

**Nondestructive Characterization and Evaluation of Embrittlement  
Kinetics and Elastic Constants of Duplex Stainless Steel SAF 2205 for  
Different Aging Times at 425 °C and 475 °C**

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## **Abstract**

In this work, an experimental study was carried out to evaluate the potential of the ultrasonic technique, with ultrasonic velocity and attenuation measurements, to assess the heat aging effects on duplex stainless steel SAF 2205, at temperatures of 425 °C and 475 °C for time periods of 12 h, 24 h, 50 h, 100 h and 200 h, as well as in the as received state of the material. Velocity measurements were calculated for both longitudinal and transversal waves. The elastic constants, Young's modulus and shear modulus, of the material were computed from the relationship between longitudinal and transversal velocities. For the ultrasonic attenuation, only longitudinal waves were considered. Despite the large scatter measurements, both ultrasonic velocity and attenuation increased with the heat aging time, particularly at 475 °C. Thus, it may be concluded that the technique used is promising and provides relevant contributions to an accurate characterization of materials and evaluation of their mechanical properties in a non-destructive manner.

**Key-words:** Aging heat treatment, Ultrasonic velocity; Ultrasonic attenuation; Young's modulus; Shear modulus; Duplex stainless steel.

## 1 INTRODUCTION

From an acoustic perspective, the majority of materials used in engineering are heterogeneous. On a microstructural level, acoustic heterogeneity in materials is essentially influenced by the grain, fiber characteristics, and any inclusions, microruptures, precipitates and pores <sup>[1]</sup>.

Acoustic heterogeneities caused by microstructural effects can be analyzed using ultrasonic techniques with the aim to: (1) detect ruptures or measure thickness or (2) identify the causes of the acoustic heterogeneities. The study of ultrasound/heterogeneity interactions is very important in both cases, especially in the later one.

The interaction of ultrasound with microstructural properties of a material can be analyzed in terms of propagation velocity variations, loss of amplitude (or attenuation) and analysis of retro-dispersed signal <sup>[1]</sup>. In the current literature, it is possible to find studies where authors applied ultrasound measurements to calculate elastic constants, analyze microstructures, texture and mechanical properties. For example, Freitas et al. <sup>[2]</sup> presented a reliable and fast nondestructive characterization of microstructural and elastic properties of plain carbon steel, based on ultrasonic testing. The authors measured the ultrasonic velocity and attenuation for relationships with the ferrite, pearlite, ferrite-pearlite, and martensite microstructures. Albuquerque et al. <sup>[3]</sup> used techniques to carry out a spinodal decomposition mechanism study on the duplex stainless steel duplex USN S31803 based on X-ray diffraction measurements and ultrasonic velocity and Silva et al. <sup>[4]</sup> evaluated the material phase transformations through the speed of ultrasound, Charpy impact energy, X-ray diffraction, hardness and microscopy.

The great majority of works that have evaluated embrittlement kinetics of the  $\alpha'$  phase in duplex stainless steels use aging temperatures up until 550 °C, because above this temperature embrittlement kinetics is too high for the formation of the  $\alpha'$  phase, and also because embrittlement occurs due to precipitation of the  $\sigma$  phase in the ferritic matrix. Besides that, several authors have been studying the relationships among the microstructural, magnetic and mechanical properties <sup>[5]</sup>. For example, Tavares et al. <sup>[6]</sup> evaluated the corrosion resistance of the UNS S31803 duplex stainless steels aged at temperatures ranging from 350 °C to 550 °C. Abreu et al. <sup>[7]</sup>, studied the consequences of aging at 400 °C and 475 °C on the  $\alpha'$  precipitation and corrosion resistance of a novel Cr–Mo ferritic stainless steel with high Mo content through X-ray diffraction and a Mössbauer spectroscopy. They concluded that this phase is related to a decrease of resistance to corrosion.

Here this work uses the ultrasonic technique, mainly ultrasonic velocity and attenuation, to monitor the embrittlement kinetics of a thermally aged duplex stainless steel SAF 2205, and to evaluate its Young's modulus and shear modulus for each heat aging time used.

The choice of the duplex stainless steel SAF 2205 for this work was essentially based on its excellent mechanical properties; mainly, its mechanical resistance and its resistance to corrosion, as well as its wide use in aggressive environments such as in chemical and petrochemical industries among others <sup>[8-12]</sup>.

When exposed to temperatures above 300 °C, the duplex stainless steel SAF 2205 becomes susceptible to new phases (precipitates), such as: the sigma phase ( $\sigma$ ), chi phase ( $\chi$ ), alpha line phase ( $\alpha'$ ), chrome nitrides ( $\text{Cr}_2\text{N}$ ), among others. Depending on the application on hand, these new phases may cause damage to the material,. Thus, to

ensure the important properties of this type of steel, it is extremely important that there are no phase transformations during its production, application or maintenance.

As such, the main aim of this work was to monitor and characterize, in an accurate and non-destructive manner, the phase transformations of a duplex stainless steel SAF 2205 submitted to heat aging treatments at 425 °C and 475 °C for time periods of 12 h, 24 h, 50 h, 100 h and 200 h, as well as the material in the as-received state. Ultrasonic velocity and attenuation measurements were taken and from these measurements, the Young's modulus and the shear modulus of the material were determined and analyzed. The longitudinal and transversal wave velocity measurements and the density of the material were taken to calculate the elastic properties. Additionally, the Rockwell C hardness test was used to macroscopically evaluate the rigidity of the material, due to phase transformations arising from the aging heat treatments that were done.

This paper is organized as follows: The next section presents: the materials used; the different heat aging conditions to which the material was submitted to; a description of the new phases; and the ultrasonic technique applied for material characterization is introduced. Section 3 presents the Results obtained and the Discussion. Finally, in Section 4, the Conclusions of the work are pointed out.

## **2 MATERIAL AND METHODS**

The duplex stainless steel used for this work was the SAF 2205, also commonly specified as UNS S31803, manufactured by Sandvik Steel (Brazil). Table 1 shows the chemical composition of the duplex stainless steel certified by the manufacturer. Eleven samples of 55x25x10 mm<sup>3</sup> were made for the experimental tests. There were five

samples for each temperature and one for the as-received state. The samples were aged at temperatures of 425 °C and 475 °C for time periods of 12 h, 24 h, 50 h, 100 h and 200 h, in an electric resistance furnace, except for the as-received sample which was kept as a reference. For each sample, the hardness value was assessed through the Rockwell C method <sup>[13]</sup>, by measuring the hardness in five random points and averaging the values obtained.

The pulse-echo technique and direct contact method were used to obtain the parameters of ultrasonic velocity and attenuation. Lubricant oil SAE W40 was used for coupling the longitudinal and transversal wave transducers. For processing, a Krautkramer ultrasound device (GE Inspection Technologies, USA, model USD15B) connected to a 100 MHz digital oscilloscope (Tektronix, USA, model TDS3012B) transferred the signals to a computer where they were stored and processed (Figure 1).

Procedures for the ultrasonic tests included: definition of the ultrasound parameters; cleaning of the sample surfaces; application of the coupling and positioning of the transducer, selection of the relevant part of the ultrasound signal; acquisition and processing of the signal. Since the major goal of this work was a relative behavior analysis and all test samples were prepared to the same surface finishing standard, the superficial transmission and reflection coefficients were considered equal for all samples.

All signals were captured with 10,000 points, at a sampling rate of 1 Gs/s. After acquisition the data was processed to determine the ultrasonic velocity and attenuation. The ultrasonic velocity measurements were obtained using longitudinal waves with frequencies of: 4 MHz, using a transducer with a 10 mm diameter from Krautkramer (Australia) - model MB4S-E; 5 MHz using a transducer with a ¼" diameter,

Krautkramer (Australia) - model MSW-QCG; and 10 MHz using a transducer with a ¼" diameter from Olympus (USA) - model V112-RM; also from transversal waves with a frequency of 5 MHz with a transducer with a ¼" diameter from Valpey Fisher (USA) - model SF052. The choice of these frequencies was based on the authors' previous experience in this kind of nondestructive testing and knowledge concerning the material under study [2, 3, 4].

For each sample, ultrasonic velocity measurements were obtained from five signals with two adjacent echoes per signal. Next, the time between the first two echoes was measured. With the propagation time of the sonic wave and the thickness of the sample, obtained with a micrometer at the same points where the ultrasound signals were obtained, the average propagation speed of the wave was determined for the sample via the equation:

$$v = \frac{2X}{\tau_0}, \quad (1)$$

where  $X$  is the sample thickness [m] and  $\tau_0$  is the traveling time [s] of the wave until the two adjacent echoes ( $B_1$  and  $B_2$ ) overlap, which is determined using:

$$\left| \int_{-\infty}^{\infty} B_1(t).B_2(t - \tau)dt \right|. \quad (2)$$

The constant 2 (two), in Equation 1, is applied because with the pulse-echo technique used here the sound travels through the sample twice before going back to the receptor [14].

Although the time of flight of the wave may be directly obtained from the oscilloscope, the echoe overlapping method was used, because it is more sensitive and guarantees maximum accuracy of the measurements [14].

The ultrasonic attenuation values were calculated based on the reduction of the amplitude of an ultrasound pulse, measured in decibels per millimeter ([dB/mm]) and given as:

$$\alpha = \frac{20}{2x} \log \frac{A_0}{A_1}, \quad (3)$$

where  $\alpha$  is the attenuation coefficient [dB/mm],  $x$  is the thickness of the sample measured in the test [mm],  $A_0$  is the amplitude of the first echo in dB and  $A_1$  is the amplitude of the second echo. Once again, constant 2 (two) in Equation 3, is due to the pulse-echo technique used. The measurements were obtained considering longitudinal waves and frequencies of 4 MHz and 5 MHz.

The elastic constants of the material under study, that is Young's modulus (E) and shear modulus (G), were calculated according to the norm ASTM E 494-2005 <sup>[15]</sup>, for measuring ultrasonic velocity in materials, with the following equations:

$$E = \frac{\rho V_T^2 (3V_L^2 - 4V_T^2)}{(V_L^2 - V_T^2)}, \quad (4)$$

$$G = \rho V_T^2, \quad (5)$$

where  $V_L$  is the longitudinal wave velocity [m/s],  $V_T$  is the transversal wave velocity [m/s] and  $\rho$  is the density of the material [g/cm<sup>3</sup>] under study which, based on the current literature was considered to be equal to 7.805 g/cm<sup>3</sup> <sup>[19]</sup>.

### 3 RESULTS AND DISCUSSION

Embrittlement of duplex stainless steels is due to phase transformations. At temperatures below 550 °C the main phase responsible for the embrittlement is the  $\alpha'$  phase and for temperatures above 550 °C the  $\sigma$  phase is responsible for the



embrittlement. In this work, embrittlement by transformation of phases in a duplex stainless steel SAF 2205 was studied at temperatures of 425 °C and 475 °C and with aging times up to 200 h, as well as in the as-received state. Hardness measurements, as well as ultrasonic velocity and attenuation measurements were carried out on all the samples.

The temperature range studied here is characterized by the decomposition of the initial  $\alpha$ -phase into two phases: one poor in chromium and the other rich in chromium; the mechanism responsible of this transformation is known as spinodal decomposition<sup>[8]</sup>. On the other hand, the  $\gamma$  phase does not suffer any transformation within this temperature range<sup>[16]</sup>. For an easier understanding of the transformations taking place during the ageing at the temperature range of 425 to 475 °C, the hardness of the material was analyzed through energy absorbed and hardness measurements, Figure 2. As has been reported in other spinodal decomposition systems<sup>[17,18]</sup>, Figure 2 shows that the hardness curve can be divided into two-stages of hardness, with a fast increase in hardness for stage I followed by a transition level. Afterwards, a second stage begins at a lower hardening rate. According to Choo et al.<sup>[17]</sup>, the two causes for the hardness at stages I and II are spinodal decomposition and the growth of organized  $\alpha'$  phase particles, respectively. The hardness growth rate at the second stage is lower, mainly due to the coalescing of the  $\alpha'$  phase particles. The data for the graphs of Figure 2 has a 95% .degree of confidence.

The measurements for ultrasonic velocity were obtained from longitudinal waves at frequencies of 4 MHz, 5 MHz and 10 MHz, and transversal waves at a 5 MHz frequency. Figures 3a and 3b show the sonic velocity measurements obtained from longitudinal waves for samples aged at 425 °C and 475 °C, respectively, at the three

frequencies. These figures show that the sonic velocity increases with the aging time, which is coherent with the results reported by Silva et al. <sup>[3]</sup> and Matsubara et al. <sup>[20]</sup>. The same behavior was observed for the three frequencies used, and a tendency for a slight increase of the ultrasonic velocities with an increase in the frequency was also observed.

For the purpose of comparison, in Figures 4a, 4b and 4c longitudinal velocities for both temperatures (425 °C and 475 °C) at each frequency are shown. The charts in Figure 4 include the error bars and reveal the behavior of the ultrasonic velocity and the longitudinal waves at each temperature. The highest velocities are associated with the samples aged at 475 °C. The increase in ultrasonic velocity with the treatment time is mainly due to the increase of the Young's modulus of the grains in phase  $\alpha$ , since the precipitation of the brittle phases occurs in this phase when exposed to these temperatures for long periods of time. In Figure 4, the transition from spinodal decomposition to the growth of particles is noticed at 20 h of aging. This reveals the higher sensitivity of the sound velocity over the hardness test.

The results of the ultrasonic velocity of transversal waves at temperatures of 425 °C and 475 °C did not present significant variations until 50 hours of aging (Figure 4d). However, at 475 °C, the transversal velocity of the waves increased at aging times greater than 50 hours. This can be explained by the fact that embrittlement due to  $\alpha$ -phase transformations in this steel is more assiduous at 475 °C, as the kinetics of phase decomposition is higher at this temperature.

The ultrasonic attenuation measurements were made at frequencies of 4 MHz and 5 MHz for the samples aged at 425 °C and 475 °C. These results are shown in Figures 5a and 5b. These figures show the behavior of ultrasonic attenuation at each

temperature pursuant to the aging time period and frequency, and reveal that the attenuation coefficients are influenced by the aging time. The attenuation coefficients values obtained for each frequency at the higher temperature were more distinct.

As already mentioned, the Young's modulus and the shear modulus of the duplex steel samples under analysis were obtained by ultrasound with the pulse-echo technique at a frequency of 5 MHz. Measurements were obtained for the as-received sample, and also for the aged samples, through the relationship between longitudinal and transversal ultrasonic velocities and the density of the material (specific mass).

The results presented in Figures 6a and 6b show that the values of the elastic constants increase with the aging time. The increase of Young's modulus with the aging time is more evident at the temperature of 475 °C than at 425 °C. This variation in the elastic constants is due to fluctuations in the composition generated by spinodal decomposition, where the  $\alpha'$  particles are formed. These are finely dispersed in the ferritic matrix of the material under analysis. For the temperature of 475 °C, the shear modulus decreases with aging up to 25 h due to the reduction of the matrix saturation followed by the formation of a coherent phase.

#### **4 CONCLUSIONS**

This work studied the potentialities of the ultrasound technique, namely the ultrasonic velocity and attenuation parameters, for the accurate and non-destructive monitoring of the phase transformation of a duplex stainless steel SAF 2205. The following conclusions can be pointed out:

- The results show that the ultrasonic parameters analyzed are sensitive to the microstructural variations in the material studied, identifying the transformation of

phase  $\alpha$  to phase  $\alpha'$  due to the different heat aging treatments to which the material was submitted.

- Ultrasonic velocity could follow the formation of the phase  $\alpha'$  in duplex steel SAF 2205. The precipitation of this phase in the ferritic matrix reduces the internal displacements. This fact is reflected in the increase in hardness, in the Young's modulus, and in the shear modulus for aging times superior to 25 hours, and consequently, in the ultrasonic velocity and attenuation values.
- The ultrasonic attenuation measurements for frequencies of 5 MHz showed better results than of 4 MHz, but further studies need to be carried out with higher frequencies.

The results obtained are promising and contribute to the characterization of the material and reveal their mechanical properties through ultrasonic parameters in a straightforward, fast and non-destructive manner.

To conclude, it is important to emphasize that this work aimed to verify the feasibility of the ultrasonic technique and parameters for practical applications, and the results were very promising. However, further studies on the influence of the dimensions of the transducer used, sample sizes, superficial transmission and reflection coefficients, are required. In addition, as the acquired data had a wide scatter behavior, studies considering a larger number of samples should be addressed in order to deduce meaningful trends and eliminate artifacts.

## **ACKNOWLEDGEMENTS**

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## FIGURE CAPTIONS

Figure 1. Experimental setup used in the acquisition and analysis of the ultrasonic signals.

Figure 2. Variation of the Rockwell C hardness against aging time at a) 425 °C and b) 475 °C.

Figure 3. Average longitudinal velocity against aging time at a) 425 °C and b) 475 °C and frequencies of 4 MHz, 5 MHz and 10 MHz.

Figure 4. Average longitudinal velocity against aging time at 425 °C and 475 °C and frequencies of a) 4 MHz, b) 5 MHz and c) 10 MHz, and d) for the average transversal velocity with frequency of 5 MHz.

Figure 5. Average ultrasonic attenuation against aging time at a) 425 °C and b) 475 °C and frequencies of 4 MHz and 5 MHz.

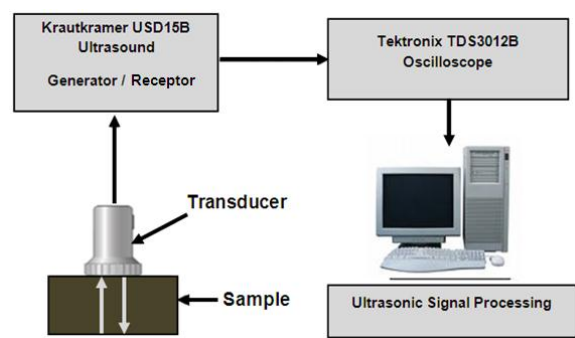
Figure 6. Variation of Young's modulus and shear modulus against aging time at a) 425 °C and b) 475°C.



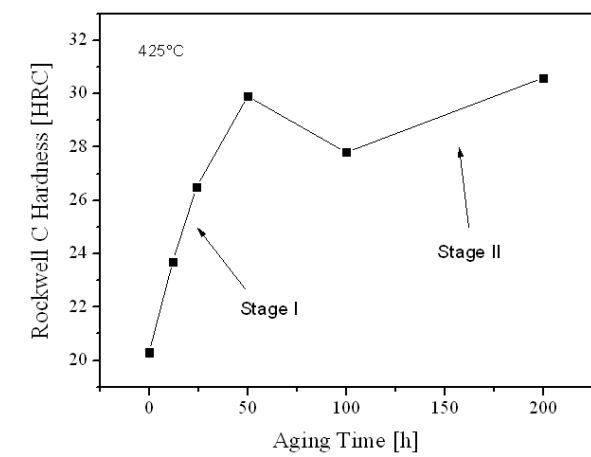
**TABLE CAPTION**

Table 1: Duplex stainless steel chemical composition as-received in wt.%.

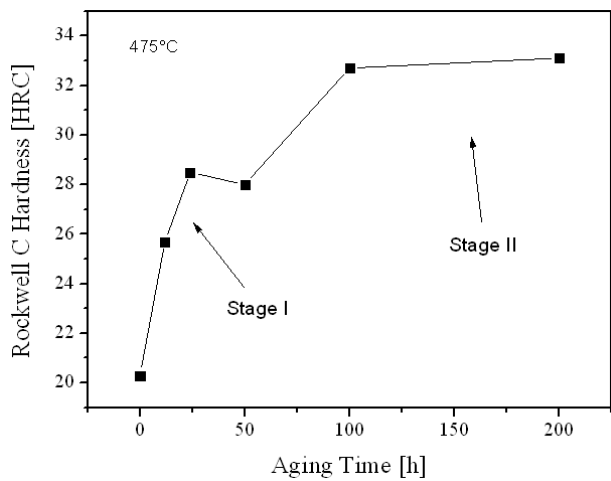
**FIGURES**



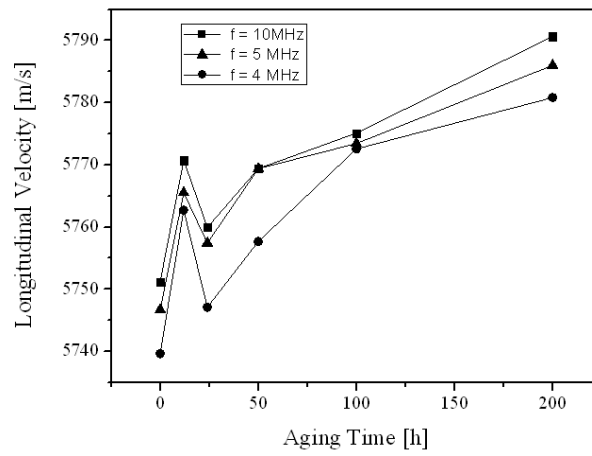
**Figure 1**



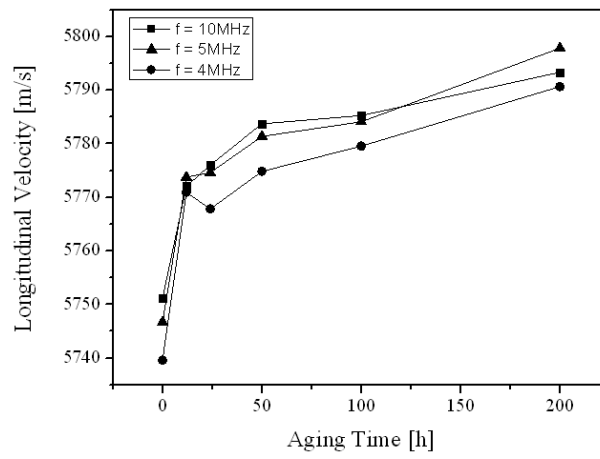
**Figure 2a**



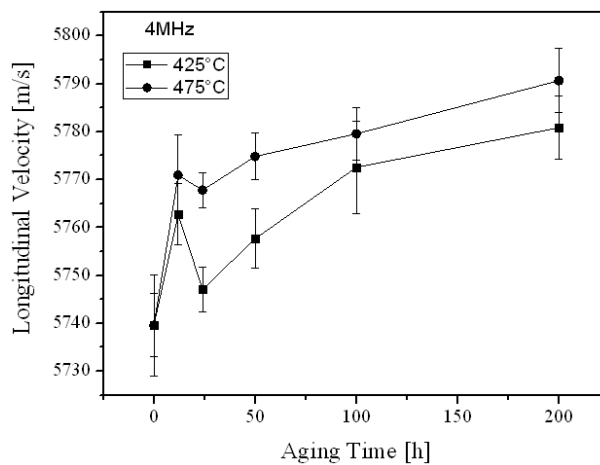
**Figure 2b**



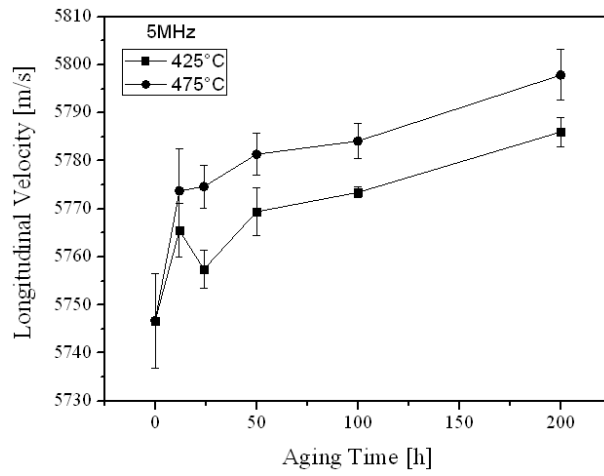
**Figure 3a**



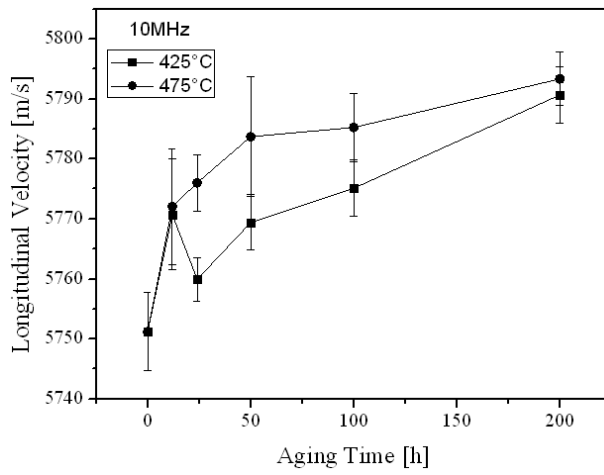
**Figure 3b**



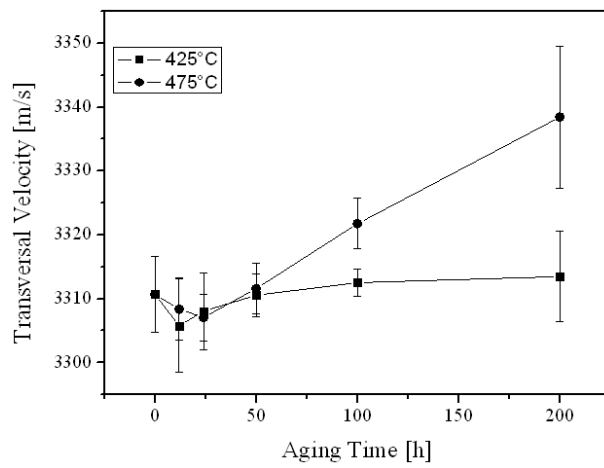
**Figure 4a**



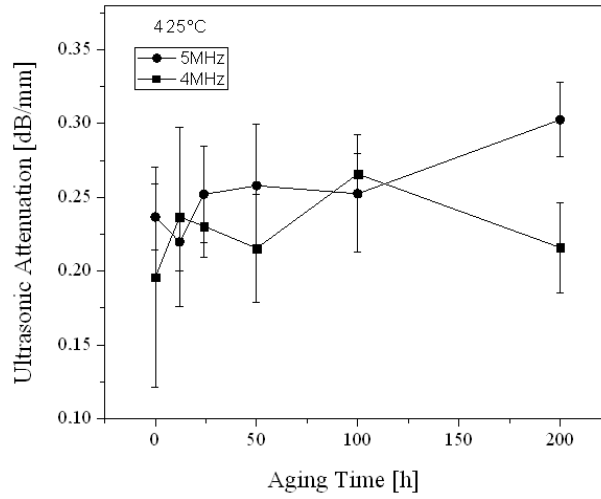
**Figure 4b**



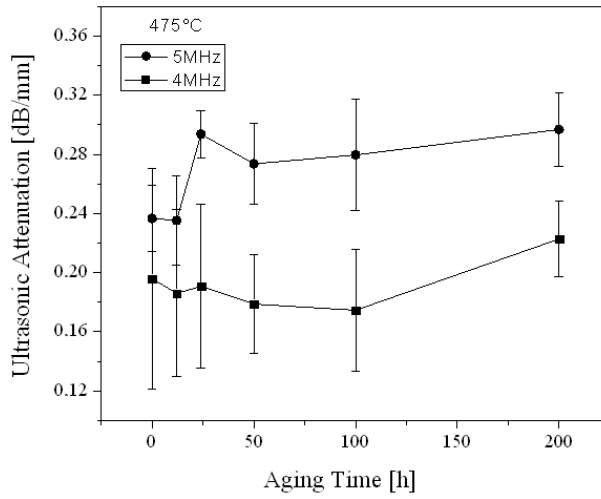
**Figure 4c**



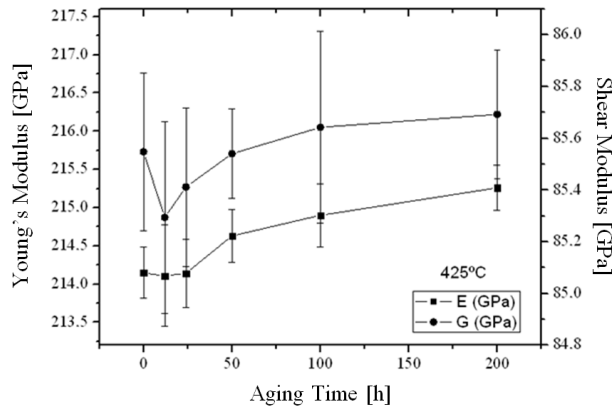
**Figure 4d**



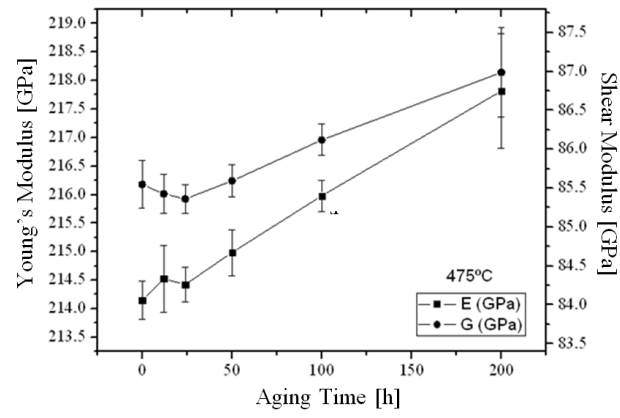
**Figure 5a**



**Figure 5b**



**Figure 6a**



**Figure 6b**

## TABLES

Table 1

Duplex Stainless Steel SAF 2205							
C	Mn	P	S	Si	Cr	Ni	Co
0.018	1.480	0.019	0.001	0.450	22.220	5.590	0.130
Cu	Mo	N	Nb	Al	Sn	Ce	Fe
0.280	3.080	0.180	0.021	0.003	0.012	0.020	66.496