Characterization of Duplex Stainless Steel Heat – Treated at 1300°C

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Abstract- Attenuation characteristics and ultrasonic velocity of duplex stainless steel test coupons held at 1300°C for 15, 30, 60 and 90 minutes period and later water quenched were found to be strongly influenced by the microstructural features. Attenuation increases with increase in grain size while longitudinal velocity is influenced by both the austenite content and the grain size. Elastic constants show better correlation with shear wave velocity than longitudinal velocity. However, for bulk modulus correlation coefficient (R) is 0.8 while for other elastic constants (R) value is more than 0.9.

Index Terms- Duplex stainless steel, Heat-treatment, Microstructure, Ultrasonic characterization, Elastic constants.

I. INTRODUCTION

A Large variety of stainless steel materials are needed in industry. Process and quality control of stainless steels to achieve the desired properties become important since competition in industry demands improved performance and longer service life. For many engineering applications Duplex Stainless Steels (DSS) are preferred because of their improved corrosion resistance and mechanical properties [1–2]. The workhorse in DSS is characterized by is chemical composition comprising chromium (~22%), molybdenum (~3%) and nitrogen (~0.18%). Due to their increased chromium and molybdenum content, DSSs offer excellent corrosion resistance in many media. DSSs have two-phase microstructure of ferrite (α) and austenite (γ) in which austenite phase is distributed as coarse region in a ferrite phase matrix [3]. Simultaneous presence of ferrite and austenite phases provides a combination of the best properties of these two phases; better corrosion resistance than austenitic stainless steel and better mechanical properties than ferritic stainless steel. Because of their superior characters, DSS are being successfully used in many fields such as structural material in critical components of nuclear power plants, chemical industries, pulp and paper industry digesters, food processing equipment, piping, tubing, and heat exchangers for the handling of gas and oil, transportation and other general engineering applications [4-6]. These advantageous are based on a special equilibrium of both phases having near equal volume i.e., $\alpha \approx 50\%$ and $\gamma \approx 50\%$ [7-9]. The phase balance in DSS is obtained by careful heat treatment as it is crucial for the mechanical properties and corrosion resistance. However, the material properties can be degraded by improper or inadvertent heat treatment. Therefore inspection of heat treated parts is an efficient way to prevent and reduce failures in industrial plants. For such applications Non-Destructive Testing (NDT) methods can offer large potential in microstructural characterization and evaluation of mechanical properties of these materials. Among the various NDT methods, ultrasonic technique can provide valuable information about the microstructures, mechanical properties, thermo-mechanical history of the material, etc. [10]. There are several studies correlating ultrasonic velocity and attenuation to microstructure of steels [1, 11–15], i.e., phases present in the microstructure and their morphology, grain size distribution, etc. Ultrasonic parameters (longitudinal & shear velocity) are also used for evaluation of mechanical properties and elastic moduli of many engineering materials [16-17].

In the present study, DSS test coupons are heat treated at 1300°C for 15, 30, 60 and 90 minutes, followed by water quenching. Longitudinal velocity and attenuation of ultrasonic are correlated to the variation in microstructure resulted due to heat treatment. Elastic constants of these heat treated DSS test coupons were also estimated from the ultrasonic longitudinal and shear wave velocities. Correlation between the ultrasonic velocities and elastic constants was also studied.

II. MATERIALS AND METHODS

SAF2205 duplex stainless steel of chemical composition shown in Table 1, received in the form of 5mm thick sheet was cut into coupons of 60mm X 50mm X 5mm. Test specimens were heated in an INDFUR electric muffle furnace to 1300°C ± 1°C and held at this temperature for 15, 30, 60 and 90 minutes followed by water quenching. No special protective environment was employed during heat treatment.

Table 1: Chemical composition of the duplex stainless steel wt. %)

Element	C	S	P	Si	Mn	Cr	Ni	Mo	N
wt %	0.02	0.02	0.03	0.6	1.4	22.2	5.9	2.9	0.15

Samples for metallographic studies were cut from the heat treated specimens using BAINCUT – M abrasive cut off machine taking care not to raise the temperature of test specimens while cutting. Metallographic samples were prepared by following the standard mechanical polishing practice. Polished specimens were then etched by immersion in Beraha's colour etchant (HCl -

20ml, H_2O - 80ml, potassium meta bi sulphite - 0.3 to 0.5mg). Microstructural study was carried out in a METSCOPE–I microscope and the images were captured using Envision 3.0 series image analyzer.

Ultrasonic tests were accomplished by the contact pulse-echo method in an Olympus Panametrics NDT Model 5800 unit using 5MHz longitudinal and shear wave probes at room temperature. A proprietary gel was used as couplant. Ultrasonic velocity was determined by measuring the time taken for the ultrasonic waves to travel through thickness of the material between the parallel faces and can be estimated from the relationship:

$$Velocity\ (m/s) = 2 * thickness\ (m) / time\ (s) \quad (1)$$

Attenuation co-efficient is measured from the logarithmic decrement of amplitude (dB) between the two consecutive back wall signals and then dividing it by the total path travelled. It is expressed in terms of dB/mm. Attenuation measurements were made using 5MHz longitudinal wave transducer. Attenuation co-efficient was calculated according to the relationship [14-15]:

$$Attenuation\ co\text{-}efficient\ (dB/mm) = 20 \log (S_1/S_2) / 2d \quad (2)$$

where S_1 and S_2 are the amplitude of two consecutive back wall echoes, and d is the thickness of test material in mm.

Ultrasonic longitudinal (V_l) and shear wave (Vs) velocities, and density (ρ) of material were used to calculate the four different elastic constants viz., Young's modulus (E), shear modulus (G), bulk modulus (K) and Poisson's ratio (ν) from the relations given as follows [16-18] :

$$E = [\rho\ Vs^2\ (3\ V_l^2 - 4\ Vs^2)] / (V_l^2 - Vs^2) \quad (3)$$
$$G = \rho\ Vs^2 \quad (4)$$
$$K = \rho\ (3\ V_l^2 - 4\ Vs^2) / 3 \quad (5)$$
$$\nu = (V_l^2 - 2\ Vs^2) / [2\ (V_l^2 - Vs^2)] \quad (6)$$

Density of DSS was assessed to be 7837 kg/m^3 using a density apparatus that works based on Archimedes's principle and this value is used in elastic constant calculations.

III. RESULTS AND DISCUSSION

The microstructural details developed in the DSS by the heat treatment cycles imposed and the corresponding variation in ultrasonic attenuation and longitudinal velocity are presented and discussed. The elastic constants were determined by using both longitudinal and shear wave velocities.

3.1. Microstructural details

The changes in microstructure due to thermal cycles can be explained and best understood from the pseudo binary diagram of stainless steels for 70% Fe-Cr-Ni [19] which is shown as Fig. 1. Duplex stainless steels primarily solidify as ferrite when they are cooled from liquid state. Above the solvus line only ferrite (α) is present. Upon cooling, below the solvus, part of the ferrite transforms into austenite resulting in two phase (α + γ) structure. Equilibrium slow cooling generally results in formation of chromium rich σ - phase. σ - phase is undesirable because of its embrittling nature and poor corrosion resistance. In order to avoid formation of σ - phase along with other undesirable secondary phases and to retain near equal volume of ferrite and austenite, usually DSSs are rapidly cooled from around 1050°C. Typical microstructure of a rolled duplex stainless steel is shown

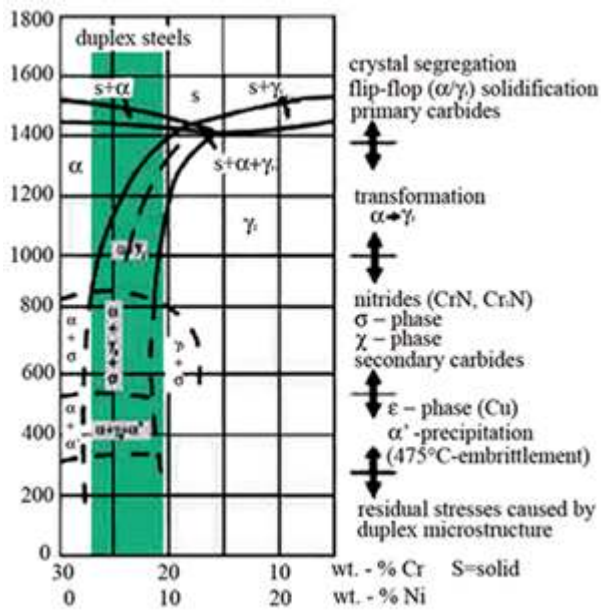

Figure 1: Pseudo binary diagram of duplex stainless steel for 70% Fe-Cr-Ni [19]

in Fig. 2. The microstructure shows banded structure with austenite islands in a continuous ferrite matrix. The dark regions are ferrite while the light etched or bright regions are austenite. Banded structure observed in the microstructure is characteristic of DSS rolled products.

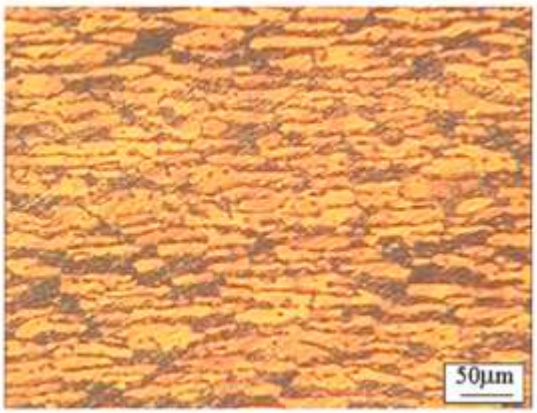

Figure 2: Optical micrograph of duplex stainless steel in as-received condition. Dark etched regions are ferrite and white regions are austenite.

Influence of increase in solution treatment temperature but below the solvus line (1300°C) there is a progressive increase in ferrite content. Microstructural changes occurred in the DSS specimens at 1300°C for different exposure period can be seen from the microstructures shown in Fig. 3. Because of increase in solution treatment temperature more ferrite and austenite is

coarsened. Most of the metallurgical transformations are diffusion controlled and therefore they are time dependent. From Fig.1 it can be seen that equilibrium amount of ferrite at 1300°C is more than that at 1050°C. When DSS is heated to 1300°C, dissolution of austenite in ferrite continues to increase with dwell time and finally reaches equilibrium. Holding the specimen at 1300°C for 15 minutes resulted in dissolution of austenite to a great extent (see Fig.3a). However, presence of undissolved elongated austenite can still be seen. Growth of ferrite grains are also outlined by the thin grain boundaries. (While observing the

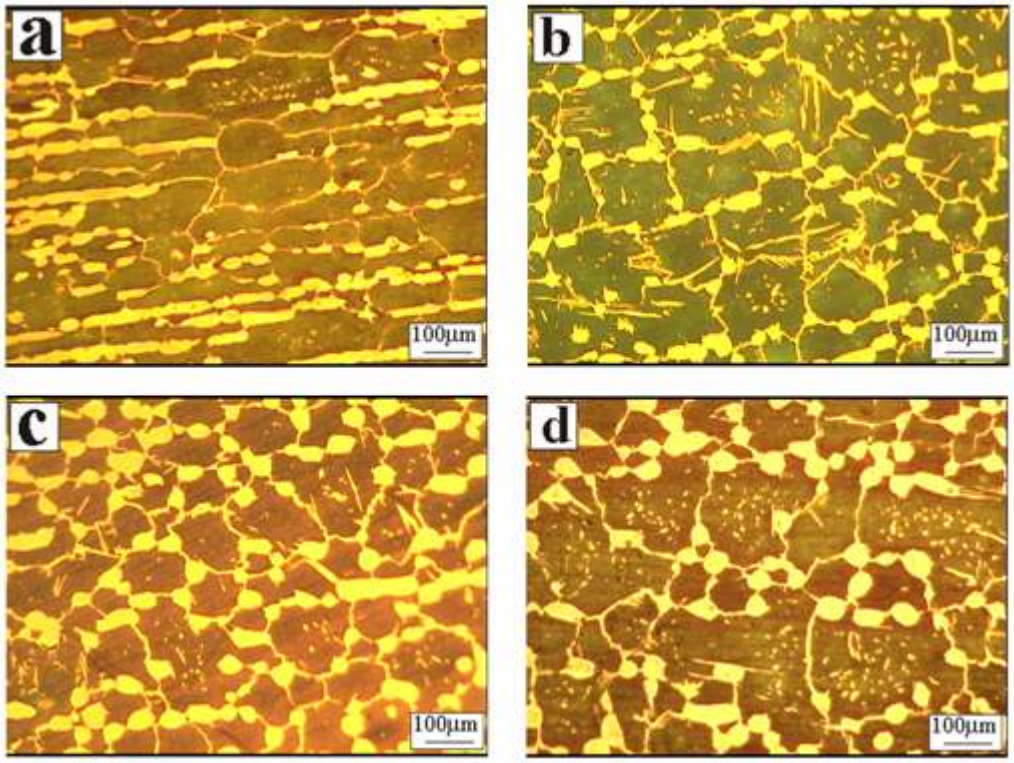

Figure 3: Microstructure of duplex stainless steel held at 1300°C for different duration,
(a) 15min (b) 30min (c) 60min (d) 90min and water quenched

microstructures it may be borne in mind that the high temperature microstructures are retained at room temperature by resorting to rapid cooling. Depending upon the nature of the alloy some transformation product can also form during rapid cooling or quenching. Duplex stainless steels can develop grain boundary austenite, widmanstätten austenite and intragranular austenite, while quenching them from high temperature. In the present study only small amount of intragranular austenite is seen. However, the amount of austenite that would have been present at 1300°C would be less than that as seen in the microstructures recorded at room temperature). Intragranular austenite that are seen as bright spots within the grains, are formed while cooling the specimens from 1300°C to room temperature. With prolonged holding at 1300°C more amount of austenite is dissolved in ferrite. Presence of coarse austenite even after 90 minutes holding indicates that 1300°C is below the solvus line for the given composition. Further it can be seen from Fig. 3, at 1300°C the equilibrium could be established within 30 minutes duration. Additional holding time results in grain growth without any change in austenite content. This can also be confirmed by the average grain diameter values estimated by intercept method which is presented in Table 2.

Table 2: Effect of holding time at 1300°C on grain size of duplex stainless steel

Holding time (minutes)	15	30	60	90
Average grain diameter (µm)	94	112	126	134

3.2 Attenuation

Attenuation refers to the loss of sound energy as the ultrasonic beam passes through the material by the combination of scattering and absorption. As described earlier attenuation is measured as logarithmic decrement between the two consecutive back wall echoes. The attenuation characteristics of the DSS specimens heat-treated at 1300°C for different durations are presented in Fig. 4.

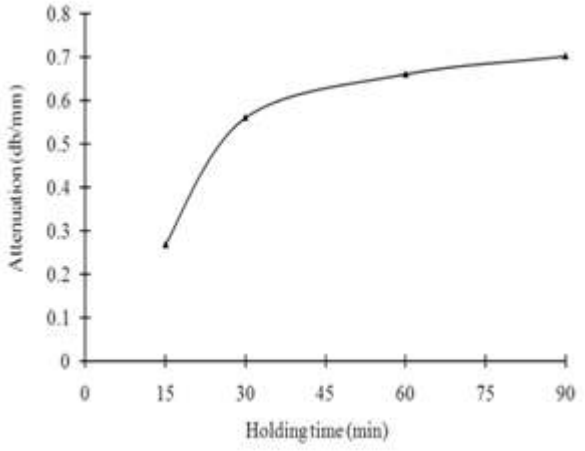

Figure 4: Dependence of attenuation coefficient of DSS on holding time at 1300°C

As the holding time increases at 1300°C, austenite dissolution in ferrite is promoted and simultaneous grain growth takes place. Fig. 5 shows that grain size increases monotonously with time at 1300°C. It can be seen from Fig. 4 attenuation also increases with increase in holding time. However, a steep rise in attenuation in the initial period is noticed, which can be attributed to growth of grains from the original fine grain size of the base material to coarse equilibrium grain size at 1300°C. From this it can be concluded that attenuation is mainly decided by the grain size and the influence of relative amount of phases and their morphology is insignificant. Dependence of attenuation in ferritic steels primarily on grain size has also been reported by Anish kumar and co-workers [20].

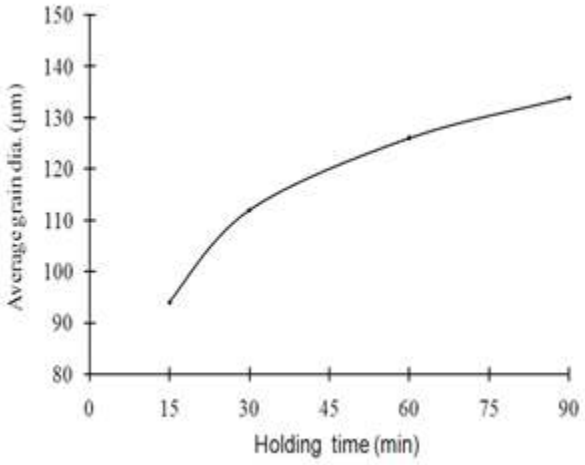

Figure 5: Effect of holding time at 1300°C on average grain size of DSS

3.3 Ultrasonic longitudinal velocity

Microstructure of an alloy decides the ultrasonic longitudinal velocity in it. The trend in change in ultrasonic velocity in DSS heat-treated at 1300°C is shown in Fig. 6. Ultrasonic longitudinal (V_l) velocity in the DSS material in as-received condition, which is characterized by near equal volume of ferrite and austenite, and fine grained structure is V_l = 5347 m/s.

The longitudinal velocity as shown in Fig.6, increases steeply at the beginning and decreases sharply with an increase in the holding time. Subsequent change in velocity with the increase in holding time is only marginal. When DSS is heated from room temperature to 1300°C expected changes in microstructure are grain growth and increase in ferrite content. However these two transformations are diffusion controlled and therefore time dependent. Kinetics of these two transformations is rapid to start with and levels off with increase in holding time at 1300°C. It has been reported that sound velocity decreases with increase in the grain size as coarse grain size causes ultrasonic waves to take a longer path to cover the material thickness and thereby decreases the sound velocity drastically [1, 21]. Therefore initially the sound velocity is expected to decrease since rapid grain coarsening takes place at 1300°C. Contrary to this sound velocity was found to increase steeply in the initial period. The other parameter that works against the influence of grain growth is increase in ferrite content by the dissolution of austenite. From this it can be inferred that the ferrite content in DSS has

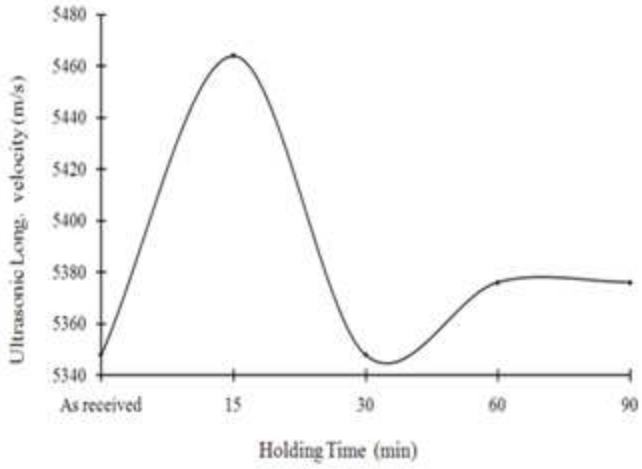

Figure 6: Variation in longitudinal wave velocity in DSS with holding time at 1300°C

stronger effect than the grain size in deciding the longitudinal velocity and also the two parameters have opposite effect on longitudinal velocity of sound. Therefore in the start of the experiment, longitudinal velocity increased with reduction in austenite even though grain coarsening reduced the velocity. With the holding time up to 90 minutes period at 1300°C the grain size continues to increase while austenite to ferrite transformation levels off. At one point of time the effect of grain size on longitudinal velocity dominates the influence of ferrite content and then onwards longitudinal velocity starts decreasing. Towards the end though the grains size continues to increase a slight reduction in ferrite content causes the longitudinal velocity to be more or less at the same value after the holding time of 60 minutes 1300°C.

3.4 Elastic constants

Ultrasonic inspection uses high frequency elastic waves to nondestructively inspect the materials. The most frequent of application of ultrasonics in material property measurement involves the study of elastic constants and related strength properties [22]. The elastic deformation can be quantified by elasticity and Poission's ratio. Additionally elastic stiffness constants are used to fully define the elastic behavior of a material [23]. Elastic constants for duplex stainless steel samples under analysis were obtained from the velocities of ultrasound that were assessed with the contact pulse-echo method at a frequency of 5MHz. These results are summarized in Table 3.

Table 3: Elastic constants of the DSS thermally soaked at 1300^{o}C for up to 90 minutes

Holding time (min)	Young's modulus (GPa)	Shear modulus (GPa)	Bulk modulus (GPa)	Poisson's ratio
As received	185	741	125	0.25
15	195	780	129	0.24
30	170	662	135	0.29
60	196	800	119	0.22
90	189	760	125	0.24

The relationship between elastic moduli and ultrasonic wave velocities was studied for 5 DSS specimens subjected to different thermal treatments. Figs. 7 to 10 shows the variation in ultrasonic wave velocities with Young's modulus (E), shear modulus (G), bulk modulus (K) and Poisson's ratio (ν), respectively. From the figures it can be seen that correlation between velocity and Young's modulus (E) or shear modulus (G) has a positive slope indicating that both E and G values increase with increase in the ultrasonic velocity while respective graph for bulk modulus (K) and Poisson's ratio (ν) show a negative slope meaning that these two parameters are inversely proportional to the ultrasonic velocity.

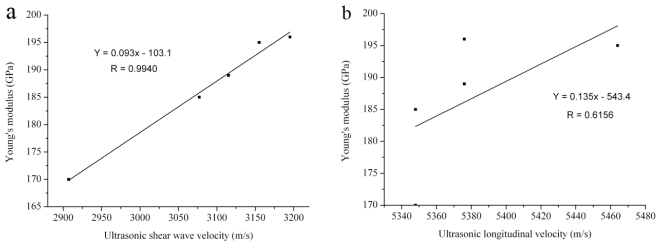

Figure 7: Correlation between (a) Young's modulus and ultrasonic shear wave velocity (b) Young's modulus and ultrasonic longitudinal wave velocity, in DSS

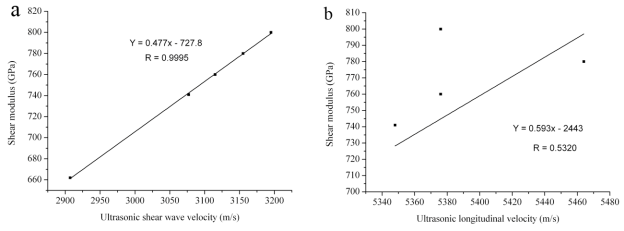

Figure 8: Correlation between (a) shear modulus and ultrasonic shear wave velocity (b) shear modulus and ultrasonic longitudinal wave velocity, in DSS

Further these graphs also show that elastic constants can be linearly correlated to ultrasonic velocities (Vs) and (V_l). However, in all the cases it is found that shear wave velocity gives a better correlation than longitudinal velocity. From the figures 7 and 8, the Young's modulus has a correlation co-efficient (R) of 0.9940 for shear velocity and 0.6156 for longitudinal velocity. For shear modulus correlation co-efficient values are 0.9995 and 0.5320 for shear wave velocity longitudinal wave velocity, respectively. Similar trend can be seen from Fig. 9 and 10 for Bulk modulus and Poisson's ratio with respect to correlation coefficient for longitudinal and shear wave velocities. Anish Kumar et al. have also reported that shear wave velocity show better correlation than longitudinal velocity with the elastic constants for isotropic solid materials [24].

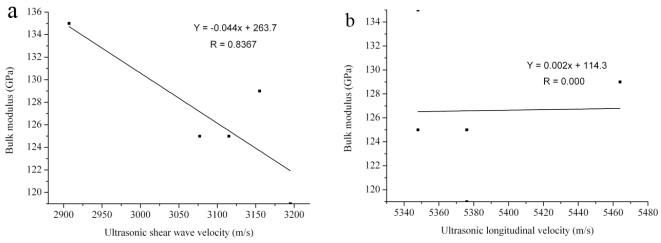

Figure 9: Correlation between (a) Young's modulus and ultrasonic shear wave velocity (b) Young's modulus and ultrasonic longitudinal wave velocity, in DSS

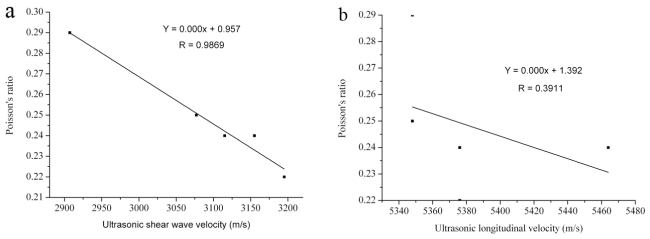

Figure 10: Correlation between (a) Poisson's ratio and ultrasonic shear wave velocity (b) Poisson's ratio and ultrasonic longitudinal wave velocity, in DSS

IV. CONCLUSION

A systematic approach to understand the relationship between the microstructure of a DSS heat treated at 1300°C and ultrasonic sound velocity and attenuation was applied.

The results show that the attenuation is mainly decided by the grain size and attenuation increases with increase in grain size. But the ultrasonic velocity is found to be dependent both on ferrite-austenite ratio and the grain size. The correlation studies on ultrasonic longitudinal and shear wave velocities with elastic constants for this DSS indicate that ultrasonic shear wave can be used for materials characterization as shear wave velocity has better correlation as compared to longitudinal wave velocity.

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