Effect of Irradiation on Austenite Phase in Thermally Aged 308 Stainless Steel Weld Metal

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Abstract. In this study, 308 stainless steel weld metals aged at 400°C for 7000h were irradiated at 400°C to different fluences with 240keV proton to study the effects of radiation on austenite phase. The microstructural change was examined by transmission electron microscope (TEM), and the hardness change was measured with a nano-indentation tester. It was found that bubbles and dislocation loops were formed in the austenite phase, and their size and numerical density increased with the irradiation dose. The hardness of the austenite phase also increased with the irradiation fluence. According to the statistical results of microstructural features, the dispersed barrier hardening model was used to correlate the hardness change with the microstructure evolution. Although the calculated value of hardness change was lower than the measured value, the variation trend of the calculated hardness values was in good agreement with that of the measured values.

1. Introduction
The failure of nuclear materials has become a key factor affecting the safe and efficient operation of nuclear power plants [1]. It is necessary to fully evaluate the service performance of structural materials. Because of its good mechanical properties and corrosion resistance [2-4], stainless steel is widely used as the main weld metal for various key structures of the reactor (e.g. core barrel welds and flux thimble tube plugs) [5, 6]. Service environment for stainless steel weld metal is extremely harsh, such as high temperature and severely neutron irradiation which can reach $10^{22}$n/cm$^2$ (E > 1MeV) at the end of the design life of the reactor [7, 8]. Under the condition of high temperature, stainless steel weld metal will undergo complex microstructure changes in the ferrite phase, such as spinodal decomposition into Fe-rich α phase and Cr-rich θ phase, G-phase precipitate formation [9-12]. Besides, irradiation can not only lead to the formation of defects by displacement of lattice atoms in materials, but also enhance the diffusion of defects and solute/impurity atoms, and the evolution of...
defects will lead to more complex microstructure configurations in materials, such as the formation of defect clusters, dislocation loops, cavities and the precipitates [13]. Under long-term service conditions, thermal aging and irradiation effects will lead to the increase of brittleness and decrease of toughness of the stainless steel weld metal, which will lead to the deterioration of mechanical properties of materials, affect the stability of structural materials, and thus seriously threaten the service safety of nuclear power units [14-16].

Previous studies have shown that the decrease of toughness of stainless steel weld metal comes from the contribution of ferrite phase, while the change of tensile strength mainly depends on the change of microstructure of austenite phase [17-19]. Although a large number of researches focused on the microstructural evolution and mechanical property changes in ferrite phase in stainless steel weld metal under different conditions [2-6, 10, 11, 17-19], little attention was paid to the radiation and thermal aging effects on austenite phase in stainless steel weld metal. In order to fully and accurately evaluate the service behavior of stainless steel weld metal, the microstructure evolution and mechanical property changes in austenite phase under different conditions also need to be systematically studied. Synergistic effects of irradiation and thermal aging exist in stainless steel weld metal under the actual service conditions. However, it is obviously unrealistic to carry out relevant research in the laboratory. An irradiation experiment after thermal aging and a thermal aging experiment after irradiation are fruitful methods to understand the basic synergistic effect between them [20].

In this study, the method of accelerated thermal aging treatment first and then ion irradiation was adopted to investigate the effect of irradiation on austenite phase in thermally aged 308 stainless steel weld metal. Microstructural evolutions and mechanical property changes were examined by TEM and nano-indentation analyses, respectively. The dose dependence of the size and number density of bubbles and dislocation loops was studied. The dispersed barrier hardening model was applied to correlate the microstructures and hardness changes of austenite phase in thermally aged 308 stainless steel weld metal.

2. Materials and methods

2.1. Materials

The materials used in this study are the 308 stainless steel weld metals, cut from the weld cladding of a mockup of the nozzle/safe-end weld joint. Table 1 lists the chemical composition of the 308 stainless steel weld metals.

| Sample | C  | Si | Mn | P  | S  | Cr | Mo | Ni | Cu | V  | Fe  |
|--------|----|----|----|----|----|----|----|----|----|----|-----|
| 308    | 0.012 | 0.42 | 1.36 | 0.011 | 0.012 | 19.70 | 0.27 | 10.06 | 0.072 | 0.045 | Bal. |

Figure 1. Microstructure of 308 stainless steel weld metal, showing wormlike δ-ferrite (δ) embedded in austenite (γ) matrix.
The weld cladding materials used were fabricated by use of argon tungsten arc welding process, followed by post-weld annealing treatment at 650°C for 1 hour. Fig. 1 shows the microstructure of the 308 stainless steel weld metals. Typical characteristics of austenite dendrites with distribution of wormlike ferrite phase were manifested in the stainless steel weld metals, and the typical width of ferrite is about 1 μm. Thermal aging treatment was conducted at 400°C for 7000 h. The ferrite content, which was measured by ferritometer, was about 14% in materials before and after the thermal aging treatment.

2.2. Irradiation

![Figure 2](image)

**Figure 2.** The damage profile and H+ distribution of proton irradiations in the thermal aged 308SS weld specimens to a fluence of $3 \times 10^{17}$ H+/cm$^2$.

Thermal aged specimens were cut with a size of 15×15×1mm$^3$ used for proton irradiation experiments. The surface of the specimen was prepared by combination of mechanical and electrochemical polishing in order to remove the influence of surface damage. All specimens were irradiated with 240 keV proton at 400°C at the ECR–320 kV High-voltage Platform in Institute of Modern Physics (IMP), Lanzhou, China. The irradiation fluences were $1.5 \times 10^{17}$ and $3 \times 10^{17}$ H+/cm$^2$. According to Monte-Carlo code SRIM 2013 using the quick damage mode with the density of 7.8 g/cm$^3$, lattice binding energy of 0 eV and displacement energies of 40 eV for Fe, Cr and Ni sub-lattices, calculated results for the fluences of $1.5 \times 10^{17}$ and $3 \times 10^{17}$ H+/cm$^2$ showed that atomic displacement reached their maximum values of about 0.3 and 0.6dpa at the depth of about 1μm for 240keV proton irradiation. The damage profile and H$^+$ distribution of proton irradiations in the thermal aged specimens to a fluence of $3 \times 10^{17}$ H+/cm$^2$ was shown in Fig. 2.

2.3. Material characterization

Using a lift-out method, TEM samples were lifted with a dual-beam focused ion beam instrument (Helios 600i, FEI). To minimize the damage layer caused by Ga ion implantation on the surface of the samples, a small energy of 2 keV was used during the final milling process. BF and WBDF characterization for all specimens were performed using FEI Tecnai G$^\text{2}$ F20 S-TWIN TEM at 200kV. The thickness of the test area was characterized by CBED method, and the thickness of about 70 nm was obtained.

Nano-indentation tests were performed using the Anton Pair Nanoindentation Tester (NHT) with a load of 2.5mN to investigate the irradiation-induced hardening behaviors of the austenite phase in thermal aged stainless steel welds. The mean values of hardness were acquired from at least 6 indentations for each sample.
3. Results and Discussion

3.1. Microstructure of austenite phase in unaged and thermally aged stainless steel weld metals

![Microstructure of austenite phase in 308 stainless steel welds](image1)

**Figure 3.** Microstructure of austenite phase in 308 stainless steel welds a) without thermal aging treatment b) after thermal aging treatment at 400 °C for 7000h.

Fig.3 shows the microstructure of austenite phase in as-received and thermally aged 308 stainless steel weld metals characterized by TEM. The typical morphology of dislocations and stacking faults was observed. Besides, there is nearly no difference between the microstructure of austenite phase in as-received and thermally aged 308 stainless steel weld metals. Fujii et al. [20] studied the hardness change of austenite phase in duplex stainless steel aged at 400 °C for up to 40000h, the results showed that the hardness of austenite phase did not change. Although the hardness of austenite phase increased after irradiation, the hardness of materials with different aging duration before irradiation remained consistent. It was indicated that the microstructure has not changed after aging at 400 °C for up to 40000h. In addition, Takeuchi et al. [21] found that all the solutes were distributed homogeneously in the austenite phase of unaged and 400 °C -10000h aged RPV weld overlay materials regardless of aging conditions by using APT. Therefore, it can be considered that the microstructure of austenite phase in stainless steel weld metals may not be changed under the thermal aging condition of 400 °C for 7000 h.

3.2. Microstructural evolution of austenite phase in thermally aged stainless steel weld metals during proton irradiation

![Microstructural evolution of austenite phase in thermally aged stainless steel weld metals during proton irradiation](image2)
Figure 4. Under-focus and over-focus TEM photographs of bubble morphology in the damage peak region of proton-irradiated austenitic phase in thermally aged stainless steel weld metal a) & b) \( \sim 0.3 \text{dpa} \); c) & d) \( \sim 0.6 \text{dpa} \).

As a typical feature, bubbles can be easily formed in austenitic stainless steels after irradiation at intermediate temperatures (\( \sim 0.3-0.5T_m \), TM is the melting point of the metal) [13]. The bubble can be effectively identified and observed with over-focus and under-focus method by using TEM [22], as shown in Fig. 4. The morphology of bubbles was very prominent in the peak damage regions of austenite phase in thermal aged stainless steel weld metals irradiated to different dose levels of about 0.3 and 0.6dpa at 400°C.

![Figure 4](image)

Figure 5. Quantitative statistical results of number density and average size of the bubble distribution in austenitic phase of thermally aged 308 stainless steel weld metal under different irradiation doses.

Different size distributions of bubbles can be seen clearly from the obtained TEM images. By the quantitative statistics of these images, the change of average bubble size and number density with irradiation dose in the peak damage regions of austenite phase in thermal aged stainless steel weld metals was obtained, as shown in Fig. 5. With the increase of irradiation dose, the bubble size and number density increased. However, considering the statistical error, the change of average bubble size was not obvious. According to the trend curve and TEM images, newly formed small-sized bubbles along with some growing bubbles were identified in austenite phase irradiated to 0.6dpa. The statistical average treatment of bubble size results in the small difference between the average bubble sizes in austenitic phase under different irradiation doses.
It has been reported that the saturated dose of bubble density in austenitic stainless steels is at doses of less than 0.5 dpa during irradiation near 400°C [23]. However, according to the experimental results, the bubble density was still increasing when the irradiation dose was up to 0.6 dpa. Unlike pure austenitic stainless steel, stainless steel weld metal contains a large number of ferrite phase. This indicates that there are a lot of phase interfaces around the austenite phase. It is well known that boundaries as the neutral sink of the defect can absorb interstitials and vacancies formed in materials by irradiation, and make these defects recombination at the interface to reduce the vacancy supersaturation concentration required for bubble nucleation [24], which leads to the increase of bubble density saturation dose.

**Figure 6.** BF and WBDF TEM photographs of dislocation loop morphology in the damage peak region of proton-irradiated austenitic phase in thermally aged stainless steel weld metal a) & b) \(\sim0.3\text{dpa}\); c) & d) \(\sim0.6\text{dpa}\)
Figure 7. Quantitative statistical results of number density and average size of the dislocation loop distribution in austenitic phase of thermally aged 308 stainless steel weld metal under different irradiation doses a) number density b) average size.

Like the case of the bubble, dislocation loops also exist widely in irradiated austenitic stainless steels, and are considered to be the key microstructure characteristics affecting the mechanical properties of materials. Based on WBDF technique with g=200, radiation induced dislocation loops formed in austenite phase in thermally aged 308 stainless steel weld metals were characterized. Fig.6 shows the morphology of dislocation loops in the peak damage regions of austenite phase in thermally aged stainless steel weld metals irradiated to different dose levels of about 0.3 and 0.6 dpa at 400°C. The size and density of dislocation loops were measured manually, and results were shown in Fig.7. With the increase of irradiation dose, the number density and the average size of dislocation loops in the peak damage region of austenite phase increase significantly, and there is no sign of saturation. Studies have shown that the number density of the dislocation loop of irradiated austenitic stainless steel generally reaches saturation at about 1 dpa, while the saturation dose for the average size of the dislocation loop is about 5 dpa [25]. The results obtained in this work are in good agreement with those of previous studies.

3.3. Correlation between microstructures and hardness changes of austenite phase in thermally aged 308 stainless steel weld metal

Figure 8. Variation of hardness of austenite phase in thermally aged stainless steel weld metal irradiated by protons with irradiating fluence.
The variation of the average hardness of austenite phase in thermally aged 308 stainless steel weld metals with the irradiation fluence is shown in Fig. 8. The hardness of austenite phase in un-irradiated thermally aged 308 stainless steel weld metal is about 3.1 GPa, which is similar to Fujii's experimental result of about 3 GPa [20]. The hardness of austenite phase increases significantly with the increase of irradiation fluence. Radiation hardening behavior is common in Fe-based alloys and has been reported frequently [26-28]. Irradiation can induce defects and their cluster formation in materials. These defects will become obstacles to the movement of dislocation, resulting in hardening of the material. Combined with the experimental results, it can be seen that the formation of bubbles and dislocation loops is the cause of hardening of austenite phase in thermally aged stainless steel weld metals after irradiation. Studies have shown that the dispersed barrier hardening model based on Orowan’s mechanism can establish a correlation between irradiated microstructure and yield strength changes [29, 30]:

$$\Delta \sigma_y = \left( \sum \Delta \sigma_k^2 \right)^{1/2} \text{ and } \Delta \sigma_k = aM\mu b(N_kd_k)^{1/2}$$

(1)

Where, $k =$ dislocation loops, black dots, cavities and precipitates, $M$ is the Taylor factor taking the value of 3.06; $\mu$ is the shear modulus, and $b$ is the Burgers vector: taking $\mu=84$ GPa and $b=0.255$ nm in this study; $N_k$ and $d_k$ are the number density and the mean diameter of type $k$ defects, respectively [30, 31]. The $\sigma$ is the hardening coefficient which is dependent on the type of defects. For stainless steels, we choose the values are 0.2 and 0.45 for bubbles and dislocation loops, respectively [32, 33]. On the basis of these calculations, the yield strength increments due to these two kinds of irradiation defects were obtained.

Figure 9. Comparison of hardness increment of austenite phase calculated by dispersion hardening model with the experimental value after proton irradiation at different fluence of thermally aged 308 stainless steel weld metal.

In addition, according to Busby’s research on the relationship between the changes of yield strength and hardness of different materials after irradiation, it is shown that there is a following relationship for austenitic stainless steels [34]:

$$\Delta \sigma_y = 3.03\Delta H_v$$

(2)

The calculated value of hardness change of austenite phase can be obtained by comprehensively using the above relationships, and results are shown in Fig. 9. As the trend of the measured values, the variation of the calculated hardness values increases continuously with the increase of the irradiation dose. However, compared with the hardness measured in the experiment, the calculated value is lower.
It is worth noting that TEM is one of the most effective means to characterize the microstructure of materials, but its resolution has some limitations. During the nucleation stage, the size of irradiation defects is relatively small, and TEM is difficult to distinguish these small defects, which leads to the low statistical value of defect number density. Besides, some studies have shown that precipitates such as Ni₃Si were formed in austenitic stainless steel after irradiation [18]. Although the relevant information of the precipitation phase was not found in this work, it was possible that its size is too small to be effectively characterized.

4. Conclusion
In this study, radiation effects on the microstructure and hardness of austenite phase in thermally aged 308 stainless steel weld metal were systematically studied. It was found that the microstructure of austenite phase in 308 stainless steel weld metal aged at 400 °C for up to 7000h has not changed. After irradiation, bubbles and dislocation loops were found obviously in the austenite phase in thermally aged 308 stainless steel weld metal. The size and numerical density of the bubble and dislocation loop increase with the irradiation dose. Nano-indentation test results indicated that the hardness of austenite phase in thermally aged 308 stainless steel weld metals was increasing with the increase of irradiation dose. Correlation between microstructures and hardness of austenite phase in irradiated samples was studied by using the dispersed barrier hardening model. The results show that the calculated value of hardness change is lower than the measured value. The reason may be due to the tiny defects and precipitates which hardly be resolved by TEM.

Acknowledgments
This study was supported by the National Key Research and Development Program under Grant No. 2017YFB0702200, the National Natural Science Foundation of China under Grant No.11675123 and 11775255. The authors would like to thank Tielong Shen from Institute of Modern Physics, Chinese Academy of Sciences for his detailed discussion.

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