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Characteristics of Ultrasonic Nonlinearity by Thermal Fatigue

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The deterioration conditions of nuclear power plants and chemical plants have raised deep concerns about the safety of high-risk structures. The safety of a structure can be secured by evaluations of breakages and lifespan in the early stage. Therefore, the material condition of a structure needs to be estimated. A nonlinear ultrasonic evaluation method is reportedly more sensitive on microscopic changes of a material than a linear evaluation method. Therefore, the nonlinear ultrasonic technique based on ultrasonic nonlinearity is considered a promising method of evaluation for breakages and lifespan. This study measured the changes in nonlinear parameters by using a nonlinear ultrasonic method, observed the microstructure of a heated material, and analyzed the relation between the size of the microstructure and the changes in the nonlinear parameters.

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1. Introduction

High strength age-hardenable Ni-Cr-Fe alloy, Inconel X-750, was originally developed for applications requiring good mechanical properties and high resistance to corrosion and oxidation at elevated temperatures.[1] Its use has extended to a variety of applications including the nuclear power industry. Inconel X-750 is extensively used both in PWRs (pressured water reactor) and in BWRs (boiling water reactor), mainly for the core internals such as bolts, springs, guide pins and other structural components requiring high strength and high resistance to stress relaxation.[2] Usually, prior to the use of the original Inconel X-750, heat treatment is applied to generate an adequate microstructure that would improve the mechanical properties. The effect of different heat treatments on the Inconel X-750 alloy has been intensively studied; however, the most recent values of the treatment parameters are only rough estimations. From this viewpoint, knowing precisely the most suitable heat treatment to improve the mechanical properties of this alloy would be beneficial.

The deterioration conditions of nuclear power plants and chemical plants have raised deep concerns about the safety of high-risk structures. The safety of a structure can be secured by evaluations of breakages and lifespan in the early stage. Therefore, the material condition of a structure needs to be estimated. In particular, structures such as power generation facilities requiring high safety needs to be evaluated by a method that can detect even small changes in material to estimate its structural safety in the early stage. An evaluation method based on the nonlinear characteristics of ultrasonic waves is reportedly more sensitive to micro-structural changes of a material more than a linear evaluation method is.

The most powerful nondestructive method for evaluating material degradation is the ultrasonic method, since the characteristics of ultrasonic wave propagation are directly related to the properties of the material.[3] Higher harmonic generation is the most common phenomenon that occurs when the waveform of an incident wave is distorted by the nonlinear elastic response of a medium; higher harmonic waves are generated in the transmitted wave.[4] Recent experimental studies and new physical models are demonstrating the potential of nonlinear ultrasonics to quantitative detection and characterization of fatigue damage in metals. This fatigue damage first appears in the form of dislocation substructures, such as veins and persistent slip bands, and the persistent slip bands accumulate at the grain boundaries to produce strain localization and, then finally, a microcrack initiates with increasing fatigue cycles.[5] Most of the work to date has been concerned with the use of longitudinal waves propagated through the bulk of a material. Jhang et al. used a longitudinal wave in the 2.23Cr-1Mo steel and analyzed its response by the bi-spectrum method.[6] Recently Ogi et al. used the surface wave resonance at the circumference of a steel rod generated by an electromagnetic acoustic transducer to monitor fatigue damage.[7]

There are a number of advantages in using surface waves in nonlinear acoustics. First, surface waves do not require access to both sides (one side for generation and the second side for detection) of a component, as is the case for most bulk wave applications; this single sided technique is particularly promising for field applications, where the availability of two parallel surfaces (each available to mount transducers) will be a limiting restriction. Second, most of the energy of surface waves is concentrated near the stress free surface, which can lead to the appearance of stronger nonlinear effects compared to what would appear with bulk waves; this behavior would be helpful in detecting fatigue damage, since fatigue damage typically initiates on the free surface of a component. Finally, Rayleigh surface waves propagate far distances, making them an ideal for evaluating large components.[8]

The purpose of this paper is to show the relationship between heat treatment and material nonlinearity. Surface waves are experimentally generated with a wedge transducer and detected with a receiver. This study measures the changes in nonlinear parameters of a material due to heat treatment-induced material nonlinearity by using a nonlinear ultrasonic method; observes the microstructure of the heated material; and shows the relation between the size of the microstructure and the changes in the nonlinear parameters.

2. Theory

A method to detect nonlinearity is receiving signals that propagate a certain distance after the incidence of ultrasonic wave of a certain frequency as a specimen. The nonlinearity of a specimen can be detected by signals that are generated when ultrasonic waves of a certain frequency have propagated a certain distance in the specimen. If the material is nonlinear, the ultrasonic wave of basic frequency becomes distorted after propagating a certain distance and the wave becomes of high frequency. In this method, it is important to select the mode that can generate the higher second harmonic wave.[4]

In order to explain the generation of higher order harmonic waves, consider a single frequency ultrasonic (longitudinal) wave propagating in a degraded material. Here, A_1 is the displacement amplitude of the fundamental frequency component, x is the propagation distance, k is the wave number, and t is time. We introduce the nonlinear Hooke's law for the degraded material, whose stress–strain relation is described by

$$\sigma = E\varepsilon(1+\beta\varepsilon+....) \tag{1}$$

where E is the Young's modulus and β is a second order nonlinear elastic coefficient. If we assume that attenuation can be neglected, the equation of motion of the longitudinal wave 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 mass density of the solid in the unperturbed state, x is the propagation distance of the sound wave, u is the displacement, and σ is the stress. Using Eqs. (1), (2) and the relationship between strain and displacement, $\varepsilon(x,t)=\partial u(x,t)/\partial t$, one can obtain the nonlinear wave equation in terms of displacement $u(x,t)$ as follows:

$$\rho\frac{\partial^2 u}{\partial t^2}=E\frac{\partial^2 u}{\partial x^2}+2E\beta\frac{\partial u}{\partial x}\frac{\partial^2 u}{\partial x^2} \tag{3}$$

In order to obtain a solution, the perturbation method is applied. For this purpose, the displacement u is assumed as

$$u=u_1+u_2 \tag{4}$$

where u_1 and u_2 represent the fundamental wave and the second order perturbation solution, respectively. If we set u_1 to be a sinusoidal wave of single frequency,

$$u_1=A_1\cos(kx-wt) \tag{5}$$

then we can obtain the perturbation solution up to the second order as follows:

$$u=A_1\cos(kx-wt)+A_2\sin 2(kx-wt) \tag{6}$$

with

$$A_2=A_1^2k^2\beta x/8 \tag{7}$$

The second term in Eq. (6) represents the second harmonic frequency component. As a result, we can explain how occurs through the propagation of ultrasonic wave in a nonlinear elastic solid. In addition, from Eq. (7), we can see that the magnitude of the second-order component A_2 depends on β, which represents the nonlinear characteristics of degraded material. Therefore, if we can measure the magnitude of β, we can evaluate the extent of degradation of a property of a material due to high-temperature exposure. For constant k and x, β can be normalized as

$$\beta'=A_2/A_1^2 \tag{8}$$

In this study, the normalized coefficient β' is defined as the ultrasonic nonlinearity parameter, and will be measured experimentally.

3. Materials and experimental methods

3.1 Materials and heat treatment

Age-hardenable Ni-Cr-Fe alloys, Inconel X-750, are complex materials and their properties can be controlled by a variety of heat treatments. In the heat treated condition, these age-hardenable alloys are composite materials consisting of several intermetallic phases bound by a metallic matrix.[9] The specimens under investigation are rectangular plates, with 5.0 mm thickness. The dimension of specimens is 30 mm × 200 mm. The chemical composition of the material is shown in Table 1. One of the specimens is original raw material. And the other three are thermally degraded samples. All of the heat treatment processes include quenched and tempered stages as indicated in Fig. 2. Fig. 1

shows the specimens used in the experiment, and their chemical composition is shown in Table 1. For the experiment, the specimen without heat treatment and the specimen with heat treatment, shown in Fig. 2, were prepared. The materials were heat treated in several different ways, varying both the tempering temperature and the aging treatment. The temperature and time of heat treatment are the same as those of quenching, but they are differently set for tempering.

Table 1 Chemical composition (%) of Inconel X-750

Al	C	Cr	Cooper	Iron	Mn
0.4~1	0.08	14~17	0.5	5~9	1
Ni	Nm	Si	Sr	Ti	
-	0.7~1.2	0.5	0.01	2.25~2.75	

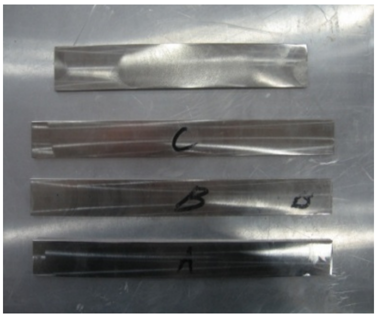


Fig. 1 Inconel X-750

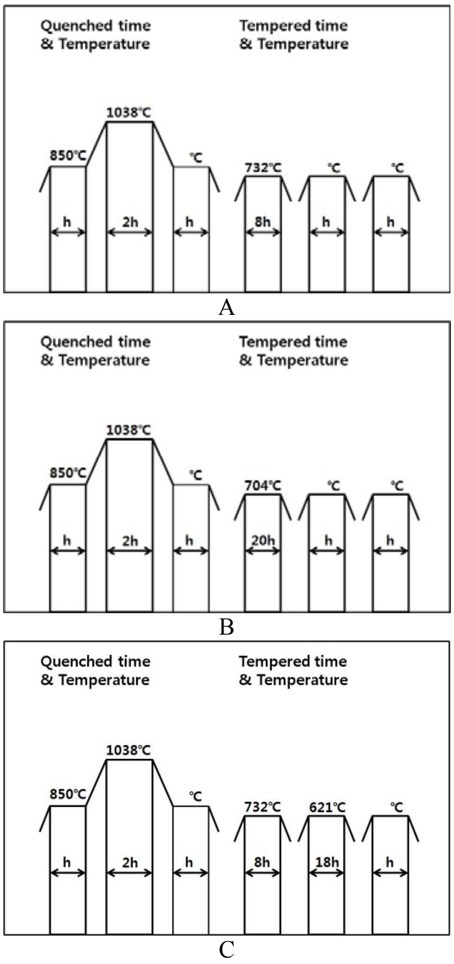


Fig. 2 Heat treatment conditions

3.2 Configuration of measurement system

Fig. 3 is the schematic diagram of the measurement system. This system uses RPR 4000 (U.S RITEC), which is a low-noise ultrasonic signal analyzer available to acquire data. A high power tone burst system with a pair of narrow band transducers is employed to generate a narrow band signal with the central frequency of 5 MHz. The input frequency is tuned at 5 MHz, while the central frequency of receiver is chosen at 10 MHz to obtain the corresponding second harmonic frequency components. Both transducers are carefully mounted with the specimens with specially designed holders. A consistent loading is applied to the pair of transducers through mechanical grips to ensure uniform bonding condition. The received signal is converted to A.D and analyzed by the PC. Upon analysis of the frequency and interpretation of the spectrum of the received wave by FFT, nonlinear parameter values were obtained. The ultrasonic wave used in this study was a surface wave. The received angle measured by Snell's law was 70^o. In addition, the heat loading condition in Fig. 4 was given for 1 cycle, in which the specimen was heated with 800^{o}C and refrigerated with 40^{o}C for 15 min. Heat loading cycles are 10 and 20. Ultrasonic nonlinearity changes depending on the heat loading cycles.

4. Result

4.1 Observation of microstructure

The microstructures of the materials subjected to different heat treatments were studied by paying special attention to the grain boundary region. The microstructure of the metal was changed during heat treatment. Figs. 5~7 show some micro structure changes that resulted from heat treatment:carbide formation is observed at the grain boundaries. The samples for SEM were ground and polished using normal metallographic means. The SEM samples were etched in an acid solution mixedat the following ratio. (15 mL HCl + 10 mL acetic acid + 5 mL HNO3 + 1~2 drops glycerol)

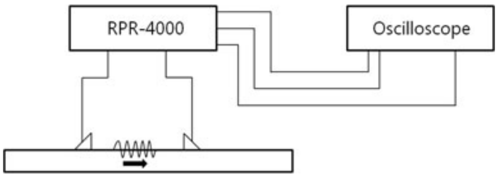


Fig. 3 Schematic diagram of measurement system

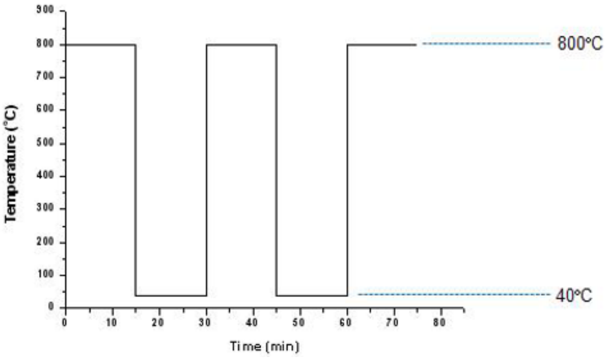
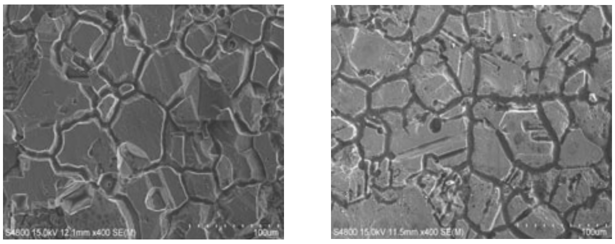
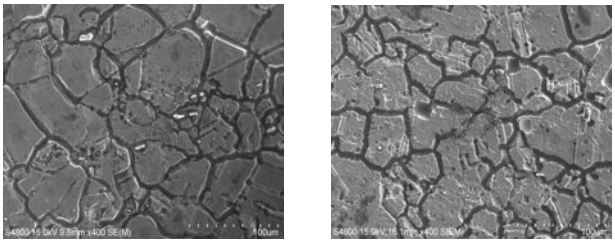


Fig. 4 Heat loading condition

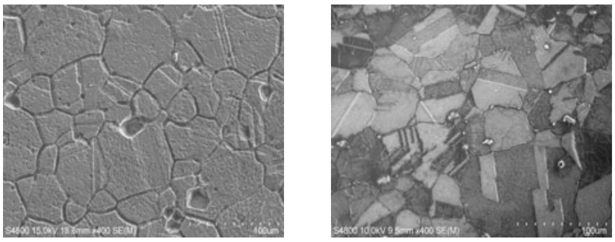


(a) Without heat treatment (b) With heat treatment A

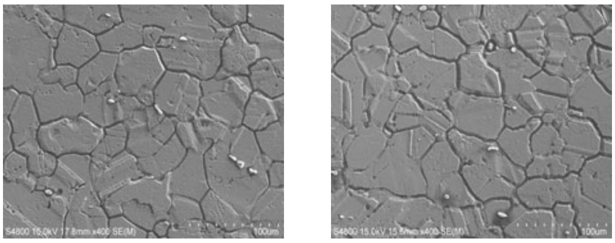


(c) With heat treatment B (d) With heat treatment C

Fig. 5 Microstructure of Inconel X-750 with different heat treatments

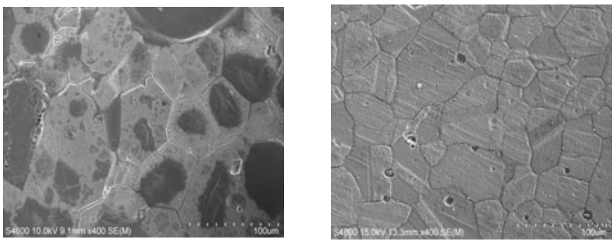


(a) Without heat treatment (b) With heat treatment A

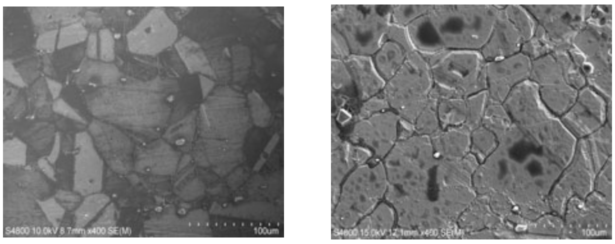


(c) With heat treatment B (d) With heat treatment C

Fig. 6 Microstructure of Inconel X-750 with different heat treatments after 10 cycles of heat loading



(a) Without heat treatment (b) With heat treatment A



(c) With heat treatment B (d) With heat treatment C

Fig. 7 Microstructure of Inconel X-750 with different heat treatments after 20 cycles of heat loading

The identified carbides were of the types MC and $M_{23}C_6$. In addition to these and the intermetallic precipitates, no other precipitates were identified. The MC and $M_{23}C_6$ carbides were found to be grain boundary precipitates. The microstructure of the Inconel X-750 alloy was strongly affected by heat treatment. The main effect of the solution tempering temperature was related to the amount of carbon dissolution from the MC carbides into the matrix.

4.2 Size of the grain boundary and the detection of nonlinear parameter

To verify that the measurements from the specimens were truly due to material inherent nonlinearity and not only due to the nonlinearity arising from the measurement system, it was necessary to show the variation of A_2 / A_1^2 with heat treatment conditions and heat loading cycles. It is important to note that a measured relative nonlinearity should include material inherent acoustic nonlinearity plus any nonlinearity from instrumentation and couplant.

Fig. 8 shows the changes in the grain boundary size of the Inconel X-750 specimen. The size of the grain boundary varies with respect to heat treatment conditions, and the C treatment condition makes the grain boundary smaller than the other treatment conditions. Furthermore, the size of the grain boundary is increased by heat loading.

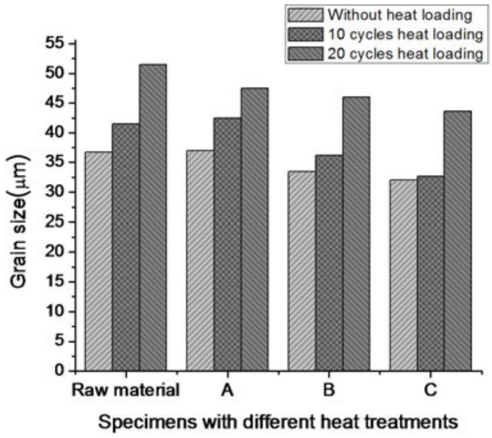


Fig. 8 Change of grain boundary size for different heat loading cycles

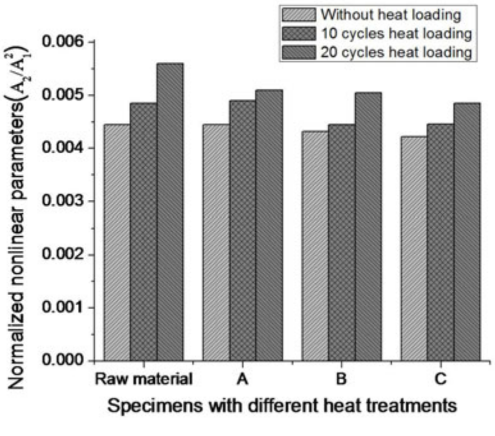
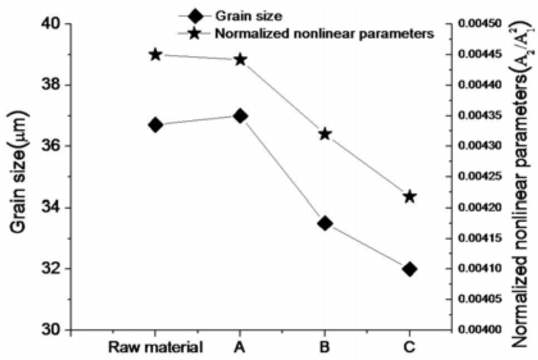


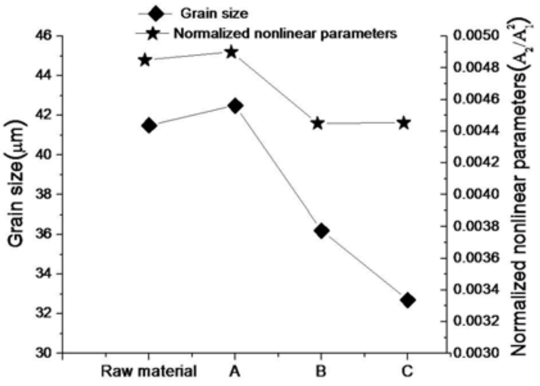
Fig. 9 Normalized nonlinear parameters of Inconel X-750 for different heat treatments and heat loading

Fig. 9 describes the changes in normalized nonlinear parameters with respect to heat treatment conditions. In Fig. 9, the size of the normalized nonlinear parameters varies according to heat treatment conditions. In addition, the sizes of the normalized nonlinear parameters are decreased. For 10 cycles of heating loading, the normalized nonlinear parameters become larger than the ones without the heating loading, and in the case of 20 cycles, the size of normalized nonlinear parameters is biggest.

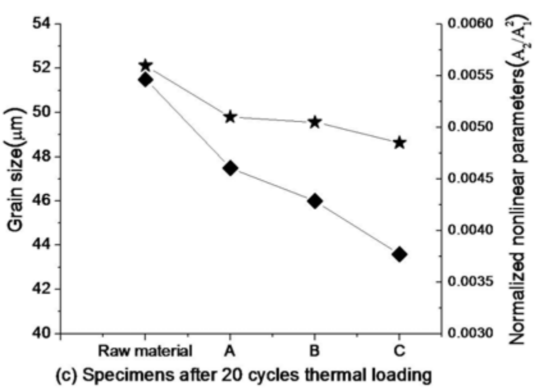
The changes in nonlinear parameters and in the grain boundary measured by FESEM show a similar trend. The sizes of normalized nonlinear parameters and the grain boundary of the specimen without heat treatment and heat loading are bigger than those of the other specimens. Also, the size of the grain boundary in specimen C, showing the smallest size of normalized nonlinear parameters, is small. Such phenomena are shown for the given heat loading conditions.



(a) Specimens without heat loading



(b) Specimens after 10 cycles thermal loading



(c) Specimens after 20 cycles thermal loading

Fig. 10 Comparison of grain size and nonlinear parameters in different specimens with various thermal loading

Heat treatment and heat loading can change the microstructure. The microstructure of the material in this paper changed after heat treatment and heat loading. Therefore, the normalized nonlinear parameter changed with respect to the heat treatment condition and heat loading.

4.3 Hardness measurements

Heat treatment can change the physical and mechanical properties of a material, which become totally different from the original material when a material is heated and refrigerated below the melting point. This study measured the changes in inclination with respect to heat treatment conditions and heat loading, and the experiment results are shown in Fig. 11. Hardness also varied depending on heat treatment conditions, and the specimen with heat treatment showed higher than the one without heat treatment. The lower the heat loading, the higher the hardness became.

As well known, material nonlinearity could be attributed to micro-structural defects such as lattice deformation or dislocation motion. Any process that alters the local atomic potential or impedes the movement of dislocations will change the microstructure and material nonlinearity. Such as internal or residual stress, microstructure non-uniformity, grain boundaries. It is not long ago that the technique become of a greater concern to researchers. According to the authors' literatures survey, the technique emerged as a promising NDE tool, just recent years. Nevertheless, the technique has been vigorously studied due to the potential to monitor early stage material damage. At present, the technique is accepted as a qualitative monitoring tool, not a quantitive one, in terms of the correlation between damage level and ultrasonic nonlinearity. In addition, the correlation can be made only in a relative manner not an absolute scale. Without a doubt, there could be many other nonlinear sources coming from experimental set-up besides material micro-damages. To assure that the nonlinearity from material damage is dominated over other sources. Only the nonlinear parameters noticeable exceeding the base ling value of the raw specimens without heat treatment after obtained over a number of times, and the average datas are plotted to alleviate the experimental uncertainly. This study aims at

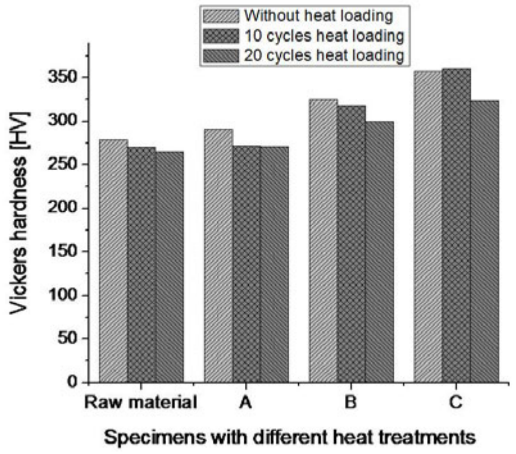


Fig. 11 Hardness of each specimen with heat treatment and heat loading cycles

investigating the thermal effect on material nonlinearity, which is very meaningful for nondestructive evaluation of thermal damage in early stage by using nonlinear ultrasonic waves.

5. Conclusion

This study measured the nonlinearity of Inconel X-750 by using ultrasonic surface waves for different heat treatment conditions, and the results are as follows.

1. The changes in nonlinear parameters due to heat treatment conditions improved the mechanical properties of the meterial.
2. Normalized nonlinear parameters varied according to heat treatment conditions, and the specimen with heat treatment showed smaller normalized nonlinear parameters than the one without heat treatment.
3. In addition, the changes in the size and the inclination of normalized nonlinear parameters and the grain boundary were observed in the heated specimen with heat loading. The changes in inclination were found according to heat treatment conditions.
4. Through the results above, the changes in normalized nonlinear parameters were found to be related to the changes in the size of the grain boundary.

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