Research on High Temperature Aging Detection Method of Heat Resistant Steel Based on Nonlinear Ultrasound Detection Technology

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Abstract. Rapid detection of high temperature aging process and early damage is very important for TP347HFG austenitic heat resistant steel, which is brittle and has short stable expansion period. Nonlinear ultrasound technology is sensitive to micro-nano scale defects and has great potential in early damage detection. The relationship curve between thermal damage degree and ultrasonic nonlinearity coefficient is obtained by using non-linear ultrasound technology, and the microstructure evolution characteristics of materials are analyzed. The results show that the ultrasonic nonlinearity coefficient of TP347HFG steel increases with aging time. The maximum point appears at 500 h and then increases monotonously. This trend is related to the formation and loss of conformal strain state. The experimental results provide a theoretical basis for the non-linear ultrasonic testing of TP347HFG steel at high temperature.

1. Introduction
The development of clean and efficient ultra-supercritical units is of great strategic significance for alleviating energy shortage and environmental pollution in China. TP347HFG steel is widely used in heating surface parts of ultra-supercritical pressure boilers, such as superheater and reheater pipes [1]. This material is a new type of 18-8 austenitic heat-resistant steel with high endurance strength and good resistance to steam oxidation corrosion. However, due to its short application time in China and lack of sufficient operation test data, and its brittleness is large and stable growth period is short, once macro-cracks occur, its growth speed is far faster than that of ordinary steel, which has great safety risks. Therefore, it is of great significance to detect early damage of TP347HFG steel.

The research on characterization of material damage by non-linear ultrasonic technology has been carried out continuously. According to the damage types, it can be divided into plastic damage [2], fatigue damage [3], thermal damage, creep damage [4], and so on. Thermal damage is one of the main factors of early damage of austenitic heat-resistant steel. Thermal damage refers to the change of material properties under stress-free or low stress state caused by temperature. Because the effects of solid solution atoms, precipitates and dislocations are more complex in the process of thermal damage, the response of ultrasonic non-linear parameters to thermal damage varies with the material [5-6].

In this paper, the principle of non-linear ultrasonic technology is briefly introduced. According to the actual demand of power plant, TP347HFG austenitic heat-resistant steel aged at high temperature is used for non-linear ultrasonic testing. Then, combined with the information of crystalline phase and precipitate, the change of non-linear ultrasonic coefficient is explained and explained from the micro...
level to prove non-linear ultrasonic coefficient. Linear ultrasonic technology has great potential in thermal damage assessment of austenitic steel.

2. Principle of Nonlinear Ultrasound Technology
Due to the existence of crystal structure, crystal defects or microcracks, solid materials generally have the characteristics of non-linearity, but the non-linearity signal is extremely weak and the traditional linear ultrasonic detection technology does not pay attention to such signals. However, when large-scale high-energy ultrasound is input into solid media, the nonlinear effect is enhanced significantly, and the propagation of ultrasound is distorted, resulting in second or higher harmonics. Nonlinear signals are sensitive to micro-and nano-scale defects and micro-cracks, which can fill the gap of traditional non-destructive testing methods in the field of micro-scale detection, and is of great significance for early damage detection of materials.

Taking one-dimensional longitudinal wave as an example, this paper briefly describes the formation of non-linear ultrasound in solids [6-7]. In general, considering the third-order accuracy, the nonlinear stress-strain relationship of solid media can be described as follows.

\[
\sigma = E\varepsilon(1 + \frac{1}{2}\beta\varepsilon)
\]  

In the formula, \(\sigma\) is stress, \(\varepsilon\) is strain, \(E\) is modulus of elasticity, and \(\beta\) is second-order nonlinear coefficient. From equation (1), we can see that the equation of motion of particle in solid medium in x direction is

\[
\rho \frac{\partial^2 u}{\partial t^2} = \frac{\partial \sigma}{\partial x}
\]  

In the formula, \(\rho\) is the density of material, \(x\) is the propagation distance of ultrasonic wave, \(u\) is the displacement of direction, \(t\) is the propagation time. Assuming that a series of compressed longitudinal waves propagating along the x-axis enter the solid medium mentioned above, combined formula (1) we can get obtain the wave equation of the longitudinal wave

\[
\rho \frac{\partial^2 u}{\partial t^2} = E \left( \frac{\partial^2 u}{\partial x^2} + \beta \frac{\partial u}{\partial x} \frac{\partial^2 u}{\partial x^2} \right)
\]  

When the ultrasonic wave entering the solid medium is a single-frequency sinusoidal wave

\[
u = A_1 \sin \omega t
\]  

Where \(A_1\) is the amplitude of the fundamental frequency wave, and \(\omega\) is the angular velocity, ignoring the sound attenuation, we can obtain the expression of the ultrasonic wave in the solid medium by using the approximate iteration method.

\[
u(x,t) = A_1 \sin(kx - \omega t) - \frac{1}{8} \beta k^2 A_1^2 x \cos 2(kx - \omega t) + \cdots
\]  

In formula (5), \(k\) is wave number. As can be seen from formula (5), the second harmonic amplitude is

\[
A_2 = \frac{1}{8} \beta k^2 A_1^2 x
\]  

Nonlinear parameters of second-order ultrasound can be obtained by transforming equation (6)

\[
\beta = \frac{8 A_2}{k^2 x A_1^2}
\]  

Formula (7) shows that \(\beta\) can be calculated from the fundamental frequency amplitude and the second harmonic amplitude. The damage and micro-defects in materials can be evaluated by \(\beta\).

3. Test device and test method
In this paper, a non-linear ultrasonic measurement system is built based on RITEC SNAP. The principle diagram of the measurement system is shown in Figure 1. The SNAP system mainly includes
a broadband RF pulse amplifier, a signal tracking receiver, a 90° phase sensitive detector, a gate integrator and multiple frequency synthesizers. It can generate high power RF pulses. The built-in superheterodyne receiver has the ability to extract hard-to-detect signals. This paper chooses the corresponding attenuator, amplifier and high frequency synthesizer. The low-pass filter module optimizes the configuration of SNAP system and integrates a sampling frequency. The UTD4202C oscilloscope with a rate of 200 MHz forms a set of non-linear ultrasonic measurement system. The non-linear ultrasonic measurement system consists of two piezoelectric ultrasonic probes, one is the transmitter and the other is the receiver. The center frequency of the transmitter is 5 MHz, the center frequency of the receiver is 10 MHz, and the diameter of the piezoelectric ultrasonic probe is 8 mm.

The samples of non-linear ultrasonic testing were aged at high temperature in box type energy saving resistance furnace (model SX 2-5-12). The holding temperature was 700°C and the holding time was 0 h, 500 h, 800 h, 1500 h and 3650 h, respectively. The samples were divided into five samples under different pressures of 9.8 N, 19.6 N and 29.4 N. Set the pressure value through the pressure sensor. Before testing, the parameters of the non-linear ultrasonic testing system need to be set. The excitation signal is a 12-cycle sinusoidal signal. Hanning modulation signal can highlight the center frequency and reduce the side lobe. The integral rate is 196 V/Vms, the fundamental wave received signal gain is set to 32 dB, the second harmonic received signal gain is set to 46 dB, and the adjustable preamplifier is set to 20 dB.

![Figure 1. Principle diagram of a non-linear ultrasonic measurement system](image)

In order to analyze the results of non-linear ultrasonic testing, TP347HFG steel samples aged at high temperature were grinded, polished and corroded by FeCl₃ hydrochloric acid solution. The metallographic structure of the samples was observed under Olympus metallographic microscope. Scanning electron microscopy (SEM) of Hitachi S-3400N and QYANTA 400 was used to observe the microstructures of the samples. The matrix and precipitated phases of the samples were analyzed by EDAX spectrometer (EDS) attached to the SEM.

4. Test results and discussion

4.1 Relationship between nonlinear coefficient and retention time
The non-linear ultrasonic measurement results of TP347HFG steel are shown in Figure 2. As can be seen from Figure 2, the non-linear coefficient of TP347HFG steel decreases rapidly after reaching its maximum at 500 h holding time. The inflection point appeared at 1 000-1500 h, and then increased monotonously with aging time. It should be noted that the nonlinearity coefficient varies with the pressure, which may be due to the ultrasonic energy transfer in the experiment. The coupling degree between the device and the sample is different.

When the holding time reaches 3500 h, the non-linear coefficient increases significantly, even exceeds the maximum point of 500 H. The non-linear coefficient is positively correlated with the aging time of the material. Therefore, in this paper, the microstructures of the test samples are studied...
by metallographic microscope and scanning electron microscope. The weave was studied and the results of the non-linear ultrasonic test were discussed from the micro-level.

4.2 Microstructure of Test Samples

The change of the non-linear coefficient is closely related to the microstructure of the sample. For thermal damage samples, the main factors affecting the non-linear effect are grain size, the number and size of precipitates. This section will start from these two aspects to analyze the influence of the microstructure of the test sample on the non-linear coefficient.

The metallographic structure of TP347HFG steel raw materials and samples with high temperature simulated aging temperature of 700 °C, aging time of 500 h, 800 h, 1500 h, 2500 h and 3650 h is shown in Figure 3.

Figure 2. Variation of Nonlinear Coefficient of TP347HFG Steel at Different Holding Time (Aging Temperature 700 °C)

Figure 3. Metallographic Structure of TP347HFG in Primitive and Age
From Figure 3, it can be seen that the original TP347HFG steel has obvious twin structure, a large number of twins, and the twin grain boundary is straight, with a small amount of abnormal coarse grains. After aging for 500 h (Figure 3(b)), no grain size was found. The number of twins decreases obviously. The grain size of aging structure at 800 h is obviously refined, and the number of twins decreases compared with that at 500 h, and tends to equiaxed. At 1500 h (Figure 3(d)), grain growth and aging were observed. The grain size of 500 h is similar, and the abnormal growth grain increases, and the number of twins is less. With the aging time prolonging, at 2500 h (Figure 3(e)), the grains increase to a great extent, and the abnormally coarse grains also increase. When aging for 3650 h, the size of grains decreases compared with aging for 2500 h, showing equiaxed grains and crystal twin almost disappearing. It can be seen that the microstructure change of TP347HFG steel at 700 °C high temperature aging is that as the aging time increases, the grains of TP347HFG steel grow rapidly after short-time refinement, and the final size tends to be stable and slightly reduced. The crystal twin number continues to decrease, and the abnormal coarse grains gradually increase.

4.3 Discussion of test results
The relationship between the microstructure of the test sample and the nonlinear effect can be explained by the principle of coherent strain. The so-called coherent strain refers to the lattice distortion that occurs when the interface between the substrate and the precipitate is at the same time on the lattice of the matrix and the precipitate. In the coherent strain state, there is a strong force between the dislocation and the precipitate. When the precipitate grows further, the coherent strain state gradually disappears and becomes a quasi-coherent strain or a non-coherent strain. The state of the coherent strain depends on the interaction of the grain boundary and the precipitate, and the grain refinement can promote the sample size to the coherent strain state, but this also requires the combination of the number and size of the precipitate.

5. Conclusion
(1) Ultrasonic nonlinear effect increases with aging time, and then increases first and then decreases, then monotonically increases. The test sample reached the coherent strain state when the aging time was 500 h, and the nonlinear coefficient showed a maximum value. After the test sample loses the coherent strain state, the nonlinear coefficient increases monotonously with the increase of aging time, which is mainly related to the increase of the number of precipitated phases and the coarsening of grain boundaries.

(2) Nonlinear ultrasonic technology has application value in the early detection and evaluation of TP347HFG under high temperature aging conditions, but it is necessary to further expand the database and increase the reliability of its prediction by mathematical modeling.

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