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Influence of Microstructural Changes' Effects on the Linear and Nonlinear Ultrasonic Parameters of Cast Stainless Steels

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Abstract: In this research, some nondestructive ultrasonic techniques were employed to inquire into the effect of microstructural changes induced by thermal aging and cold work on the ultrasonic response. As thermal embrittlement is a risk to the safety of nuclear power plants, a nondestructive detection method has to be developed for on-site monitoring. The austenitic stainless steel with δ -ferrite specimens were used to study the behavior of microstructural changes caused by age-treating and cold work and then examined by the velocity, attenuation, and nonlinear ultrasonic technique. The variations of the linear and the nonlinear ultrasonic parameters were related to the microstructural changes. Additionally, the experimental results suggest that the ultrasonic nonlinearity parameter of cast stainless steel is determined by the microstructure evolution caused by spinodal decomposition and the phase precipitation process.

Keywords: microstructural change; nonlinear ultrasonic; cast stainless steel; nondestructive examination

1. Introduction

The cast stainless steel with a two-phase microstructure of austenite (γ) and δ -ferrite have extensively been applied in nuclear industry for a long time because of their great corrosion resistance, high mechanical strength, and good weld ability [1,2]. Some of the cast stainless steels, CF3, CF3M, CF8, CF8M, and CF20, have been used in the power plant for valves, pump casings, and the primary loop piping of pressurized water reactors (PWR). Even though these stainless steels have excellent performance, there are various mechanisms that may lead to the degradation of cast stainless steel during reactor service. One of the mechanical property degradation mechanisms is thermal embrittlement [3–5]. The high temperature is a major concern for the cast stainless steel pipe of power plants due to the microstructural changes in the ferrite phases. The spinodal decomposition is produced after a long-term isothermal aging treatment at the range 300–500 °C [6–8]. The reaction of decomposition can be represented as the δ -ferrite transformed into a chromium-rich α' phase and an iron-rich α phase.

Even though just a phase transformation happened in the ferrite phases, the brittleness is a gradual increase in the whole cast stainless steel component. When the α' phases are precipitated, the hardness of ferrite phases would increase and become brittle. If the ferrite phases are brittle, the interface between austenite (γ) and ferrite has the potential to lead to cracking. Under the irradiation condition, the irradiation would cause further hardening and make the phase become more brittle. A crack may initiate a repetitive process during a long operation time, affecting cast stainless steel component integrity.

In the light of this, the detection and evaluation of microstructural changes of cast stainless steel has become more important with the growing demands of power plants. Up to now, the destructive tests in laboratories have commonly been used for phase evaluation, these techniques are time-consuming and costly. It is of great interest to develop some non-destructive techniques for real time detection and evaluation of microstructural change. In the previous studies, ultrasonic-based non-destructive testing (NDT) techniques were employed for monitoring the materials [9–12]. The ultrasonic method is a powerful nondestructive way of evaluating material since the characteristics of ultrasonic waves are related to the properties of the material. In this research, some ultrasonic methods were used to evaluate the microstructural change of cast stainless steel.

In recent years, an inspection method known as nonlinear ultrasonic technique has proved to be sensitive enough for detection of material micro-damage and evaluation of change of microstructure. A theoretical model was proposed by Kim et al. [13] to relate the nonlinear ultrasonic parameter to the local strain. The results obtained by Chen et al. [14] on damage of granite samples show that the nonlinear parameter is found to have a positive correlation with the loading level. Moreover, the experimental measurements by Matlack et al. [15] indicate a decrease in the ultrasonic nonlinearity parameter with increasing heat treatment time. The results obtained by Li et al. [16] show that the nonlinear ultrasonic parameter has a close relationship with structural cracks. Wang et al. [17] employed nonlinear ultrasonic measurements to characterize the creep damage in ASME T92 steel welded joints.

The traditional ultrasonic method is based on linear theory and normally determines the properties of a material by measuring some ultrasonic parameters, such as velocity, attenuation, transmission, and reflection coefficients. However, it is difficult to detect and evaluate the evolution of microstructure by the conventional ultrasonic technique. However, the nonlinear ultrasonic technique overcomes this limitation. It transfers time-domain signals into frequency-domain to analyze the information of frequency which would be related to the discontinuities of material. This phenomenon is due to the incident wave distorted by the nonlinear elastic response of material so that the higher harmonic waves are generated in the transmitted wave [18]. This nonlinear wave behavior appears in the form of dislocation substructures, such as slip bands. The slip bands would accumulate in the grain or at the grain boundaries which may cause strain localization. A number of investigators have studied the nonlinear ultrasonic technique to measure the effect of dislocation density. In the case of precipitate, the microstrain induced plasticity would change between the precipitate and the matrix. In addition, the precipitate would interact with dislocations which lead to changes in the nonlinear ultrasonic parameter [19]. It is expected to be much more sensitive to microstructure than the linear ultrasonic wave. From the studies above, the nonlinear ultrasonic technique shows the potential for characterizing the microstructural changes.

The generation of higher order harmonic waves in a solid material is related to the nonlinear relationship between the stress and strain of material and can be expressed by the nonlinear Hook's law as shown in Equation (1) [20]

$$\sigma = E\varepsilon + E\beta\varepsilon^2,\tag{1}$$

where σ is stress, ε is strain, *E* is Young's modulus, and β is the nonlinear acoustic coefficient. Considering the case of a single frequency ultrasonic longitudinal wave propagating in a material, and assuming that the attenuation can be neglected, the equation of motion for longitudinal waves in the material can be represented by

$$\rho \frac{\partial^2 u}{\partial t^2} = \frac{\partial \sigma}{\partial x},\tag{2}$$

where ρ is the material density, *u* is the displacement, and *x* is the propagation distance. The relationship between strain and displacement for a one-dimensional longitudinal wave is given as

$$\varepsilon = \frac{\partial u}{\partial x},\tag{3}$$

Substitution of Equations (1) and (3) into Equation (2) yields

$$\rho \frac{\partial^2 u}{\partial t^2} = E \frac{\partial^2 u}{\partial x^2} + 2E\beta \frac{\partial u}{\partial x} \frac{\partial^2 u}{\partial x^2},\tag{4}$$

In order to obtain a solution, the displacement is assumed as

$$u = u_0 + u', \tag{5}$$

where u_0 is a solution with $\beta = 0$ and u' is the first order perturbation solution. Then, the solution of Equation (4) can be obtained as

$$u = A_1 \cos(kx - wt) - \frac{\beta A_1^2 k^2 x}{8} \sin 2(kx - wt),$$
(6)

where A_1 , k, and w are the amplitude of the fundamental frequency component, the wave number, the angular frequency, respectively. The amplitude of the second harmonic wave can be described as

$$A_2 = \frac{\beta A_1^2 k^2 x}{8},$$
 (7)

Therefore, the parameter β , which is also called the nonlinear parameter, can be evaluated from A_2 , as shown in Equation (8)

$$\beta = \frac{8A_2}{k^2 x A_1^2},$$
(8)

In this paper, the frequency of longitudinal wave and the transmitting distance are fixed. The parameter β is proportional to $\frac{A_2}{A_1^2}$. For convenience, a relative nonlinear ultrasonic parameter is adopted and determined as

$$\beta' = \frac{A_2}{A_1^2},\tag{9}$$

Therefore, the variation of microstructural change could be characterized by measuring A_1 and A_2 .

This is a feasibility study intended to characterize microstructural change of the cast stainless steel using the linear and nonlinear ultrasonic techniques. Measurements of the ultrasonic parameters were carried out on the cast stainless steel specimens with different aging times and cold work. The relationship between ultrasonic parameters and the material property were correlated based on the ultrasonic responses to the variations of aging times.

2. Materials and Experiment

The materials used in this work were static cast austenitic stainless steel to the dimensions of $20 \times 20 \times 10$ mm. Two kinds of samples were statically cast from high ferrite (HF) and low ferrite (LF) content cast steels (Table 1). The ferrite contents are 22.83% for HF and 7.48% for LF, respectively. The samples of HF and LF cast stainless steels were solution treated-annealed at 1040 °C for 1.5 h and cooled in the furnace and then age-treated at 385 °C for 2400, 6000, and 30,000 h. The unaged sample (0 h) for each condition was kept as a reference. To obtain samples with microstructural change from cold work, two cold rolled samples made from static cast stainless steel were tested. The specimens deformed with 10% were prepared for phase observation and ultrasonic analysis. For observation of microstructure evolution caused by thermal ageing and cold working, light microscope (LM), hardness measurement (Rockwell and Vickers methods, load of 25 g, five Rockwell measurements for one sample) and SEM/EBSD method (Oxford Instruments Plc, Abingdon, UK) were carried out to characterize changes of microstructure.

Table 1. Chemical composition of cast stainless steels (wt%).

	С	Cr	Ni	Si	Mn	Р	S	Fe
LF	0.048	18.79	8.67	0.42	1.2	0.067	0.005	Bal
HF	0.045	21.66	8.44	0.44	1.1	0.063	0.007	Bal.

For linear ultrasonic measurement, Figure 1 shows the commercial ultrasonic thickness gauge (Krautkramer Branson, Lewistown, PA USA) and a calibration block for velocity measurement, and Figure 2 shows the schematic sketch of the experimental setup for attenuation measurement. The arm with a weight sensor attached would push on the transducer with the same weight. The weight tested by an electronic scale is shown in Table 2. From Figure 2, there was just a reflect echo from the bottom. Therefore, an ultrasonic thickness gauge was used and calibrated by a calibration block to avoid the zero point error. The velocity could be calculated by measuring the velocity of the calibration block and the thickness of samples. The velocity *v* is given as

$$v = v_0 \times \frac{x}{x_0} \tag{10}$$

where v_0 is the velocity of the calibration block, x is the real thickness of the sample, x_0 is the measured value the of thickness. The amplitude of waves would decay during propagation. However, it is an ordeal to calculate the attenuation coefficient from the only echo. A relative attenuation parameter γ is defined as

$$\gamma = \frac{I_0}{I_1} \tag{11}$$

where I_0 is the amplitude of the unaged samples (0 h), and I_1 is the amplitude of the aged/cold rolled samples.



Figure 1. The commercial ultrasonic thickness gauge and a calibration block.

For nonlinear ultrasonic measurement, Figure 3 shows the schematic sketch of the experimental setup. A high power tone-burst amplifier RITEK SNAP RAM-5000 (RITEC Inc., Warwick, RI, USA) was used to excite a longitudinal piezoelectric transducer with a central frequency of 5 MHz to generate 10 cycles tune burst signals. A longitudinal piezoelectric transducer with a central frequency of 10 MHz was used to receive the fundamental and second harmonics of ultrasonic waves. The fast Fourier transform (FFT) was used to convert the first reflect echo of the received signal to the frequency space for all measurements. Figure 4 shows the amplitude of the fundamental and second harmonics from FFT. To control the amount of the coupler, a precise syringe was used to inject 2 mL of gel for each

measurement. To maintain alignment parallelism, the transmitting and receiving transducers were fixed during the course of measurement. The center of samples was measured for three times at the same position to calculate the average value. The signal was recorded by a 2.5 GS/s oscilloscope and stored in a computer. In order to confirm the nonlinear signals which are due to the material nonlinearity, the nonlinear acoustic signal was calibrated by increasing the input voltage. Figure 5 shows the amplitude of the second harmonic wave is linearly related to the square of the amplitude of the fundamental frequency component with the increased excitation voltage. Therefore, it could be concluded that the measured nonlinearity would be reliable.



Figure 2. The sketch of the experimental setup for attenuation measurement.

Table 2. The weight tests by an electronic scale.



Figure 3. The sketch of the experimental setup for nonlinear ultrasonic measurement.



Figure 4. The amplitudes of the fundamental and second harmonics from the fast Fourier transform (FFT).



Figure 5. Second-harmonic amplitude A_2 versus the square of the fundamental wave amplitude A_1 with an increase of input voltage.

3. Results and Discussion

3.1. Morphology and Hardness Test

It is well-known that cast stainless steels suffer thermal embrittlement when exposed to temperatures in the range 300–500 °C due to the decomposition of ä-ferrite into chromium-rich α' phase and iron-rich α phase. Figure 6 shows the microstructure evolution of the specimens age-treated at 385 °C for 2400, 6000, and 30,000 h and the unaged samples (0 h), respectively. It can be seen that the island-shaped (LF) or the network-like (HF) ä-ferrite islands are dispersed in the austenite (γ) matrix, and that the specimens show no significant changes of metallographic features with aging time from 0 to 30,000 h. To make better sense of the aging effect, the Vickers and Rockwell hardness tests were carried out. Figure 7 shows the hardness values of the ä-ferrite and ã phase. The hardness values of the ä-ferrite in the unaged samples were HV 183 (LF) and HV 190 (HF). For the specimens age-treated for 2400 and 6000 h, the hardness increased to HV 256 (LF), HV 276 (HF) and HV 306 (LF), HV 391 (HF), respectively. After prolonging the aging time to 30,000 h, the hardness values showed no significant change. Figure 8 shows the Rockwell hardness tests with aged time from 0 to 30,000 h. For the specimens age-treated, the hardness increased with aged time both HF and LF samples. The increase in hardness was attributed to the spinodal decomposition and precipitation [4–7]. To have a comparison of the change in the microstructure, two cold rolled specimens machined from

the HF and LF static cast stainless steel were analyzed and measured by EBSD and the hardness test. Figure 9 shows EBSD of the HF and LF specimens deformed to 10% cold work, respectively. The slip bands were noticed within the austenite matrix after cold work. Figure 10 shows the hardness values for 10% deformed HF and LF samples. For the specimens machined, the hardness increased both HF and LF samples. The increase in hardness was due to the increase in dislocation density.



Figure 6. Cont.



Figure 6. Cont.



Figure 6. Cont.



Figure 6. Microstructure evolution of specimens age-treated at 385 °C for various periods of time, (a) low ferrite (LF) 0 h; (b) LF 2400 h; (c) LF 6000 h; (d) LF 30,000 h; (e) high ferrite (HF) 0 h; (f) HF 2400 h; (g) HF 6000 h; (h) HF 30,000 h.



Figure 7. Cont.



Figure 7. Cont.



Figure 7. Cont.



Figure 7. Microstructure and hardness test indentations of specimens age-treated at 385 °C for various periods of time, (**a**) LF 0 h; (**b**) LF 2400 h; (**c**) LF 6000 h; (**d**) LF 30,000 h; (**e**) HF 0 h; (**f**) HF 2400 h; (**g**) HF 6000 h; (**h**) HF 30,000 h.



Figure 8. Variations of HRB hardness measurement with aging time.



Figure 9. Cont.



Figure 9. Cont.



Figure 9. EBSD of 10% deformation, (**a**) LF 10% 200×; (**b**) LF 10% 1000×; (**c**) HF 10% 200×; (**d**) HF 10% 1000×.





3.2. Ultrasonic Velocity Tests and Characteristics

To grasp the main points of the interaction between microstructural change and ultrasonic signal, the ultrasonic velocity and attenuation measurements were carried out. Figures 11 and 12 show the variations in ultrasonic velocity with aging time and deformation. There was an increase trend in ultrasonic velocity both HF and LF. In addition, the velocity increases both of the deformed samples.

According to the hardness test and velocity test, the increase in the hardness was observed to increase with aging time up to the 30,000 h. These results are coherent with the results reported by Freitas et al. [21].

The experimental measurements by Freitas et al. indicated that the increase in ultrasonic velocity is due to the increase of the Young's modulus and attributed to the spinodal decomposition. This result reveals that the ultrasonic velocity may have the potential to monitor the microstructural change.



Figure 11. Variation of velocity with aging time.





3.3. Ultrasonic Attenuation Tests and Characteristics

Figures 13 and 14 show the variations in attenuation with aging time and deformation. There were the same trends that the attenuation parameter ã decreases with aging time from 0 to 6000 h, and increases slightly from 6000 to 30,000 h. For the specimens machined, the ã decreased both HF and LF samples.

The attenuation of ultrasonic depends on damping, grain size, and scattering of the material [22]. From Figure 6, the grain sizes are almost the same with aging time from 0 to 30,000 h. In the ultrasonic

aspect, the interface between two different phases may induce reflection and transmission. As the metallographic features did not show significant changes, the grain sizes and the scattering would not be the main factors in the attenuation. Some studies have shown that the primary mechanism of aging embrittlement is spinodal decomposition, and the second is the precipitation of G phase [4–7], that is to say, the variety in attenuation is due to the change of damping which may attribute to the spinodal decomposition and precipitation. It could be observed from the deformated samples that the attenuation parameter ã decreases because of the deformation which causes the tangling of multiplied dislocation and consistent with the conclusions obtained through present investigations [23].



Figure 13. Variation of relative attenuation with aging time.



Figure 14. The relative attenuation with 10% deformation for HF and LF samples.

3.4. Ultrasonic Nonlinearity Tests and Characteristics

For a better understanding of the relationship between microstructure and nonlinear ultrasonic parameter (β), the magnitudes of 5 and 10 MHz were measured to give an estimated \hat{a}' value with Equation (9). Figures 15 and 16 show the changes in the \hat{a}' values for cast stainless steel specimens with aging time and deformation. It is observed from Figure 15 that the variations in \hat{a}' for HF and LF specimens with aging time are in a similar trend. The value was noted to increase in the early stage of the aging process. After that, a decrease with aging time was observed. For the specimens machined, the \hat{a}' increased both HF and LF samples which were in reasonable agreement with the present investigation [24]. This suggests that the variations in the microstructural change would affect the nonlinear ultrasonic parameter.

In order to elucidate the relationship between microstructures and nonlinear ultrasonic parameter, cast stainless steels with two different ferrite contents were used. From Figure 15, it can be seen that the HF-0h and LF-0h specimens show variations in ultrasonic nonlinearity. This implies that the variation of phase content will induce the change of ultrasonic nonlinearity. According to Xiang et al. [25], a stress-dependence model has been proposed to depict the change in the ultrasonic nonlinearity parameter with the applied stress

$$\Delta\beta \propto |\sigma| \tag{12}$$

where $\Delta\beta$ is the change of nonlinearity parameter, and σ is the applied stress. The stress generated by the change of lattice structure can be calculated by the following expression

$$\sigma \approx 2\mu\varepsilon' f_p \tag{13}$$

where f_p is the volume fraction of the dispersed phase, μ is the shear modulus of the matrix, and ε' is the coherency strain. For assuming a two-phase material, the coherency strain resulting from a phase embedded into the other phase matrix is described as

$$\varepsilon' = \frac{3K\delta}{3K + 2E(1+\nu)} \tag{14}$$

where *K* is the bulk modulus of the embedded phase, δ is the embedding parameter, *E* is the Young's modulus of the matrix, and ν is the Poisson's ratio of the matrix. The embedding parameter δ can be used to represent the relationship between embedded phase and the matrix as

$$\delta = \frac{2(a_e - a_m)}{(a_e + a_m)} \tag{15}$$

where a_e is the lattice parameter of the embedded phase and a_m is the lattice parameter of the matrix. The nonlinear ultrasonic parameter evaluated by the precipitate-matrix model is a function of the volume fraction of precipitates and the misfit parameter. As an example, there is a stainless steel containing austenite and ferrite phases. The lattice parameter of austenite is 0.36 nm, and that for ferrite is 0.28 nm. When the content of ferrite increases, a variation of δ is obtained for the steel with the ferrite grains dispersed in the austenite matrix. This indicates that a phase embedded into the matrix would change the nonlinear ultrasonic parameter. From Figure 15, the ferrite content increases with the nonlinearity decrease. In the similar way, the nonlinearity would change by either the phase transformation of spinodal decomposition in the ä-ferrite islands due to the aging or the dislocation in the austenite matrix due to the deformation. From Figure 15, the aging behaviors could be separated into two stages. The nonlinear parameter curve shows different trends in stages I and II, corresponding to the different states of microstructural change. From Gutierrez-Vargas et al. [26], the study shows that the nonlinearity parameter decreases due to the phase precipitation. It means that some precipitations would decrease the nonlinearity parameter. Similarly, the increase of ferrite phases in the austenite matrix would decrease the nonlinearity. According to previous studies [27,28], the phase transformation

would rely mainly on the spinodal decomposition at the beginning during the aging treatment at 300–500 °C and the intermetallic compound would precipitate significantly after a long-term aging time. From K. Fujii et al. [29], the study showed that the spinodal decomposition did not change significantly from 10,000 to 40,000 h and indicated that the G-phase precipitate formation could be observed by atom probe tomography (APT) when the cast austenitic stainless steel thermally aged at 400 °C for 40,000 h. Therefore, an increasing nonlinearity for stage I may follow by the spinodal decomposition stage. Subsequently, a decrease in the nonlinearity was observed in the second stage, that may be attributed to the precipitation of intermetallic compound (G-phase). From Figures 13 and 15, the figures show that variations in the nonlinear parameter and attenuation with aging time follow a similar trend. Since both the attenuation and the nonlinear parameter could present the spinodal decomposition and the precipitates in the ä-ferrite, it suggests that the ultrasonic system can be used to evaluate the properties of the microstructural change.



Figure 15. Variations of nonlinear parameters for HF and LF with aging time.



Figure 16. The nonlinear parameters with 10% deformation for HF and LF samples.

4. Conclusions

The effects of microstructural changes by aging and deformation on the ultrasonic were examined. The results show that the aging and deformation would change both of the linear ultrasonic parameters and the nonlinear ultrasonic parameter. The measurement results further verify the relationship between microstructural changes (spinodal decomposition, precipitation, and dislocation) and ultrasonic nonlinearity of cast stainless steel. Characterization of microstructural changes in cast stainless steel by ultrasonic is under study. From the results, the feasibility of the ultrasonic technique is verified. When the practical applications were considered, the ultrasonic measurements are simple, fast, and non-destructive. In addition, as the ultrasonic parameters would reveal the mechanical properties, the ultrasonic techniques are highly recommended in applied fields of material science. These ultrasonic techniques are important for monitoring material properties of industrial components and are prospective methods for on-site inspections.

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