Spectral Analysis of Ultrasonic Lamb waves applied to the study of the Intermetallic phase presence on plates of AISI 430 Ferritic Stainless Steel submitted to Isothermal Treatments.

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Abstract
Ferritic stainless steels appear as a substitute for austenitic due to absence or low nickel content. However, when subjected to thermal processes such as welding, structural variations may occur due to partial formation of martensite and precipitation of carbides, which are responsible for embrittlement and sensitization of the material that reduces the mechanical properties and especially corrosion resistance. Ultrasonic Lamb waves as a nondestructive evaluation of plates can become an effective tool on the diagnosis of macro-structural changes besides the advantage of spreading over long distances. On this work, it was carried out an experimental study of the interaction of the S₀ propagation mode of Lamb waves on plates treated isothermally. Tests were performed in transmission-reception setup and processed on the time and frequency domains. The results show that Lamb waves can be useful in the characterization of ferritic stainless steel plates in order to qualify and quantify the presence of intermetallic phases.

Keywords: Nondestructive Testing, Ultrasonic Lamb Waves, Ferritic Stainless Steel, Materials Characterization, Intermetallic Precipitation.

1. Introduction
The immunity to stress corrosion cracking, high heat conduction and low thermal expansion are some of the properties more important of ferritic stainless steel, that when they are compared to austenitic stainless steels make them more attractive in the commercial and economical point of view, because the lack or the low percentage of nickel at the alloy makes it cheaper. Nevertheless, the susceptibility to sensitization and the structural embrittlement in welding process are some limitations for their use mainly in chemical and petrochemical industries [1].

The ultrasonic nondestructive testing has been widely used in the inspection of materials to guarantee the structure integrity and to avoid failures due to changes in microstructure [2]. Heterogeneities, coarse grain size and intermetallic phases of precipitation, due to thermic process, turn the interpretation of the results difficult [3]. Among the techniques used, guided Lamb waves are known as an advanced inspection technique that allows analyzing great areas quickly and efficiently because of the long range propagation on plate-like structures. The propagation of this type of wave is very dispersive [4].

The objective of this work was to analyze and study the influence of intermetallic phases precipitation and transformations on the microstructure that occur on steel AISI 430 after isothermal treatments on temperatures between 600°C and 1100°C. It was done ultrasonic testing by immersion, using Lamb Guided Waves. To verify changes in frequency specters of the mode S₀ due to thermal treatments applied. The signals were processed using software MATLAB®. It is expected to create a new method to inspect the microstructural integrity of the stainless steel plates thermally treated with the guided Lamb wave.
2. Theory

2.1 Ferritic Stainless Steels

The ferritic stainless steels are alloys of Fe-Cr (11 to 26% in weight of Cr) and content of carbon in general above 0.1%. At room temperature, they are formed basically of a ferrite matrix ($\alpha$), with Body-Centered Cubic (BCC) structure. Others metals are presented as alloy elements at the stainless steels and Chrome is the most important and its presence is indispensable to ensure some properties as corrosion resistance, because it is due to this chemistry element that the passivation is guaranteed to this kind of materials [5]. The 430 ferritic steel, as an example, when it is subjected to thermal processes as welding it is possible to see the formation of martensite from the austenite due to high temperatures mainly in grain’s boundary when the material is cooled. That increases hardness and loss of ductility in the material and its intermetallic precipitates on the region near to the weld fillet called Heat-affected Zone (HAZ). Figure 1 shows the Fe-Cr equilibrium diagram where it is marked the formation zone of the ferritic steels.

![Fe-Cr diagram](image)

Figure 1. Fe-Cr diagram shows the region with commercial ferritic steels stressed [6].

The embrittlement of this kind of steel is explained by Demo et al [7], also called material sensitization. The authors describe the reaction mechanism of the material in a determined temperature. When the ferritic stainless steel are heated in temperatures above 950°C there is the dissolution of precipitates rich in chrome (carbides $M_23C_6$ and/or nitrites $Cr_2N$) in the ferritic matrix. When heated between 500°C and 900°C it can occur fast precipitation of carbides and chrome nitrites, causing the relief of the supersaturated ferritic matrix. In the range of 700°C and 950°C, simultaneously the precipitation there is the diffusion of chrome in direction to the poorest areas, leading the steel to recuperate the intergranular corrosion resistance. In the range between 500°C and 700°C, there is fast precipitation of rich phases in Cr resulting in a poor region causing the sensitization.

2.2. Guided Lamb Waves

In Lamb waves, a large number of particle vibration modes are possible with specific energy quantities (specific modes), which depends substantially of some factors, as: pulse system, the
ultrasonic beam incidence angle, transducer central frequency, the bandwidth frequency and others parameters described in [8, 9]. Figure 2 shows the propagation modes most common: the symmetric and asymmetric modes. The complex movement of the particle is similar to the elliptical orbit to the surface.

Figure 2. (a) Propagation of Lamb waves in a plate of thickness d; (b) symmetric mode; (c) asymmetric mode.

The velocity guided Lamb waves is not only dependent on the material (like longitudinal, shear and surface waves) but also the thickness of the material and frequency. Dispersion curves are used to describe and predict the relationship between frequency, phase velocity and group velocity, incidence angle, mode and thickness [9]. These curves are originated from solutions that satisfy boundary conditions of the wave equation for a determined system and are described, as seen before, in terms of Lamé Constants. The solutions can be found numerically through a data set of constants related to the materials properties. Figure 3 shows dispersion curves for an aluminium plate with 0.5 mm thickness and immersed in water.

Figure 3. Dispersion curves for aluminium plate with 0.5 mm thickness immersed in water. The modes showed are symmetrical (S) and asymmetrical (A).

Through the analysis of the Lamb waves propagation modes dispersion curves in function of frequency-thickness, it was determined the frequency range of interest and the incidence angle to be applied on the practical experiments to ensure the less dispersive guided wave propagation mode only. Araujo et al [4] selected the propagation mode $S_0$ due to their non-dispersive characteristic on the 0.85 MHz-mm region to inspect the aluminium plates with defects on immersion testing. The researchers analyzed the simulated dispersion curve by Disperse Software® [10], evaluating the phase velocity variation, group velocity and attenuation of the fundamental propagation modes $A_0$ and $S_0$. 
2.3. Ultrasonic Spectral Analyses

The digital signal processing techniques applied to ultrasonic Lamb waves are tools with high importance in information distinction related to the damages presented in structures like plates. Echogram analysis is capable to extract information that could not be analyzed on time domain. There are parameters in phase or amplitude that demonstrates specific characteristics of determined defects through spectral analysis [11]. The use of the Fast Fourier Transform (FFT) algorithm for the application of the Discrete Fourier Transform (DFT) preserves the frequency amplitude of each component as its phase after the ultrasonic pulse interacts with the material, behaving like a “digital impression” for the material [11].

Yanxun et.al [12] illustrate the FFT application as a result of microstructural degradation identification in plates of a ferritic league (Fe-Cr) in high temperatures, using non-linear ultrasonic Lamb waves in the contact experimental setup. They identified, through spectral components analysis (FFT), the differences on the frequency specters related to the microstructural characteristics in several temperatures and succeed in characterizing them.

3. Methodology

3.1. Specimens

It was made 7 specimens of standard ferritic stainless steel AISI 430 (composition: C-0.12%; Si- 1.00%; Mn – 1.00%; P – 0.040%; S – 0.030%; Cr – 16.00-18.00%; N – 0.12 max.). All of the specimens were confectioned with the dimensions of 300 x 150 x 1.5 mm [13]. Table 01 shows the thermal conditions and the time that each specimen was subjected.

<table>
<thead>
<tr>
<th>Material</th>
<th>Specimens</th>
<th>Temperature (ºC)</th>
<th>Exposition Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferritic AISI 430</td>
<td>S_NT</td>
<td>As received</td>
<td>No treatment</td>
</tr>
<tr>
<td></td>
<td>S_600</td>
<td>600</td>
<td></td>
</tr>
<tr>
<td></td>
<td>S_700</td>
<td>700</td>
<td></td>
</tr>
<tr>
<td></td>
<td>S_800</td>
<td>800</td>
<td>2 hours</td>
</tr>
<tr>
<td></td>
<td>S_900</td>
<td>900</td>
<td></td>
</tr>
<tr>
<td></td>
<td>S_1000</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>S_1100</td>
<td>1100</td>
<td></td>
</tr>
</tbody>
</table>

For the ferritic steel samples, the S_NT specimen (No Treated) remained as received. For the others samples it was realized thermal treatments for secondary phase precipitation [13]. For the S_600, it was aimed to find a small quantity of carbides precipitation. On the S_700 specimen the main goal was to verify little microstructural change, grains recrystallization and precipitation on grain’s boundary [14], what characterize material sensitization. This mechanism is seen until the temperature of 800ºC, after this temperature the structure becomes duplex (ferrite and martensite). Higher than 900ºC, the specimens, S_900, S_1000, is expected to have duplex matrix with little carbides precipitation. On the S_1100, it is only found duplex matrix made by the dispersion of martensite on ferrite. The choosing of the temperatures and the respective transformation mechanisms can be verified in figure 4.
3.2 Metallographic Testing

The ferritic steel 430 samples used in the metallographic testing were embedded in Bakelite, sanded by sandpaper of 220, 320, 400, 600 and 1200 mesh, polished with diamond paste 3µm and 1µm and alumina 0.3µm. The steel samples were etched for 40 seconds by the solution of Vilella containing: 1g of picric acid, 5mL of HCl, 100mL of ethanol and 50mL of water. It was used to delineate carbides particles of second phase, phases δ and σ [15]. After this stage, an optical microscope model UNIOMET and a photographic camera model Nikon D50 allow the capture and storage of images related to the microstructure revealed by the testing.

3.3 Ultrasonic Inspection by Immersion in Pitch-Catch Configuration

The transducer-receptor was placed in line in relation to the transducer-issuing. The inspection was made by using a pulse generator Olympus®, model 5077PR, with transducers Olympus Panametrics®, model V303-SU, diameter of 12.7 mm, central frequency of 0.9 MHz and bandwidth of 0.51 MHz. The signals were collected in one digital oscilloscope Tektronix®, model TDS2024B, sampling frequency of 250 MHz with an interface to a microcomputer to store the signal.

The plate sweeping was done in steps of 4 mm travelling a distance of 32 mm in each sample, distance sufficient to sweep the entire area without edge interference. Figure 5 shows the positions of the equipment, sensors and the experimental scheme of inspection by immersion.
in the configuration of transmission-reception for which were applied the Fast Fourier Transform to determine the frequency spectral of which sample in other to evaluate the microstructural changes by guided Lamb wave according to [4].

### 3.4 Dispersion Curves

The dispersion curves were simulated using Disperse Software® [10] designed to calculate curves of multilayer structures. Through the analysis of the simulated dispersion curves of the phase velocity against frequency-thickness was determined the frequency and the incidence of angle beam to be used in the experimental work in order to generate only the fundamental Lamb waves modes.

### 3.5 Spectral Analysis of Lamb Ultrasonic Signals

The evaluation of the influence of microstructure and grain size, further the precipitates on the wave propagation was made on frequency domain. The specters of the collected signals of specimens with and without thermal treatment were obtained with the application of the Fast Fourier Transform (FFT) through MATLAB® software and plotted in superimposed way, for analyze the differences between the frequency components in each specimen subjected to isothermal treatments.

### 4. Results and Discussions

#### 4.1 Simulation Disperse Curves

In figure 6 are shown the simulated dispersive curves related to immersed testing for specimens by Disperse Software®. $S_0$ mode was selected because it is less attenuating than $A_0$ mode in a frequency-thickness band less dispersive for the realization of the practical experiments. Like shown in figure 6, the incident angle more adequate for the ultrasonic sensors was 16° for all specimens.

![Figure 6. Dispersion for stainless steel: (a) Phase velocity; (b) Group velocity; (c) Incidence angle; (d) Attenuation](image)

#### 4.2 Microstructural Analyses

Figure 7(a) shows the microstructure of the specimen as received, S_NT. The grain boundaries are not defined clearly, this can occur due to the difficult of the etching attacks the surface because of the high quantity of chrome. This makes a corrosion protector coat as observed in the microscopy of Farina thesis [13]. Figure 7(b) shows the microstructure according to the work done by Farina in his thesis [13]. It is showed the presence of...
precipitates in grain’s boundary what is not observed in S_NT specimen, figure 7(a).

Figure 7. (a) Optical Microscopy of S_NT steel obtained by attack with Vilella etching with a scale of 25 µm; and figure; 7 (b) with a scale of 10µm [18].

Figures 8 present the micrographs of the S_600 specimen subjected to a isothermal treatment of 600°C for 2 hours seen in figure 8(a); and 600°C for 5 hours done by Farina [13] in figure 8(b). It is possible to see that there are almost no differences in both cases in relation to the non-treated state; however it can be observed a small precipitation of carbides in grain’s boundary in both cases.

In figure 9(a), it can be seen the microstructure of S_700 specimen thermic treated at 700°C for 2 hours; and (b) the specimen treated at the same temperature for 5 hours as cited by Farina [13]. A small coarsening of the grains is observed in relation to the previous specimen. There is a higher concentration of precipitates in grain boundaries seen in both specimens.

In figures 10, (a) the microstructures of the S_800 specimen subjected to isothermal treatment of 800°C for 2 hours and (b) the S_900 specimen subjected to a temperature of 900°C for the same time. Both specimens presented a relative increase of the grainsize in relation to previous specimens. There are also intergranular precipitates points in both cases. It was not possible to see martensite phase as seen in Farina [13].
Figure 9. Optical Microscopies of S_700 specimen obtained by attack of Vilella etching (a) with a zoom of 200X; (b) with a scale of 5µm [13].

Figure 10. Optical Microscopies of (a) S_800 specimen; and (b) S_900 obtained by attack of Vilella etching with a zoom of 200X.

Figures 11 shows the microscopies of (a) S_100 and (b) S_1100 specimens subjected to thermal treatment for 2 hours. In 1000°C, it was verified that there is not precipitates in the matrix nor in grain’s boundary, nevertheless a significant increase of grain size can be observed.

Figure 11. Optical Microscopies of (a) S_1000 and (b) S_1100, obtained by attack of Vilella etching with a zoom of 200X.

In 1100°C, it is possible to verify through figure 11(b) the recrystallization of the grains besides intergranular precipitation. In none of the specimens was verified the presence of a duplex matrix as described by Farina [13].
4.3 Spectral Analysis

Figure 12 shows the frequency specters obtained via FFT from $S_0$ Lamb mode signals in all of the specimens.

![FFT](image)

Figure 12. Frequency specters of $S_0$ mode, obtained by the inspection of all the specimens with thickness of 1.5 mm by immersion testing and pitch-catch configuration.

Around the central frequency of $S_{NT}$ specimen, it is observed a decrease of the peak of amplitude until $S_{1000}$ specimen what can be due to phase precipitation (carbides) besides the gradual increase of grain size verified in the micrographics. The $S_{1100}$ specimen had a spectral behavior close to $S_{800}$ specimen what is probably explained to the similar granular microstructure.

5. Conclusions

The evaluation of the spectral content trough ultrasonic Lamb waves for the different specimens in different temperatures allowed in a satisfactory way to characterize the specimens according to the microstructural changes. It was observed the decrease of amplitude for $S_{600}$, $S_{700}$, $S_{800}$, $S_{900}$, $S_{1000}$ specimens besides the changes on the frequency peaks for $S_{900}$ and $S_{1000}$. The testing showed to be consistent because the spectral similarity between $S_{1000}$ and $S_{800}$ specimen is confirmed based on the similarity in the micrographics too. The martensitic phase was not presented as a justification for spectral changes because it was not characterized by optical microscopy. The spectral changes can be occurred by new contours of the precipitates besides the progressive increase of grain size on the matrix.

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