Research Article

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Investigation of the microstructure evolution in TP347HFG austenitic steel at 700°C and its characterization method

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Abstract: This article reports the microstructure evolution in TP347HFG austenitic steel during the aging process. The experiments were carried out at 700°C with different aging time from 500 to 3,650 h. The metallographic results show that the coherent twin and incoherent twin are existed in the original TP347HFG grains, while they gradually vanished with the increase of the aging time. After aging for 500 h, a lot of fine, dispersed particles precipitated from the matrix, but they disappeared after aging for 1,500 h. When the aging time extend to 3,650 h, the precipitates appeared apparently coarse in TP347HFG steel, which include the $M_{23}C_6$ and σ phase; besides, the microhardness of TP347HFG also changes during the aging, which was closely related to the effect of dispersion strengthening and solution strengthening. The results of the nonlinear ultrasonic measurement reveal that the β' of TP347HFG steel was also changed with the aging time. It first increased at 0–500 h, then reduced later, and increased finally at 1,500–3,650 h. The variation of β' in TP347HFG was influenced by a combined effect of the twin microstructure and the precipitate phase, which indicate

that the nonlinear ultrasonic technique can be utilized to characterize the microstructure evolution in TP347HFG.

Keywords: TP347HFG, twin structure, precipitates, nonlinear ultrasonic technique

1 Introduction

In the electric utility industry, there is a growing demand for reduction of energy consumption and CO₂ gas emission in power stations. To enhance the heat efficiency, most of the countries are developing the large capacity ultra-supercritical (USC) units. By increasing the steam temperature up to 600°C, the economic efficiency of generating units can be increased significantly. In the meantime, materials that possess the higher heat resistance are required to meet the demands of such severe service conditions [1–3].

9% Cr ferritic heat-resistant steels, such as the T/P91 (9Cr–1Mo–V–Nb) steel and T/P92 (9Cr–0.5Mo–1.8W–V–Nb) steel, have been recognized as the ideal materials for USC components because they possess high creep resistance, good ductility, and corrosion resistance. However, when the service temperature was more than 620°C, it has been reported that there was limited resistance of 9% Cr steels to steam oxidation and gas-side corrosion. However, the austenitic heat-resistant steels often possess a higher Cr content (>18%) [4–6]; therefore, they have a stronger oxidation resistance, which could be employed at the high-temperature section of the pipeline system.

New type of austenitic heat-resistant steel TP347HFG (18Cr–12Ni–Nb), developed on the basis of TP347H (18Cr–10Ni), is being promoted as the superheaters and reheaters due to its good combination of the elevated creep strength, oxidation resistance, and hot corrosion resistance. Compared with TP347H, the addition of strong carbide-/nitride-forming elements (i.e., niobium (Nb)) in TP347HFG effectively increases the creep rupture strength because the dispersive distributed fine NbCrN and Nb-rich

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carbonitrides in the matrix can play a role in precipitation strengthening [7,8].

Currently, studies related to the microstructure evolution and mechanical property change mainly focused on TP347H. However, with respect to the evolution of precipitates in TP347HFG at high temperature, there is still lack of research in this area. Considering austenitic steel with high content of Cr, the carbides usually coarsened at grain boundaries, which is unfavorable for mechanical properties; therefore, more research should be devoted to this area. This study investigated the evolution of precipitates and the change of micro-hardness of the TP347HFG. It was found that during aging, both precipitation area fraction and the micro-hardness presented a nonmonotonic variation trend. Besides, a sensitive nondestructive testing (NDT) method, i.e., nonlinear ultrasonic technique, was utilized to explore the characterization of microstructural degradation of this material. The results showed that the nonlinear parameter β' is sensitive to the microstructure change of TP347HFG. It is hoped that the research could provide valuable insights into the industrial applications of TP347HFG steel.

2 Experiments

2.1 Aging experiments and nonlinear ultrasonic measurements

The chemical composition of TP347HFG is presented in Table 1. The aging samples were cut from the TP347HFG pipe and machined into the dimension of 40 mm of length, 12 mm of width, and 10 mm of thickness. The aging experiments were performed on the resistance furnace (SX2-5-12, Yinhe, china). The aging temperature was 700°C, and the holding time was 500, 800, 1,500, 2,500, and 3,650 h.

After aging experiments, a Ritec-SNAP 5000 ultrasonic system was used to investigate the nonlinear ultrasonic response in TP347HFG specimens (see Figure 1(a)). This system was composed of a 50 Ω termination, a 6 dB attenuator, two FDK-X diplexers, and three direct digital

synthesizers (see Figure 1(b)). These function modules can realize the filtration of transmitted signals. Besides, this system contains a gated amplifier and a pair of integrators to process the information from the super-heterodyne phase-sensitive receiver. A pair of transducers (SIUI, $D = 6$ mm) are used in this study (see Figure 1(c)): 5 MHz (transmitter) and 10 MHz (receiver). To improve the accuracy of measurement, a fixed device with a pressure sensor was laid out. Thus, the pressure of the transducer on the specimen can be constant (9.8 N) during the measurement.

2.2 Microstructure observation of the TP347HFG specimens

After nonlinear ultrasonic experiments, the microstructure of each specimen was observed. The TP347HFG specimen were grinded, polished, and then etched using $\text{FeCl}_3\text{-HCl}$ solution for observation under optical microscope (OM; Olympus PMG3) and scanning electron microscope (SEM; HitachiS-3400N). The composition analysis of the precipitated phase was performed by using the energy-dispersive spectrometer (EDS; X-MaxN 20) to obtain the contents of the element. Besides, TEM samples were prepared through the extraction replica technique, the statistics of the area fraction of precipitates was performed on the TEM images, and the selected area electron diffraction (SAED) pattern was utilized to analyze the characteristic of precipitates in TP347HFG. The transmission electron microscope used in this study is JEM-2100HT (LaB₆ electron-tube heater) with 200 kV accelerating voltage.

3 Results and discussion

3.1 Microscopic structures observation under optical microscope

Figure 2 shows the OM images of the original and aged TP347HFG specimens. The matrix of TP347HFG was composed of the equiaxed austenite grains with the average size of nearly 22 μm , but the size of grains is heterogeneous,

Table 1: The chemical composition of TP347HFG steel (wt%)

Element	C	Cr	Si	Ni	Mn	P	S	Nb	N	Fe
TP347HFG	0.09	18.57	0.47	11.80	1.56	0.022	0.004	0.61	0.04	Bal.

Figure 1: Diagram of nonlinear ultrasonic measurements of TP347HFG steels.

with the large grain size of about $38\ \mu\text{m}$ and the small one of about $12\ \mu\text{m}$. Meanwhile, twin structures can be clearly observed in the grains of the original TP347HFG specimen, and it can also be subdivided into the coherent twin and incoherent twin. These twins formed at the annealing heat treatment (i.e., solution treatment) [9], which depends on the slip of stacking fault for nucleating and extending; After aging for 500 h, the grain size has not changed much, and the coherent twin and incoherent twin are still existed in the TP347HFG microstructure. Nevertheless, when the aging time exceeded 800 h, the intragranular twins gradually disappeared and the twin boundaries began to turn unclear, which is shown in Figure 2(b)–(e). After

aging for 3,650 h, no apparent grain coarsening can be found in the TP347HFG microstructure since the average grain size remains at $18\text{--}24\ \mu\text{m}$, which indicates that the austenite grain structures are relatively stable during the thermal aging at 700°C . However, the coherent twin were almost breaking down, which have no longer traversed the grains, and only a small portion of incoherent twin residue in the grain. Vanishing of the twin structures could be mainly owed to the migration and degradation of stacking fault during the aging process [9]. Besides, a certain amount of second-phase particles is distributed inside the grains of TP347HFG (see Figure 2(e) and (f)); however, due to the limitation of the resolution, it is difficult

Figure 2: The microstructure observations of TP347HFG steels during the thermal aging: (a) origin, (b) 500 h, (c) 800 h, (d) 1,500 h, (e) 2,500 h, and (f) 3,650 h.

to investigate the characteristic of precipitates and its evolution behaviors through the optical microscope observation.

3.2 Microscopic structures observation under scanning electron microscope

To figure out the evolution behaviors of precipitates in TP347HFG during the aging, the SEM observations and EDS analysis were performed. The SEM image of the original TP347HFG is shown in Figure 3(a). Before aging, it can be observed that there were relatively few precipitates existed in the grains or grain boundaries of the TP347HFG microstructure. However, after aging for 500 h, there were many tiny primary particles precipitating from the matrix, which is clearly shown in Figure 3(b). Those particles can be divided into the intragranular precipitates and intergranular precipitates. The intragranular precipitates are distributed dispersedly in the TP347HFG matrix, and their size is relatively small. On the contrary, the intergranular precipitates possess a considerable larger dimension, and they prone to aggregate at the junctions of grain boundary or triple grain boundary. The previous study [10] showed that the grain boundary junction was a kind of plane defect with large lattice distortion energy. Therefore, the carbon atoms were easier to diffuse at this place so as to fulfill a combination reaction with other elements. In addition, EDS results showed that some intergranular precipitates contain higher content of Nb (point 4), so that they could be the carbide particle of Nb(C,N).

After aging for 800 h, the fine, dispersed intragranular precipitates can still be observed in the TP347HFG matrix, but they were no longer there when aging time increased to 1,500 h. Instead, the precipitated particles appear apparently coarse, and the size of both intragranular precipitates and intergranular precipitates increased significantly. This phenomenon is the classical Ostwald Ripening [11], which is a spontaneous process of energy transition. Generally, the small particles are kinetically favored, while large precipitates are thermodynamically favored. Initially, many small particles precipitated in the TP347HFG matrix (see Figure 3(b) and (c)) as the small precipitates could nucleate more easily. However, those tiny particles have a larger surface area to volume ratio, which are energetically less stable. Consequently, many small precipitates gradually disappear except for a few that grow larger, at the expense of the small precipitates [12] (see Figure 3(d) and (e)). Eventually, some tiny particles transformed into the large ones (i.e., with the greater

volume to surface area ratio), so that a lower energy state can be achieved. During aging, the overall process of the evolution behaviors of precipitates in TP347HFG is clearly shown in Figure 3. Besides, after aging for 800 and 1,500 h, the precipitates such as $M_{23}C_6$ and σ phases have occurred (see EDS of Point 7 and Point 10), it should be note that the σ phase is a kind of intermetallic compound, which was composed of the similar element fraction of Fe and Cr. In addition, after aging for 3,650 h, the coarsening of the Nb(C,N) carbides can be clearly observed, and the content of Nb element was obviously increased in those precipitates (see Figure 3(f)).

To investigate the evolution of precipitates in TP347HFG in a quantitative manner, the statistics of the area fraction of precipitates was performed on the TEM images (see Figure 4(a)). This process was carried out by using the image analysis software (IPP-6.0, see Figure 4(b)). More than five different viewing fields were randomly chosen in each aging specimen. The area fraction of precipitates was equivalent to the total area of precipitates divided by the area of the field of view [10,13], and the error bar was also given.

Figure 4(c) shows the variation of area fraction of precipitates in TP347HFG steel during the aging. It can be observed that the area fraction of precipitates increased at the first 500 h and then decreased within the range of 800–1,500 h. After 1,500 h, the area fraction of precipitates increased continuously. Here, the change rule of precipitates was consistent with the earlier SEM observation. It is not difficult to understand that the increase in the area fraction of precipitates for the first time (0–500 h) was due to the precipitation of many tiny primary particles in the TP347HFG matrix. Then, Ostwald Ripening occurred, and many small precipitates faded away, so that the area fraction of precipitates presented a descend trend (800–1,500 h). Later, the precipitates appeared apparently coarse (2,500–3,000 h), which contributes to the increase of area fraction.

In the meantime, the micro-hardness of TP347HFG steel during the aging displayed a trend of earlier increase, later decrease, and then level off (see Figure 4(d)). The increase of the micro-hardness of TP347HFG at the early aging stage (0–800 h) can be owed to the dispersion strengthening of the precipitated particles. These tiny primary particles existed in the grains and grain boundaries, which can play a role in obstructing the glide and climb of the dislocation, thus resulted in the increase of the micro-hardness of TP347HFG. However, as time went on, alloying elements such as the Cr and Nb inevitably precipitated from the TP347HFG matrix. Thus, the effect of solution strengthening has diminished, which causes the decrease of the micro-hardness of TP347HFG.

Figure 3: The SEM observations and EDS analysis of TP347HFG steel during the thermal aging: (a) origin, (b) 500 h, (c) 800 h, (d) 1,500 h, (e) 2,500 h, and (f) 3,650 h.

Figure 4: The statistics of area fraction of the precipitates and the hardness measurement of TP347HFG: (a) TEM images, (b) statistics process and the use of image analysis software, (c) the variation of area fraction of precipitates, (d) the hardness change in TP347HFG during the aging, and (e) the analysis of precipitates by using the SAED pattern.

Besides, the selected area electron diffraction (SAED) pattern was utilized to analyze the characteristic of precipitates in TP347HFG. Digital micrograph software was used here to calibrate the diffraction pattern. The SAED results further confirmed that, after aging for 3,650 h, there were $M_{23}C_6$ phase and σ phase precipitated in the TP347HFG matrix. Nevertheless, the σ phase presented a striped shape, and this could induce the stress concentration at the σ phase/matrix interface, which

was unfavorable for the microstructural property of TP347HFG steel.

3.3 Nonlinear ultrasonic measurements

Since the characteristics of ultrasonic signal can reflect the inherent property of metallic materials as the ultrasonic wave travels through, the characterization of the

materials degradation based on the ultrasonic method has been used widely during these years [14–17]. Nevertheless, it is worth noting that most of the conventional ultrasonic methods such as wave velocity method or attenuation method are only available for detecting those defects with larger dimensions ($\Phi > 1$ mm, i.e., gross defect), but regarding the degradation of microstructure in materials that happened in premature stages (e.g., precipitates coarsening or dislocation structure change), the effectiveness of conventional ultrasonic methods have been confirmed to be quite low [18].

The nonlinear ultrasonic technique is an emerging alternative approach, which has been recognized as a powerful method to characterize the microstructural feature of materials. For instance, Hikata *et al.* [19] investigated the nonlinear effect in an aluminum single crystal, and they found that the dislocation movement under the action of stress will contribute to the variation of the nonlinear parameter; Valluri *et al.* [20] studied the nonlinear response in a 99.98% pure copper, and they pointed out that the appearance of micro-void during the creep process could also lead to the increase of the nonlinear parameter. Xiang *et al.* [21] investigated the nonlinear effect in a ferritic Cr–Ni alloy steel, and they revealed that the precipitation of the second phases during the process of thermal degradation will result in the change of ultrasonic nonlinearity. In conclusion, all these studies indicated that the nonlinear ultrasonic technique is sensitive enough to probe the microstructural change in metals, which can be unitized to characterize the degradation of material that occurred in early stages.

3.3.1 The principle of the ultrasonic nonlinearity

The main difference between the conventional ultrasonic techniques and the nonlinear ultrasonic technique was that the latter could reflect the frequency change of the input signal, while the former could not. Thus, the degradation of material can be determined by monitoring how much amplitude of the higher harmonic wave generated in the transmitted wave [10].

Generally, the nonlinear wave equation could be expressed as follows:

$$\rho \frac{\partial^2 u}{\partial t^2} = E \frac{\partial^2 u}{\partial X^2} \left(1 + 2\beta \frac{\partial u}{\partial X} \right), \quad (1)$$

where u is the displacement vector, t is the time, ρ is the medium mass density, β is the second-order nonlinear elastic coefficient (i.e., nonlinear parameter), and X is the wave propagation distance.

Consider there is a plane wave $A_1 \cos(kX_1 - \omega t)$, A_1 is the fundamental wave amplitude, k is the wave number, and ω is the angular frequency, and then, the approximate solution of displacement can be obtained after two iterations:

$$\begin{aligned} u_1 &= -\frac{1}{8} \beta k^2 A_1^2 X_1 + A_1 \cos(kX_1 - \omega t) \\ &+ \frac{1}{8} \beta k^2 A_1^2 X_1 \cos[2(kX_1 - \omega t)] + \dots \\ &= A_0 + A_1 \cos(kX_1 - \omega t) + A_2 \cos[2(kX_1 - \omega t)] + \dots \end{aligned} \quad (2)$$

From this solution, the amplitude of the second harmonic (i.e. A_2) can be obtained as follows:

$$A_2 = \frac{1}{8} A_1^2 k^2 X_1 \beta. \quad (3)$$

Figure 5: The curve of the frequency spectrum of the TP347HFG steel: (a) origin specimen and (b) aging for 3,650 h.

Therefore, the nonlinear parameter β can be written as follows:

$$\beta = \frac{8A_2}{k^2 X_1 A_1^2}. \quad (4)$$

Because the X_1 and k are constant during the measurement, to simplify the calculation, it can also use β' to replace β :

$$\beta' = A_2/A_1^2. \quad (5)$$

3.3.2 Results of nonlinear ultrasonic measurement

Figure 5 shows the curves of the frequency spectrum of the TP347HFG steel before and after the aging experiments. It can be recognized that the frequency spectrum comprises both 5 and 10 MHz output frequency signals, which correspond to the fundamental wave (A_1) and second harmonic wave (A_2), respectively. Figure 5 shown that the second harmonic amplitude (A_2) of TP347HFG increased significantly after aging for 3,650 h (here, it should be noted that the fundamental wave amplitude (A_1) also increased after the aging experiments).

In general, the numerical value of the second harmonic (A_2) can be extracted to assess the degradation of materials. However, a tooth-like nonlinear waveform (around the frequency of 10 MHz) was generated during the Fourier transform, which could lead to the data oscillation of the amplitude of second harmonic. To assess the degradation degree and calculate the nonlinear parameter of TP347HFG steel more reliably, the signal processing was carried out on the second harmonic value, the average of the amplitude of the frequency component from 9.9 to 10.1 MHz (i.e. nine points) was taken, and this value could represent the overall level of the second harmonic in each TP347HFG specimen.

Figure 6 shows the variation of nonlinear parameter β' of the TP347HFG steel before and after the aging experiments, the nonlinear parameter was calculated from equation (5), and the error bar showed the maximum and minimum value of each specimen. Figure 6 shows that the nonlinear parameter did not exhibit a monotonic increase or decrease in TP347HFG during the aging. However, it increases first (0–500 h), declines later (500–800 h, 800–1,500 h), and then increases at the final (1,500–3,650 h). This result is somewhat like the change rule of the area fraction of precipitates (see Figure 4(c)); however, previous studies [17–19] revealed that the nonlinear effect in metals can be affected by multiple factors; apart from the coarsening of precipitates, the appearance

Figure 6: The change of the nonlinear parameter β' in TP347HFG during the aging experiment.

of micro-void and the change of dislocation density or structure were also essential for the generation of nonlinear effect. In this study, the variation of the nonlinear parameter in TP347HFG may influence by a combined effect of the change of the twin microstructure and the precipitates.

As shown in Figure 7, in the first 500 h, the twin structure has not changed too much since coherent twin and incoherent twin were still there. However, a lot of tiny particles precipitating from the matrix could give rise to the nonlinear effect in TP347HFG. Marino et al. [22] studied the evolution of precipitates in a 9% Cr ferritic steel, and they revealed that the nonlinear parameter was sensitive to the change of the density of precipitates. As the dimension of the precipitated particles is far less than the wavelength of the ultrasonic wave, the phenomenon of sound scattering can happen, which could alter the initial propagation path of the ultrasonic wave. Thus, the multiplication of precipitates in TP347HFG steel could result in the distortion of the wave propagation, which gives rise to the nonlinear effect in the aged 500 h specimen.

When aging time increased to 800 h, compared with the former, there was less change in the density of the precipitates (see Figure 4(c)), while a part of twins (both coherent twin and incoherent twin) has begun to degrade. Previous studies [17,19] verified that the content of dislocation in metals could also be a significant factor for the ultrasonic nonlinearity. Meanwhile, the twin boundary can be viewed as the dislocation wall, which was composed of a set of partial dislocations. Therefore, the degradation of the twin structure could lead to the decline of the nonlinear effect in metals, and this might be the appropriate interpretation for the decrease of nonlinear parameter from the range of 500 to 800 h. Besides, the

Figure 7: Illustration of microstructure variation of the TP347HFG steel during the aging experiment.

nonlinear parameter decreased further when aging time increased to 1,500 h. This can be attributed to the combined effects of the twin degradation (see Figure 2(d)) and the decreased content of precipitates (i.e. the decrease of area fraction of precipitates, see Figure 4(c)).

After aging for 1,500 h, the nonlinear parameter β' did not drop any more, but increases continuously with the increase of the aging time (1,500–3,650 h). At this stage, the coarsening of precipitates (e.g., $M_{23}C_6$ phase and σ) may play a more important role in the change of the ultrasonic nonlinearity than the twin degradation. Cantrell and Yost [23] revealed that the growth of precipitates embedded in a surrounding matrix will cause a local stress field and thus would trigger the motion of

dislocation segments and give rise to a significant change in the nonlinear effect. In addition, precipitates can also be viewed as the body defect. When an ultrasonic wave was propagating through the material, the change of the propagating direction of ultrasonic wave would happen on the precipitates/matrix interface, which could cause the effect of the reflection or refraction of the ultrasonic wave. Apparently, the probability of the distortion of ultrasonic wave will increase with the increase in the size of precipitates, which lead to the increase in the nonlinear parameter. Based on the aforementioned results, it can be recognized that the nonlinear ultrasonic response is sensitive to the microstructure change of TP347HFG steel during aging.

4 Conclusions

In this study, the evolution of precipitates in TP347HFG steel during aging at 700°C was investigated. Meanwhile, the nonlinear ultrasonic technique was utilized for characterizing the microstructure change of the TP347HFG steel. The main conclusions can be summarized as follows:

- (1) Coherent twin and incoherent twin are existed in grains of the original TP347HFG specimen. After aging for 800 h, the intragranular twins gradually disappeared. When the aging time increased to 3,650 h, the coherent twin structures were almost break down, which no longer traversed the grains, and there was only a small portion of incoherent twin residue in the grain.
- (2) Many fine, dispersed precipitates can be observed after aging for 500 h, but they disappeared when aging time increased to 1,500 h. With the extend of aging time, the precipitate particles appeared apparently coarse, which is a phenomenon of the classical Ostwald Ripening.
- (3) The micro-hardness of TP347HFG was also changing during the aging experiment. The increase of the micro-hardness of TP347HFG steel at the early stage (0–800 h) was due to the dispersion strengthening. With the increase of aging time, the effect of the solution strengthening diminished and the micro-hardness of TP347HFG have declined.
- (4) The nonlinear parameter of TP347HFG steel increases first (0–500 h), declines later (500–1,500 h), and then increases finally (1,500–3,650 h) during the aging process. The variation of the nonlinear parameter in TP347HFG was influenced by a combined effect of the change of the twin microstructure and the precipitates.

In conclusion, these results manifest that the nonlinear ultrasonic technique is sensitive enough to probe the microstructure evolution in metal, which can be utilized for the characterization of microstructural feature of TP347HFG steel.

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