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Effect of Aging Treatment on Properties and Post-weld Microstructure of Stainless Steel Containing Niobium

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Abstract: The effects of different aging processes on the precipitated phase, mechanical properties, molten salt corrosion resistance and post-weld microstructure of 347H stainless steel were studied. The results show that a large number of precipitated phases appear in the crystal after aging at 700 °C for 400 h. After aging for 3000 h, the number of precipitated phases increases and most of them are gathered at the grain boundaries. There are two forms of precipitates, one is the coarse precipitate rich in Cr, and the other is the smaller precipitates mainly consisting of NbC. After aging at 700 °C for 30 min, the yield strength and tensile strength of the samples at room temperature and 593 °C increase, but the elongation decreases. The corrosion results in nitrate at 565 °C show that the corrosion products of the aged samples are the same as that of the original samples, which are Fe₂O₃, Fe₃O₄, MgCr₂O₄, MgFe₂O₄, FeCr₂O₄ and NaFeO₂. The proportion of Fe₃O₄ that is dense and well bonded to the subtrate in the original sample is higher than that in the aged sample, so the corrosion resistance is better. At 700 °C, the aging time has no obvious effect on the microstructure after welding.

Key words: aging; 347H stainless steel; precipitated phase; mechanical properties; molten salt corrosion; welding property

Solar energy is found as the most promising renewable green energy due to its large reserves, easy access, low utilization cost and no pollution. Concentrated solar power (CSP) is considered to be the most promising generation technique for renewable energy power due to its low photothermal power generation cost and high thermal utilization efficiency^[1]. The principle of CSP is to heat the heat transfer fluid (HTF) inside the concentrator and to reflect the sunlight onto the receiver by the concentrator. The HTF then passes the heat through the heat exchanger to transfer the heat to the feed water, which is converted into high temperature and high pressure steam. Finally, the steam drives the steam turbine to generate electricity. Due to the existence of heat storage systems, power generation can be achieved at night or in cloudy days, bringing significant economic advantages to solar power plants^[2-3]. At present, the widely used HTF medium is molten salt. Among them, nitrate molten salt is widely used in CSP power plants because of its high density, medium specific heat, low chemical reactivity and low cost, as well as good thermal conductivity, diffusivity and low heat loss coefficient, for example, the binary mixture of solar salt-sodium nitrate and potassium nitrate (60:40, wt%)^[3-5].

In the CSP system with molten salt as HTF, the operating temperature of the cold tank is 290 °C, and the operating temperature of the hot tank is higher than 550 °C. Due to the high working temperature, the construction of thermal storage containment components requires the use of stainless steel materials, in which austenitic stainless steel is the main candidate material^[6–7]. It is well known that binary nitrates are unstable above 600 °C in oxidizing atmosphere^[8], and the combination of high operating temperature and salt composition presents a severe corrosive environment^[9]. Molten nitrates contain oxidizing substances, such as nitrate, nitrite ions and oxygen (from air), as well as impurities including commercial salts, such as chlorides, nitrites and sulfates, which can cause corrosion of structural materials^[8–10]. In this

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case, it is important to evaluate the corrosion resistance and service life of the stainless steel to optimize material selection, as failure of the stainless steel can lead to severe damage and cost increase in the operation of photothermal power plants^[11].

347H stainless steel is a chromium-nickel-niobium austenitic stainless steel. Due to the presence of niobium element, it has good intergranular corrosion resistance, structural stability and excellent oxidation resistance. In addition, it also has good welding performance. It is mainly used to manufacture high temperature superheater, reheater and various high temperature and high pressure pipe fittings of large power generation boilers with subcritical and supercritical pressure parameters^[12–13]. Due to the complex and harsh application environment, 347H is required to have excellent corrosion resistance and good high temperature performance. In particular, the resistance to molten salt corrosion and creep resistance under high temperature is particularly critical, which directly affects the service life and safety of molten salt storage tanks^[14]. 347H stainless steel is based on 304H and contains an appropriate amount of Nb element. The Nb element combines with the C atom in the stainless steel to form NbC, which has a pinning effect on the grain boundary and prevents the C atom from combining with Cr to form intergranular Cr₂₃C₆, thereby improving the intergranular corrosion resistance. However, the control of microstructure such as grain size and precipitation after processing is an important challenge affecting the overall performance^[15-16]. Therefore, for achieving the required performance of the metal parts, in addition to the selection of appropriate materials and forming process, it is also necessary to consider the heat treatment, which is essential.

Aging treatment refers to the heat treatment process in which the material is treated by solid solution, quenched from high temperature or deformed by a certain degree of cold processing, and placed at higher temperatures or room temperature to maintain its shape, size and performance over time. Generally speaking, after aging, the hardness and strength are increased, while the plasticity and internal stress are decreased^[17-18]. At present, there are few studies on the high temperature behavior and molten salt corrosion behavior of 347H stainless steel after heat treatment. In addition, in the application process of 347H steel plate in high temperature molten salt storage tank of photothermal power station, the high temperature mechanical properties need to be improved by aging treatment. However, due to the influence of weld microstructure on the weld quality of post-weld aged steel plate, further research and verification are needed to ensure the safety of the tank under molten salt storage conditions.

In this study, through Thermo Calc simulation software, metallographic microscope (OM), universal mechanical testing machine, scanning electron microscope (SEM) equipped with energy disperse spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS) and X-ray diffractometer (XRD), the effects of aging treatment at different temperatures and durations on the precipitates, mechanical properties, molten salt corrosion resistance and post-weld microstructure of 347H stainless steel were studied. The influence law and mechanisms of aging process on the microstructure and mechanical properties of 347H stainless steel and its weld were summarized. At the same time, the effect of aging treatment on the corrosion performance of 347H stainless steel in nitrate at 565 °C was investigated, which laid a foundation for the application of 347H stainless steel in different industrial fields.

1 Experiment

This study conducted experimental research on 347H cast billets smelted on the production site. The specific process flow is as follows: electric furnace-AOD converter smelting→LF furnace refining→continuous slab casting→ slab grinding-heating furnace heating-medium thick plate rolling-annealing and acid washing-inspection and storage. The chemical composition of 347H smelting is listed in Table 1. Thermo Calc software was used to calculate the equilibrium phase diagram of 347H heat-resistant stainless steel and to determine the equilibrium phases at different temperatures. 30 mm×30 mm×10 mm samples of 347H stainless steel were taken under different aging processes, which were polished by 400#, 800#, 1200#, 1500# and 2000# sandpaper in sequence, then polished, washed with alcohol and blow dried. The samples were corroded in ferric chloride hydrochloric acid solution for 8 min. The precipitated phases were observed by FESEM and EDS. The mechanical properties were tested by a universal testing machine. Molten salt was adopted for static immersion corrosion test, with a mass ratio of sodium nitrate to potassium nitrate at 6:4. Put the mixed nitrate into the crucible, and set the temperature of the box type resistance furnace to 565 °C. As the experimental temperature reached the expected temperature, the sample was placed in the crucible for 120, 240 and 360 h. After the experiment was completed, remove the sample and wash it with deionized water and alcohol for 5 and 10 min before weighing. SEM was used to observe the corrosion products, XPS was used to analyze the valence state of the corrosion products, and XRD was used to determine the phase of the corrosion products.

2 Results and Discussion

2.1 Effect of aging on precipitated phases

Fig. 1 shows the solidification equilibrium phase diagram and precipitation phases of 347H stainless steel. It can be seen that the main equilibrium phases of 347H stainless steel include austenite phase (γ), ferrite phase (α), Nb(C, N), σ phase, Laves, M_5 SiN, $M_{23}C_6$, NbNi₃, Z phase, etc. The phase composition of each precipitated phase at different

Table 1 Chemical composition of 347H smelting billets (ASTM A480/A480M) (wt%)

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С		Si	Mn	Р	S	Ni	Cr	Nb
0.05	50	0.52	1.14	0.025	0.0006	9.13	17.46	0.54



Fig.1 Phase diagram of 347H solidification equilibrium (a) and precipitation phases under 347H equilibrium phase diagram (b)

temperatures in equilibrium is shown in Table 2.

From Table 2, it can be seen that at 1300 °C, the Cr element content in the α phase is 21.6wt%, the Ni element content is 6.34wt%, the Cr element content in the γ phase is 17.43wt%, and the Ni element content is 9.46wt%. Nb (C, N) mainly contains Nb, C and N elements, with a Nb content of 88.09wt%, C element content of 6.83wt% and N element content of 4.45wt%. At 700 °C, compared with 1300 °C, the Cr element content decreases to 17.19wt%, the Ni element content in the γ phase continuously increases to 12.77wt%, and the element composition of Nb (C, N) shows an increase in C element content and a decrease in N element content. Laves phase mainly consists of Cr and Mo elements, $M_{23}C_6$ mainly contains Cr, Mo and C elements, with a Cr content of up to 68.45wt%. M₅SiN mainly contains Cr, Ni and Si elements. The appearance of Mo element in the 347H precipitation phase is mainly due to the presence of a small

amount of Mo element when adding the alloy. Ref. [6-8] studied the precipitation of phases at different aging temperatures and durations, and found that NbC, σ and Fe₂Nb phases appeared after long-term aging at 700 °C.

Fig.2 shows the metallographic structure of 347H at 700 °C for different durations. It can be seen that it is a typical austenitic structure, and precipitates are suspected to be NbC with agglomeration phenomenon. After long-term aging at 700 °C, the metallographic structure of 347H plate slightly increases with prolonging the aging time, and a large number of precipitates appear at the grain boundaries. With the increase in aging time, the precipitates increase and some become coarse. After long-term aging at 700 °C, the metallographic structure of the plate slightly increases with prolonging the aging time. After aging at 700 °C, most precipitates appear at the grain boundaries of 347H sheet, and a lot of precipitate detachment marks appear at the metallographic corrosion state grain boundaries^[18–20].

Fig.3 shows SEM images at 700 °C for different aging time. Many precipitates appear on the 347H plate after long-term aging at 700 °C. With prolonging the aging time, the precipitates increase and some become coarse. There are two types of precipitates at the grain boundary, one is coarse and easily corroded, and the other is relatively small. As shown in Fig.4, there are more 347H precipitates at the grain boundary.

Fig. 5 shows the EDS line scanning results of the precipitation phase. Both coarse and fine precipitates appear at the grain boundaries, in which coarse precipitates are easily corroded, resulting in many precipitation detachment marks at the grain boundaries. These coarse precipitation phases are rich in Cr, and fine precipitates are rich in Nb and C. Based on the phase composition at different temperatures in Table 2, the coarse particles at 700 °C have higher Cr content and no significant increase in Ni content, indicating the presence of Nb, which may be Laves phase. The fine particles have higher C and Nb content, and the aging temperature is within the temperature range in which chromium, iron and other carbides are completely dissolved (400-825 °C), which is lower than the temperature at which niobium carbide is completely dissolved $(750-1120 °C)^{[21]}$. Therefore, fine particles are NbC.

2.2 Effect of aging on mechanical properties

To investigate the effect of aging on the properties of 347H,

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Temperature/°C	Phase	С	Cr	Cu	Fe	Mn	Мо	Ν	Nb	Ni	Si
	α	0.02	21.60	0.28	69.04	1.15	0.62	0.01	0.52	6.34	0.42
1300	γ	0.04	17.43	0.54	69.97	1.33	0.36	0.02	0.32	9.46	0.52
	Nb(C, N)	6.83	0.52	0	0.03	0	0.09	4.45	88.09	0	0
	α	0	17.19	0.10	78.39	0.37	0.24	0	0	3.46	0.23
	γ	0	13.45	0.80	70.45	1.68	0.21	0	0	12.77	0.64
700	Nb(C, N)	8.64	0.52	0	0	0	0	2.47	88.38	0	0
/00	Laves	0	17.32	0.10	35.75	0.03	43.73	0	2.57	0.50	0
	M_5 SiN	0	45.31	0	1.21	0	5.42	4.33	0	35.04	8.69
	$M_{23}C_{6}$	5.17	68.45	0	7.64	0.05	18.49	0	0	0.20	0

Table 2 Phase composition at different temperatures (wt%)



Fig.2 Metallographic structures of 347H at 700 °C for different durations: (a) original steel plate, (b) 400 h, (c) 1000 h, and (d) 3000 h



Fig.3 SEM images of samples aged at 700 °C for different durations: (a) original steel plate, (b) 400 h, (c) 1000 h, and (d) 3000 h



Fig.4 Morphologies of precipitated phase: (a) 1000-fold and (b) 5000-fold



Fig.5 Microstructure of precipitates (a); EDS line scanning results of coarse precipitates along line A in Fig.5a (b) and fine precipitates along line B in Fig.5a (c)

mechanical testing was conducted at 700 °C for 30 min. Fig.6a and Table 3 show the mechanical properties at room temperature. Compared with the original samples, the yield strength $(R_{p0,2})$ and tensile strength (R_m) of the aged sample increase, while the elongation decreases. This is mainly due to the dispersed distribution of small precipitates of 347H in the matrix, which hinders the slip and climb of dislocations and requires greater external force during deformation, thereby improving strength and hardness, while reducing elongation. Fig. 6b and Table 3 show a comparative analysis of high temperature performance at 593 °C. At 593 °C, compared to the pinning effect of precipitates, temperature plays a dominant role in the performance. The higher the temperature, the lower the activation energy of dislocation slip. A lower activation energy of dislocations means that dislocations are more likely to move and the material is more prone to plastic deformation, resulting in a higher elongation^[22-24]. The yield strength and tensile strength of the aged sample increase, while the elongation decreases, indicating that the precipitated

Table 3 Mechanical properties of 347H stainless steel

Temperature	Sample	R _m /MPa	R _{p0.2} /MPa	Elongation/%
Room	Aged	615.6	224.0	63.1
temperature	Original	604.0	213.6	66.2
502.00	Aged	397.3	144.9	37.0
593 °C	Original	374.5	131.1	41.4



Fig.6 Mechanical properties at room temperature (a) and 593 °C (b)

phase also has an impact on the high temperature performance at the same temperature.

Fig. 7 shows the fracture morphology of the aged sample and the original tensile sample. Both tensile fracture morphologies exhibit a large number of ductile dimples, which is a typical plastic fracture^[25-27]. The toughness of the



Fig.7 Tensile fracture morphologies of aged sample (a) and original tensile sample (b)

original sample is smoother than that of the aged sample, mainly due to the presence of many precipitated phases such as NbC and Laves in the aged sample. The interface between different phases may affect the coordination of plastic deformation, thereby affecting the surface morphology of the toughness. The elongation of the original sample at room temperature is about 4% higher than that of the aged sample, and the plasticity of the original sample is better than that of the aged sample.

2.3 Effect of aging on corrosion behavior

Three main aspects that affect molten salt corrosion are the microstructure of stainless steel, impurities in molten salt and chemical activity. The effect of aging on grain size is not significant, but it increases the carbides and nitrides of niobium, preventing the precipitation of Cr element in stainless steel. Fig.8 shows the corrosion morphology of aged samples and original samples in molten salt at 565 °C for 240 and 360 h.

The XPS spectra of the aged sample are shown in Fig.9a. It can be seen that element Fe has strong characteristic peaks, and the main product of corrosion is Fe oxide. Matrix elements such as Mn, Cr and Si also participate in the corrosion process, forming corresponding oxides. In addition, some impurity elements in molten salts, such as element Mg, also exist in the form of oxides, contributing to the corrosion resistance of alloys. However, due to their low content, their effectiveness is limited. According to the XPS manual, peak fitting is performed on each element to obtain fine spectra of Fe 2p, O 3d, Cr 2p and Mg 1s. In the spectra of Fe 2p, there are three peak positions for Fe^{3+} , Fe_3O_4 and Fe_2O_3 . The position with a binding energy of 713.2 eV $(2p_{3/2})$ corresponds to Fe^{3+} , the position with a binding energy of 711.5 eV (2p_{1/2}) corresponds to Fe₂O₃, and the position with a binding energy of 709.2 eV (2p_{3/2}) corresponds to Fe₃O₄. The spectra of O 1s

exhibit two peak positions with binding energies of 531.3 and 529.1 eV, corresponding to OH⁻ and O²⁻, respectively. In the spectrum of Mg 1s, 1303.5 eV is the characteristic peak of element Mg, and 1303.9 eV is the characteristic peak of Mg²⁺. The characteristic peak intensity of Fe₃O₄ in aged sample is relatively high, and it has a more effective corrosion product layer that resists molten salt corrosion.

The XPS peak fitting of original samples in Fig.9b shows that the corrosion products are the same as that of aged samples, but the characteristic peak of Fe_3O_4 is higher than that of aged samples, and it also has a higher intensity of Mg^{2+} characteristic peak, which can prove the precipitation of spinel with more Mg. The characteristic peak of strong Na indicates that the corrosion layer contains sodium ferrite structure. The original sample has better resistance to molten salt corrosion than the aged sample.

Fig. 10 shows the XRD patterns of the original sample and the aged sample of 347H stainless steel after corrosion in nitrate. The corrosion products of the two samples are hematite (Fe₂O₃), magnetite (Fe₃O₄), some spinel structures (MgCr₂O₄, MgFe₂O₄ and FeCr₂O₄) and sodium ferrite (NaFeO₂). This shows that the material has a chemical reaction with the high temperature nitric acid molten salt, and the formation process of each corrosion product is passed.

Corrosion of metals in contact with molten salts of nitric acid is mainly caused by oxygen, which is due to the decomposition of nitrates into oxygen and nitrogen oxides by Eq. (1) at high temperatures^[28-29]:

$$NO_3^{-}+2e^{-}\leftrightarrow NO_2^{-}+O^{2-}$$
(1)

This reaction will cause the iron element in the stainless steel matrix to be oxidized, and in general, it is gradually oxidized from the low valence state of iron to the high valence state. However, since the formation temperature of FeO is greater than 570 $^{\circ}$ C, it is only possible to generate magnetite



Fig.8 Morphologies of aged samples (a–b) and original samples (c–d) after molten salt corrosion at 565 °C for different durations: (a, c) 240 h and (b, d) 360 h



Fig.9 XPS spectra of aged sample (a) and original sample(b)

 (Fe_3O_4) and hematite (Fe_2O_3) in the molten salt at 565 °C, as shown in Eq.(2–4)^[30]:

$$Fe+O^{2-} \leftrightarrow FeO+2e^{-}$$
 (2)

 $3FeO+O^{2-} \leftrightarrow Fe_{3}O_{4}+2e^{-}$ (3)

$$2Fe_{3}O_{4}+O^{2} \leftrightarrow 3Fe_{2}O_{3}+2e^{-1}$$
(4)

Impurities in commercial grade nitrates are considered to be important activators of molten salt corrosion behavior^[31]. The commonest impurities are magnesium, perchlorate, carbonate and sulfate. The evaluation results of the Solar Two plant firstly pointed out that magnesium nitrate is thermally decomposed to form gaseous nitrous oxide by-products, and MgO is formed above 480 °C, as shown in Eq.(5)^[32]:

$$Mg(NO_3)_2 \leftrightarrow MgO + 2NO_2 + \frac{1}{2}O_2$$
(5)

The MgO formed by the reaction further interacts with iron to form magnesium ferrite. Element Mg replaces element Fe in Fe₂O₃ to form a very stable compound MgFe₂O₄, as shown in Eq.(6)^[33]:

$$MgO+Fe_2O_3 \leftrightarrow MgFe_2O_4 \tag{6}$$

Sodium ions in the molten salt easily penetrate into the spinel structure and form NaFeO₂ by replacing hematite (Fe₂O₃), as shown in Eq. $(7-8)^{[34]}$:

$$2Na^{+}+O^{2} \leftrightarrow Na_{2}O \tag{7}$$

 $Na_2O + Fe_2O_3 \rightarrow 2NaFeO_2$ (8)

In high temperature mixed nitrates, the Cr element on the



Fig. 10 XRD patterns of corrosion products of 347H stainless steel after corrosion in molten nitrate salt at 565 °C for 360 h

surface of the stainless steel substrate is firstly oxidized to Cr_2O_3 (Eq.(9)), and then reacts with the oxide of iron to form ferrochrome spinel (FeCr₂O₄), as shown in Eq.(10)^[35]:

$$\operatorname{Cr}+3\operatorname{O}^2 \longrightarrow \operatorname{Cr}_2\operatorname{O}_3 + 6e^-$$
 (9)

$$Cr_2O_3 + FeO \rightarrow FeCr_2O_4$$
 (10)

The presence of MgFe₂O₄ is found in the XRD pattern, which is because the impurities Mg²⁺ and O²⁻ in the molten salt enter the Fe₂O₃ layer, as shown in Eq.(11). At the same time, Mg²⁺ and O²⁻ penetrate into the inner layer and react with Cr₂O₃. The spinel structure of MgCr₂O₄^[36-37] form, as shown in Eq.(12):

$$Fe_2O_3 + Mg^{2+} + O^2 \rightarrow MgFe_2O_4$$
 (11)

$$Cr_2O_3 + Mg^{2+} + O^2 \rightarrow MgCr_2O_4$$
(12)

The corrosion products of spinel structure can help to improve the corrosion resistance. For example, FeCr₂O₄ has a certain density and adheres to the surface of the substrate, which hinders the direct contact between the impurities and the substrate and thus protects the substrate to a certain extent, but it allows element Fe to diffuse outward and O2- to transport inward^[38-39]. MgCr₂O₄ can reduce the activity of chromium, thereby reducing the diffusion and loss of chromium^[40]. It is considered that NaFeO₂ has no effect on protecting the matrix^[41]. The iron oxides in the corrosion products are Fe_3O_4 and $Fe_2O_3^{[7,42]}$, in which Fe_3O_4 has a dense structure and is closely bonded to the inner layer, thus effectively protecting the matrix, while Fe₂O₃ is loose and porous, which cannot effectively prevent the diffusion of free oxygen in the molten salt to the matrix, so the corrosion resistance to molten salt is poor^[43-46]. In the XRD pattern, the diffraction peak intensity of Fe₃O₄ in the corrosion product of the original sample is higher than that of the aged sample, which indicates that there are more Fe₂O₄ in the corrosion layer of the original sample. Therefore, the original sample shows better corrosion resistance than the aged sample.

In addition, EDS point scan analysis was conducted on the corrosion products of the original sample and the aged sample. The results are shown in Fig.11 and Table 4. The main elements of points 1 and 2 are O, Fe, Cr, Mg and Na, among which, the contents of Cr, Mg and Na elements are close. The ratio of O to Fe at point 1 is closer to 1.5: 1, indicating



Fig. 11 SEM morphologies of 347H stainless steel after corrosion: (a) aged sample and (b) original sample

Table 4 EDS spot scanning results of corrosion products in Fig.11 (at%)

Point	0	Fe	Cr	Mg	Na	Bal.
1	50.5	31.8	3.9	5.1	3.7	5.0
2	49.1	35.7	3.7	5.8	2.4	3.3

 Fe_2O_3 , while the ratio of O to Fe at point 2 is closer to 4:3, indicating the Fe_3O_4 . By analyzing the ratio of O to Fe, it can be seen that Fe_3O_4 accounts for a large proportion in the corrosion products of the original sample, and dense Fe_3O_4 with more content can better protect the matrix. A small amount of corrosion layer shedding is observed in Fig. 11a, which is because Fe_2O_3 , which is loose and porous and not tightly bonded to the matrix, falls off at high temperature. Whereas the original sample containing more Fe_3O_4 has no obvious shedding.

Fig. 12 shows the corrosion kinetics curves of molten salt. It can be found that the corrosion rate shows a trend of first increasing and then decreasing. The aged sample has a high resistance to molten salt corrosion, mainly because the corrosion products of Fe_3O_4 in the original sample are higher than that in the aged sample, and the diffusion rate of oxygen atoms slows down, resulting in better resistance to molten salt corrosion of original sample than that of the aged sample.

2.4 Welding properties

During the on-site manufacturing process of hightemperature molten salt storage tanks, manual welding is required to weld the steel plates. In order to study the influence of the base material on the welding performance, the laboratory simulated shielded metal arc welding (SMAW) was conducted, and E347H was used as the filler metal electrode during welding. The base material thickness is 8 mm, and no



Fig.12 Corrosion kinetics curves of 347H samples

heat treatment is performed after welding. The welding process parameters of SMAW method (double sided welding and forming; Φ 4.0 mm welding rod E347H) are shown in Table 5.

The metallographic structure of the welded joint between the aged template and the original template is observed. Fig. 13a-13b show the metallographic structure of the weld metal (WM) and heat affected zone (HAZ) of the aged sample after SMAW. Fig. 13c-13d show the WM and HAZ of the original SMAW sample. It can be seen that WMs of the aged sample and the original sample are typical skeletal high temperature ferrite and austenite structure. The morphology of the WM is mainly determined by the composition of the filler metal. The presence of high temperature ferrite can effectively avoid solidification cracks and ensure good welding performance of the material. The grain size of HAZ structure decreases sequentially from the fusion line to the base metal^[47].

The sensitivity to intergranular corrosion of the welded joints between the aged sample and the original sample was tested, as shown in Table 6. Both the aging template and the original template show severe grain boundary corrosion when evaluating intergranular corrosion sensitivity.

Fig. 14 shows the intergranular corrosion cracks observed after testing the intergranular corrosion sensitivity of aged and original samples. It can be seen that there is obvious knife-shaped corrosion in the area close to the fusion line, which is mainly due to the complete dissolution of carbides in the high temperature HAZ close to the melt pool. During cooling, chromium-rich carbides precipitate faster than NbC, resulting in a chromium-deficient sensitization zone near the fusion line and severe intergranular corrosion.

Table 5 SMAW process parameters

Parameter	Current/A	Voltage/V	Time/s	Board length/mm	Heat input/kJ·mm ⁻¹
Bottom	140	30	90	500	0.756
Filling	150	30	110	500	0.990
Front cover	150	30	120	500	1.080
Reverse cover	150	30	110	500	0.990



Fig.13 WM (a, c) and HAZ (b, d) of aged sample (a-b) and original sample (c-d)

Table 6 Intergranular corrosion rate of SMAW samples

Sample	Corrosion time/h	Corrosion rate/g \cdot m ⁻² ·h ⁻¹
Aged	120	97.13
Original	120	97.15



Fig.14 Intergranular corrosion morphologies of welding samples: (a) aged sample and (b) original sample

3 Conclusions

1) A large number of precipitated phases appear after longterm aging at 700 $^{\circ}$ C. With prolonging the aging time, the number and size of precipitated phases increase. The coarse precipitated phase is rich in Cr element, and the relatively fine precipitated phase is NbC.

2) After aging at 700 $^{\circ}$ C for 30 min, the yield strength and tensile strength of the samples at room temperature and 593 $^{\circ}$ C increase, but the elongation decreases.

3) The corrosion products of the original samples in nitrate are the same, but there are denser and more uniform Fe_3O_4 in the corrosion layer of the original samples, so it shows better corrosion resistance than the aged samples.

4) The effect of aging treatment on the microstructure and intergranular corrosion resistance of welded joints is not significant.

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时效对含铌不锈钢性能及焊后组织的影响

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摘 要:研究了不同时效工艺对347H不锈钢析出相、力学性能、耐熔盐腐蚀性能和焊后组织的影响。结果表明,700℃时效400h后, 晶体内部会出现大量析出相;时效3000h后,析出相数量增多,并大量聚集在晶界处;析出物有2种形态,其中粗大的析出物富含Cr, 而较小的析出物以NbC为主。700℃时效30min后,试样在室温和593℃时的屈服强度以及抗拉伸强度均升高,但延伸率降低。在 565℃硝酸盐中的腐蚀结果表明,老化试样与原始试样的腐蚀产物相同,均为Fe₂O₃、Fe₃O₄、MgCr₂O₄、MgFe₂O₄、FeCr₂O₄和NaFeO₂。 原始试样中致密均匀、与基体结合较好的Fe₃O₄的比例高于时效试样,因此耐腐蚀性较好。在700℃时,时效时间对焊后组织无明显 影响。

关键词:时效; 347H不锈钢; 析出相; 力学性能; 熔盐腐蚀; 焊接性能

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