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CHARACTERIZATION OF MICROSTRUCTURE AND DETERMINATION OF ELASTIC PROPERTIES IN SAF 2205 DUPLEX STAINLESS STEEL USING ULTRASONIC MEASUREMENTS

R. Jayachitra*

Department of Physics, Urumu Dhanalaksmi College, Tiruchirappalli, Tamil Nadu, India

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ABSTRACT

Microstructural changes in duplex stainless steel due to changes in annealing temperature are characterized by ultrasonic pulse-echo technique and optical microscopy. Duplex stainless steel was subjected to a series of heat treatments from 1100°C to 1350°C, followed by water quenching. The microstructure after heat treatment at 1350°C resulted in coarse grained ferrite, which favored formation of Widmanstatan austenite with fast cooling. Micro hardness and optical microscopy results are correlated with ultrasonic longitudinal and shear wave velocities, attenuation and ferrite count (%) in the specimens. Ultrasonic velocities and micro hardness decrease with annealing temperature in the 1100°C - 1200°C range, while they increase slightly beyond 1200 up to 1350°C. Ultrasonic attenuation exhibits an opposite behavior to velocity and hardness. Fast Fourier Transform exhibit a decrease of the spectral amplitude in specimens with high heat treatment temperature. The results show that the use of Ultrasonic measurements to correlate the ultrasonic parameters with the microstructures and hardness is very fast and reliable.

KEYWORDS: Duplex stainless steel, Microstructure, Micro hardness, Ultrasonic characterization, FFT.

INTRODUCTION

In the recent times a number of new materials have been developed to the needs of the different industrial sectors. Stainless steels have been playing a major role in the areas of chemical and petrochemical processing industries, nuclear power industries etc., owing to their excellent properties, especially corrosion resistance and mechanical properties. 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 its chemical composition comprising of chromium (~22%), molybdenum (~3%) and nitrogen (~0.18%). DSS are an intermediate class between ferritic and austenitic stainless steels. DSS having two phase structure (α) ferrite and (γ) austenite. Simultaneous presence of ferrite and austenite phases provides a combination of the best properties of these two phases, like good corrosion resistance and improved mechanical properties [3,4]. DSS are employed for a wide variety of applications as structural material in critical components of nuclear power plants, chemical industries, oil and gas sectors, paper and pulp industries, transportation and other general engineering applications because of higher strength, superior resistance to stress corrosion cracking and better weld ability [5,6]. The outstanding properties of DSS are mainly due to the phase balance i.e., $\alpha \approx 50\%$ and $\gamma \approx 50\%$, [7,8,9]. The phase balance in DSS is obtained by careful heat treatment as it is crucial for the mechanical properties and corrosion resistance. However for large industrial applications of duplex stainless steels the base metal is subjected to a series of thermal cycles. As a result, complex microstructural transformations occur, affecting the α/γ phase balance in the steel. In the heat affected zone, the microstructure undergoes both rapid heating and cooling cycles, which drive the α/γ transformation to varying levels of completion [10].

In-service degradation with heat treated parts is one of the most critical factors determining the structural integrity of the elevated-temperature components in power plants, chemical plants, and oil refineries the world over. They undergo progressive damage over time. To save energy and improve thermal efficiency, the steam pressures and operating temperatures in the components have been increased. As a consequence, material degradation is



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accelerating. Therefore, an in-service assessment of the state of the damage is important for ensuring safe operations, predicting remaining life, and promoting life-extending programs. For this assessment, a nondestructive technology (NDT) have been recently gaining more importance due to the rapid development in technology and due to several types of materials and systems that could be tested without causing any damage or destruction. Among the various NDT methods, ultrasonic technique can provide valuable information about the microstructures, mechanical properties, thermo-mechanical history of the material, etc. Moreover, microstructure variations cause the scattering of the sound wave causing an increased attenuation of the material. This technique's field of operation depends on the knowledge of the material's sound attenuation coefficient. The propagation of ultrasonic wave in polycrystalline materials are subject to interact with microstructural components such as specks, inclusions, porosities, microcracks, corrosion, among others, causing sound attenuations and variation in the speed of sound propagation on the material in analysis [11]. The wave propagation speed and energy losses through interactions with the microstructure are the fundamental factors of the material's ultrasonic characterization [12,13]. There are several studies correlating ultrasonic velocity and attenuation to microstructure of steels [1,14-17], i.e., phases present in the microstructure and their morphology, grain size distribution, etc. This investigation undertakes an interdisciplinary research to enhance NDT capabilities to assess microstructure and mechanical characteristics of SAF 2205 duplex stainless steel. The grain size of a material is an important engineering parameter which influences its mechanical properties such as fatigue, creep, yield strength etc. [14,18], have proposed a new approach to the degree of sensitization of the AISI 304 stainless steel. Ultrasonic parameters (longitudinal & shear velocity) are also used for evaluation of mechanical properties and elastic moduli of many engineering materials [19,20]. Studies made on SAF 2205 duplex stainless steel using the new approach indicated that the average grain size could be correlated with combined measurements of attenuation and amplitude on the Fast Fourier Transform(FFT). These studies have been mainly focused on identification of small microstructural changes with strong effect on the chemical and mechanical properties.

In the present study, ultrasonic parameters have been employed to characterize various microstructural features in SAF 2205 duplex stainless steel. In order to generate specimen with different microstructures the DSS was heat treated at 1100, 1200, 1300 or 1350°C for 30 minutes, in an electrically heated muffle furnace, followed by water quenching. The changes in ultrasonic velocities are able to characterize the micro hardness variation with phase transformation due to heat treatment. The effect of heat treatments on microstructures, grain size and mechanical properties are investigated through the measurement of ultrasonic wave propagation technique and Fast Fourier transform spectral analysis. Elastic constants of these solution treated DSS test specimens were also estimated from the ultrasonic longitudinal and shear wave velocities, and correlation between the ultrasonic velocities and elastic constants were investigated.

EXPERIMENTAL PROCEDURE

Specimen preparation and heat treatment

The chemical composition of SAF 2205 duplex stainless steel is given in Table-1. Five specimens of DSS type SAF2205 were obtained in the form of 5mm thick sheet were cut in to coupons of 60mm x 50mm x 5mm. Corrosion and Mechanical properties of DSS are more decided by the microstructure and relative amount of the phases present than on the composition of the alloy. In turn, microstructure and phases present are governed by the heat treatment cycle [21]. Test specimens were heated in an INDFUR electric muffle furnace to temperatures 1100, 1200, 1300 (or) 1350°C \pm 1°C for 30 minutes followed by water quenching. No special protective environment was employed during heat treatment. Samples for metallographic studies were cut from the heat treated specimens using BAINCUT-M abrasive cut off machine. Care was taken not to raise the temperature of test specimens, while cutting as it might affect the microstructure. The silicon carbide paper was used in achieving the proper grinding of the specimen.

Table 1. Chemical Composition of SAF 2205 type Duplex Statiless Steel (Wi. %)									
Element	С	S	Р	Si	Mn	Cr	Ni	Mo	Ν
Wt %	0.02	0.02	0.03	0.6	1.4	22.2	5.9	2.9	0.15

Table 1. Chemical Composition of SAF 2205 type Duplex Stainless Steel (Wt. %)



The polishing was done on polishing cloth using alumina powder dissolved in water at a reasonable proportion. Light pressure was applied until the surfaces were free of scratches. Polished specimens were then etched by immersion in Beraha's colour etchant (HCl - 20ml, H₂O - 80ml, potassium meta bi sulphite - 0.3 to 0.5mg). The samples were cleaned, dried and then examined under the microscope. Microstructural study was carried out in a METSCOPE–I microscope and the images were captured using Envision 3.0 series image analyzer. The ferrite percentage was measured by Fisher Feritescope MP30.

Micro hardness Measurements

Vicker's hardness of all the specimens was measured using a Zwick hardness tester at a test load of 1000g at room temperature. A diamond indenter was used to make an indentation on the polished surface of the specimen. For each specimen, the hardness number was calculated from at least five indentations and the average value is reported. The following equation is used for the estimation of the hardness.

Vicker's hardness number, VHN =
$$1.854 \text{ P/d}^2$$
 (1)

where d is the length of diagonal of the indentation (in mm) and P is the applied load (in kg)

Ultrasonic Velocity Measurements

Ultrasonic testing parameters are significantly affected by changes in microstructural (or) mechanical properties of materials. The contact pulse-echo technique was used to obtain the parameters of ultrasonic velocity and attenuation at room temperature using an Olympus Panametrics NDT Model 5800 unit .In this method, the transducer operated at a fundamental frequency of 5MHz longitudinal and shear wave probes. The experiment setup is shown in Figure-1. The transducer was placed in contact with the specimen with a thin layer of ultrasonic gel couplant to ensure proper coupling of energy to the specimen.



Figure 1. Schematic of the experimental set-up for ultrasonic measurement

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) (2)

The ultrasonic attenuation co-efficients were measured based on the reduction of the amplitude of an ultrasonic pulse. 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, Attenuation co-efficient (dP/mm) = $20 \log (S_1/S_2)/2d$ (3)

Attenuation co-efficient (dB/mm) = $20 \log (S_1/S_2) / 2d$ (3)

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.



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Ultrasonic longitudinal (V₁) 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 (v) from the relations given as follows [22,23]:

(4)
(5)
(6)
(7)

Density of DSS was assessed to be 7837 kg/m³ using a density apparatus that works based on Archimedes's principle and this value is used in elastic constant calculations.

RESULTS AND DISCUSSION

The microstructural details developed in the DSS by the heat treatment cycles are imposed, and the corresponding variation in ultrasonic attenuation and longitudinal velocity are presented and discussed. 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 ($\alpha + \gamma$) 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. SAF 2205 duplex stainless steels are heat -treated at 1050°C to obtain near equal amount of ferrite and austenite, and followed by quenching in water to avoid formation of intermetallics and secondary phases that will have adverse effect on toughness and corrosion resistance. Influence of increase in solution treatment temperature on the resultant microstructure can be explained based on Fe-Cr-Ni pseudo binary diagram [5] for 70% Fe, as shown in Fig. 2(a). Typical microstructure of DSS in the as-received condition is shown Fig. 2(b). 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.

When the heat-treatment temperature is above 1050° C but below the solvus line there is a progressive increase in ferrite content which can be seen from Fig. 2(c) and (d) depicting microstructure of DSS solution treated at 1100° C and 1200° C, respectively. At temperatures above the solvus line only single phase i.e., α is present and with cooling some amount of austenite can be formed along the grain boundaries Fig. 2(e). Higher solution treatment temperature viz, 1350° C resulted in a coarse grained ferrite that favored formation of austenite in specific planes, with fast cooling, known as Widmanstatten austenite and this structure is shown in Fig. 2(f). Thus the heat treatment cycles could result in microstructures of different ferrite-austenite ratio, morphology and varying grain size.





Figure. 2 (a) 70% Fe–Cr–Ni pseudo binary diagram. Micro structural aspect of DSS after heat treatment; (b) M1, (c) M2, (d) M3, (e) M4 and (f) M5.

Ultrasonic waves in a polycrystalline material are attenuated by scattering at structural boundaries and absorption due to dislocation damping, thermo elastic loses, and magnetic hysteresis loss, [13]. One of the ultrasonic parameter is signal strength or the peak height of half cycle having maximum amplitude, called echo height. The echo height depends upon the solution treatment temperature very appreciably. Ultrasonic attenuation characteristics and ferrite count in DSS solution treated at different temperatures is plotted as a graph in Fig. 3.



Figure. 3 Variation in ultrasonic attenuation and ferrite number with different heat treated temperatures for DSS.



It can be seen from the graphs, that the attenuation is nearly the same for DSS in the as-received condition and for DSS heat-treated at 1100°C and 1200°C. But attenuation started increasing steeply after 1200°C i.e., for 1300°C and 1350°C. When DSS is heated above the solvus line, it is taken into a single phase region where it experiences grain coarsening even for small further increase in temperature. This significant increase in ultrasonic attenuation is attributed to the formation of fully ferrite microstructure at higher heat treatment temperature, and a significant increase in grain size was observed. Ultrasonic attenuation measurements are influenced by grain size distribution, and usually a small number of large grains may totally change the attenuation. However, a steep rise in attenuation in the initial period is noticed, which can be attributed to the growth of grains from the original fine grain size of the base material to coarse equilibrium grain size at 1300°C. [13] have shown in their studies on ferritic steels that attenuation in creases with increase in grain size. Similar results are seen in our studies also, and therefore it is evident that attenuation in heat-treated DSS is decided by the heat treatment temperature; attenuation remains more or less constant for temperature within two phase ($\alpha \ll \gamma$) region, but increases rapidly with increase in temperature in the single α phase region. This effect is mainly due to changes in grain size.

Figure 4(a) and (b) show the first backwall echo and its FFT spectrum for the DSS heat treated at different temperature, respectively. It is clear from the FFT, that there is a sharp decrease in amplitude from the base material to heat treated material as seen in Fig. 4(b). As the grain size increases more attenuation takes place and the ratio of peak height changes, due to increase in the attenuation at higher heat treatment temperature.



Figure. 4 Variation in (a) first backwall echo and (b) FFT spectrum with different heat treated temperatures for DSS.

Fig. 5 shows the effect of heat treatment on hardness and ferrite percentage of DSS. The hardness of the material decreases with increasing heat treatment temperature [24]. For the sample heat treated below solvus line, the hardness decreases in the α -phase region. As the heat treatment temperature increases, the hardness increases, which are attributed to the increase in the amount of harder phase (Ferrite) formed. This increase is due to solid solution hardening and arises from the slight differences in size of the nickel, iron and chromium atoms [25]. The solid solution hardening of Ni and Mo of the ferrite phase, the internal strain hardening between ferrite and austenite due to different coefficient of thermal expansion, and few inter-diffusion of alloying elements during heat treatment at particles boundary might be a few explanation for why the mechanical properties of DSS increase as compared with a mono phase austenitic stainless steel [27]. Above 1300°C, the grain size increased rapidly, a lower hardness was observed with a further increase in temperature. [26], reported that the microhardness of ferrite is higher than that of austenite. The effect of microstructure on mechanical properties is dominated by the amount of ferrite phase present. The ferrite content and its morphological parameters as related to heat treatment temperature are summarized in Table 2. The ferrite content increases in the material with increasing temperature, as indicated by the pseudo-binary diagram of the Fe-Cr- Ni system. In this diagram, the composition of the alloy is displayed in terms of chromium and nickel equivalents, which relate to the relative power of the various alloying elements as α and γ stabilizers.



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 Table 2. Ultrasonic velocity, attenuation, ferrite count and hardness of SAF2205 duplex stainless steel at

 different heat treatment temperatures.

				Longitudinal	Shear wave	Attenuation	
Specimen	Hoat traatmont	Microhardness	Ferrite No.	wave	velocity at	coefficient	
	meat treatment	(VHN)	(%)	velocity at	5MHz (m/s)	at 5MHz	
				5MHz (m/s)		(dB/mm)	
M1	None	306	55.06	5347	3077	0.2116	
M2	1100 °C/30 min/WQ	262	59.33	5263	3086	0.2497	
M3	1200 °C/30 min/WQ	259	86.12	5236	3125	0.2279	
M4	1300 °C/30 min/WQ	260	92.06	5291	2873	0.3522	
M5	1350 °C/30 min/WQ	267	97.36	5208	2483	0.8943	



Figure. 5 Variation in hardness and ferrite number with solution treated temperature.

Ultrasonic velocity and micro hardness changes in DSS heat treated at different temperature are characterized with 5MHz longitudinal and shear wave transducer and the trend is given in Fig. 6 (a) and (b). For DSS solution treated at temperatures in the two phase region decrease in longitudinal velocity and hardness is seen with increase in temperature, while the shear velocity remains almost constant.



Figure. 6 Variation in ultrasonic (a) longitudinal (b) shear wave velocity and micro hardness with solution treatment temperature.

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. When the solution-treatment temperature is above the solvus, initially fine ferrite grains are formed and they grow in their size with increase in temperature. Fine grain ferrite formed just above the solvus temperature resulted in an increase in the longitudinal velocity followed by reduction in both longitudinal and shear velocity with

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grain coarsening as the solution treatment temperature in the single phase region is increased. The sharp decrease in the shear velocity at 1300°C is attributed to the increase in the grain size with increase in temperature, which is reflected in the variation of the hardness also. 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 [18,28,29], reported that the ultrasonic velocity could follow the formation of α phase in SAF 2205 DSS. [30] reported the correlation between microstructure, ultrasonic attenuation and hardness of tempered martensitic stainless steel. It is evident from this observation that the best mechanical and ultrasonic characteristics of DSS are obtained, after heat treatment, in which all the secondary phases-carbide and intermetallic –originating from the as cast microstructure are dissolved at high temperatures. This result shows that the ultrasonic velocity in solution-treated DSS is influenced both by the phase content and the grain size.

The most frequent application of ultrasonic in material property measurement involves the study of elastic constants and related strength properties [31]. 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 [32]. Elastic constants for duplex stainless steel samples under analysis were obtained from the ultrasonic velocities that were assessed with the contact pulse-echo method at a frequency of 5MHz. These results are summarized in Table 3. 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 (v), respectively. From the figures 7 and 8, 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 (v) show a negative slope, meaning that these two parameters are inversely proportional to the ultrasonic velocity. Further these graphs also show that elastic constants can be linearly correlated to ultrasonic velocities (Vs) and (V₁). However, in all the cases it is found that shear wave velocity gives a better correlation than longitudinal velocity. The Young' modulus has a correlation co-efficient (R^2) of 0.997 for shear velocity and 0.317 for longitudinal velocity.

Spaaiman	Young's	Shear	Bulk	Poisson's
specimen	modulus(GPa)	modulus(GPa)	modulus(GPa)	ratio
M1	185	741	125	0.25
M2	184	746	117	0.24
M3	187	765	112	0.22
M4	167	648	133	0.29
M5	130	483	148	0.35

Table 3. Elastic constants of the DSS thermally soaked at 1300°C for up to 90 minutes



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

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For shear modulus correlation co-efficient values are 0.999 and 0.263, for shear wave velocity and longitudinal wave velocity, respectively.



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

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.



Figure 9. Correlation between (a) bulk modulus and ultrasonic shear wave velocity (b) bulk modulus and ultrasonic longitudinal wave velocity, in DSS

[19], have also reported that shear wave velocity show better correlation than longitudinal velocity with the elastic constants for isotropic solid materials.



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

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CONCLUSION

Ultrasonic attenuation, longitudinal velocity and shear velocity, micro hardness, ferrite percentage in DSS heattreated in two phase ($\alpha \& \gamma$) and single phase (α) regions were studied. The results show that the attenuation is mainly decided by the grain size and attenuation increases steeply with increase in grain size. Ultrasonic attenuation exhibits an opposite behavior to velocity and micro hardness. 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 the DSS indicate that ultrasonic shear wave can be used for materials characterization and shear wave velocity has better correlation as compared to longitudinal wave velocity. In general, the outcomes are very promising and ultrasonic measurements could detect significantly the phase transformations and development of modulus of elasticity successfully.

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