MEASUREMENTS OF ACOUSTIC NONLINEARITY IN COLD-ROLLED AND HEAT-TREATED 304 AUSTENITIC STAINLESS STEEL USING NONLEANER ULTRASOUND

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MEASUREMENTS OF ACOUSTIC NONLINEARITY IN COLD-ROLLED AND HEAT-TREATED 304 AUSTENITIC STAINLESS STEEL USING NONLEANER ULTRASOUND

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LIST OF SYMBOLS AND ABBREVIATIONS

∂ partial derivative operator

δ variational operator

β nonlinearity parameter

ϵ strain tensor

δ_{ij} Kronecker delta

T shear stress tensor

σ stress

ρ mass density

ω angular frequency

Δ Laplace opeator

Ω Domain or range

c wave velocity

C_R Rayleigh wave speed

C_s shear wave speed

C_L longitudinal wave speed

d_{ij} Euclidean distance matrix

e exponential function

f frequency

f_i body force per mass vector

i imaginary unit

k wave number

n_i normal vector

t time

t_i traction vector

u displacement along first coordinate axis

u_i displacement vector

x direction of propagation

A amplitude or Area

A_1 amplitude of first harmonic wave

A_2 amplitude of second harmonic wave

C_{ijkl} higher order stiffness tensor

D_{ijkl} higher order stiffness tensor

E Young's modulus of elasticity or Larganian/ Green tensor

F_i force vector

I identity tensor

N number of precipitates

P non symmetrical tensor

S surface or uniform grid size

U internal work or approximated displacement

V volume

W external work

T_R recrystallization temperature

T_m melting temperature

max maximum

min minimum

s seconds

EPR electrochemical potentiodynamic reactivation

SS stainless steel

DRX dynamic recrystallization

HAZ heat affected zone

SUMMARY

In energy industry, austenitic stainless steels have been used in a number of different applications. Their excellent mechanical properties, good formability, and high corrosion resistance make them a popular candidate material. Among other, SS 304 is the most common stainless steel. However, it has one disadvantage that the material is susceptible to sensitization. The sensitization occurs when the material is exposed to a certain temperature even for a short duration, where chromium and carbon forms chromium carbide precipitates along the grain boundaries. The formation of the precipitates causes chromium depletion and ultimately leads to intergranular stress corrosion cracking (IGSCC). The heat affected zone (HAZ) of weld in 304 SS structures is usually the most probable locations for sensitization. The IGSCC is of great interest because most cracking in high temperature pipes, such as water reactor pipes, initiates in HAZ due to the sensitization.

Nondestructive evaluation methods based on nonlinear ultrasound such as the second harmonic generation technique are highly sensitive to material damage at microstructure level. The second harmonic generation technique measures the change of the second harmonic frequency in the initially monochromatic wave, and relates property changes in the tested materials to the measured nonlinearity parameter, β. This paper evaluates microstructures of cold rolled and heat treated stainless steel 304 by using nonlinearity parameter, β. Using longitudinal waves, the change of microstructural properties of cold rolled 304 SS is first evaluated. Next, combined effect of cold rolled and heat-treated stainless steel is evaluated

using the same technique. The results are then compared with those from previous works [1] [2] [3] [4] [18] [19], which have examined each effect individually. The result shows that the cold work causes drastic increases in nonlinearity of 304 SS due to the plastic deformation and formation of dislocations. Also, the cold rolling affects the sensitization in different ways depending on the percent cold rolled of 304 SS. The heat treatment at 675 °C induces two different effects in this material: sensitization and recrystallization. With different percent of cold rolling, the combined effect of cold rolling and heat treatment result differently on the nonlinearity of 304 SS, depending on domination of either sensitization or recrystallization.

Chapter 1.

Introduction

1.1Motivation

In energy industry, austenitic stainless steels have been used in a number of different applications. Their excellent mechanical properties, good formability, and high corrosion resistance make them a popular candidate material. Among other, SS 304 is the most common stainless steel. However, it has one disadvantage that the material is susceptible to sensitization. The sensitization occurs when the material is exposed to a certain temperature even for a short duration, where chromium and carbon forms chromium carbide precipitates along the grain boundaries. The formation of the precipitates causes chromium depletion and ultimately leads to intergranular stress corrosion cracking (IGSCC).

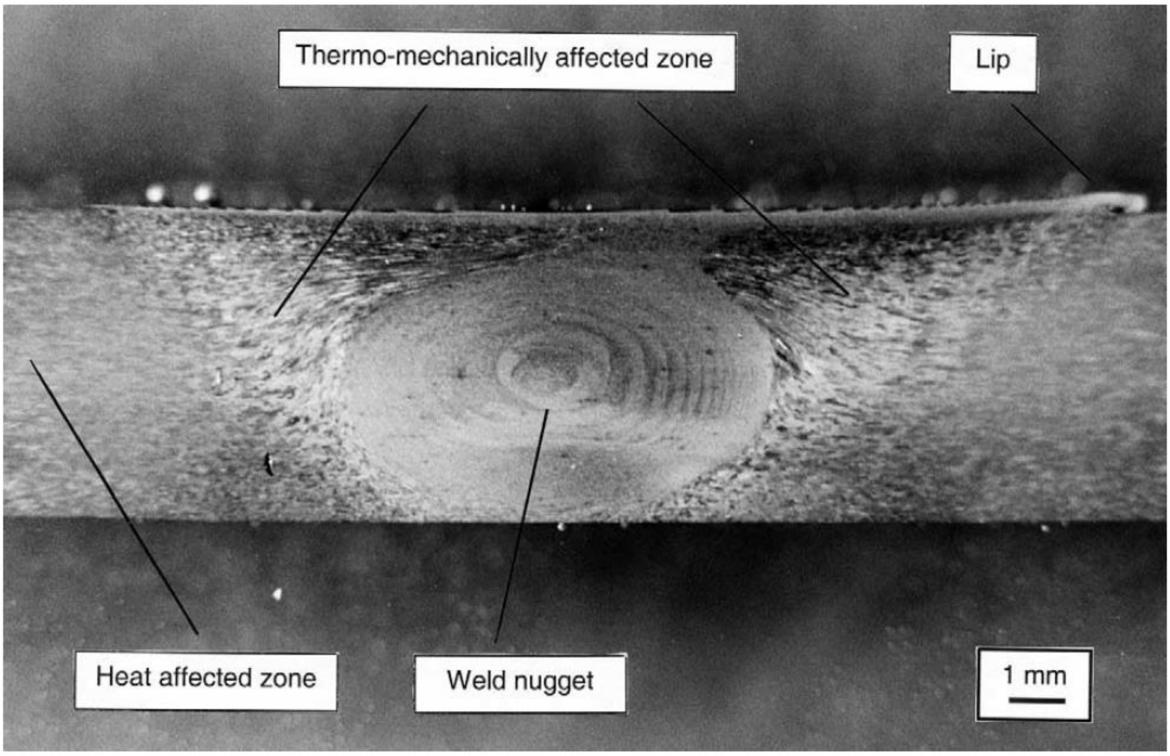


Figure 1. Cross section of welded joint with Heat Affected Zone (HAZ) [5]

As can be seen in Figure 1, the heat affected zone (HAZ) occurs when two materials are joined by welding. While welding of two parts, material parts to be welded experience high temperature for certain amount of time, and the materials' properties are changed by the heat. This is a great concern in all the construction and mechanical engineering industries, but especially in nuclear power plants because the main cause of the failure takes place in HAZ due to the IGSCC, which is induced by the sensitization happening at HAZ due to the heat effect. The sensitization temperature for stainless steels is between 450 °C to 850 °C and it is important to notice that many applications of the stainless steels are used at this temperature, which have potential danger of IGSCC causing microcracks, ultimately leading to macrocracks and individual failures or in unfortunate cases the failure of the whole structure with great cost of replacements.

Among other techniques, the quantitative nondestructive evaluation (QNED) is one way to prevent the failure of components. Up to the present, there are many researches evaluating the effect of sensitization, chromium carbide precipitation on 304 SS using electrochemical [7], Rayleigh surface wave [2] [6] [20]and longitudinal wave [1] measurement techniques. In addition, there are also some evaluations of heat-treated steel, ferritic and reactor pressure vessel steel, using longitudinal waves [8] [9].

1.2 Objective

There are two main parts in this research: cold work and heat treatment of 304 SS. First, cold work is widely used in many industries and the effect of cold work is that it changes the microstructure of a material. To verify the sensitivity and effectiveness of the nonlinear ultrasound (NLU) measurement technique, the cold worked 304 SS will be evaluated using the longitudinal NLU wave measurement. The second research objective is to quantify the combined effect of cold work and heat treatment on 304 SS. To evaluate this, prior cold worked 304 SS will be heat treated. This will determine the relationship between cold work and the heat treatment of 304 SS. Then these results will be compared with previous work of individual effects of cold work and heat treatments [1] [2] [6] [20] which will clarify how cold work can affect the heat treatment differently than the individual heat treatment affected without the experiencing of plastic deformation on the 304 SS.

1.3 Thesis structure

There are 6 chapters in this thesis. Chapter 1 provides the motivation, objective of the research and structure of the thesis. Chapter 2 brings the basic ideas and introduction of cold work, heat treatments of 304 SS. In this chapter each cold working technique will be discussed. Also, each heat treatment effect, such as annealing, recrystallization, and sensitization will be discussed. In chapter 3, wave propagation in materials will informed with equation of motion and leading towards derivation of nonlinearity parameter β, longitudinal and shear wave propagation and reflection. Chapter 4 will introduce some previous works from other research to provide comparable results. This will include results of cold worked and sensitization and recrystallization of 304 SS from other research groups. Chapter 5 will provide measurement and procedure of the experiments including sample preparation to signal processing. In chapter 6, the result and the data analysis will be provided. In chapter 7, the conclusion of the thesis is presented.

Chapter 2

Cold work and Heat treatment of Stainless steel

2.1 Cold work

In many industries and other places using metals, the original shape does not always come in desired way. To change the length, shape and strength to the desired mode, post process in necessary. The process will include cutting, hardening, bending, heating, etc. These post process will have different effect on the material which causes changes in their microstructure and properties. The main focus of the research is on cold working, work hardening process, especially cold rolling, (which will be discuss in detail in next section, cold rolling (2.1.1)).

The Cold working is widely used in steel and aluminum to reshape to the desired body. The reshaping process is called ‘cold’ because it is proceeding below the metal’s recrystallization, (which will be discuss in next section, recrystallization (2.2.3)), temperature. This process causes plastic deformation and because of the crystalline structure change, the material become resistant to additional deformation, the active crystals will decrease their movement due to reduction of the spaces. There are some advantages along with the disadvantages. With cold worked materials, it had hardened and have increased tensile strength. Also, the surface condition and finish are not affected if the process is done in appropriately. However, the ductility is decreased which means it is susceptible to bending for other reforming process. In table 1, different types of cold working techniques are shown.

Table 1. Different cold working techniques [10]

Rolling	Bending	Shearing	Drawing
Extrusion	Angle Bending	Blanking	Tube drawing
Coining	Roll Forming	Slitting	Metal Spinning
Forging	Flanging	Lancing	Embossing

2.1.1 Cold rolling

Among different types of cold working techniques, the cold rolling is one of the popular process used in many industries such as power generation industry. [10] As can be seen in Figure 2, the specimen or the working plate is moving through two rolling mills to reduce their thickness to desired shape and volume.

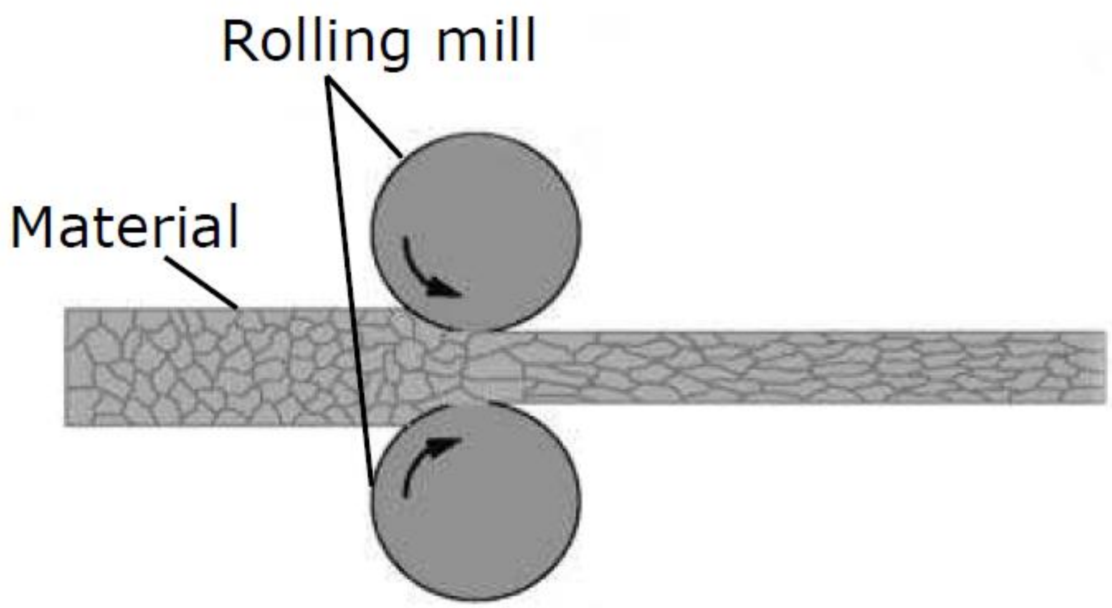


Figure 2. Cross section of cold rolling mills [11]

The advantages of the cold rolling process are follows,

1. Absent of oxidation due to room temperature operation.

2. Relatively quick procedure with potential of massive production and automation.

3. Fine surface finish with clean mills.

4. Availability of thin plate production.

The percent change in thickness usually indicates the percent cold rolled of the material. In this research the maximum percent cold rolled is about 40% due to the frequency and the cycles of the signal, (which will be discussed in future chapter 4, signal processing (4.5)).

2.2 Heat treatment of Stainless Steel

In many situations, especially in any industries, the heating effect on stainless steel parts is unavoidable due to purpose of using the stainless steel, for its resistivity of high temperature. There are variety of heat treatments out there which will affect the materials differently at different temperatures. For some heat treatments the material can experience 'damage' of the microstructure or it can even provide some energy to self-heal the material itself. Among those heat treatments, four different heat treatment will be discussed, Annealing, sensitization, desensitization and recrystallization.

2.2.1 Annealing of Stainless Steel

Annealing is one type of heat treatment where it helps to remove stress by decrease number of dislocations. It is often used for many different metals, such as copper, silver, but especially for stainless steel. By doing annealing, the reduction of hardness and increment of ductility occurs and material becomes more workable. The process of annealing is to heat the material well above the recrystallization (2.2.3) temperature and allow certain time to remove any prior effect and cooling down, both slowly air cooling with room temperature or fast cooling by quenching with water or oil works.

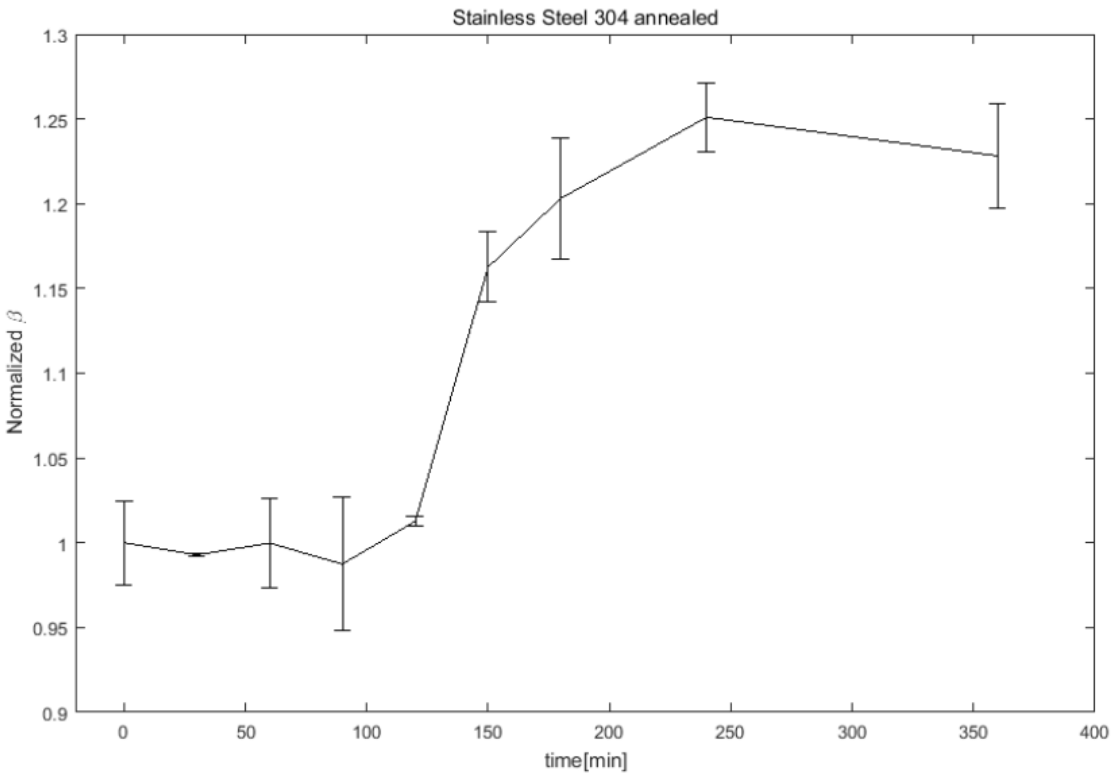


Figure 3. Normalized β Over Heat Treatment times at 675 °C for Annealed 304 SS [2]

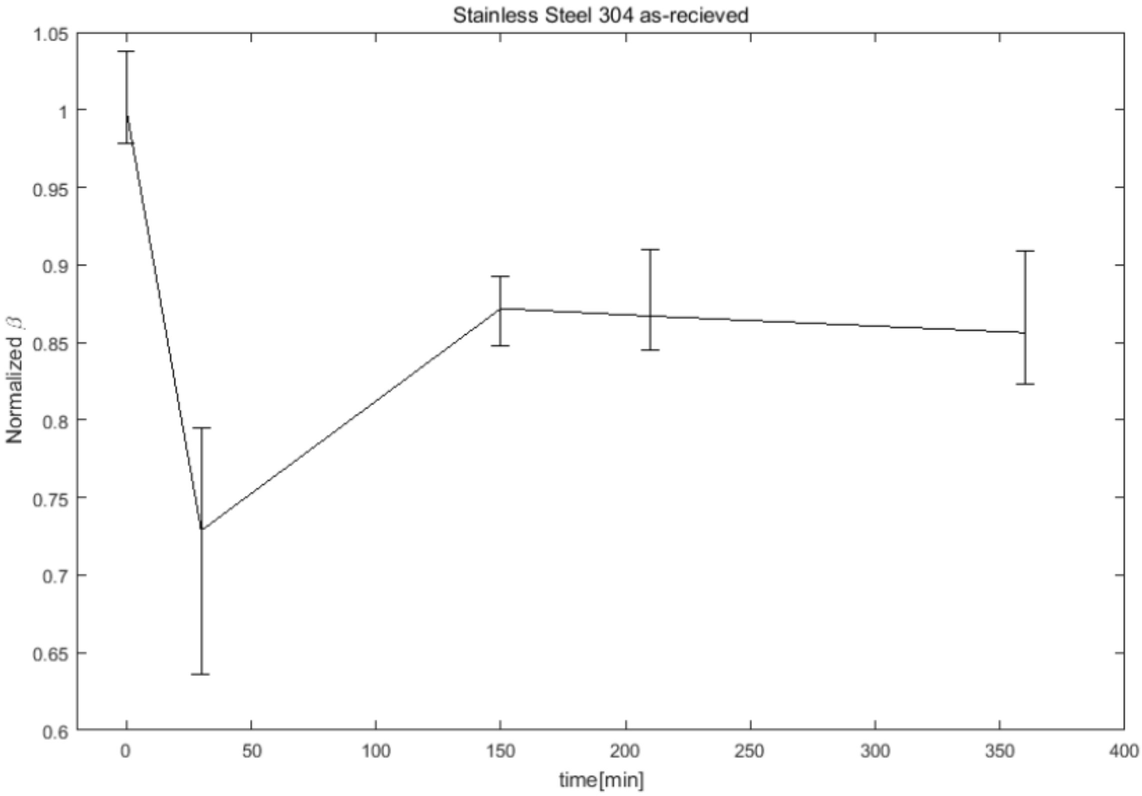


Figure 4. Normalized β Over Heat Treatment times at 675 °C for ‘As Received’ 304 SS [2]

The purpose of the annealing in this research is to remove all the prior effect, especially stress due to production, on the ‘as received’ specimens, so all the specimens have same starting point for future treatments. In Figure 3, and Figure 4 the same heat treatment had been applied to same 304 SS specimens at same temperature. However, the result for ‘as received’ has unpredictable result due to its prior effect on the material such as production, etc. [2] [6] [20]

In Metallurgy prospective, there are different phases in steel, austenitic, martensitic and ferritic. For each phases of steel, the annealing temperature and crystalline phase is different. For Austenitic stainless steel, 304 SS, the annealing temperature range is between 1050 °C to 1120 °C. However, for Martensitic steel, at this temperature, hardens, oppose to softening Austenitic steel.

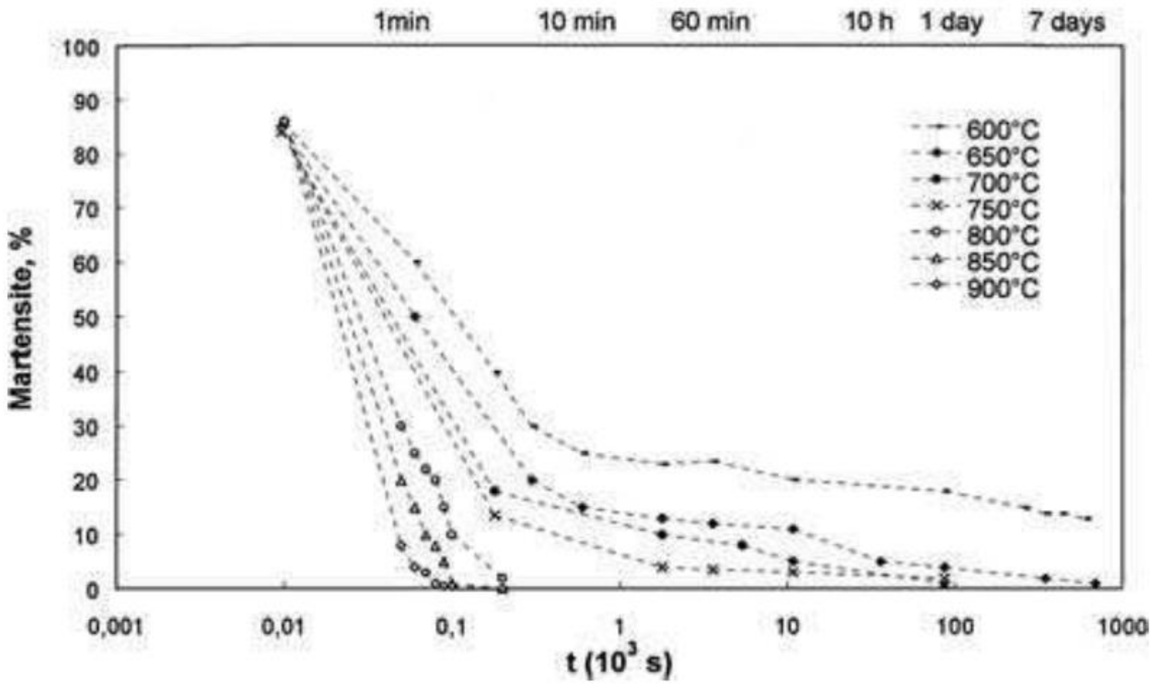


Figure 5. Effect of time and temperature during annealing on martensite induced 304 SS by cold work [12]

In figure 5, the cold worked 304 SS was annealed at different temperature and time. And the percent martensite, very closely related to dislocations and stress in the materials, are evaluated over different temperature and time. As can be seen, for the temperature at 900 °C for 1 hour of time, the percent martensite becomes zero, which proves that if all the specimens are being treated with annealing, the stress level for all specimens will become same and becomes comparable.

2.2.2 Sensitization of stainless steel

Sensitization occurs when chromium and carbon forms chromium carbide precipitation along the grain boundaries with heat energy in stainless steels. For stainless steels, the sensitization occurs at temperature between 450 °C to 850 °C with certain amount of time, which in industries, welding process at HAZ experiences. This will ultimately result in intergranular stress corrosion cracking (IGSCC) and cause failure of components and the whole structure.

Not only the temperature affects the sensitization but there are many different factors that can affect the sensitization, such as cold rolling. In Singh et al. [7], the cold rolling process creates space between each grain and lead the chromium solutes to gather and induce the sensitization. In figure 6, micro-structure of AISI 316 specimens at different holding times at temperature of 700 °C is shown. The precipitated chromium is dark lines and these lines are present in the grain boundaries. For longer holding times at same temperature creates more dark lines, which proves that there are more chromium precipitants. In figure 7, The effect of carbon content in a material on sensitization is shown, and as carbon content increases, the time requires to sensitization occur reduces at different temperature.

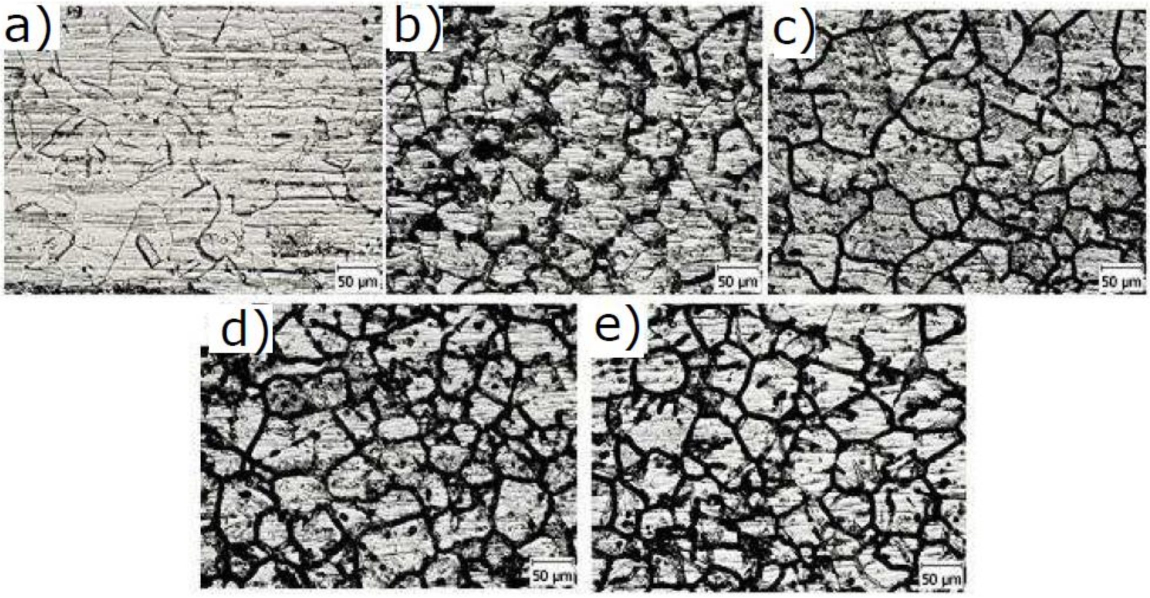


Figure 6. Micro-structure of AISI 316 specimens at different holding times at temperature of 700 °C for a) 15 min b) 30 min c) 60 min d) 300 min e) 600min [13]

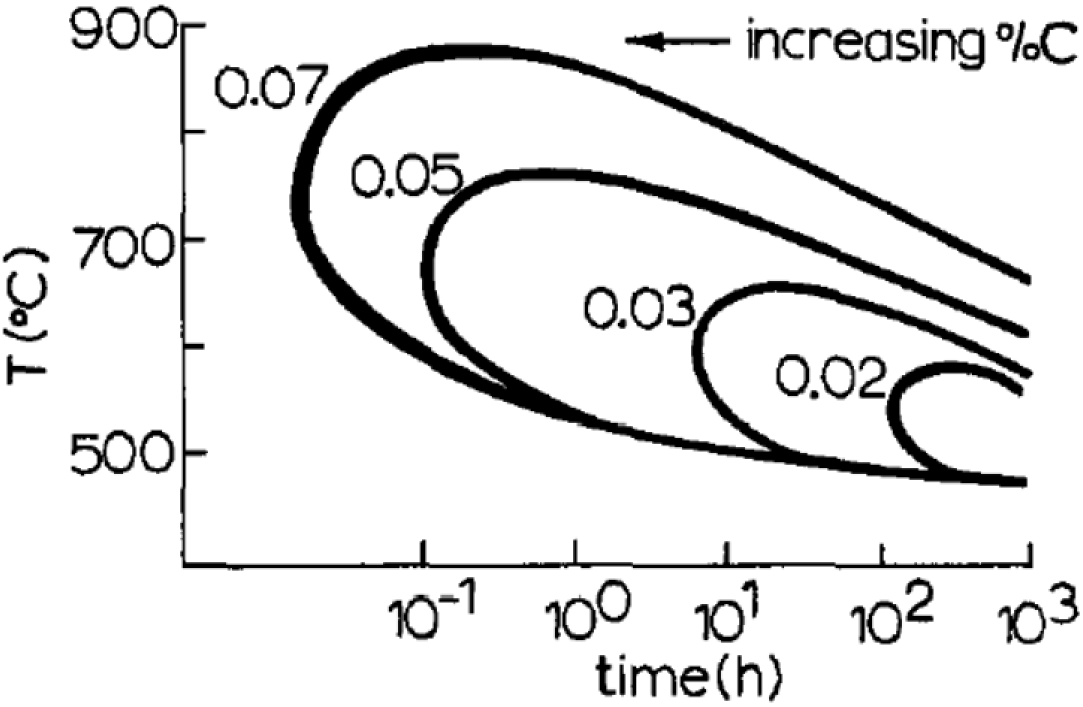


Figure 7. Effect of carbon content in a material on sensitization [14]

As mentioned, the sensitization is very complex and it is affected by different factors such as,

1. Temperature and its holding time
2. Carbon content in a material
3. Grain shape, orientation and size
4. Phases of steel and material composition
5. Cold work
6. Desensitization effect (will be discuss in next section (2.2.3))

2.2.3 Desensitization of stainless steel

Desensitization is one of self-healing process after sensitization, which occurs at same temperature range 450 °C to 850 °C held at certain amount of time like sensitization. The factors that affect the desensitization is same as sensitization as discussed previously (2.2.2).

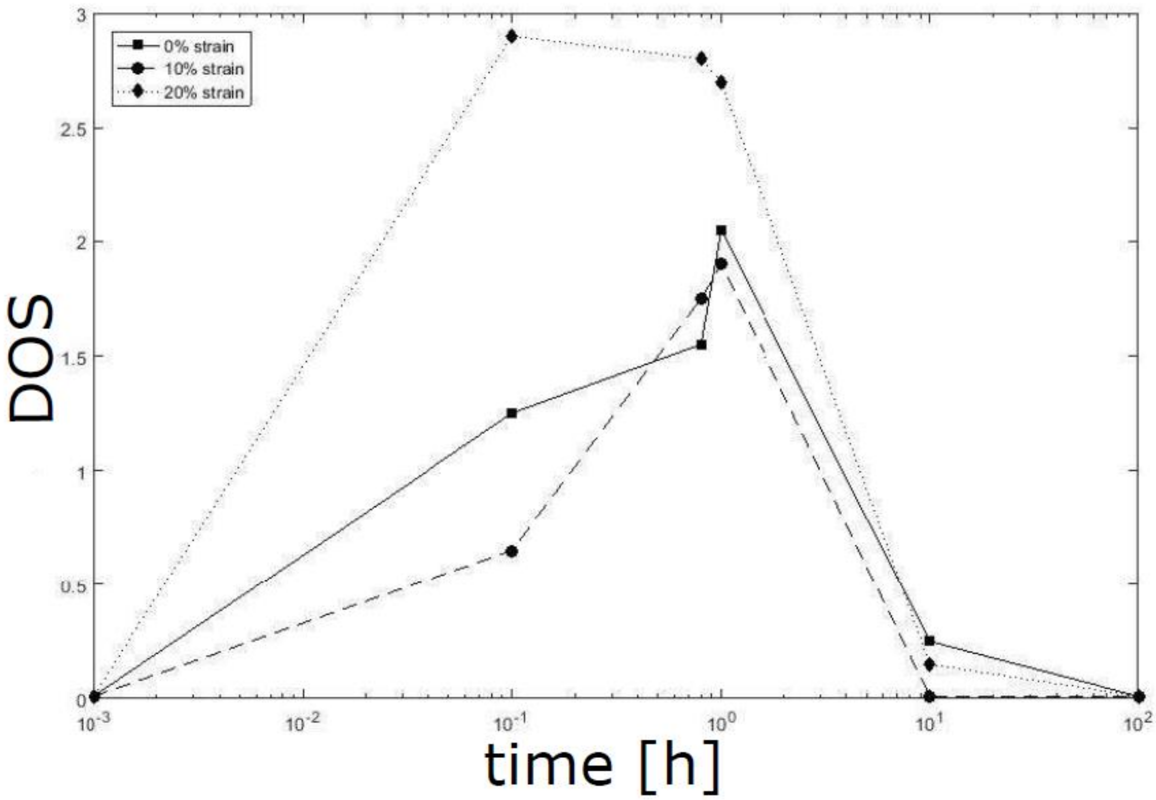


Figure 8. Degree of sensitization (DOS) over time for 304 SS at temperature 675 °C for different deforming stain. [15]

As can be seen in Figure 8. The degree of sensitization (DOS), which quantify the sensitization present in the material, increases to maximum point at certain time, but decreases and become zero after about 10 hours of holding time. With this study, one thing important is to sensitize the material well, the correct time and temperature is the key to successfully makes the material maximally sensitized.

2.2.3 Recrystallization

Recrystallization is healing process that occurs on a material by making new crystals from previously 'damaged' or deformed crystals. Figure 9 shows recrystallization of deformed brass with strain hardening. As can be seen in Figure 9, material, brass, that experiences plastic deformation has dislocations and point imperfections. With this opportunity and enough heat energy, the atoms rearrange themselves to form in more perfect array.

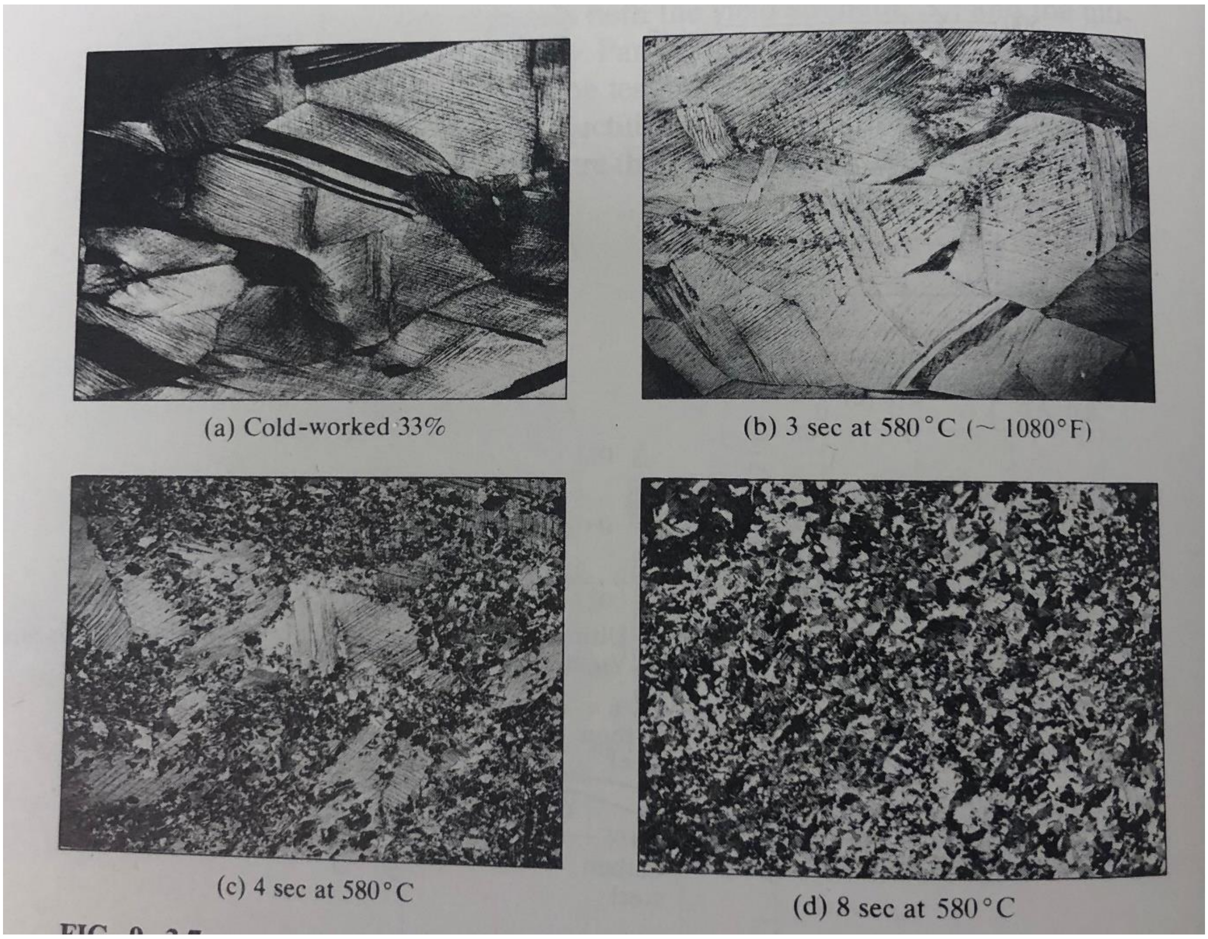


Figure 9. Recrystallization of Strain-Hardened Brass (X40) [16]

The important point hear is that the “recrystallization process requires movement and rearrangement of atoms.” [16] Which means with higher temperature and increase in holding time result in more recrystallization to occur. Also, the recrystallization temperature for different materials are different. The recrystallization temperature is usually in range of temperature depend different materials and it is clearly calculated with equation (1) for pure metals like steel between 400 °C to 700 °C. [17] However, for the mixed metals like 304 SS does not have clear recrystallization temperature range.

$$T_R = 0.3\, T_m \; to \; 0.6\, T_m \tag{1}$$

Where T_R is the recrystallization temperature and T_m is the melting temperature of a material. (The recrystallization temperature for 304 SS will be discuss in detail in chapter 4.2 previous work section.)

Chapter 3

Wave Propagation in Materials

First of all, the nonlinear ultrasound technique that this research use is base on the relationship between first and second harmonic of wave propagated in materials. In this chapter, the nonlinearity parameter, β will be mathematically derived using equation of motion and explain how this parameter can be used to see the effect of cold rolling, and several heat treatments.

3.1 Equation of Motion

Starting with the general from of the linearized theory, equation (2) can be generated.

$$\int_S t\, dA + \int_V \rho f\, dV = \int_V \rho \ddot{u}\, dV \tag{2}$$

Where S is close region of surface with boundary S and area A, V is volume, u is displacement, and f and t are body force per unit mass. The next equation is the "Cauchy-Stress formula" equation (3) with stress tensor τ and orientation n.

$$t_l = \tau_{kl} n_k \tag{3}$$

Equation (2) and (3) can be then written as equation (4)

$$\int_S \tau_{kl} n_k\, dA + \int_V \rho f_l\, dV = \int_V \rho\, \ddot{u}_l dV \tag{4}$$

From equation (4) and gauss theorem, equation (5) can be generated

$$\int_V (\tau_{kl,k} + \rho f_l - \rho\, \ddot{u}_l)\, dV = 0 \tag{5}$$

The volume, V is arbitrary and the integrand of it is continuous which can generate equation (6) and the Cauchy's first law of motion can be defined.

$$\tau_{kl,k} + \rho f_l = \rho\, \ddot{u}_l \qquad (6)$$

Then the general relationship of stress tensor and strain tensor is defined in equation (7)

$$\tau_{ij} = C_{ijkl}\varepsilon_{kl} \qquad (7)$$

Using the Hook's law, equation (7) can be rewritten as equation (8)

$$\tau_{ij} = \lambda\varepsilon_{kk}\delta_{ij} + 2\mu\varepsilon_{ij} \qquad (8)$$

Where λ and μ are lame constants and defined as equation (9) where E is young's modulus and υ is Poisson's ratio

$$\lambda = \frac{E\upsilon}{(1+\upsilon)(1-2\upsilon)} \qquad \mu = \frac{E}{2(1+\upsilon)} \qquad (9)$$

By using equation of motion (6) ,equation (8), and equation of strain displacement relation equation (10) we can define equation of motion, equation (11)

$$\varepsilon_{ij} = \frac{1}{2}(u_{ij} + u_{j,i}) \qquad (10)$$

$$\mu u_{i,jj} + (\lambda + \mu)u_{j,ji} + \rho f_i = \rho\ddot{u}_i \qquad (11)$$

With the vector form, The equation (11) becomes equation (12)

$$\mu\nabla^2 u + (\mu + \lambda)\nabla\nabla \mathrm{u} = \rho\ddot{u} \qquad (12)$$

For longitudinal stress, the the body force and stress tensors depend only on one direction and variable, then the equation of motion becomes equation (13)

$$(2\mu + \lambda)u_{1,11} + \rho f_1 = \rho\ddot{u}_1 \quad (13)$$

Equation (13) leads, then to the velocity of longitudinal wave, equation (14) which is parallel to the direction of propagation and shear wave, equation (15) which is rotational waves.

$$\text{c} = c_L = \sqrt{\frac{\lambda+2\mu}{\rho}} \quad (14)$$

$$\text{c} = c_s = \sqrt{\frac{\mu}{\rho}} \quad (15)$$

3.2 Derivation of the nonlinearity parameter, β

From the equation of motion, suppose a sinusoidal longitudinal wave with single frequency and angular frequency ω. The wave is propagating through the thickness of an isotropic, nonlinear material in x-direction. The equation of motion then can be rewritten as equation (16)

$$\rho\frac{\partial^2 u}{\partial t^2} = \frac{\partial \sigma_{xx}}{\partial x} \quad (16)$$

where σ_{xx} is the normal stress in the x-direction. Because the material has a quadratic nonlinear relationship between stress and strain, the normal stress can be defined as equation (17)

$$\sigma_{xx} = \sigma_o + E_1\frac{\partial u}{\partial x} + \frac{1}{2}E_2(\frac{\partial u}{\partial x})^2 \quad (17)$$

Where σ_o is stress that was preexisted, E_1, E_2 are second and third order elastic constant. The original materials' nonlinearity parameter, β has relationship with the second and third order elastic constant which can be defined as equation (18)

$$\beta = -\frac{E_2}{E_1} = -\frac{3C_{11}+C_{111}}{\sigma_o+C_{11}} \tag{18}$$

By considering equation (16), (17), (18), equation (19) can be determined in one direction

$$\frac{\partial^2 u}{\partial t^2} = C_L^2\left[1-\beta\frac{\partial u}{\partial x}\right]\frac{\partial^2 u}{\partial x^2} \tag{19}$$

The practical solution to the wave equation, equation (19) is given in equation (20)

$$u_{out} = A_1\sin(kx-\omega t) + \frac{\beta A_1^2 x k^2}{8}\sin(2kx-2\omega t) + \cdots \tag{20}$$

The solution to the wave equation can go on to higher orders with $2\omega, 3\omega$ ... but the amplitude of second harmonic generation is given by equation (21)

$$A_2 = \frac{\beta A_1^2 x k^2}{8} \tag{21}$$

By rewriting equation (21) with isolating β, equation (22) can be defined

$$\beta = \frac{8A_2}{xk^2A_1^2} \propto \frac{A_2}{xA_1^2} \tag{22}$$

Where nonlinearity parameter β is proportional to the amplitude of second harmonic generation over amplitude of first harmonic times propagation distance. This nonlinearity parameter β is very closely related to the dislocations and stress. With this equation (22) it can be seen that the relative nonlinearity parameter β can be easily determined with first and second harmonic generated amplitudes.

Chapter 4.

Previous works on Stainless Steel 304

In this chapter, three different individual effects on 304 SS done previously from other groups and one combined effect on 304 SS will be discussed. By which all the other groups used different techniques to measure the effect of cold rolling only, sensitization only, and combined cold work and sensitization. In addition, in this chapter the recrystallization effect, recrystallization temperature of the 304 SS will be discussed in detail.

4.1 Effect of cold rolling using longitudinal wave and nonlinear ultrasound on 304 SS

Viswanath et al. [1] made some conclusion in ‘Nondestructive assessment of tensile properties of cold worked AISI type 304 SS using nonlinear ultrasonic technique’ on how the cold rolling on 304 SS can be measured using longitudinal wave and nonlinearity parameter, β. In his study, the 304 SS specimens used have chemical compositions, and dimensions as shown in Table 2.

Table 2. Chemical composition and dimensions of 304 SS specimen

C	0.08%
Cr	18.00%
Ni	10.50%
Mn	2.00%
Si	1.00%
S	0.002%
P	0.003%
Dimensions	120mm X 40mm X 12.4mm

Six specimens were then prepared with solution annealed at 1323 K, then reduced the thickness from 10% to 47% using the cold rolling. In Figure 10, the results of the longitudinal measurement, nonlinearity parameter, β, tensile strength, and yield strength respect to each percent increase of cold rolling is shown. The result demonstrates that with increase in percent cold roll, the nonlinear parameter, β, also increase dramatically. With 10 % cold rolling, the β increased up to about 18% and with 47% cold rolling, the b increased up to nearly 170% compare to solution annealed non-cold rolled specimen.

The increase in β is due to formation of dislocations, grains and dislocation of double walls, and α’ martensite as discussed in previous chapter, (2.1). With this result one can conclude that the longitudinal wave measurement technique is effective and sensitive to cold rolling of 304 SS with using nonlinearity parameter β.

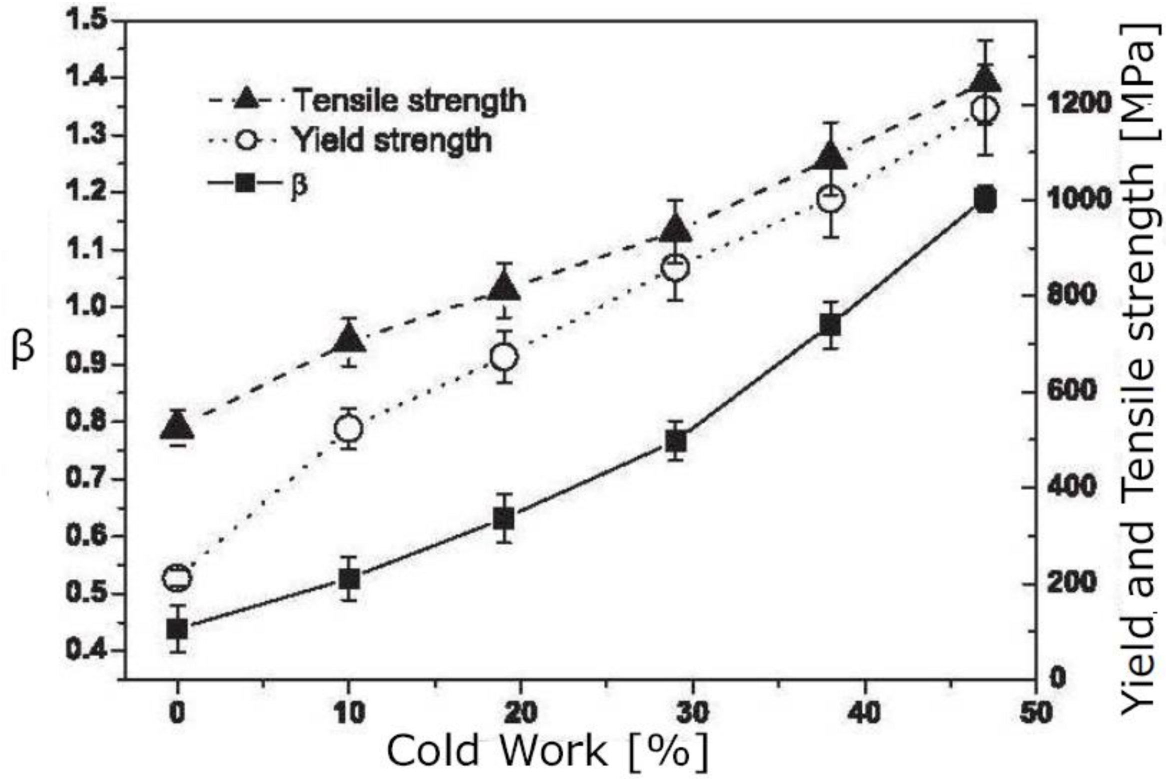


Figure 10. measurement result of cold rolling on 304 SS with nonlinearity parameter, β, tensile strength, and yield strength with respect to percent cold rolled [1]

4.2 Effect of sensitization using Rayleigh surface wave and nonlinear ultrasound on 304 SS

In Doerr [2] thesis, he evaluated effect of sensitization on 304 SS by using Rayleigh surface wave and quantify it by using the nonlinearity parameter β. First, the specimens were prepared with annealing in same oven with same time to bring the specimens to the same level. Then the specimens were put in the oven at 675 °C for different amount of holding time to sensitize differently.

Because the Rayleigh surface waves, the surface finish is very important for the contact condition of transducers and wave propagation, after the sensitization, the specimens were polished to have mirror surface finish, hand sand up to 2500 GRIT. Figure 11 shows the result of Rayleigh surface wave measurement.

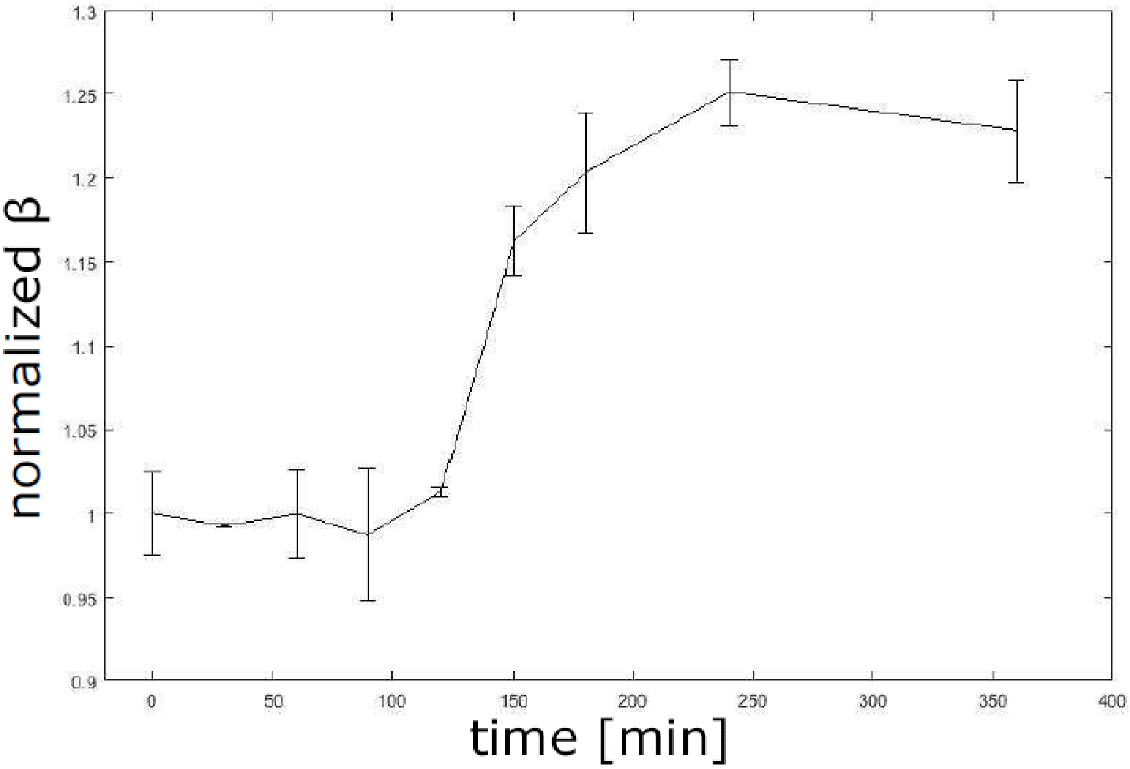


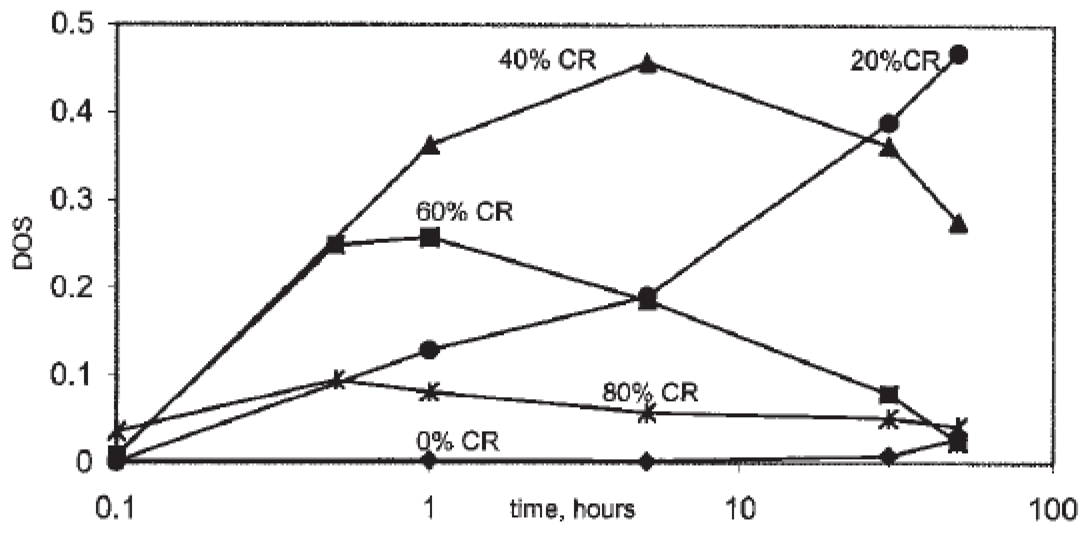
Figure 11. Nonlinearity parameter β of sensitized 304 SS with different holding time [2]

From Figure 11, one can conclude that the Nonlinearity parameter β increases dramatically after 150 min and stops at the maximum peak at 250 min and stays at constant after. At 250 min, the specimens are fully sensitized and there is no more of chromium carbide precipitates forms. As mentioned in (2.2.3), after longer holding time, the specimens will experience desensitization effect and the Nonlinearity parameter, β will decrease until it reaches zero. Also, from this result, the initializing sensitization need some time to start where there is almost no change between holding time of 0 to 150 min.

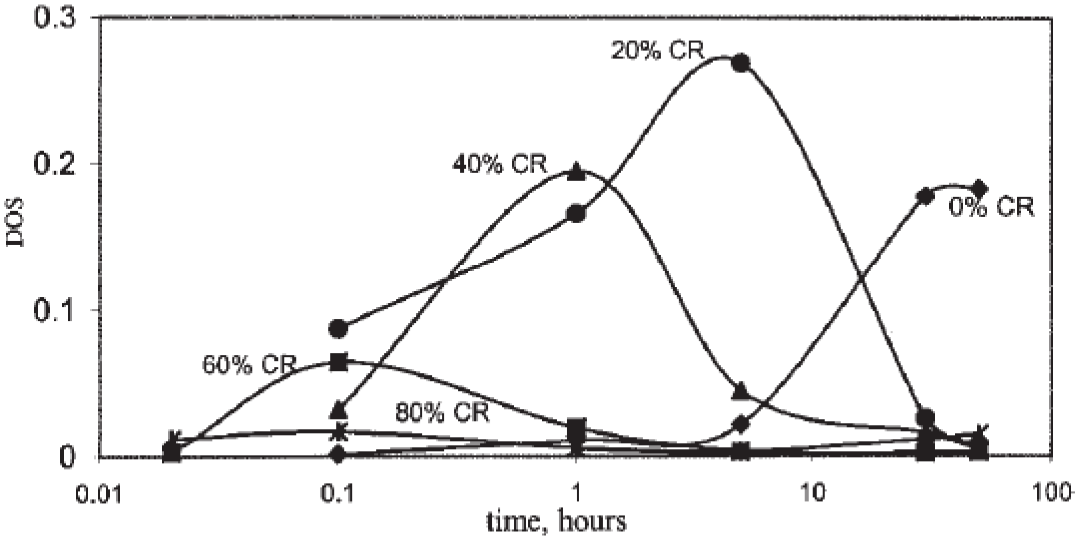
From this study, one can conclude that the sensitization, formation of chromium carbide precipitates, can be effectively measured by the nonlinear Rayleigh surface wave. Also, the nonlinearity parameter, β is very sensitive and can quantify the effect of sensitization on 304 SS.

4.3 Combined effect of cold rolling and sensitization

In one study by Singh et al. [7], the group evaluated effect of both cold work and sensitization combined. They used AISI 304 SS plate of thickness with 8mm. They first annealed the plate and reduced the thickness to 20%, 40%, 60% and 80% with cold rolling. Then each different cold rolled specimens were sensitized at 600 °C and 700 °C with different holding time. By using the electrochemical potentiokinetic reactivation (EPR) test, they observed the degree of sensitization (DOS).



(a) 600 °C



(b) 700 °C

Figure 12. Effect of deformation on DOS values at (a) 600 °C (b) 700 °C with different holding time [7]

Figure 12 shows DOS of different percent cold rolled with varying holding time. For 600 °C, Figure 12 (a), the interesting point is that DOS of 40 % cold rolled specimen increases and the maximum DOS is way higher then 60% or 80% cold rolled specimens. In addition, for 700 °C, Figure 12 (b), maximum DOS of 40 % is way higher than those of 60 % and 80 %. Also, the 20 % cold rolled specimen has highest maximum DOS. This is very interesting because the assumption before this result was that higher percentage of cold rolled, deformation, will have more effect on the sensitization.

4.4 Effect of Recrystallization on 304 SS

As mentioned in previous chapter (2.2.3), the recrystallization happens when there is imperfection in the arrays of atoms in materials with enough heat energy to rearrange to make it perfect. The recrystallization temperature for pure metals can be easily calculated using equation (1), but mixed metals like 304 SS is not valid to use this equation.

To determine the recrystallization temperature, one can assume to use closest pure metal to 304 SS, which is steel and calculate the recrystallization temperature, about 400 °C to 700 °C. [17] Manshadi et al. [3] investigated this temperature using hot torsion at different temperatures, 600 °C to 1200 °C. In Figure 13, the result of stress, strain curve at different temperature is shown.

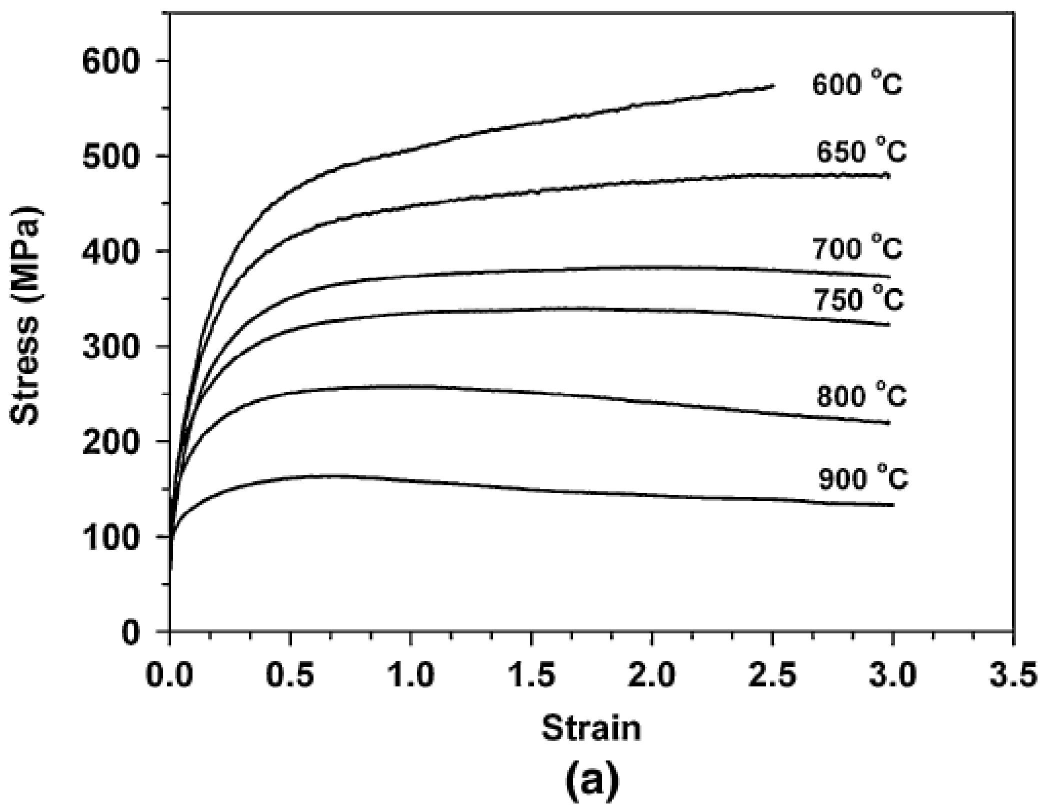


Figure 13. Stress and strain curve of hot torsion at different temperature [3]

The dynamic recrystallization (DRX) is one of evidence to notice the recrystallization temperature of mixed metals. The DRX flow curve is typically formed in stress and strain curve where as strain increases, a peak stress appears followed by long steady state. At 900 °C, it is clear that the typical DRX flow curve is shown in Figure 13, and as temperature decreases, the DRX flow curve does not appear below 700 °C which means the boundary temperature of the recrystallization is about 700 °C with strain growing, stress remains the same which shows marginal DRX flow curve phenomenon.

Also, to note that recrystallization temperature is an approximated temperature where the microstructure and strength of material changes drastically which means below this temperature, there is still recrystallization happening but not as much as in the recrystallization temperature range.

Chapter 5.

Specimen preparation and measurement

5.1 Material

The specimen used in this research is AISI 304 stainless steel. This material is widely used in many places such as mechanical facilities, engineering industries and nuclear power industries because of its resistivity of high temperature and corrosion. However, by working with this material, a lot of times it is weakened by different treatments and modification of this material to use as desired situations, such as cold work and heat treatments. These treatments and modifications can change the strength and properties of the material and ultimately, failure of the material can happen. To avoid the failure, this research is focusing on this material and how the different treatment effects the 304 SS. The chemical composition the material used in this research is given in table 3. Since 304 SS is mixed metal, the chemical composition may differ, but in this research the most important composition is the carbon and chromium for the sensitization.

Table 3. Chemical composition of AISI 304 SS

C	Cr	Ni	Mn	Cu	S	P
0.0570%	18.0850%	8.1300%	1.7585%	0.3855%	0.0010%	0.0310%

5.2 Material preparation

The original specimen 'as received' had dimensions of 6 in x 2 in x 0.610 in where 0.61 inches is the thickness. Then the specimen was cut in 8 different pieces of same size, 1.5 in x 1 in x 0.61 in as shown in figure 14. using wire EDM to avoid any stress of heating effect during the process. The dimension is chosen this way in two reason. For the thickness, the important point is that it should be thick enough to have at least 15 cycles of the signal when it is reduced to 40 % thickness. (this will be discussed in detail in next section 5.4 Signal Processing.) The other dimension, length and width, is cut to fit to the longitudinal measurement set up and have enough area to put the transducer on both sides. (the measurement set up will be discussed in detail in next section 5.3 Measurement instruments and set up)

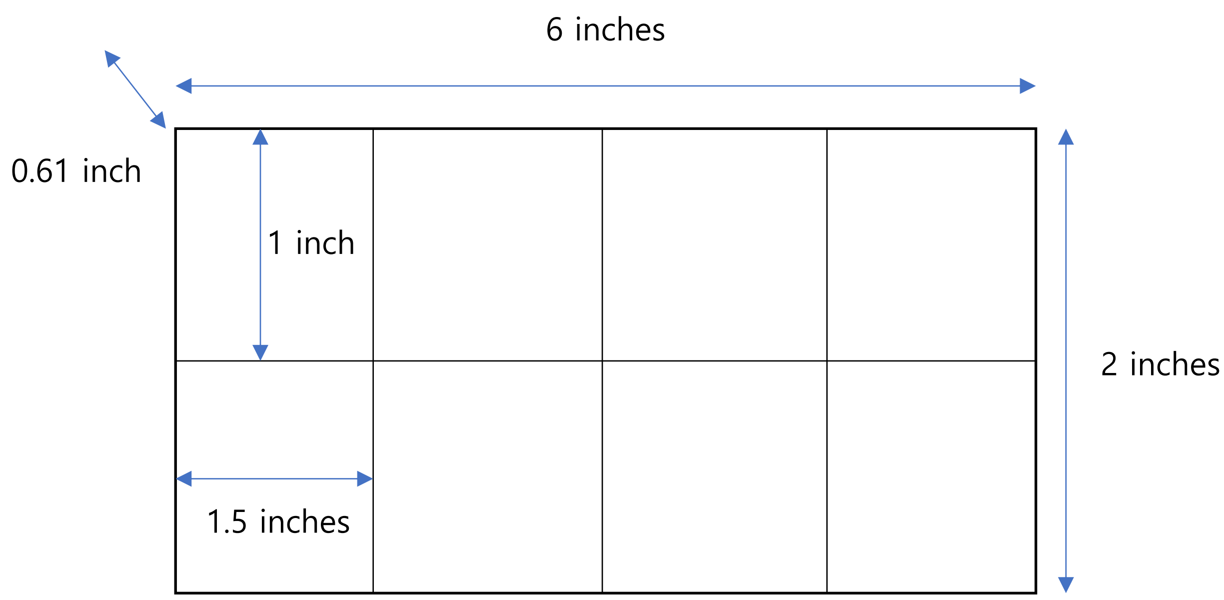


Figure 14. Dimensions of specimens

Once the size of the specimens was fixed, the specimens were annealed in 1080 °C for 1 hour of period. This allowed the specimens to have same level of starting point in which it eliminated the previously existing stress, dislocations, etc. that might affect the future measurements. After the annealing, the specimens had poor surface conditions due to oxidation and other minor chemical reactions during the annealing process and cooling. Therefore, the surface was hand polished from GRIT size of 50 to 1500. The surface preparation was done carefully using the hand sanding because the machine sanding can change some material property due to the heat effect from the fraction occurring during the process. Then, first measurements using the longitudinal nonlinear ultrasound measurement were performed to obtain the original state of nonlinearity parameter, β.

After gathering nonlinearity parameter, β of the pure state, cold rolling was performed. Out of eight, six specimens were cold rolled, ~5%, ~10%, ~15%, ~20%, ~30%, ~40% as seen in table 4. The cold machine was very old and the huge nod controlling the gap between two mills, shown in figure 2., did not have any digits. Therefore, it was very hard to control the % cold roll as desired. However, the cold rolling process was done carefully to fulfill the desired changes for different specimens. The surface condition after the cold rolling process was fine, the measurement could be done without polishing. However, for the better condition, all the cold rolled specimens were cleaned with oil to remove any dust and other impurities.

Table 4. Thickness change after cold rolling process of each specimens

Sample	Desired cold rolled	Original thickness [in]	Cold rolled thickness [in]	Actual % cold rolled [%]
A	~5%	0.611	0.576	5.728
B	~10%	0.610	0.545	10.656
C	~15%	0.609	0.514	15.600
D	~20%	0.610	0.471	22.787
E	~30%	0.610	0.430	29.500
F	~40%	0.608	0.361	40.625

After the cold rolling, the second measurement were done to ensure that only cold rolling data points were gathered for future comparison. Once the measurement of cold rolling was done, all the cold rolled specimens, A to F, and one un-cold rolled specimens were put in the oven at 675 °C for 3 hours, to fully sensitize. [2] inside the stainless bag to minimize the oxidation affect during the sensitization and cooling process. As did after the annealing process, after the sensitization, the surface condition for all specimens were poor even with the stainless bag. Therefore, the hand polishing was required. Again, the surface of all specimens was hand polished to 1500 GRIT size for the longitudinal nonlinear ultrasound measurement.

5.3 Measurement instruments and procedure

5.3.1 Function generator and amplifier

In this research, computer software call “SNAP system for RM-116 from RITEC,” and “RITEC Advanced Measurement System Ram-5000,” were used to both perform and control the function generator and amplifier. The signal was induced to the transducer from this system to travel through the specimens each time. It operates tone burst signal at 5 MHz, using the 5 MHz transducer to start the signal. (Will be discuss in detail in next section 5.3.2) The signal created with this system was set at 15 cycles with 1.1ms of burst delay. Then the output amplified level was set from 17 to 43, which varies from about 4 V to about 18.5 V peak to peak at traveling each specimen. This acoustic energy is enough to generate and obtain the change of second harmonic waves.

5.3.2 Oil coupled transducer

Two different transducers were used in this research, 5 MHz and 10 MHz transducers. Both transducers were Panametrics piezoelectric narrow band transducer which can create longitudinal waves. Once the signal is created by the function generator and amplified using the previous system, 5 MHz transducer creates signal that travels through the specimen, and the 10 MHz transducer receives and capture the first and second harmonic waves. For the contact condition, the oil was applied between both transducers and coupling the specimen.

5.3.3 Oscilloscope

The oscilloscope was used to visualize and save the incoming signals from the received transducer, 10MHz. The oscilloscope captures average of 512 waveforms over one time-domain signal and the sampling rate is 500Ms/s. Once the signal is visualized by the oscilloscope, it was saved as csv. File for further signal processing using MATLAB.

5.3.4 Longitudinal measurement setup

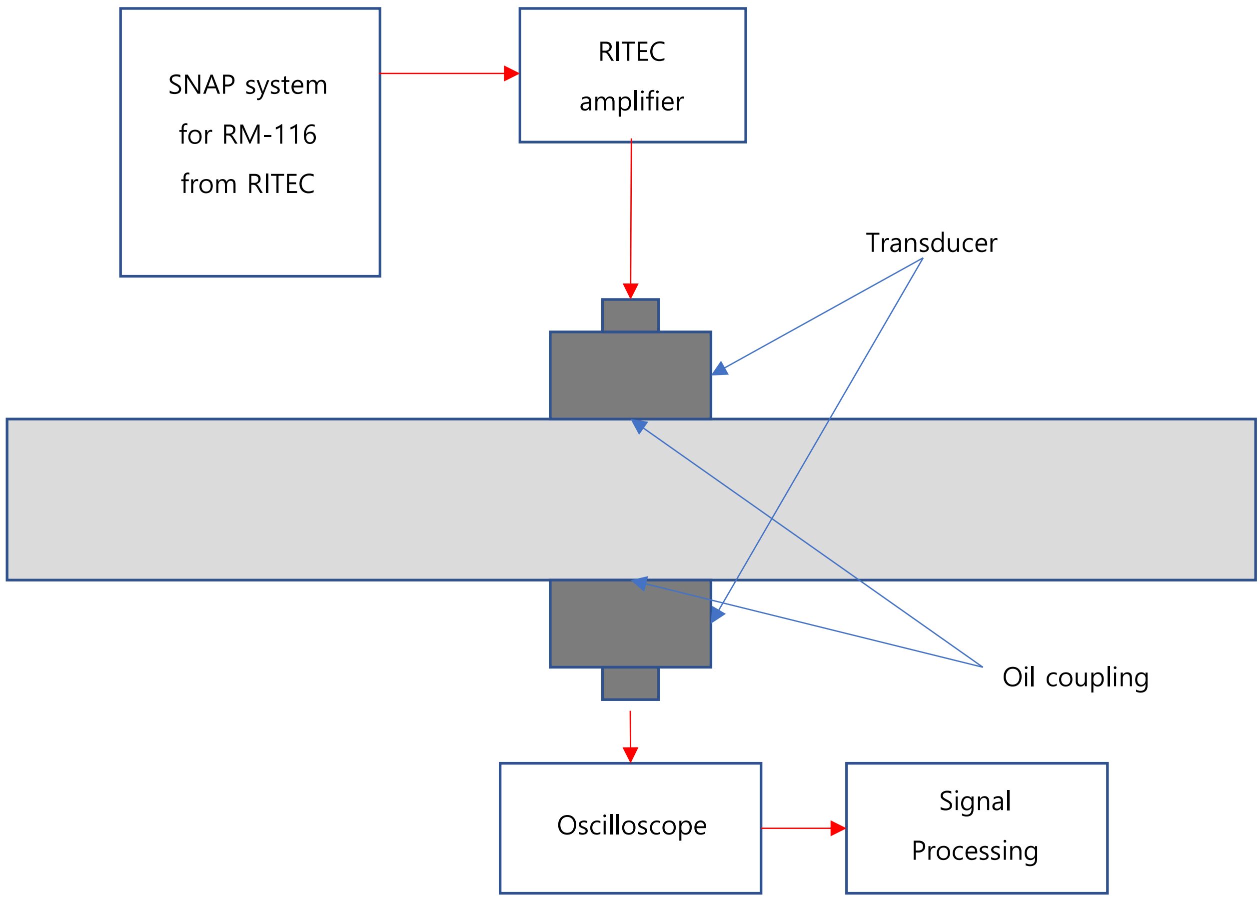


Figure 15. Longitudinal wave measurement setup

As can be seen in the Figure 15, The signal is controlled by the "SNAP system for RM-116 from RITEC," and RITEC amplifier to create different signals with increasing amplitudes. Once the signal is sent to the 5 MHz transduce, the signal travels through the specimen and the 10 MHz transducer receives it and send it to the oscilloscope for visualization. The oil coupling between the transducers and the specimen are for the better contact conditions. At oscilloscope, the signals are averaged 512 times and saved as csv. Files to further signal processing.

5.4 Signal Processing

First of all, the contribution of each instruments, function generator, amplifier, transducer and oscilloscope, need to be considered. The each instruments have their own nonlinearity that effect the materials' nonlinearity. It is not possible to measure how much each instrument effect the nonlinearity of the material. Therefore, the relative nonlinearity β', (equation 23) can be used instead of nonlinearity parameter β by using equation 22 as provided in previous chapter 3.2

$$\beta' = \frac{A_2}{xA_1^2} \tag{23}$$

To determine the β' the amplitude was varied from 17 output level to 43 output level and saved at each csv. files. Then using the MATLAB, out of 15 cycles, bad signals were eliminated as shown in Figure 16., only selected part between 2 red dots were picked.

However, there should be at least 8 cycles of signal to ensure the accuracy of β' [8]

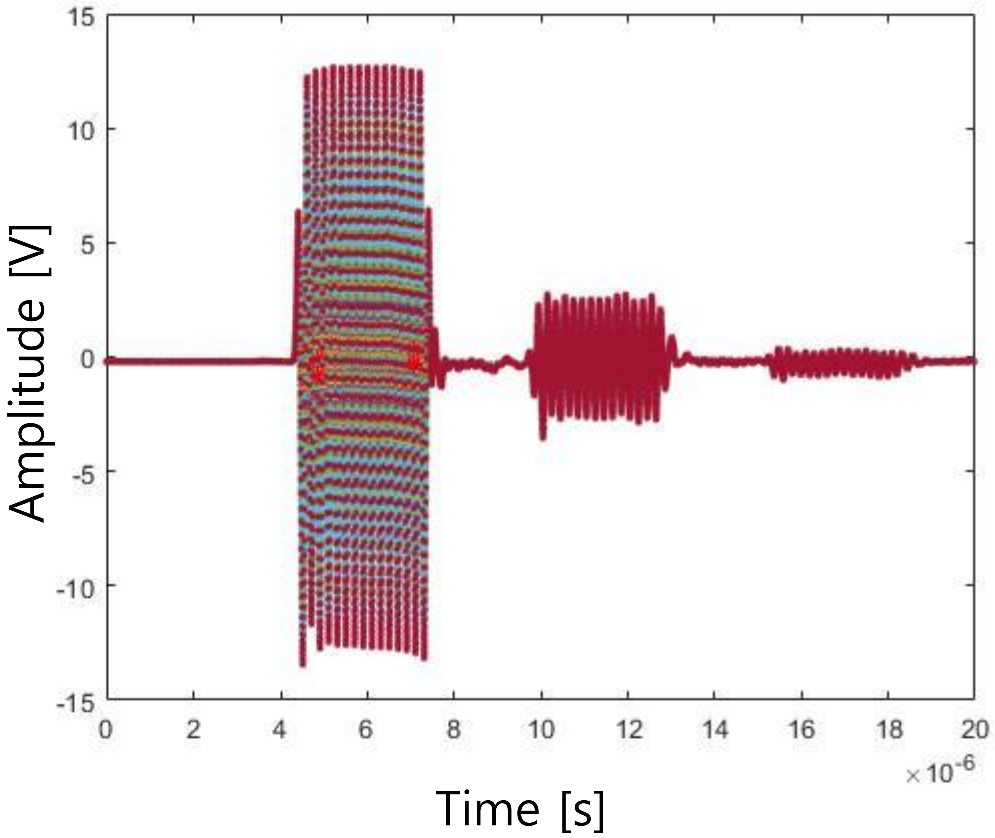


Figure 16. Time domain signal

Then each signal was transformed from time domain to frequency domain using the Fast-Fourier-Transform (FFT), as shown in Figure 17. The fundamental and second harmonic waves, A_1 and A_2 are well displayed the clear peaks. From different amplitude out levels, Figure 18 can be generated. Figure 18 shows the relationship between square of the first harmonic amplitude and second harmonic amplitude over the change in amplitudes. Form this, we can easily think that the slope of the plot is the $\frac{A_2}{A_1^2}$ and by dividing by x, the propagation distance or thickness of the specimen, the relative nonlinearity parameter β' is obtained.

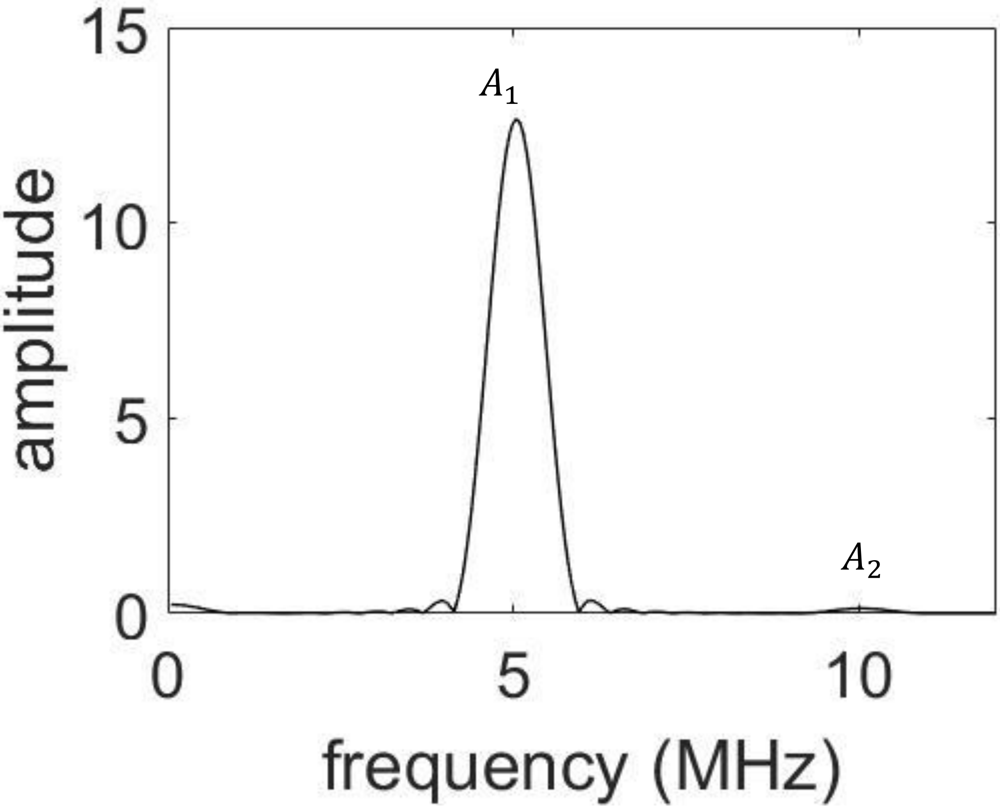


Figure 17. Frequency Domain signal using FFT

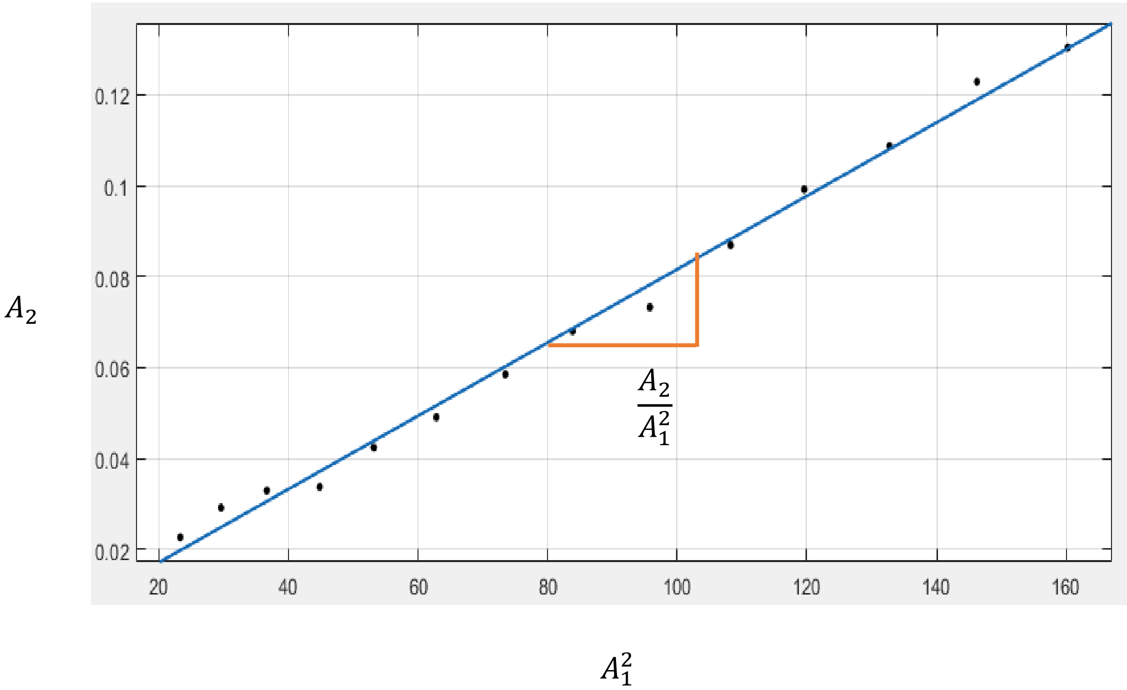


Figure 18. Relationship between square of first harmonic and second harmonic amplitudes

Chapter 6.

Results and Discussion

In this chapter, the results of the longitudinal wave NLU measurements of cold rolling only and cold rolling and heat treatment combined on 304 SS are presented. Then the results are compared with previous work to validate. The specimens experienced three different phenomena: cold rolling, sensitization and recrystallization in this research and the results are as follows.

6.1.1 Longitudinal measurement of cold rolled 304 SS

The series of longitudinal wave NLU measurements are made after cold rolling each specimen with reducing to different thickness after annealing process. As shown in table 4, each specimen was cold rolled close to the desired percent change in thickness. However, the cold rolling machine was not precise, and the actual thickness does not exactly match with the desired thickness.

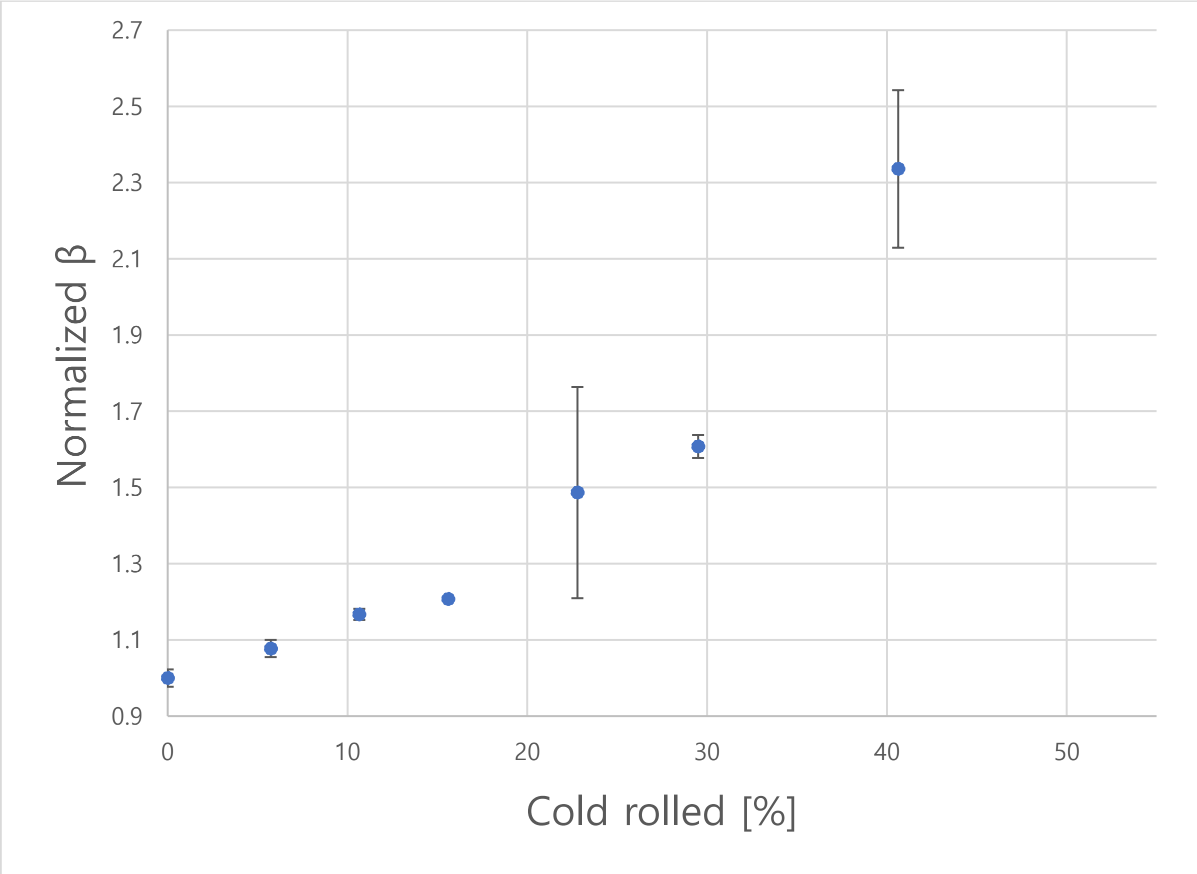


Figure 19. Percent change in β with change in percent cold rolled after effect of cold rolling alone

Table 5. Result for cold rolled 304 SS

Cold rolled [%]	averaged A2/A2^2 (x10^-3)	Propagation distance x	averaged β (x10^-3)	STD	normalized β
0	1.002	0.611	1.640	0.023	1.000
5.728	1.026	0.5805	1.767	0.023	1.077
10.656	1.044	0.545	1.915	0.015	1.167
15.6	1.018	0.514	1.981	0.000	1.207
22.787	1.149	0.471	2.439	0.277	1.487
29.5	1.134	0.43	2.637	0.030	1.608
40.625	1.383	0.361	3.832	0.207	2.336

Figure 19 and table 5 shows the result of normalized β in terms of percent change in cold rolling. First, the $\frac{A_2}{A_1^2}$ was calculated from the slope as seen in figure 18. The slope was calculated with trend line with R^2 over 95%, in most cases, R^2 was above 99%. Because the propagation distance is same as the thickness of the specimen, propagation direction is alone the thickness of the specimen, to calculate nonlinearity parameter, β, the $\frac{A_2}{A_1^2}$ needs to be divided by the thickness of each specimen. In addition, the scattering or the standard deviation of nonlinearity parameter, β, is shown in Table 5 with the highest 27%, which shows the repeatability of this measurement is valid.

As can be seen from these results, for 5 % cold rolled specimen, about 8 % change in the measured nonlinearity, β. Also, for 10 % cold rolled specimen, the nonlinearity increased by about 16 %. For 40 % cold rolled specimen, the nonlinearity, β changed more than twice that of the original specimen. From figure 19, it is clear that the in β parameter of the cold rolled specimens increases monotonously with the increase in percent cold rolling.

These results are well matched with the theory of cold work that was described in previous chapter. (2.1 Cold Work) As cold rolling changed the thickness of the specimens, the specimen experiences plastic deformation. With this plastic deformation, the microstructure of the specimen is affected, and the dislocations and stress are introduced. These effects occur even more with increase in more plastic deformation, in this case increase in percent cold rolling which effect the nonlinearity of the specimen. With 40% plastic deformation using the cold rolling, the nonlinearity parameter, β changed by 133% which demonstrates that the nonlinear longitudinal NLU wave measurement technique is very sensitive and effective to capture the behavior of cold work.

6.1.2 Comparison of result of longitudinal wave on cold rolled specimen to previous study

As mentioned in the previous chapter, (4.1), Viswanath et al. [1] evaluated longitudinal measurement of cold rolled 304 SS. The result from this study and the result from this research is put together in Figure 20, for easy comparison.

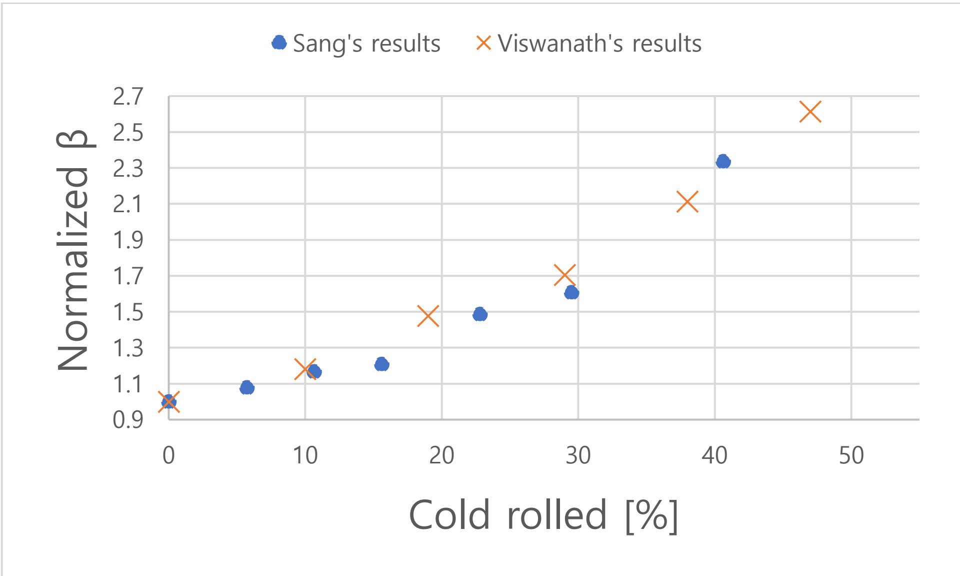


Figure 20. Comparison of result from previous work of longitudinal measurement of cold rolled 304 SS

Despite the fact that the two studies have different cold rolling steps were taken, Viswanath et al. [1] had 0%, 10%, 19%, 29%, 38%, and 47% where as in this research, 0%, 5.7%, 10.7%, 15.6%, 22.8% 29.5% and 40.6%, the result in figure 20 shows that the trend line of the normalized nonlinearity parameter β over the change of percent cold rolled match perfectly. This approved that the measurement of longitudinal wave to determine the nonlinearity parameter β is valid.

6.1.3 Longitudinal measurement of cold rolled and heat treatment combined 304 SS

After the cold rolling measurement, the six specimens and one pure specimen, which did not experience any changes of thickness with cold rolling, were put in the oven at 675 °C for 3 hour and cooled with air at room temperature for 1 day. The temperature 675 °C and the holding time of 3 hours should give the specimens fully sensitized. [2] This is different than other work on sensitization, [2] [6] [13] [10], where here the holding time was fixed to fully sensitize the material and, to obtain the effect of cold rolling of 304 SS to maximum sensitization.

After the sensitization, the surface condition of all specimens was very poor because of the oxidation effect on the surface. Therefore, all specimens were hand polished with increments of 50, 80, 120, 240, 300, 600, 800, 1000, to 1500 GRIT size. The polishing was done carefully with hand to avoid any other heating effect or damage to the specimen. Then the longitudinal wave measurement was performed as it was done after the cold rolling of the specimens.

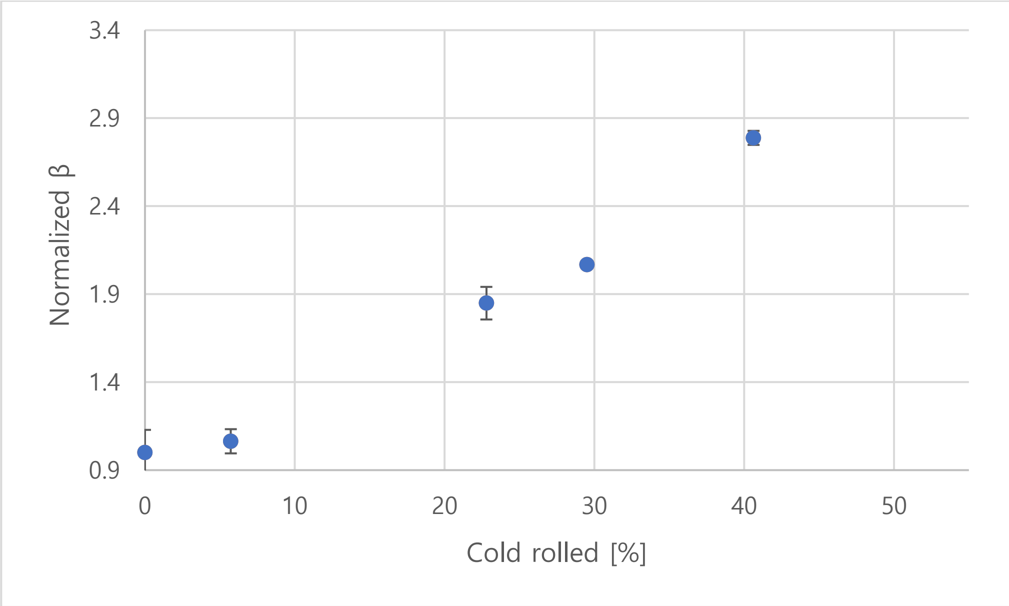


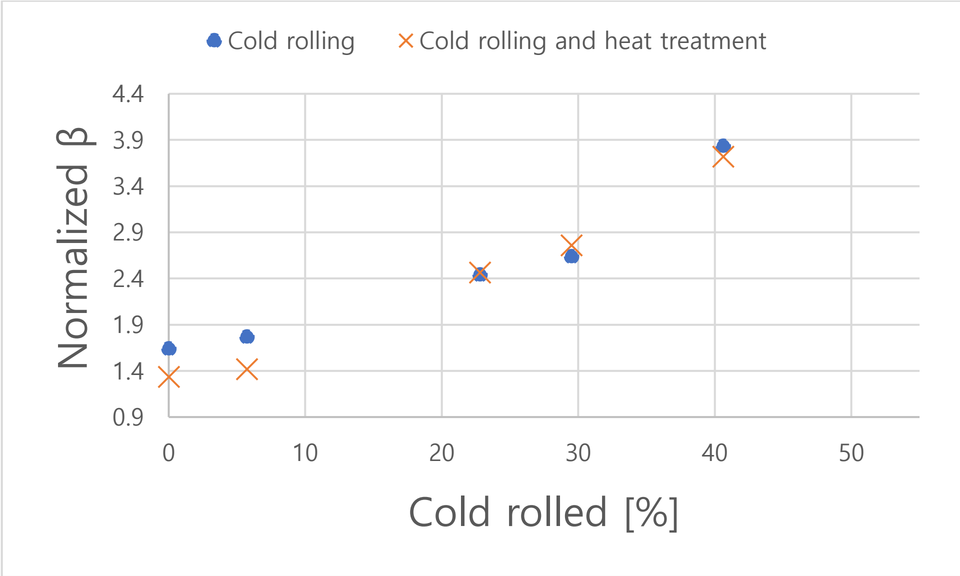
Figure 21. Percent change in β with change in percent cold rolled after cold rolling and heat treatment effect combined

Table 6. Result of cold rolled and heat treated 304 SS

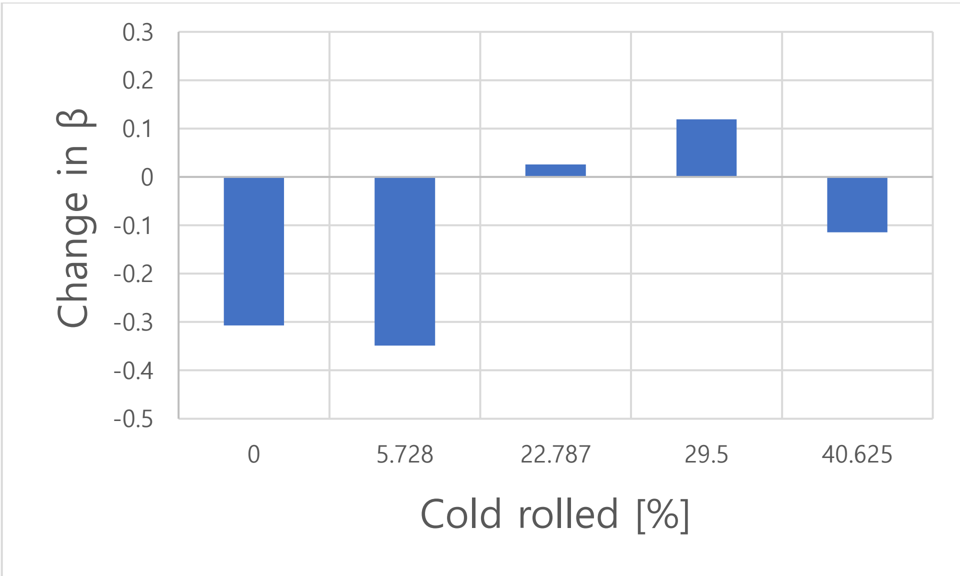
Cold rolled [%]	Averaged $\frac{A_2}{A_1^2}$ $(X\ 10^{-3})$	Propagation Distance x	Averaged β $(X\ 10^{-3})$	STD	Normalized β
0	0.8147	0.611	1.333	0.01288	1
5.728	0.8236	0.5805	1.419	0.00687	1.0641
22.787	1.161	0.471	2.465	0.09260	1.8487
29.5	1.185	0.43	2.757	0.00208	2.067
40.625	1.342	0.361	3.717	0.004	2.788

The results of cold rolled and heat treated 304 SS is provided in Figure 21, and Table 6. Like the effect of cold rolling only, the averaged $\frac{A_2}{A_1^2}$ were calculated using the trend line with R^2 was above 95%, for this case again the most R^2 for the trend line were above 99%. The nonlinearity parameter, β, is function of propagation distance, in which the thickness, averaged $\frac{A_2}{A_1^2}$ were divided with the thickness of each specimen. Also, the scattering or the standard deviation of nonlinearity parameter, β, is shown in Table 6 with highest 9%, which shows the repeatability of this measurement is also valid.

As can be seen from the result, the heat treatment made the percent change in nonlinearity parameter, β. For ~5% cold rolled, the β increased about 6.4%, for ~23% cold rolled, the β increased about 85%, for ~30% cold rolled, the β increased about 107% and for ~40 % cold rolled, the β increased about 180%. However, it is important to note that the specimens below 20 % cold rolled have actually decrease in increment of the β compared to cold rolled only effect, for example, ~5% cold rolled, the cold rolling only effected about 7.7% increase in β whereas the cold rolling and heat treatment combined made the increase in β only 6.4%. The specimens above 20% cold rolled, on the other hand, increase in increment of the β, for example, ~40 % cold rolled, the cold rolling only effected about 133% whereas the cold rolling and heat treatment combined made the increase in β about 180%.



(a)



(b)

Figure 22. Change in β of cold rolled only to cold rolled and heat treated combined with different percent cold rolled (a) scatter plot (b) bar graph

Table 7. Averaged β of cold rolling only, cold rolling and heat treatment combined and change of β between the two.

	Cold Rolling Only	Cold Rolling and Heat Treatment Combined	Change in β	
Cold rolled [%]	averaged β (x10^-3)	averaged β (x10^-3)	Change in β (x10^-3)	Change in β [%]
0	1.6405	1.3334	-0.3071	-18.72
5.728	1.7674	1.4188	-0.3486	-19.72
22.787	2.4391	2.4650	0.0259	1.06
29.5	2.6372	2.7566	0.1194	4.53
40.625	3.8319	3.7175	-0.1145	-2.99

In figure 22 and table 7, the difference of β of specimens that experience only cold rolling and cold tolling and heat treatment combined are shown. Specimen that experienced low cold rolling, below ~20% cold rolling, actually decreased the nonlinearity parameter β after the heat treatment effect. However, for specimen that experienced higher cold rolling, about ~20% cold rolling, increased the nonlinearity parameter β after the heat treatment effect.

At the temperature of 675 °C, the specimen experiences two different heating effects. The first one is the sensitization, where chromium meet carbon with enough heating energy, they form chromium carbides precipitants which then it leads to the chromium depletion and the nonlinearity of the specimen increases over holding time. Since the holding time was fixed at 3 hours, which makes the specimen fully sensitized. [2] After this effect of sensitization, the nonlinearity of the specimen should be at a maximum, which means that the nonlinearity parameter β should increase. However, with combination effect of cold rolling and sensitization, this was not true. As mentioned in the previous chapter, (4.3) Singh et al. [7] conducted an experiment of sensitization after cold rolling on several specimens, and by using EPR test, the degree of sensitization (DOS) of different percent of cold rolled specimens at 600 C and 700 C were presented.

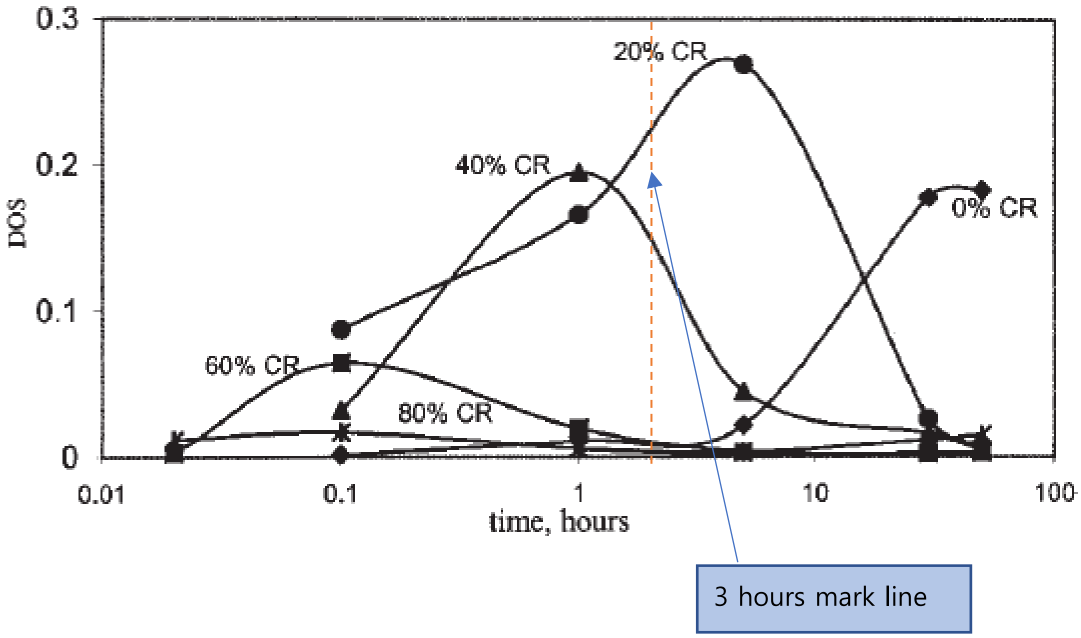


Figure 23. Recreated DOS of different percent cold rolled 304 SS over time with 3 hours mark line

The result from Singh et al. [7] was recreated with 3 hours mark line in figure 23 for comparison of result from this thesis where the heat treatment was held at 675 °C with holding time of 3 hours. According to these results, at 700 C, the DOS of 0% cold rolled specimen did not see any changes at 3 hours. Also, DOS of 20% cold rolled specimen increased dramatically at 3 hours, even increased more than 40% cold rolled.

This result from Singh et al. [7] may have slightly different temperature than the heat treatment temperature that the specimens in this thesis experiences but will have very similar effect of sensitization at different prior cold rolled percent. Also, the increments of percent cold rolled are different, for example, there is not existing data of 30 % cold rolled, but we can assume that these will have similar behaviors.

The second heating effect that the specimens experienced at the temperature of 675 °C is the recrystallization. As mentioned in the previous chapters, (2.2.3), (4.2), the recrystallization temperature for the mixed metal cannot be determined using the equation (1), relationship between the melting temperature and the recrystallization temperature. However, according to the study from Manshadi et al. [3], the recrystallization temperature range of 304 SS can be assumed to start around 700 °C with the DRX flow curve behavior, and the recrystallization happens below the recrystallization temperature but not as drastically as the recrystallization temperature range. [16]

Therefore, when different percent cold rolled specimens were heat treated at 675 °C, these specimens experienced both sensitization and recrystallization effect. For the case of ~20% below cold rolled specimens, the DOS does not increase with the sensitization, but the recrystallization is present. This lead to decrease in nonlinearity of the specimen because the recrystallization effect can be considered as self-healing and this effect dominated the sensitization effect. However, for the case of ~20 % cold rolled specimens, the DOS is very high at 3 hours mark line. The recrystallization is also affecting the ~20 cold rolled specimen, but the DOS dominates the recrystallization effect and the nonlinearity of the specimen should increase.

The result from the longitudinal wave measurement after combined effect of cold rolling and heat treatment matches with this theory. As can be seen in Figure 22, 20 % below cold rolled and heat-treated specimens have negative change in nonlinearity parameter β from cold rolled only specimens. For the cases of 20% above cold rolled and heat-treated specimens have positive change in nonlinearity parameter β from cold rolled only specimens. This result matches perfectly with the theory behind the heat treatments of sensitization and recrystallization effect happening at the same time.

Chapter 7.

Conclusion

This research used a nonlinear ultrasonic (NLU) procedures using longitudinal waves to observe the change of nonlinearity and microstructure of 304 SS due to cold rolling, plastic deformation, and heat treatment effect. The nonlinearity parameter β was calculated with the first and second harmonic waves generated by the longitudinal wave which propagated through the specimen of 304 SS after each effect, cold rolling, and heat treatment combined.

For the measurement of cold rolling effect, the result was presented with the change of nonlinearity parameter β with respect to percent cold rolled of 304 SS. The result showed that the nonlinearity parameter β is very sensitive to cold rolling, especially above ~20% there was drastic increase in β. The increase in β with respect with the increase in percent cold rolling has increased monotonously. Also, the result was then compared with the previous work from Viswanath et al. [1] and the result from this thesis and from the previous work matched perfectly proving that the longitudinal wave measurement technique is valid, and the nonlinearity parameter β is very sensitive to cold rolling.

For the measurement of combined effect of cold rolled and heat treated 304 SS, the specimens were first cold rolled with different percent reducing in thickness, then these specimens were heat treated at 675 °C for 3 hours, where the specimen should have enough time and heat energy to fully sensitized. However, at this temperature of 675 °C, 304 SS experienced two different heating effects, sensitization and recrystallization.

The nonlinearity of 304 SS that experienced only sensitization should increase due to the formation of chromium carbide precipitates and depletion of chromium. When the holding time for heat treatment of 304 SS is within the sensitization, below holding time for desensitization which is about 10 hours, as holding time increases, the degree of sensitization (DOS) also increases. In addition, 304 SS with experiencing more sensitization, nonlinearity of the 304 SS increases which means nonlinearity parameter β also increase. However, the 304 SS experiencing cold rolling and sensitization combined at 700 °C, the DOS changes in a completely different fashion with increasing holding time. For ~20 % below cold rolled specimens, the DOS does not change with the increase of holding time, almost zero at 3 hours line. For ~20 % cold rolled specimens, the DOS increases with the increase of holding time, highest DOS among 40%, 60%, 80% cold rolled.

In addition, the recrystallization is considered self-healing, which movement of unorganized atoms with heat energy makes the new crystals in the microstructure to form more perfect arrays. At 675 °C for 3 hours, the cold rolled specimens, with unorganized microstructure due to formation of dislocations, experiences both sensitization and recrystallization effect. For ~20 % below cold rolled specimens, the recrystallization effect dominates the sensitization effect because the DOS of ~20 % below cold rolled are almost zero at 3 hours. For the 20 % cold rolled specimens, the sensitization effect dominates the recrystallization effect because the DOS of 20% cold rolled is very high. From the result of combined cold rolling and heat treatment effect measurement prove this theory with increasing nonlinearity parameter β for 20% cold rolled and heat treated specimen, and decrease in nonlinearity parameter β for ~ 20 % below cold rolled and heat treated specimen, which concludes that the effect of cold rolling and heat treatment has a complex relationship rather than a directly proportional relationship.

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