

Effect of Precipitation on Intergranular Corrosion Resistance of 430 Ferritic Stainless Steel

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Abstract: With Nb-Ti-stabilized 430 ferritic stainless steel (NTS430FSS) and SUS 430 ferritic stainless steel (SUS430FSS) as experimental materials, the influence of precipitation on intergranular corrosion resistance was investigated. A series of aging treatment were carried out. The free-exposure corrosion test and double loop electrochemical potentiokinetic reactivation (DL-EPR) test with a scan rate of 1.67 mV/s at 26 °C were applied to evaluate the intergranular corrosion (IGC) resistance. Metallographic observation, scanning electron microscope (SEM), transmission electron microscope (TEM) with energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analysis were conducted. The results show that IGC occurred in SUS430FSS aged above 700 °C, while it occurred in NTS430FSS as the temperature was improved to 1 050 °C. The critical degree of sensitization I_7/I_8 reaches 0.305 in SUS430FSS, which is higher than that of NTS430FSS, i.e. 0.010, aged at 950 °C for 2 h. The TEM, EDS and XRD results show that a large amount of $Cr_{23}C_6$ precipitates with size of 60 nm×22 nm are located at the SUS430FSS grain boundaries as chains. With the addition of Nb and Ti and reduction of C, the amount of precipitates reduces significantly in NTS430FSS. A majority of $Cr_{23}C_6$ were replaced by TiC and NbC. Only a small amount of spherical TiC ($R=186$ nm) and square TiN (312 nm×192 nm) with Nb and Cr adsorbed are left along grain boundaries. Due to the dual stabilization of Nb and Ti, the precipitation of $Cr_{23}C_6$ is restrained, the chromium depleted region is avoided and accordingly the resistance to the intergranular corrosion is improved.

Key words: 430 ferritic stainless steel; intergranular corrosion; double-loop electrochemical potentiokinetic reactivation; carbonitride extraction; precipitation

In recent years, the ferritic stainless steel (FSS) attracts more and more attention due to price rising of austenitic stainless steel. As reported by Chen^[1], FSS has been applied to high temperature fields, including automotive exhaust systems. Intergranular corrosion (IGC) caused by aging treatments occurs in the process of service. According to the research^[2], the maximum operating temperature of the exhaust manifolds can be over 600 °C.

Researches showed that the IGC mechanism of FSS was similar to that of austenitic stainless steel^[3-5]. The existed mechanism of IGC is that chromium compounds such as carbide and nitride precipitates separate out at the grain boundaries at first, leading to the chromium depleted zone, and finally IGC is fully developed by the electrochemical potential difference between the chromium depleted zone and the matrix^[6-9].

According to the study of Farahat^[10], Ti and Nb were used in the austenitic stainless steel to improve mechanical properties. The effects of Nb and Ti on ferritic stainless steel was investigated by Qu et al.^[11]. To prevent the precipitation of chromium compound at the matrix and grain boundaries, the amount of interstitial elements, C

and N, should be reduced as lower as possible. Stabilizing elements such as Ti and Nb can also be added to react preferentially with C and N rather than chromium. Ti and Nb can form a variety of precipitates, such as Ti(C,N), Nb(C,N), Fe_2Nb and Fe_3Nb_2C .

In present paper, in order to obtain a new type of 430 stainless steel with excellent corrosion resistance and explore the corresponding mechanism, Nb-Ti-stabilized 430 ferritic stainless steel with 0.075 9 mass% Nb-0.112 mass% Ti and SUS 430 ferritic stainless steel were investigated.

1 Experimental Materials and Method

1.1 Tested materials

The studied steel was smelted in a high frequency induction vacuum furnace and then forged to slabs. The slabs were hot-rolled at 900–1 200 °C, and then cold-rolled to sheets with thickness of 2 mm. NTS430FSS and SUS430FSS were annealed at 950 °C and 830 °C for 90 s, respectively. The chemical compositions of the materials are listed in Table 1.

Table 1 Chemical composition of tested materials

Material	C	Mn	Cr	Ni	Nb	Ti	Fe	mass%
SUS430FSS	0.071	0.198	16.2	0.235	—	—	Balance	
NTS430FSS	0.040	0.218	16.9	0.224	0.076	0.112	Balance	

1.2 Corrosion tests

The free-exposure corrosion test proposed by ASTM A763 (American standard) was performed in copper sulfate solution. The aging treatment varied from 500 °C to 1 300 °C. The samples (10 mm×10 mm×2 mm) with the surface ground to the 2 000 grit by silicon carbide paper were contacted electrically with copper balls and immersed in boiling solution of (16 mass%) H₂SO₄ + (6 mass%) CuSO₄ for 24 h. The microscopic observation was conducted for IGC.

Based on the actual service conditions, the aging treatment of samples used in DL-EPR test is listed in Table 2. The samples (10 mm×10 mm×2 mm) were polished up to 2 000 grit by silicon carbide paper as well. The modified concentration of electrolyte was (0.14 mol/L) H₂SO₄ + (0.002 5 mol/L) KSCN. The Hg-HgSO₄ electrode and platinum electrode were served as reference and auxiliary electrodes, respectively. The specimen was anodically polarized from the corrosion potential (−1 000 mV) to 200 mV with scan rate of 1.67 mV/s at 26 °C. Then the polarization was reversed at the same rate. The test finished at the corrosion potential measured initially. The maximum current density in the anodic (I_a) and reverse (I_r) curves were measured respectively. The degree of sensitization was estimated by the value of I_r/I_a [12–15].

Table 2 Conditions and serial number of the samples used in DL-EPR tests

Serial number	Material	Heat treatment
11	SUS430FSS	Sensitized at 650 °C for 2 h
12	NTS430FSS	Sensitized at 650 °C for 2 h
13	SUS430FSS	Sensitized at 950 °C for 2 h
14	NTS430FSS	Sensitized at 950 °C for 2 h

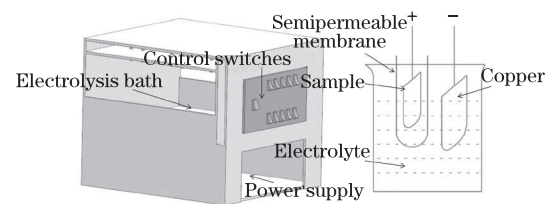
A carbonitride extraction device was invented to analyze the ingredient and amount of precipitates, as shown in Fig. 1. The efficiency and accuracy were improved. The electrolyte was (125 mL) HCl + (16 g) C₆H₈O₇ + (375 mL) H₂O and the current density was 0.02 A/cm². The powder obtained by the device was analyzed by XRD (18KW D/MAX2500V+/PC).

2 Results and Discussion

2.1 Free-exposure corrosion tests

In order to probe into the sensitization zone of IGC, free-exposure corrosion tests were conducted. The results

are shown in Fig. 2. There is no evidence of IGC in NTS-430FSS aged below 1 000 °C, while the grain boundaries appeared gradually when aged at 1 050 °C. The phenomena of IGC is obviously observed above 1 100 °C. In SUS430FSS, grain boundaries begin to emerge when aged above 700 °C. Fig. 2 indicates that improving aging temperature could not avoid IGC.

**Fig. 1** Schematic of the electrolytic extraction device

Compared with NTS430FSS, the surface of SUS-430FSS is etched seriously after free-exposure corrosion test. Copper particles can be observed on the surface of SUS430FSS, which implies a severe corrosion reaction.

Fig. 3 presents the time-temperature-sensitization (TTS) curves of NTS430FSS and SUS430FSS plotted according to the free-exposure corrosion test results. These curves indicate that the sensitive zone is raised by the addition of Nb, Ti and the decrease of C. However, IGC could not be prevented completely.

Fig. 4 indicates the microstructure morphology of specimens after free-exposure corrosion test. The black areas, where serious corrosion took place in Fig. 2, are the expansion of grain boundaries. Fine precipitates are distributed in the matrix of NTS430FSS rather than aggregated at the grain boundaries, as indicated by A in Fig. 4(a). The grain boundaries of NTS430FSS are corroded, as indicated by B in Fig. 4(a). Precipitates located in those areas are identified as a compound of Ti, Nb, Fe, Cr and C (TiC). Severe intergranular corrosion takes place in SUS430FSS. The EDS results of the separated island (as indicated by C in Fig. 4(b)) reveal that the level of Cr and C is very high.

2.2 DL-EPR tests

The DL-EPR test was used to evaluate the degrees of sensitization. The results are presented in Fig. 5. I_a , I_r and R_a ($R_a = I_r/I_a$) that reflect the degree of sensitization are listed in Table 3. The values of I_a are nearly the same for all the specimens, while the values of I_r are different from each other. The corresponding R_a values of I1, I2, I3 and I4 are $I2 < I4 < I1 < I3$ and are well reflected by OM micrographs, as shown in Fig. 6. There are deep grooves

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