (ferrite) diminishes considerably and one expects that, as a result, the intensity of the Barkhausen noise also should diminish in a corresponding manner. It can also be seen that, for longer treatment times at 800°C, the reaction is very advanced, and there are regions in which the ferrite has already been entirely consumed. At 900°C, however, the reaction is not nearly as advanced after 24 hours as it is at 800°C, indicating that the "nose" of the C-curve that characterizes sigma formation kinetics is below 900°C (this will be further addressed in the "Barkhausen Measurements" section). The morphology of formed sigma phase does not follow typical characteristics of cooperative growth, such

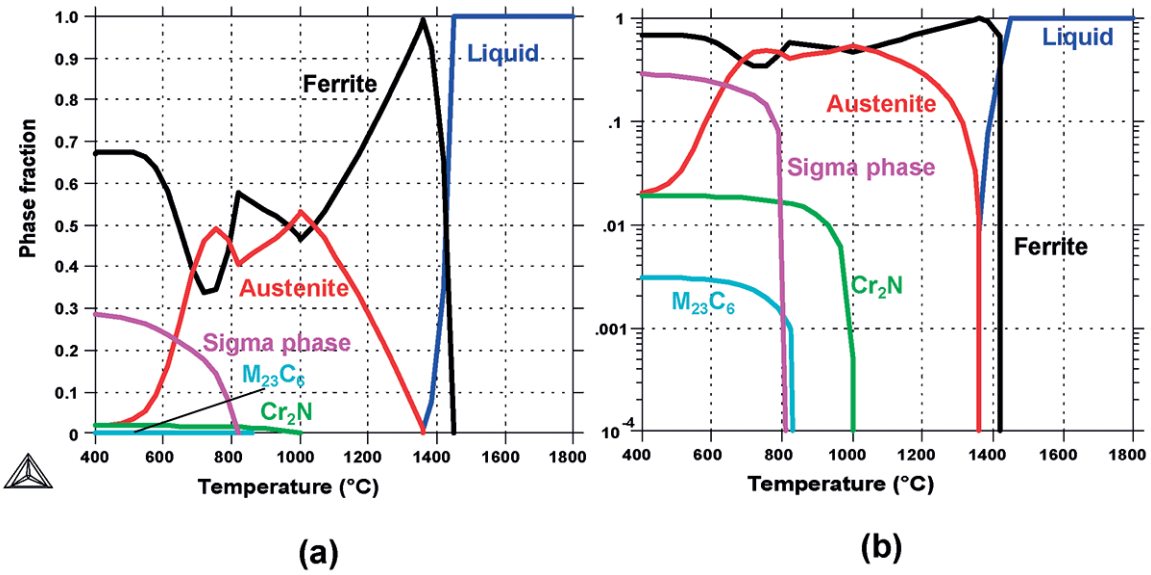

Figure 3. Calculated phase map (Mass fraction) for the material used in the present study as a function of temperature; a) Phase fraction in linear scale; b) phase fraction in log scale.

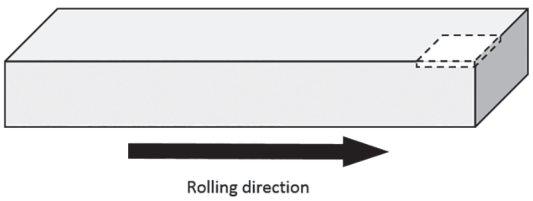

Figure 4. Schematic of sample extraction from the as received bar.

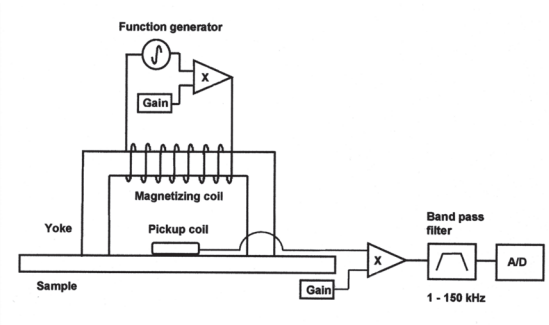

Figure 5. Experimental setup for acquisition of Barkhausen Noise[29,30].

as lamellar structures, and it is possible that small fractions of untransformed ferrite are trapped inside sigma colonies, which makes it possible that even after long treatment times some Barkhausen signal may be detected at slightly higher levels than the background noise.

3.1 Barkhausen noise measurements

The characteristic signal Barkhausen signal for the as received condition is shown in figure 8. As a consequence of the more than 50% volume fraction of ferrite, the signal-to-noise ratio found is quite strong. The Barkhausen noise signals of heat-treated samples are given in figures 9 (800 °C) and 10 (900 °C). It is clearly noticeable that the signal intensity is much higher in the as-received sample than in the heat-treated ones. While the maximum voltage value observed in the as-received condition is close to 2 V, the highest voltages even for samples treated for 1h is approximately 0.5 V, indicating a strong decrease in intensity even for very small quantities of sigma. The variation of the intensity of RMS signal with time is shown in figures 11 to 13. The decrease of signal intensity is much steeper for smaller times, and the proportional decrease (24 h compared to 8 h) becomes very slight over time, indicating that the major part of the transformation has already taken place after 8 hours.

The decrease in Barkhausen signal is smaller at 900°C. This is due to a kinetic issue. The formation of sigma phase follows a classic C-type curve. The nose of the sigma C-curve for this steel is at approximately 850°C, as reported by Magnabosco[4] in a previous study. Therefore, the treatments at 900°C lies above the nose and a slower kinetic is expected. Based on the TTT curve presented by Magnabosco[4], the time it takes to attain a 40% volume fraction of sigma at 900°C is 10 times longer than at 800°C. It should be remembered that above the nose of the C-curve, the decrease in reaction kinetics can be quite significant even for small variations of treatment temperature. The sample treated at 800°C was slightly below the nose, and the reaction kinetics is indeed expected to be significantly faster than at 900°C.

The decrease of the volume fraction of α-Fe (ferrite) in the Feritscope MP30 (Fischer) in treatments for 1, 8 and 24 h, at 800 and 900 °C are shown in Figures 12 and 13. The

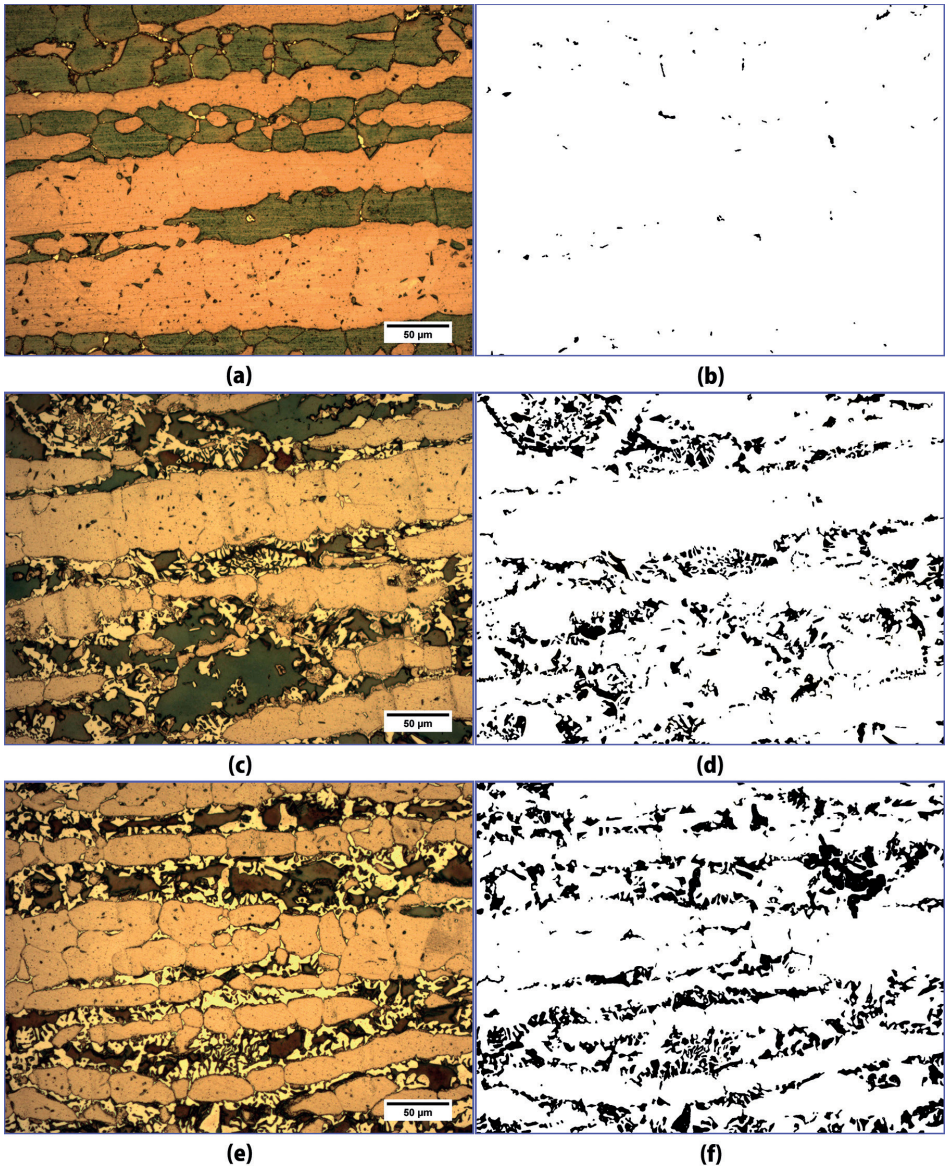

Figure 6. Micrographs and binary separation of sigma phase (sigma is black in the binary images) of samples treated at 800°C – a) optical micrograph, 1h; b) binary separation of sigma, 1h; c) optical micrograph, 8h; d) binary separation of sigma, 8h; e) optical micrograph, 24h; f) binary separation of sigma, 24h.

decrease in ferrite volume fraction is attributed not only to the increase of sigma phase, but also to other paramagnetic or less ferromagnetic phases, as nitrides, chromium carbides and especially the secondary austenite. These phases are also harmful to the corrosion resistance of the steel, since some of them are chromium traps, and reduce the availability of chromium for the formation of the passive layer.

The kinetics of sigma formation measured in this study is consistent with that reported by Magnabosco[4] and with the results of Elmer et al.[2] obtained using In-situ Synchrotron X-Ray Diffraction. More important than the kinetic measurements, however, is the indication that the Barkhausen noise signal can be adequately used to assess the decrease in ferromagnetic phase over time, thus making it a valuable non-destructive technique.

The correspondence between the microstructure and the magnetic response of stainless steels, including its variation during phase transformations, was the object of a series of studies by Tavares and co-workers[31-33]. Their work was done using measurements of saturation magnetization, remanence, coercive force and magnetic transition temperature, and included the magnetic response as a function of ferrite/austenite proportion[31], high and low temperature embrittlement[32] and specific measurements aimed at sigma phase formation[33]. In the latter case, the Ferritscope and Magnetic saturation

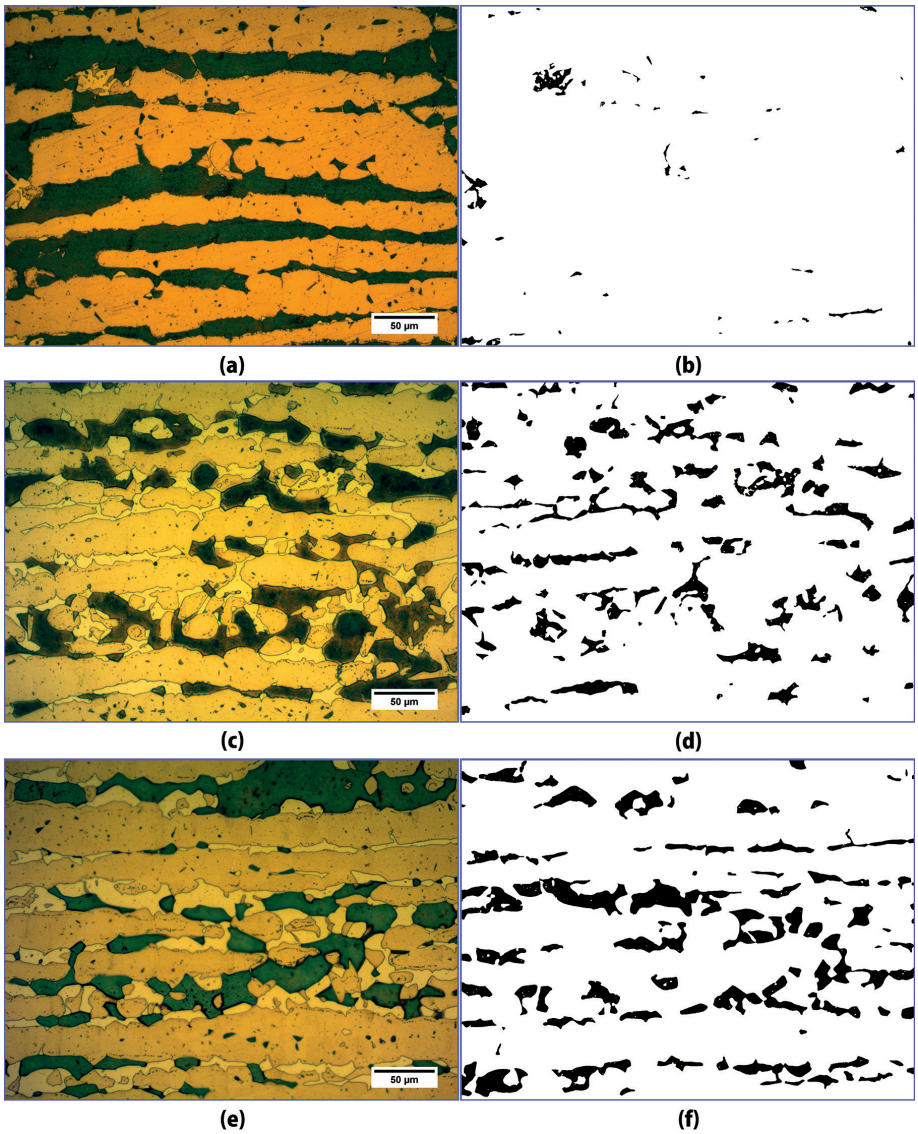

Figure 7. Micrographs and binary separation of sigma phase (sigma is black in the binary images) of samples treated at 900°C – a) optical micrograph, 1h; b) binary separation of sigma, 1h; c) optical micrograph, 8h; d) binary separation of sigma, 8h; e) optical micrograph, 24h; f) binary separation of sigma, 24h.

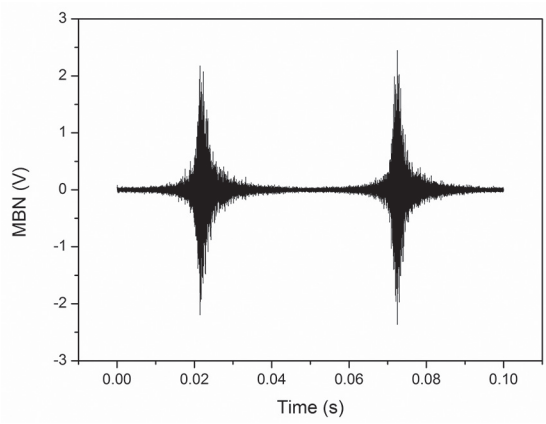

Figure 8. Barkhausen noise of the as received sample.

measurements were used, and the authors observed that both methods are sensitive to small fractions of sigma.

Mezaros and Szabo[34] also reported the change of magnetic parameters that takes place with the change in ferromagnetic phase volume fraction. The authors, however, did not try to associate a variation in ferrite fraction with the magnetic response measured. Instead, the change was measured as a function of heat treatment temperature. The magnetic measurements based on maximum magnetic permeability showed a linear relation when compared to Vickers hardness measurements. It must be noted, though, that several characteristics that are affected by the heat treatment may influence the hardness value, and they affect the Barkhausen noise signal in different ways. The authors noted that a

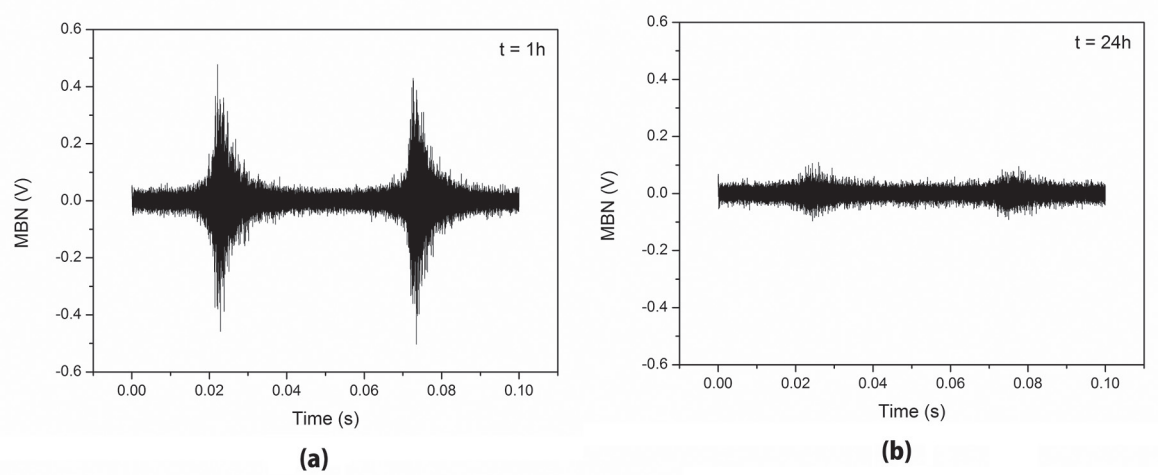

Figure 9. Barkhausen noise of samples treated at 800°C – a) 1h; b) 24h.

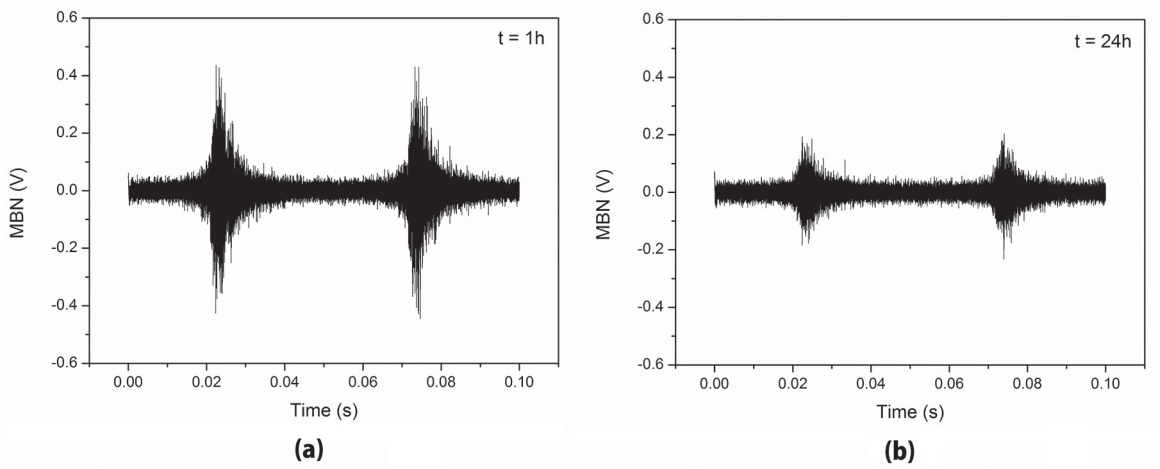

Figure 10. Barkhausen noise of samples treated at 900°C – a) 1h; b) 24h.

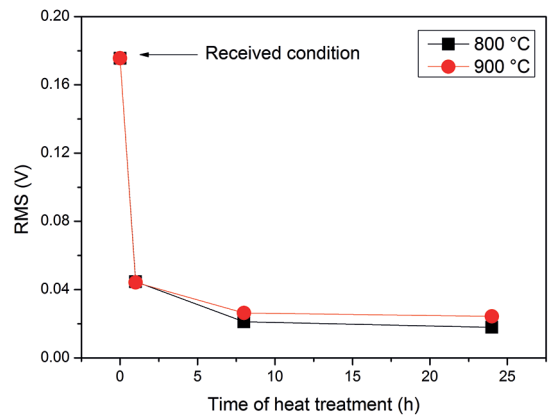

Figure 11. Variation of the intensity of Barkhausen Noise signal with time during isothermal hold at 800°C and 900°C (includes as received condition).

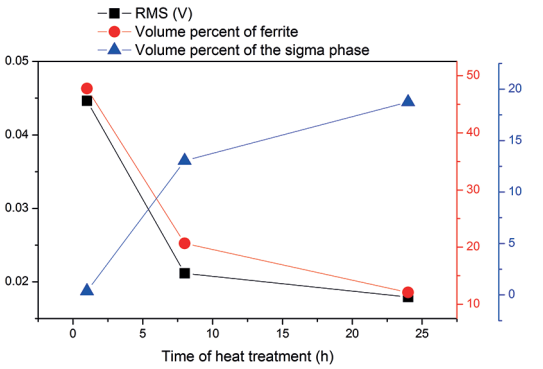

Figure 12. Variation of the intensity of Barkhausen Noise signal (RMS), Ferrite volume percent (measured with Fischer feritscope) and Volume percent of the sigma phase (measured with metallography) with time during isothermal hold at 800 °C.

correlation between Barkhausen Noise and hardness is not a reliable one. However, the correlation with microstructure is much more straightforward. Ginsztler et al.[25] pointed out the direct correlation that can be made between the Barkhausen noise signal profile and the amount of ferromagnetic phase. The results presented here are in good agreement with those

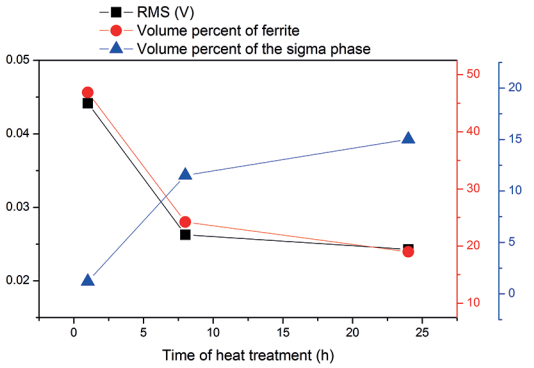

Figure 13. Variation of the intensity of Barkhausen Noise signal (RMS), Ferrite volume percent (measured with Fischer feritscope) and Volume percent of the sigma phase (measured with metallography) with time during isothermal hold at 900 °C.

presented by Ginsztler et al.[25] Also, the results presented confirm the observations of Normando et al.[24], that sigma phase formation can be accurately followed using only non-destructive techniques, which allows for inspections of facilities in the field. However, a calibration curve (corresponding microstructure vs. Barkhausen noise signal) is critical to perform accurate microstructural evaluations using Barkhausen Noise as a non-destructive technique.

Blaow et al.[19] pointed out that microstructures in carbon steels associated with magnetic softness were associated with higher peaks. Also, these authors reported that the presence of strain might lead to a separation of the Barkhausen signal peak into three different peaks. Based on this result, it is possible to state that, in the present study, sigma formation takes place without the onset of significant elastic strain during the reaction.

The results presented here are in good agreement with the aforementioned works, and present a clear indication that Magnetic Barkhausen Noise is a technique that can be used for non-destructive evaluation of microstructural changes taking place in duplex stainless steels during field service. Using the measured volume fraction of ferrite obtained from samples treated at various times, a calibration curve can be plotted, relating the fraction of ferrite and sigma phase as a function of the intensity of magnetic noise signal. It must be kept in mind, though, that sigma is not the only phase that can be formed at high temperatures. The magnetic measurements will only point out the decrease in ferrite content, without giving any clues on the products formed or what reaction mechanism is taking place. Chromium nitrides and chi phase, for example, may be formed, but only the ferrite decrease will be detected. In order to know what phases are likely formed, other classical studies (metallographic evaluation and quantification of phases) are needed. Based on the decrease of ferrite content, and aided by kinetic studies, it should be possible to estimate the amount of sigma plus other phases based on Magnetic Barkhausen noise. Besides the estimation of the amount of phases present, the correct calibration of the magnetic noise curves as a function of microstructure will also allow for the capture of a magnetic noise intensity curve that describes the progress of the decomposition of ferrite into σ and austenite, thus allowing for in situ estimation of predicted life of components subjected to work conditions in which the formation of intermetallic phases may take place. The same principle used in this study to evaluate the formation of sigma phase could also be used to evaluate reactions leading to other phases in different work conditions.

4. Conclusions

The progress of sigma phase formation during isothermal treatments conducted at 800°C and 900°C can be followed using magnetic measurements. The formation of sigma phase causes a measurable decrease in the intensity of Magnetic Barkhausen Noise.

The formation of small fractions of sigma phase can be detected using Magnetic Barkhausen Noise as a non-destructive evaluation, and the method presents high sensitivity over time.

A calibration curve can be plotted relating the fraction of ferrite and sigma phase as a function of the magnetic signal intensity. This curve can be used as a non-destructive evaluation method in industrial environments.

5. Acknowledgements

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