Experimental Mechanics (2013) 53:775–781
DOI 10.1007/s11340-012-9681-6

# Assessment of Heat Treated Inconel X-750 Alloy by Nonlinear Ultrasonics

**W. Li · Y. Cho · J. Lee · J.D. Achenbach**

Received: 29 February 2012 / Accepted: 11 September 2012 / Published online: 3 October 2012

**Abstract** The nonlinear ultrasonic technique is known as a promising tool for monitoring material states related with micro-structural changes, with improved sensitivity compared to conventional nondestructive testing techniques. It is well known that degradation of material properties is generally accompanied by the increase of material nonlinearity. However, the trend has been rarely investigated in the opposite way for improved material properties. In this paper, nonlinear ultrasonic waves are used to assess the material condition of heat treated Inconel X-750 alloy based on the nonlinear acoustic parameters. Material property testing is conducted to compare the influence of heat treatment for comparison with the nonlinear parameter based prediction. The material properties of specimens are improved by applying heat treatment, with significant decreases in the acoustic nonlinearity. The better the mechanical property achieves via heat treatment, the smaller the acoustic nonlinearity becomes. It can be concluded that the nonlinear acoustic technique can be used to evaluate the effect of heat treatment nondestructively, and to optimize the process, thus providing another indication of the feasibility of using the nonlinear ultrasonic technique for material characterization.

**Keywords** X-750 Inconel alloy · Heat treatment · Nonlinear ultrasonic wave · Material property

## Introduction

Nonlinear ultrasonic technique has been proposed as a promising method to determine changes in the microstructure of materials [1]. From a practical viewpoint, one typical nonlinear phenomena is the generation of second harmonics in an ultrasonic test, i.e., the formation of a harmonic double the frequency of the fundamental input frequency, which results from waveform distortion in the time domain. Recently, nonlinear ultrasonic approaches have been reported in numerous studies to assess the micro-damage of materials [2–4], and characterize material degradation [5, 6]. It has been shown that nonlinear ultrasonic waves can be used for the quantitative evaluation of material nonlinearity and hence are sensitive indicators for early stage damage detection. Compared to conventional linear ultrasonic measurements, some additional care is needed to detect material nonlinearity via the acoustic nonlinearity to alleviate experimental uncertainty. It has been reported that material nonlinearity could be attributed to micro-structural defects such as lattice deformation or dislocation motion [7]. Any process that alters the local atomic potential or impedes the movement of dislocations will change the microstructure and material nonlinearity [8, 9]. However, most previous efforts have been limited to the use of nonlinear ultrasonic approach for characterization of material degradation, the opposite trend for improved material properties has rarely been investigated. Material properties can be improved by the application of heat treatment to the specimens to generate an improved micro-structure. In this paper, nonlinear ultrasonic waves are used to evaluate the material nonlinearity of heat treated Inconel X-750 alloy and assess the improved material properties. Inconel X-750 alloy is a nickel based alloy. It shows a good resistance to corrosion and oxidation together with good strength [10]. It is widely used in the power industry. For example, the lock plate of a

---

W. Li · Y. Cho (✉) · J. Lee · J.D. Achenbach
School of Mechanical Engineering, Pusan National University,
10511, San 30, Jangjeon-dong, Geumjeoun-gu, Busan, 609-735,
South Korea
e-mail: mechcyh@pusan.ac.kr

J.D. Achenbach
Center for Quality Engineering and Failure Prevention,
Northwestern University,
Evanston, IL 60208, USA

**Table 1** Chemical components of Inconel X-750

| Al | C | Cr | Copper | Iron | Mn | Ni | Nm | Si | Sr | Ti |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.4~1 | 0.08Max | 14~17 | 0.5Max | 5~9 | 1Max | – | 0.7~1.2 | 0.5Max | 0.01Min | 2.25~2.5 |

gas turbine is made of this material to protect the root of turbine blade from severe operation conditions such as high temperature and pressure. A proper heat treatment can help enhance material properties to a desired level. Before the original X-750 alloy is used in applications, heat treatment is applied to form a microstructure that improves the mechanical properties [11]. The better properties provided by the heat treatments are attributed to slip homogenization and restriction of localized plastic deformation parallel to the boundaries [12]. A technique to monitor its continuously varying mechanical properties on-line has been of high interest but, at the same time, very challenging from a nondestructive testing viewpoint. This is because, in general, conventional linear feature based ultrasonic techniques are known to be insufficiently sensitive to detect such early stage material property changes.

The objective of this paper is to investigate the correlation between heat treatment and material nonlinearity and evaluate the material nonlinearity change with improved material properties by the use of nonlinear ultrasonics. The proposed procedure can be used to evaluate the heat treatment effects nondestructively, and to optimize the heat treatment parameters.

## Measurement of Ultrasonic Parameters

### Nonlinear Acoustic Parameter Measurement

To detect material nonlinearity, a single frequency ultrasonic wave is launched into the specimen, and the signals of the ultrasonic wave after a certain propagation distance are received. The ultrasonic wave is distorted due to material nonlinearity, and consequently, higher harmonics are generated. Thus, the received signal is composed of not only the fundamental frequency wave but also second or higher harmonics frequency wave. In this work, the physical effect monitored in nonlinear ultrasonic measurements is the generation of higher harmonics. The measurement of harmonic generation for micro-structural characterization is typically aimed at determining the value of nonlinear acoustic parameter $\beta$. The nonlinear parameter is related to the amplitudes of the fundamental wave and second harmonics [13].

$$\beta = \frac{8A_2}{{A_1}^2k^2x} \tag{1}$$

where $A_1$ and $A_2$ are the amplitude of fundamental wave and the second harmonic wave respectively. $k$ is the wave number and $x$ is the wave propagation distance. In experiments, the quantity $\widehat{\beta}$ is measured as follows,

$$\widehat{\beta} = \frac{A_2}{A_1^2} \propto \beta \tag{2}$$

The ratio can be tested experimentally. Thus, the material nonlinearity can be evaluated by detecting the fundamental and the second harmonic amplitudes.

### Linear Acoustic Parameters Measurement

Linear ultrasonic tests based on the measurement of velocity and attenuation are carried out for material characterization depending on the three different heat treatments.

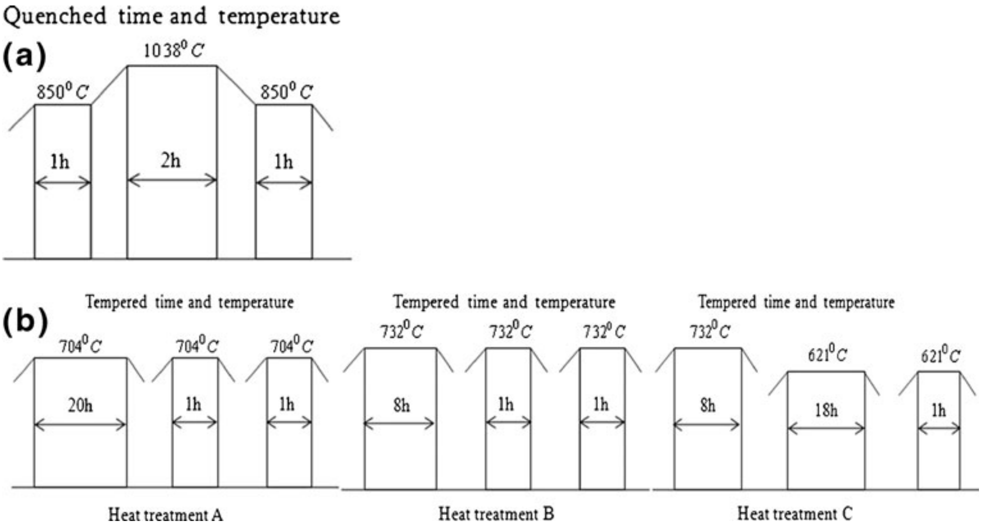


**Fig. 1** Heat treatment processes (a) same quenched stage and (b) different tempered stages

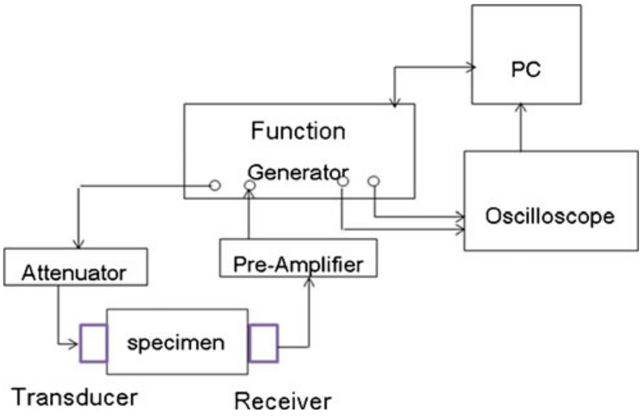


**Fig. 2** A block diagram of the ultrasonic nonlinearity measurement system

When ultrasonic wave decays with propagation distance, attenuation can also be counted as a factor affected by material condition [14]. It refers to the amplitude reduction ratio of wave signal in the logarithmic scale with respect to propagation distance between two neighboring positions as defined in equation (3). In this work, the conventional method by the pulse-echo configuration is used to measure ultrasonic wave attenuation. The attenuation is denoted by a coefficient $\alpha$ that depends on frequency $f$ as follows

$$\ln\frac{A_{1e}}{A_{2e}} = 2\alpha d \tag{3}$$

where $A_{1e}$ and $A_{2e}$ are the amplitude of the first and second ultrasonic echo respectively. $\alpha$ is the attenuation coefficient and $d$ is the thickness of specimen.

Ultrasonic wave velocity is mainly influenced by material microstructure and material properties. For homogenous materials, the ultrasonic longitudinal wave can be defined as shown in equation (4),

$$c_l = \left[\frac{E}{\rho}\,\frac{1-\upsilon}{(1+\upsilon)(1-2\upsilon)}\right]^{1/2} \tag{4}$$

where $c_l$ is longitudinal wave speed, $E$ is Young's modulus, $\rho$ and $\upsilon$ are density and Poisson ratio of material respectively. It shows that the ultrasonic velocity is not frequency dependent but influenced by material property.

## Experiment

### Specimens and Heat Treatments

The specimens under investigation are rectangular plates. The thickness of all specimens is 5.0 mm and the dimensions are 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 heat treated samples. All of the heat treatment processes include quenched and tempered stages. The temperatures and times are the same during the quenched stage but different in the tempered stage, as shown in Fig. 1. Tempering involves reheating the quenched alloy to a temperature below the eutectoid temperature and then cooling it. During this process, the alloy is heated to dissolve and to distribute alloying elements uniformly.

### Experimental Setup

The second harmonic is the wave that is characterized by double the frequency of the one for the incident wave. The equipment setup to monitor the nonlinear ultrasonic wave is shown in Fig. 2. The RAM 10000 system (RITEC) is used in this work. A 5 MHz piezoelectric transducers (PZT) is employed to generate a signal at the center frequency of 5 MHz. An attenuator and an amplifier are connected to the sender and receiver, respectively. The center frequency of the receiver is set at 10 MHz to obtain the corresponding second harmonic frequency components. Both transducers are carefully placed on each side of the specimens with holders designed to ensure uniform coupling conditions. A Hanning window is imposed on the steady state part of the

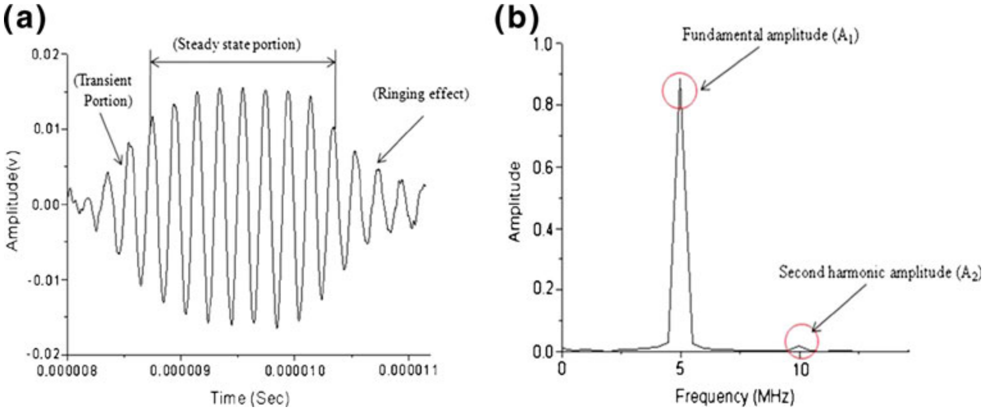


**Fig. 3** (a) time domain signal and (b) frequency spectrum (FFT) of the signal

signal and signals are digitally processed by using Fast Fourier Transformation (FFT) to obtain amplitudes $A_1$ at the fundamental frequency and $A_2$ at the second-harmonic double frequency, respectively. As shown in Fig. 3, a typical ultrasonic signal obtained in experiments is provided as examples.

As shown in equation (1), the nonlinear parameter of the ultrasonic longitudinal waves is represented as the function of the ratio of the second harmonic amplitude ($A_2$) to the square of the fundamental amplitude $\left(A_1^2\right)$ for a fixed wave number and propagation distance. This value represents the acoustic nonlinearity being correlated with the material nonlinearity of each specimen for different heat treatment. The measured parameters are normalized to their initial values to display relative changes for the convenience of interpreting the data.

Conventional linear ultrasonic tests based on the measurement of velocity and attenuation are also carried out to compare the sensitivity of linear and nonlinear acoustic features for material characterization depending on the three different heat treatments. The pulse-echo configuration is used to measure ultrasonic wave attenuation. The same frequency ultrasonic wave is incident into the specimens to check the amplitude reduction ratio. The wave speed is determined by reading the time of flight from the oscilloscope. The wave velocity is not frequency dependent but influenced by material property.

In addition, static uniaxial tensile material property testing is conducted to evaluate the influence of heat treatment in terms of the enhancement of hardness and ultimate strength.

## Result and Discussions

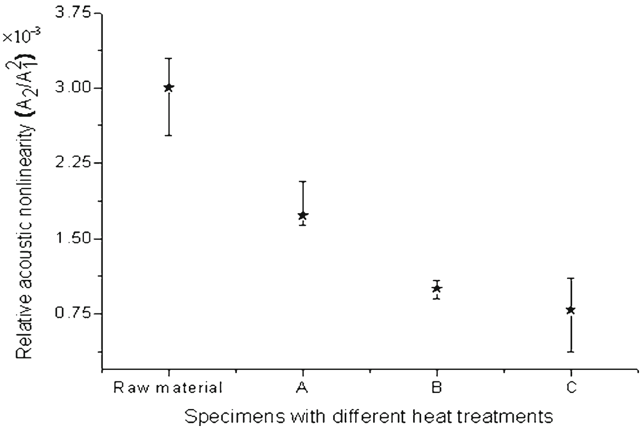


**Fig. 4** Variation of normalized acoustic nonlinearity of specimens with different heat treatments

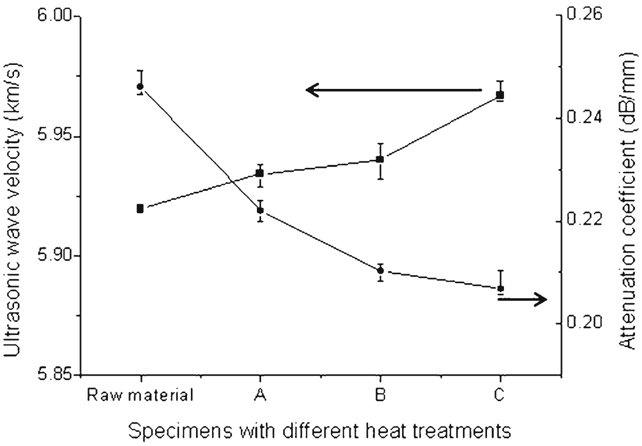


**Fig. 5** Variations of linear acoustic parameters (wave velocity and attenuation) in specimens with different heat treatments

Figure 4 presents the variation of nonlinear acoustic parameters for the specimens with different heat treatments. Compared to the virgin specimen without heat treatment, all three heat treated specimens show distinctly lower nonlinear acoustic parameters, and the acoustic nonlinearity decreases monotonically as the heat treatment condition changes from (a) to (c). Furthermore, the deviation in the acoustic nonlinearity is much more significant than those of other conventional ultrasonic features such as attenuation and velocity. As seen in Fig. 5, the variations of wave velocity and attenuation of ultrasonic wave in specimens with different heat treatments are presented. It shows that the attenuation variation range is from 0.246 dB/mm to 0.207 dB/mm in the specimens, and wave velocity variation range is from 5.92 km/s to 5.97 km/s. Neither of them shows remarkable change after heat treatments. One of the main goals of this study is to compare the sensitivities of different linear and nonlinear acoustic parameters to heat treatment

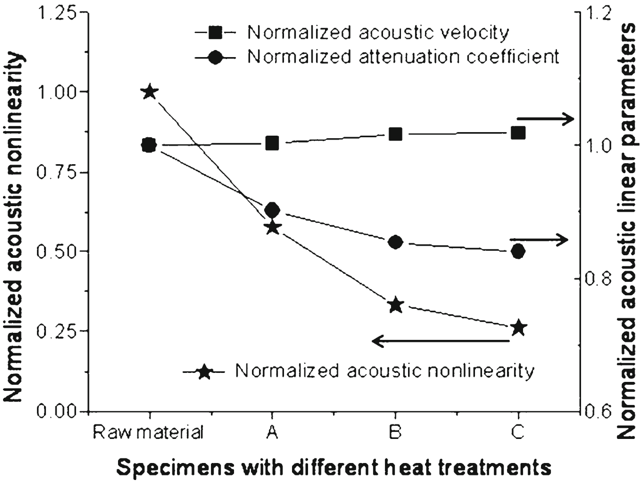
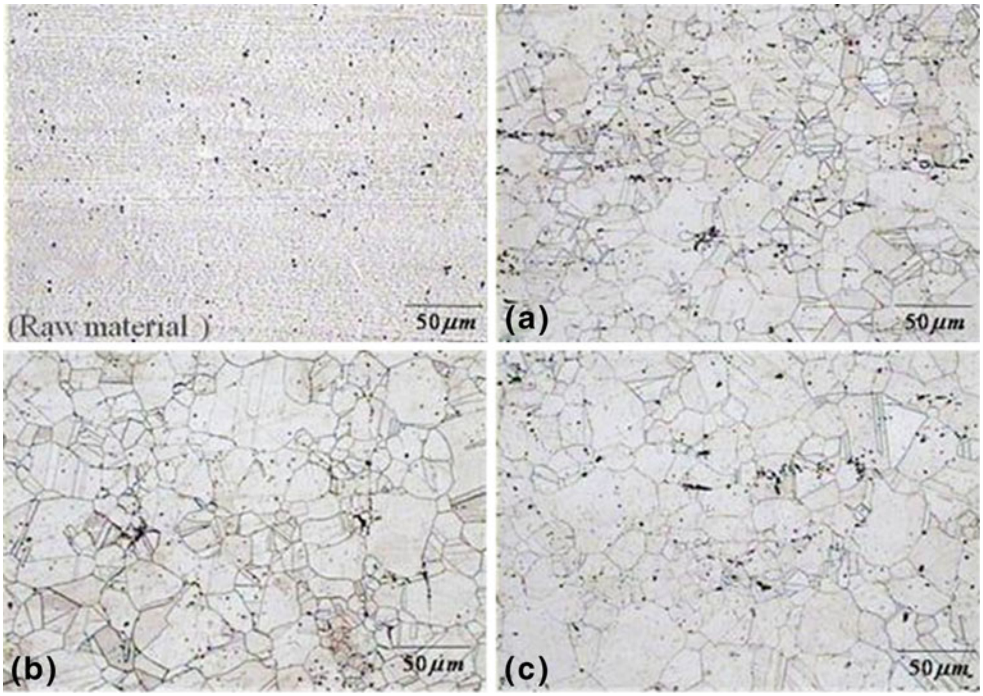


**Fig. 6** Comparison of sensitivity of acoustic nonlinear parameter and linear parameters to heat treatment effect; the acoustic nonlinearity, wave velocity and attenuation coefficient are normalized by the reference values of the raw material, $\overline{\beta}_{Raw} = 3 \times 10^{-3}$, $C_{Raw} = 5.92km/s$ and $\alpha_{Raw} = 0.246dB/mm$, respectively

**Fig. 7** Micro-structural pictures of specimens with different heat treatments



effect. The comparison of sensitivity of linear and nonlinear parameters is shown in Fig. 6. The measured acoustic parameters in the heat treated specimens have been normalized to their initial values in raw material, to display relative changes for the convenience of interpreting data. The wave velocity increased by 0.8 % and the attenuation decreased by 16 % after heat treatment C. However, the acoustic nonlinearity seems to provide more stable and noticeable features with a maximum of an over 70 % difference from the one of the virgin material, and this is very likely detectable in a reliable manner. The data tends to decrease following a parabolic pattern with an approximately 40 % drop in acoustic nonlinearity between before and after heat treatment.

To supplement the features observed in the nonlinear ultrasonic testing, pictures of micro-structural changes of the specimens are also obtained by optical microscope, as illustrated in Fig. 7. The pictures display agreement with the prediction from the acoustic nonlinearity tests, based on the data representing clear changes in grain structure before and after heat treatment. However, it is not obvious from the pictures that the heat treatment condition (c) can actually render a better mechanical property than the other two

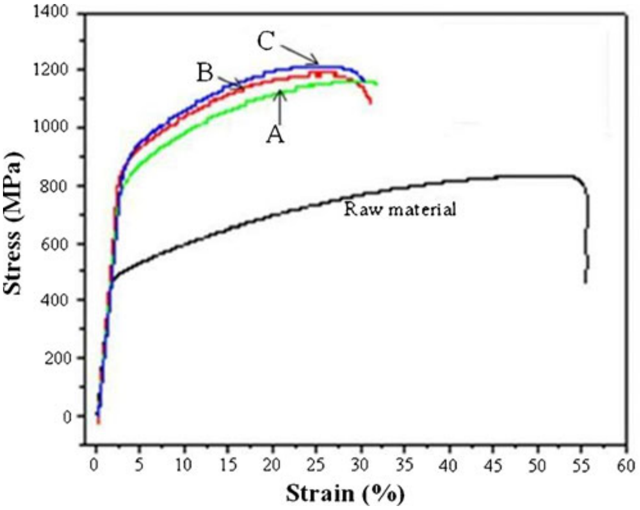


**Fig. 8** Stress and strain relationships of specimens with different heat treatments

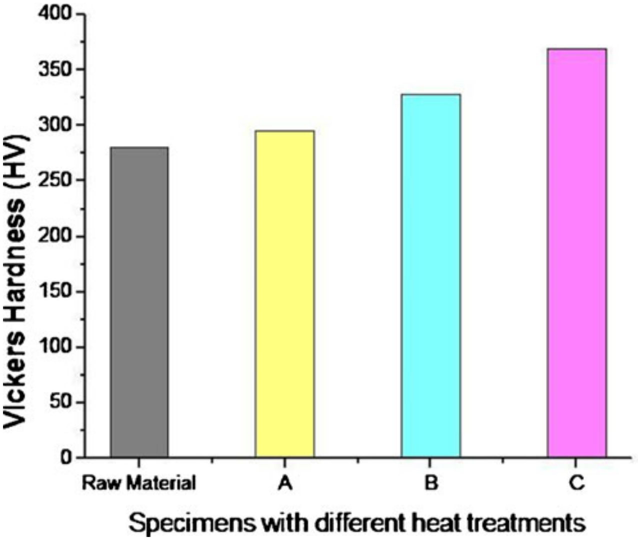


**Fig. 9** Hardness variation of specimens with different heat treatments

conditions (a) and (b), as can be shown by the ultrasonic tests.

In addition to the microstructure pictures, the results of mechanical property testing are also presented to clarify the more detailed correlation between heat treatment condition and acoustic nonlinearity. From a practical viewpoint, the enhancement of material property is determined in terms of the increase in ultimate strength and surface hardness. The data from the uniaxial tensile and the Vickers hardness tests are presented in Figs. 8 and 9, respectively. As seen in Fig. 8, the ultimate strength of the specimen with the heat treatment (c) condition appears to be higher than that of the other two cases, (b) and (a). Although the linear stress-strain relationship is almost identical for the three cases, it is noted that the nonlinear mechanical behaviors of the four specimens are quite unique depending on heat treatment conditions. This indicates that the nonlinear acoustic feature resulting from the nonlinear mechanical behavior can be a sensitive feature as predicted from the equations. (1)~(2). In Fig. 9, the surface hardness is used to distinguish the specimens after heat treated condition (c), (b) and (a) from each other much more clearly. Hardness has been used to evaluate the change in dislocation density for comparison to nonlinearity measurement [15]. As illustrated in Fig. 9, the increase of hardness would correspond to the decrease of dislocation density. This result has a good agreement with the ultrasonic nonlinearity measurement. Heat treatment is applied to form a microstructure that improves the mechanical properties [11]. The better properties provided by the heat treatments were attributed to slip homogenization and restriction of localized plastic deformation parallel to the boundaries.

As a result, overall the data trend matches quite well with that observed in the results based on the use of acoustic nonlinearity. According to the material property tests, micro-structure pictures and the nonlinear ultrasonic monitoring results, the heat treatment (c) appears to be the best process of the three conditions. The quenching condition is identical, but the three cases differ slightly in tempering temperature and time. Nevertheless, the nonlinear ultrasonic assessment can precisely predict the variation in mechanical behavior induced by the different heat treatments. This verifies that the nonlinear ultrasonic technique can be a promising tool for monitoring material enhancement as well as degradation. In addition, the result of this study provides a firm physical insight that it is possible to directly correlate acoustic nonlinearity with material nonlinearity in a reliable manner in spite of the existence of other experimental nonlinear sources arising from sensors and instruments.

## Conclusion

This paper investigates the change of acoustic nonlinearityof X-750 inconel alloy under three different heat treatment conditions. The material nonlinearity of the specimens is correlated with the acoustic nonlinearity to assess the mechanical property enhanced by the heat treatments. The experimental results show that heat treatment can help the formation of an adequate microstructure, resulting in improved mechanical properties. Material nonlinearities of specimens are also changed. The sensitivity comparison of linear and nonlinear ultrasonic parameters to material characterization is also discussed, the conventional linear ultrasonic techniques based on the use of wave velocity and attenuation are carried out. The results show that acoustic nonlienarity is a much more sensitive indicator of micro-structral changes of matereials than conventional ultrasonic features. The results of uniaxial tensile testing and the pictures of micro-structure support the data trend predicted by the nonlinear ultrasonic technique with improved sensitivity over the conventional linear methods. The present approach can be applied to the optimization of heat treatment conditions and on-line monitoring of the change of microstructure of heat treated materials.

**Acknowledgement** This work was supported by the National Research Foundation of Korea (NRK) grant funded by the Korea government (MEST) (No. 2011-0020812). The authors would also like to express appreciation to professor S. Krishnaswamy and professor J. Qu at Northwestern University, in the U. S, for their invaluable technical advice.

## References

1. Dace GE, Thompson PB, Brash LJH et al (1991) Nonlinear acoustics, a technique todetermine micro-structural changes in material. In: Thompson DO, Chimenti DE (eds) Review of progress in quantitative nondestructive evaluation. Plenum press, New York, pp 1685–1692
2. Cantrell JH (2003) Fundamentals and application of nonlinear ultrasonic nondestructive evaluation. In: Kundu T (ed) Ultrasonic nondestructive evaluation. CRC Press, Florida, pp 363–434
3. Cantrell JH, Yost WT (2001) Nonlinear ultrasonic characterization of fatigue microstructures. Int J Fatig 23:487–490
4. Nagy PB (1998) Fatigue damage assessment by nonlinear ultrasonic material characterization. Ultrasonics 36:375–381
5. Jhang KY (2000) Applications of nonlinear ultrasonics to the NDT of material degradation. IEEE Trans Ultrason Ferroelectr Freq Control 47:540–548
6. Li W, Cho Y, Achenbach JD (2012) Detection of thermal fatigue in composites by second harmonic Lamb waves. Smart Mater Struct 21:085019
7. Cantrell JH (2004) Sub-structural organization, dislocation plasticity and harmonic generation in cyclically stressed wavy slip metals. Proc R Soc Lond A460:757–780
8. Kim JY, Qu J, Jacobs LJ et al (2006) Acoustic nonlinearity parameter due to micro-plasticity. J Nondestruct Eval 25:28–36
9. Li W, Lee J, Cho Y (2010) Study of ultrasonic nonlinearity in heat treatment material. KSME 34:751–756

10. Li W, Cho Y, Hyun S (2012) Characteristics of ultrasonic nonlinearity by thermal fatigue. Int J Precis Eng Man 13:935–940
11. Floreen S, Nelson JL (1983) The effects of heat treatment and composition on the stress corrosion cracking resistance of Inconel alloy X-750. Met Trans 14:133–139
12. Ferreño D, Gorrochategui I, Sánchez L, Gutiérrez-Solana F (2004) Optimisation of heat treatment for improvement of IGSCC properties of an X-750 alloy. Eng Fail Anal 11:799–810
13. Norris AN (1998) Finite-amplitude waves in solids. In: Hamilton MF, Blackstocks DT (eds) Nonlinear acoustics. Academic, New York, pp 267–269
14. Achenbach JD (1975) Wave propagation in elastic solids. Elsevier, New York
15. Zinck AA, Krishnaswamy S (2010) Ultrasonic nonlinearity measurements on rolled polycrystalline copper. In: ThompsonD O, Chimenti DE (eds) Review of progress in quantitative nondestructive evaluation. Melville, New York, pp 1404–1409