Ultrasonic Characterization and Sensitization of Austenitic Stainless Steels

Dr. R. Jayachitra[1]
[1] PG and Research Department of Physics
[1]Urumu Dhanalaksmi College, Tiruchirappalli, Tamil Nadu, India.

Abstract- *Austenitic stainless steels are most commonly used in a wide variety of industries including power, chemical, petrochemical process and nuclear. The wide application of this steel in the industry is greatly affected by the sensitization phenomena in the grain boundary region. Austenitic stainless steels grade AISI 304 and AISI 316 test specimens were heat treated to 650°C ±1°C and held at this temperature for 15 min, 30 min, 1hour, 2 hours and 4 hours followed by water quenching. Ultrasonic velocity and attenuation are the important parameters, which are required for the ultrasonic non-destructive technique of material characterization. The microstructural changes with different heat treatment conditions are correlated. The AISI 304 grade developed a high degree of sensitization, whereas 316 grade did not develop sensitization after heat treatment. The changes in ultrasonic velocity and attenuation are used to characterize the variation in microstructure resulted due to heat treatment. Shear waves are found to be more sensitive to changes in hardness than longitudinal waves in 316 grade, whereas the longitudinal velocity plays a major role in the extent of sensitization in 304 grade than the shear wave velocity.*

Keywords- Austenitic stainless steel, Heat-treatment, Microstructure, Ultrasonic parameters.

I. INTRODUCTION

Austenitic stainless steels have excellent corrosion resistance, good weldability and oxidation resisting qualities have a wide variety of home and commercial application. [1]. Austenitic stainless steels are preferred in construction materials of the various components required in chemical, petrochemical, food industry, Heat exchanger, nuclear industries and many other industries due to their excellent corrosion and high temperature properties. Austenitic stainless steels contain 16-25% chromium, more than about 6% nickel and a very low percentage of carbon, about 0.1% or less. As the nickel content increases in steel, the amount of austenite present of the solution - treatment temperature increases. At high temperatures, the face centered cubic (fcc) crystal structure of iron is stable and the term used to describe this phase is austenite. Nickel is added to steel in order to retain the fcc structure to room temperature and below. Chromium alloying enhances corrosion resistance. Above 12% chromium steel is considered stainless steel. The wide application of austenitic stainless steel in the industry is greatly affected by the sensitization phenomena at the grain boundary region. The sensitization behavior of austenitic stainless steels is greatly influenced by several metallurgical factors such as chemical composition degree of prior deformation, grain size, aging temperature time. Sensitization occurs at temperature between 500°C to 800°C [2]. One to hundreds of hours, chromium rich carbides trend to precipitates at grain boundaries. Carbide precipitation due to a heat treatment can result in severe chromium depleted zones near the grain boundary area, referred to as sensitization [3,4]. The material properties can be degraded by improper heat treatment. Therefore the inspection of heat treated parts is an efficient way to prevent and reduce failures in industrial plants. For such applications Non-Destructive techniques (NDT) have been recently gaining more importance due to the rapid development of 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. [5]. There are several studies correlating ultrasonic velocity and attenuation to the microstructure of steels [6,7], i.e., phases present in the microstructure and their morphology, grain size distribution, etc. Our investigation undertakes to enhance NDT capabilities to assess and mechanical of AISI 304 and AISI 316 austenitic stainless steels. In the present study, AISI 304 and AISI 316 grade stainless steel has been heat treated at 650°C for 15 min, 30 min, 1 hour, 2 and 4 hrs., followed by water quenching. Ultrasonic parameters are correlated to the variation in microstructure and material hardness.

II. EXPERIMENTAL PROCEDURES

The chemical compositions of the investigated steel are given in Table 1.

Table 1: Chemical composition of AISI 304 and 316 stainless steel (wt. %)

Material	C	S	P	Si	Mn	Cr	Ni	Mo	Cu	Co
AISI 304	0.046	0.015	0026	0.401	1.45	18.32	8.04	-	-	-
AISI 316	0.058	0.006	0.024	0.414	1.46	16.71	10.08	1.92	0.495	0.25

The test specimens were heated in an electric muffle furnace to 650°C ±1°C and held at this temperature for 15 min., 30 min, 1 hour, 2 & 4 hrs, followed by water quenching. Microstructural study was carried out using Envison 3.0 series image analyzer.

Ultrasonic tests were accomplished by the contact pulse echo method using 5MHz broadband normal beam shear and longitudinal wave probes. The selected probes are of contact type with 0.25” and 0.5” dia for longitudinal and shear respectively. 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. Ultrasonic velocity can be estimated from the relationship.

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

Ultrasonic attenuation effects in material are important due to the reason that the signal amplitude reduced by attenuation can affect the quality of the image produced and thereby affecting the quality of results. Attenuation co-efficient should be calculated by considering the effects of reflection, scattering and absorption in the material. Attenuation measurements were made using 5MHz longitudinal wave transducer. Attenuation co-efficient was calculated according to the relationship.

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

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. Vickers hardness values were measured at the five different points, and then a mean hardness value for each group was determined.

III. RESULTSAND DISCUSSION

3.1 Microstructural characterization:

The sensitization behavior of austenitic stainless steels is greatly influenced by several metallurgical factors such as chemical composition, degree of prior deformation, grain size, aging temperature and time [8-9]. During this high temperature exposure, depletion of Cr of less than 12% occurs in the region adjacent to the grain boundary due to the precipitation of a continuous network of chromium-rich Cr23C6 carbides [10-12]. Sensitized grain boundaries provide connected networks for degradation. Such forms of degradation often limit the service life of austenitic stainless steel components [13-17].

The rapid oxalic acid etch test was used for analysis of the grain boundary sensitization development. The results of microstructural examination are depicted in Figure 1 and Figure 2, for types 304 and 316, respectively. Figure 1 (A1) shows the microstructure of 304 grade stainless steel in the as received condition. In Figure 1, elongated islands with step structure of Cr-rich δ ferrite are observed. From Figure 1, it is observed that, when the sample is heated at 650°C, sensitization did not occur at 15 mins of holding time, but observed at holding times of 30 mins to 4 hrs. Figure 1 (A2) (aged at 15 mins), also showed the step structure as in unaged base metal, with slight carbide precipitation inside the austenitic grains. Both as received and 15 mins aged specimen did not show any intragranular attack as seen in Figure 1 (A1 and A2). The frequency of intergranular carbide precipitation increases with the holding time, and the delta ferrite is no longer distinguished. Figure 1 (A3 to A6) reveals that aging at 650°C, for 1 hr and 2 hrs promotes severe sensitization of the material. These are “ditch” structures characteristic of completely sensitized materials. In our case, a fully ditched structure is visible, since the carbides are completely dissolved in an electrolytic etching in oxalic acid solution. The black patches observed in the microstructure form the depleted zone. It could be observed that the width of the depleted zone increased with time, leading to a flatter chromium profile near the grain boundaries at 650°C for 30 mins to 4 hrs.

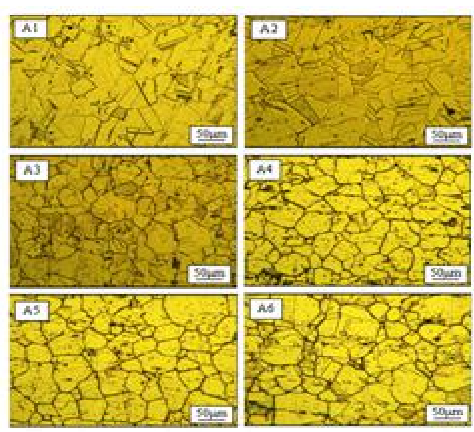


Fig. 1 : Microstructure of AISI 304 stainless steel heat treated at 650°C for different holding time : (A1) as received, (A2) 15 mins (A3) 30 mins (A4) 1 hr (A5) 2 hrs and (A6) 4 hrs

Most of the metallurgical transformations are diffusion controlled and therefore they are time dependent. When austenitic stainless steel is heated to 650°C, chromium depletion continues to increase with dwell time (upto 2 hrs), and finally reaches equilibrium. In our case, at 650°C the equilibrium could be established within 4 hrs. The microstructure shows that the surfaces of type 304 developed a high degree of sensitization as is indicated by heavy intragranular attack (A3 to A6). Even some twin boundaries are attacked indicating precipitation after heat treatment.

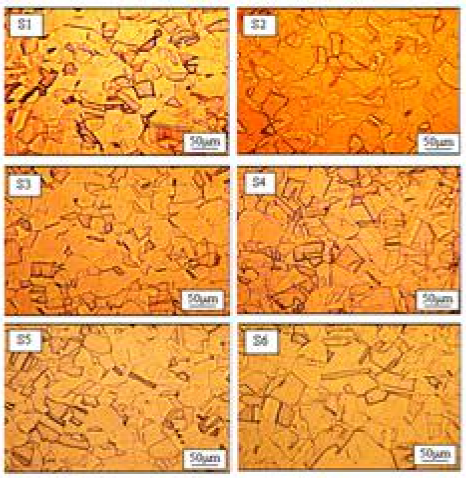


Fig. 2 : Microstructure of AISI 316 stainless steel heat treated at 650°C for different holding time : (S1) as received, (S2) 15 mins (S3) 30 mins (S4) 1 hr (S5) 2 hrs and (S6) 4 hrs

Figure 2 (S1-S6) shows the photomicrographs of the AISI 316 grade stainless steel in the as received condition, and heated at 650°C followed by water quenching for 15 mins, 30 mins, 1 hr, 2 hrs and 4 hrs. Chromium depleted zones are absent in all the specimens shown in the Figure 2. The unaged and aged specimens, that were unaffected by annealing showed a step structure. It is clear from the microstructure that 316 grade stainless steel did not develop sensitization after heat treatment at 650°C followed by aging. It is due to the fact that in 304 grade stainless steel (Figure 1), aging, increase dislocation density and leads to phase transition to deformation induced martensite, which has been detected on surfaces (Figure 1 (A3 and A4)). However, the presence of molybdenum in 316 type stainless steel did not have a form of martensitic, and therefore is much more resistant to sensitization even when heat treated at 650°C for 2 hrs. V. Kain et al. [18] reported that the stability of these molybdenum bearing and higher nickel content in 316 grade compared to 304 grade, against martensite formation is also indicated by lower Md30 temperatures. Moreover, the presence of copper reduces the grain boundaries and hence the possible nucleation sites for chromium carbide precipitation become less. This results in improvement in resistance to sensitization. Even if there is chromium carbide precipitation along the susceptible grain boundaries, the greater extent of chromium depletion can be tolerated because of the role played by copper in improving the passivity [19-26]. It was reported [27] that, among the various noble alloying additions, copper is considered to be superior in accumulating on the alloy surface. This may be due to the presence of copper in solid solution.

3.2 Grain size:

Grain size is one of the most important factors that can affect intergranular corrosion. The microstructures used to study the effect of grain size on the precipitation of Cr - rich carbides, which were obtained from grain growth at 650°C are shown in Figure 1 and 2. The grain size was calculated by counting method for different heat treated samples of 304 and 316 grade stainless steel, and the results are tabulated in Table 2 and 3. The values for 304 grade stainless steel show that for higher aging time of 2 hrs and 4 hrs, the grain size increased rapidly, which may be attributed to faster carbide precipitation, after sensitization has been initiated and progressed. The total grain boundary surface area available for carbide precipitation becomes important in sensitization.

Table 2: Ultrasonic velocity, attenuation, grain size and hardness of AISI 304 austenitic stainless steel at different heat treatment

Specimen	Heat treatment at 650°C	Grain Size (µm)	Micro hardness (VHN)	Longitudinal velocity (m/s)	Shear velocity (m/s)	Attenuation (dB/mm)
A1	None	76.32	184	5848	3185	0.3872
A2	15 mins/ WQ	78.74	256	5952	3279	0.4152
A3	30 mins/ WQ	63.72	269	6093	3195	0.3485
A4	1 hr. / WQ	60.72	216	5988	3257	0.3841
A5	2 hrs./WQ	70.50	175	5930	3215	0.4133
A6	4 hrs./WQ	69.50	181	5993	3226	0.4283

The sensitization is caused by carbide precipitation at the grain boundaries, which requires that the carbide nuclei at the grain boundary attain a critical size. According to classical phase transformation theory, in a finer-grained material, heterogeneous sites are larger in number, and therefore, the sensitization would occur earlier; this has been borne out by the work of Beltran et al. [28]. On the other hand, the growth and size of these carbides determine the extent of Cr depletion around the grain boundary. The proliferation of subcritical carbide nuclei in the fine-grained material also means that the C availability in each nucleus is restricted, due to sharing with other nuclei. Consequently, the carbide growth is faster in larger-grained material (having more C available per nuclei) due to the smaller number of nuclei [2].

Table 3: Ultrasonic velocity, attenuation, grain size and hardness of AISI 316 austenitic stainless steel at different heat treatment

Specimen	Heat treatment at 650°C	Grain Size (um)	Micro hardness (VHN)	Longitudinal velocity (m/s)	Shear velocity (m/s)	Attenuation (dB/mm)
S1	None	60.03	196	5941	3315	0.3866
S2	15 mins/ WQ	59.48	188	6061	3252	0.1978
S3	30 mins/ WQ	58.59	232	5971	3166	0.1090
S4	1 hr. / WQ	69.32	174	5946	3166	0.1257
S5	2 hrs./WQ	70.81	182	5941	3389	0.1872
S6	4 hrs./WQ	71.12	185	5971	3385	0.2446

Therefore, in 304 grade stainless steel the degree of sensitization in larger-grained material is greater after sensitization has been initiated and progressed. The finer-grained material behaves as if it contains less C compared to the coarser-grained material. In fact, if the grains are fine enough, there may not be any sensitization. However, in the 316 grade stainless steel, though heat treatment of 650°C and subsequent aging for different time promotes the growth of grains, it did not have any effect on sensitization at 650°C. This shows that grain size has a dominant effect in controlling sensitization in 304, which it did not produce significant effect in the degree of sensitization in 316 grade stainless steel.

3.3 Hardness:

The variation of hardness with different heat-treatment for 304 and 316 grade stainless steel is reported in Table 2 and 3, respectively. From the table, it is clear that in the case of 304 grade stainless steel, the hardness increase from 184 HV to 269 HV in the first 30 mins of aging. Since, the martensite is a harder phase, its formation is also indicated by hardness measurements. After 30 mins the hardness decrease, which may be attributed to the precipitation as Cr23C6 carbides, since as an interstitial solute carbon promote considerable solid solution hardening of the austenite. Figure 1(A5 and A6) shows that the carbide precipitation is most intense at 2 hrs and 4 hrs of aging. The presence of Mo and Co in 316 grade steel gives a large value of hardness for as received specimen compared to 304 grade steel. Depending on the composition, the aging time seems to have different effects on the hardness of 304 and 316 grade stainless steel. In particular, for 304 grade, the increase of aging time leads to an increase of the hardness, which prolonged up to 30 mins, and thereafter decrease in the severely sensitized region. However, in the case of 316 grade the hardness increase up to 30 mins, and after a pejorative start, hardness returns with in the untreated material range, when the aging time is prolonged.

3.4 Ultrasonic parameters:

When the ultrasonic wave propagates through the medium, some part of energy is attenuated through different mechanism like thermal loss, scattering, absorption, electron-phonon interaction, phonon-phonon interaction, and magnon-phonon interaction etc., called as ultrasonic attenuation. The ultrasonic attenuation correlates several physical properties like elastic Guruneisen parameter, thermal conductivity, thermal relaxation time, acoustic coupling constant, thermal energy, specific heat, particle size, density, Debye average velocity, and concentration etc [29]. Thus, the material can be characterized by the knowledge of ultrasonic parameters under different physical conditions. Ultrasonic NDT of material characterization is used for the determination of (a) elastic constants (Shear modulus, Bulk modulus, Young modulus and lame modulus), (b) microstructure (grain size, texture, density, etc.), (c) discontinuity (porosity, creep damage, fatigue damage etc.), and mechanical properties (tensile strength, shear strength, hardness etc.). The new work in this field also provides the characterization of advanced and smart materials.

Longitudinal and shear wave velocity measurements were made in the 304 and 316 stainless steel specimens annealed at 650°C, followed by water quenching for 30 mins to 4 hrs of aging. Table 2 and 3 reports the variation in longitudinal and shear wave velocity, for 304 and 316 grade stainless steel for different aging time, respectively. In 304 grade stainless steel, the variation of longitudinal velocity with annealing time exhibited a large increase (≈ 150 m/s) after annealing at 650°C, for 15 mins and 30 mins of aging, followed by a drastic drop (≈ 120 m/s) in the severe sensitization region, and then increased slightly (≈ 60 m/s) at 4 hrs, due to onset of desensitization. However, in case of 316 grade stainless steel, the longitudinal velocity increased (≈ 120 m/s) immediately after heat treatment at 15 mins due to partial recrystallization, followed by a slight decrease (≈ 90 m/s) at 30 mins of aging, due to retardation of recrystallization, and reached saturation for prolonged aging up to 4 hrs. There is no evidence of recrystallization from 1 hr to 4 hrs of aged sample. Shear wave velocity measured in 304 grade specimen exhibited a slight increase (≈ 95 m/s) after heat treatment and aging at 15 mins, followed by a decrease (≈ 80 m/s), due to onset of sensitization, and slightly increased and remained saturated in the severe sensitization region. However, in the case of 316 grade stainless steel, the shear wave velocity decreased (≈ 60 m/s) immediately after heat treatment due to partial recrystallization and remained saturated at 30 mins to 1 hr of aging time, and thereafter, increased drastically (≈ 230 m/s), which may be due to increase in the grain size and variation in the hardness of the material. The change in the shear wave velocity is almost different as compared to that of the longitudinal wave velocity during the aging process. Variation in shear wave velocity with aging time is opposite to that of the longitudinal wave velocity. It is interesting to note that in the case of 316 stainless steel, in the recovery regime shear wave velocities show a drastic increase, which may be attributed to the distortion of the lattice caused by point defects and dislocations, during prolonged aging.

At room temperature, ultrasonic absorption is mainly due to dislocation damping, thermo elastic losses and magneto-elastic losses [30]. By the consideration of frequency, wavelength and grain sizes, Rayleigh scattering region is most applicable in the present study. Hence, the scattering coefficient is more sensitive to the grain size in the sensitized region, and Rayleigh scattering is most applicable in this region. The degree of sensitization of the stainless steel can be studied by combined measurements of attenuation and the amplitude of the main peaks in the FFT spectra using 5 MHz transducer. The combined assessment of attenuation measurements at 5 MHz and the FFT spectrum amplitude analysis of the main peaks provide a predictive criterion for the sensitization evaluation of the steel.

Table 2 and 3 show, respectively, the attenuation values in the 304 and 316 grade stainless steel materials. It was noted from the table that by a simple comparison of the attenuation coefficients a differentiation between the heat treatments is not plausible. For the employed frequency at which the signal was satisfactorily acquired, an increase of the attenuation coefficients with the increase of the aging time is observed. On the other hand, carbides with a small average diameter should not be detected by an ultrasonic wave, and it would be expected that the isolated carbide presence should not considerably modify attenuation coefficients at relatively low frequency value as employed in this study. However, the observed change of attenuation in 304 grade stainless steel could be explained by the coalescence of the precipitates as follows. The increase of the attenuation coefficients could be related to the formation of a quasi-continuous carbide wall at each grain boundary. Because of the high difference in acoustic properties between austenite and chromium carbides, an additional reduction of the wave intensity is expected. Thus, moderate or high carbide coalescence in coarse grain materials can justify a detectable increase of the attenuation values using 5 MHz frequency.

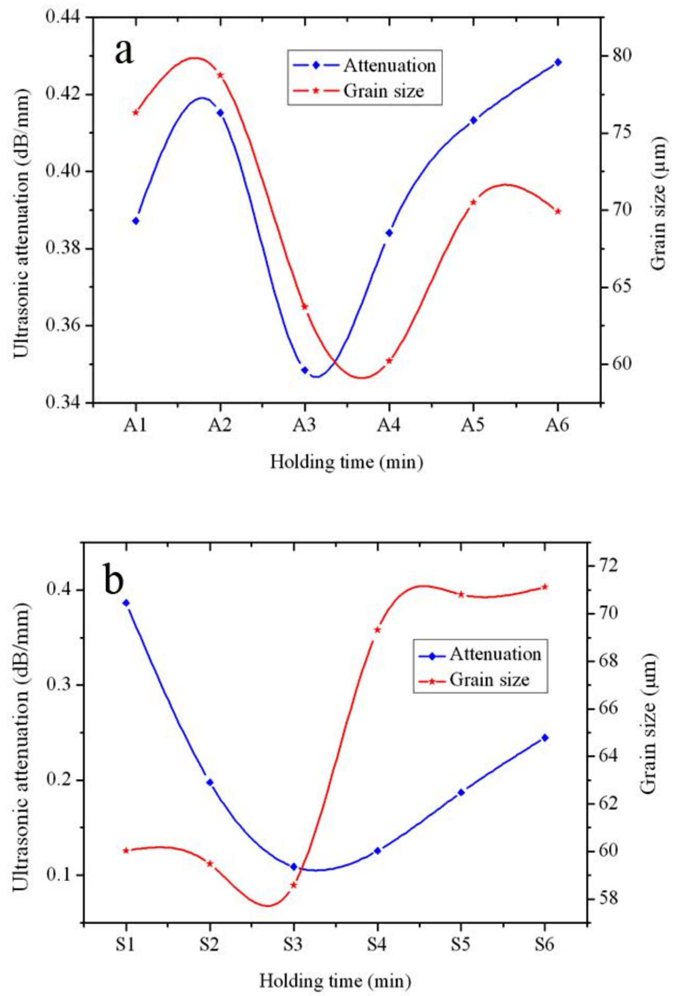


Fig. 3 : Variation in ultrasonic attenuation and grain size of (a) AISI 304 and (b) AISI 316 stainless steel at different heat treatments

Figure 3 (a) and (b) shows the variation in attenuation and grain size of 304 and 316 grade stainless steel for different heat treatments respectively. After heat treatment of austenitic stainless steels, both precipitation of chromium carbides and other microstructural changes are expected, especially modification of the average grain size. As observed in Figure 3 (a), a significant increment of the grain size takes place after heat treatments at 650°C (A4 and A5). The attenuation coefficients obtained in this study show that the grain size is not decisive when sensitization phenomena originated from heat treatment at 650°C are considered. The results summarized in Table 2 support the hypothesis that the attenuation increase in A4 and A5 specimens, mainly arises due to an extensive carbide precipitation at grain boundaries, and it is not originated from the scattering attenuation caused by a variation of the grain size.

Figure 4 to 7 shows the variation in first back wall echo and FFT spectrum for 304 and 316 grade stainless steel, respectively, in the longitudinal and shear wave mode, which suggest different responses of the tested materials. FFT spectra shown in Figure 4 (b) present peaks at frequency ranges, which are almost independent of the heat treatment. On the other hand, the main peak height of each spectrum seems to be strongly affected by the material condition. For the specimens exposed to heat treatments a considerable increase of the intensity of the main frequency with the increment of the aging time is observed. This is not contradictory with the observed increase of the attenuation reported in

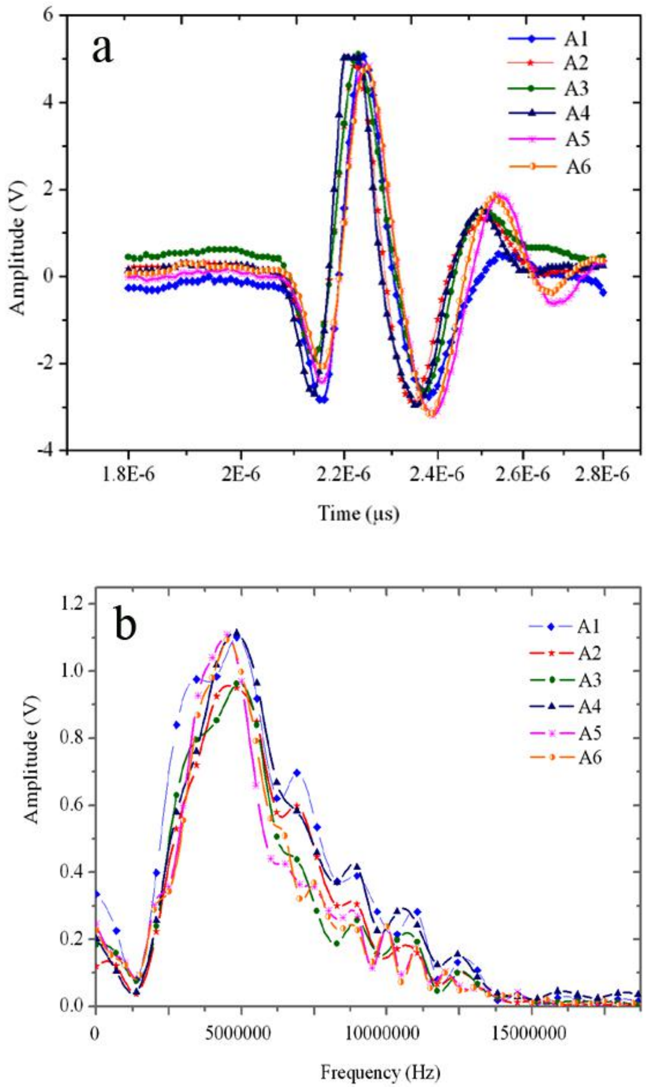


Fig. 4 : Variation in (a) first backwall echo and (b) FFT spectrum with different heat treatments, for 304 grade stainless steel in the longitudinal mode

Table 3, due to the following argument. The reported attenuation shows the intensity reduction of the ultrasonic wave through the whole thickness covered by the beam, that is different from the information provided by the FFT spectra, which show comparative values concerning wave energy transmission at specific frequencies. The increase of the amplitude for long aging times should not be correlated with precipitation or coalescence of carbides at grain boundary, but with the homogenization achieved by the matrix as a consequence of the long exposure at high temperature [31].

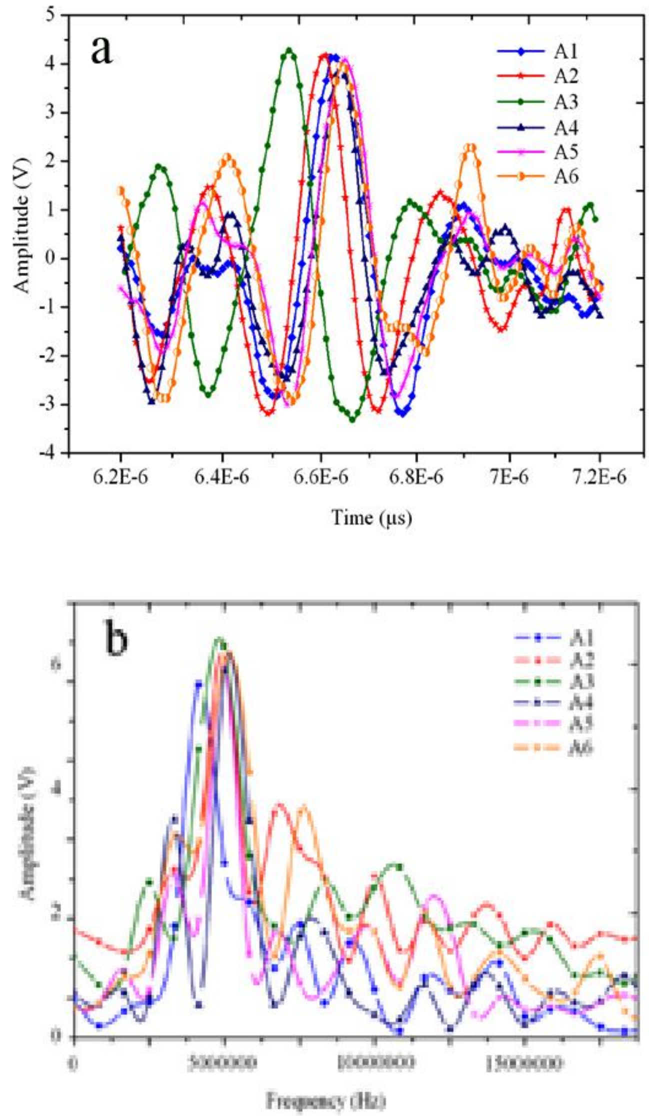


Fig. 5 : Variation in (a) first backwall echo and (b) FFT spectrum with different heat treatments, for 304 grade stainless steel in the shear mode

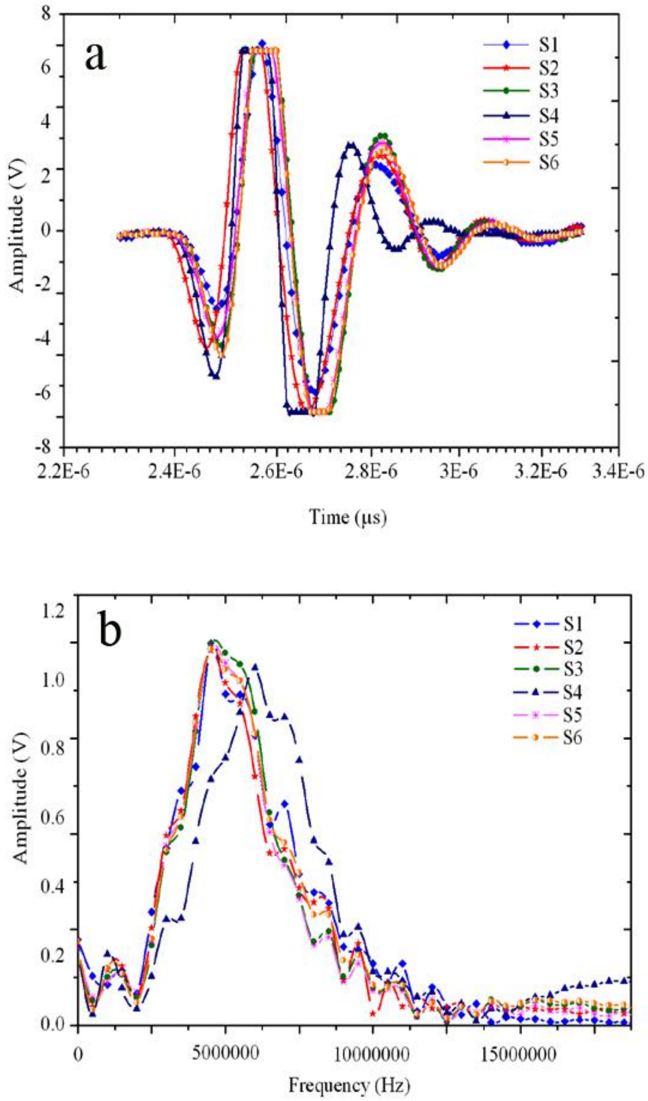
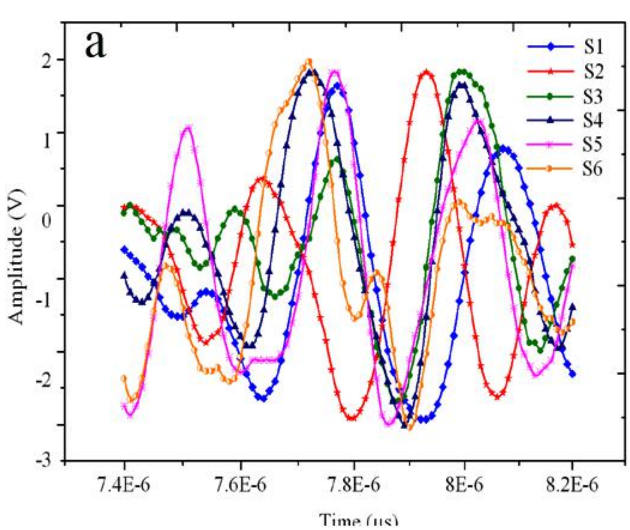


Fig. 6 : Variation in (a) first backwall echo and (b) FFT spectrum with different heat treatments, for 316 grade stainless steel in the longitudinal mode



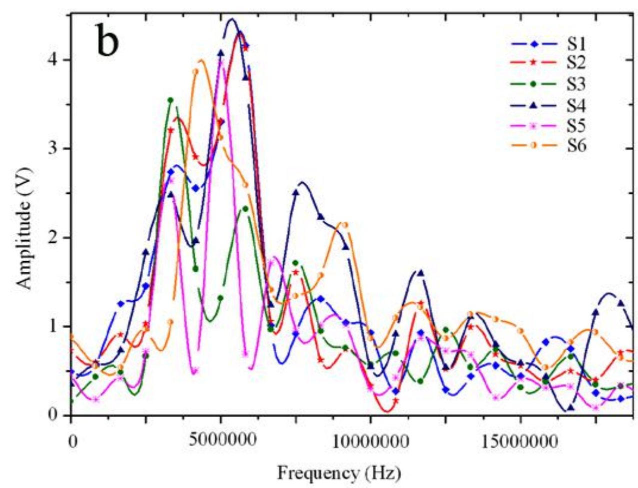


Fig. 7: Variation in (a) first backwall echo and (b) FFT spectrum with different heat treatments, for 316 grade stainless steel in the shear mode

In the case of 316 grade stainless steel Figure 3 (b), the attenuation decrease after heat treatment up to 30 mins of aging, due to the presence of fine grained specimen. After 30 mins of aging the attenuation started increasing gradually, due to the presence of coarse grained specimen at large aging times of 1 hr, 2hrs and 4 hrs. This attenuation in 304 grade stainless steel is decided by both the extent of sensitization and grain size, while in the case of 316 stainless steel it is decided only by grain size. Moreover, as the grain size and attenuation increase after 1 hr (S4 to S6), the peak frequency shifts towards lower value, as it is observed in Figure 7 (b).

IV. CONCLUSION

The above investigation, it was observed that the AISI 304 grade stainless steel developed a high degree of sensitization, whereas 316 grade stainless steel did not develop sensitization after heat treatment at 650°C followed by aging. Attenuation in 304 grade stainless steel is decided by both the extent of sensitization and grain size, while in the case of 316 stainless steel it is decided only by grain size. The sensitization behavior of austenitic stainless steels is greatly influenced by chemical composition, grain size, aging temperature and time. Grain size has a dominant effect in controlling sensitization in 304, while it did not produce significant effect in the degree of sensitization in 316 grade stainless steel.

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