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Electrochemical corrosion resistance of CeO_2-Cr/Ti coatings on 304 stainless steel via pack cementation

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Abstract: The pack cementation was employed to improve the electrochemical corrosion resistance of 304 stainless steel via CeO_2-Cr modified Ti coatings. Continuous coatings were formed on 304 stainless steel surface by this method. A series of electrochemical experiments were carried out to investigate the corrosion resistance of 304 stainless steel, Ti coating and CeO_2-Cr/Ti coatings. The sample surface was investigated by scanning electron microscopy (SEM). The phases of sample surface were detected by X-ray diffraction (XRD). It was concluded from all the outcomes that the corrosion resistance of the samples could be sorted in the following sequence: CeO_2-Cr/Ti coatings>Ti coating>304 stainless steel.

Keywords: electrochemical corrosion resistance; rare earths; Ti coating; 304 stainless steel; pack cementation

Austenitic stainless steel is one of the most widely applied ferrous materials for medical and nuclear industry for its excellent corrosion resistance, mechanical properties and ductility[1]. Among which, 304 stainless steel has been frequently utilized in the industry due to its excellent resistance of corrosion and reasonable mechanical properties[2]. Nevertheless, the bare matrix affects the resistance when exposed to salt solutions. Therefore, 304 stainless steel is limited in electrochemical corrosion resistance in many fields[3–7]. According to the recent research about corrosion of the ferrous materials, a series of corrosion protective methods, such as injecting inhibitor, cathode protection, and surface treatment have been proposed[8]. In Soltani and Tavakkoli's research, the silybum marianum was put into use as corrosion inhibitors of 304 stainless steel in 1.0 mol/L HCl solution. Good results were obtained[9]. Wu et al. prepared TiO_2 sol by peracetic titanate sol-gel method, and TiO_2 thin film was prepared on 304 stainless steel by dip-ping-pulling method. The results show that the TiO_2 film had better mechanical corrosion resistance property[10]. A method of double-glow plasma alloying technique to form a Mo alloying layer on 304 stainless steel also brought satisfactory results[11].

The pack cementation is a coating-deposition process with an *in situ* chemical vapor deposition (CVD) process by halide salts such as NH_4Cl, NaF[12–14]. It is normally used in surface modification for its low cost, simple process and ability to be applied to complex shapes[15]. Lin et al.[16,17] obtained a high-performance surface on P110 steel via pack cementation. The results indicated that the coatings were beneficial in terms of valid thickness and microhardness. Zheng et al.[18] fabricated silicon film on Nb_4AlC_3 to improve the oxidation resistance. As a result, the Si pack cemented Nb_4AlC_3 shows excellent oxidation resistance up to 1200 ºC. Pack cementation could be used for corrosion protection of 304 stainless steel in salt solution.

The decrease in the corrosion resistance is caused by heavy precipitations on the grain boundaries. The common method to prevent the material from intergranular corrosion is to add a stabilizer such as Ti and Nb[19]. In addition, rare earths (RE) such as La, Ce, Y, or their oxides have been reported to be effective in thickening the coatings[20]. So in this work, the coating of CeO_2-Cr was added into the Ti coating to prepared CeO_2-Cr/Ti coatings by means of pack cementation. The microstructure and the corrosion properties of such coatings were thoroughly investigated.

1 Experimental

1.1 Materials

For pack cementation, the substrates were cut with the dimension of Φ16 mm×5 mm plates by an electro-spark wire-electrode cutting machine. The chemical compositions of the 304 stainless steel are listed in Table 1. The specimen surface was ground with waterproof abrasive paper down to 1500#, polished using diamond paste down to 5000#, and then ultrasonically cleaned in acetone.

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Table 1 Chemical compositions of 304 stainless steel

Elements	C	Mn	P	S	N	Si	Cr	Ni	Fe
wt.%	0.08	2.00	0.035	0.03	1.0	1.00	18.00	8.00	Bal.

1.2 Preparation of CeO_2-Cr/Ti coatings

In the first step, the pack powder mixture was prepared with 49 wt.% titanium sponge (99.7%) which acts as Ti source, 50 wt.% Al_2O_3 (hard-burned) as inert filler, and 1 wt.% I_2 (99.8%) as activator. The powders were mixed sufficiently by ball-milling for 1 h using ZrO_2 balls. The substrate was buried in the powder in a stainless steel crucible. To prevent the mixtures from the oxygen, glass powder was placed above the mixtures. The schematic diagram is shown in Fig. 1. The crucible was put into the electric furnace, which was heated to 150 ºC and held for 0.5 h to remove the moisture. Ti pack cementation process was conducted under a temperature of 900 ºC for 6 h, and the heating rate was 10 ºC/min. Then the crucible was taken out and cooled down to room temperature in air. After Ti pack cementation, the specimen was polished and cleaned for the next step—CeO_2-Cr/Ti coatings. The specimen was buried in another pack powder mixture, consisting of 45 wt.% Al_2O_3 (hard-burned), 3 wt.% NH_4Cl as activator, 7 wt.% CeO_2 and 45 wt.% CrN. Adding CrN is feasible for the reason that unstable nitride like CrN and Cu_3N will resolve when suffers a heat treating, thereby, it will make the specimen and mixture exposed to a nitrogenous atmosphere at a certain pressure[21]. After the same treatment in temperature and experimental period like step 1, it had been considered more necessary to ultrasonically clean the specimen in acetone. An X-ray diffraction (XRD) with the 0.154 nm Cu Kα line as the excitation source was employed to examine the crystal structure of the coatings. The surface morphologies and compositions of samples were observed using scanning electron microscopy (SEM, QUANTA 250FEG).

1.3 Electrochemical corrosion resistance tests

Electrochemical corrosion resistance measurements were carried out on pack cementation coatings using electrochemical tests including open circuit potential measurements, cyclic potential dynamic polarization and impedance curve in 3.5 wt.% NaCl, which was conducted with electrochemical measurement system (CHI600E). The open circuit potential test was measured for 1800 s. Subsequently, the polarization curves were obtained with a scan rate of 1 mV/s in the range from –1 to 1 V. In order to obtain further information on the characteristics of the coatings, electrochemical impedance spectrum (EIS) tests were performed. Before the electrochemical experiments, it was necessary to pack the sample with 703 Silicone rubber with a copper interconnect stretching out. An exposed area of 1 cm^2 was remained to contact the solution. Then the specimens of bare matrix and coatings were measured as a working electrode (WE), with the Pt acting as the counter electrode and the saturated calomel electrode (SCE) as the reference electrode.

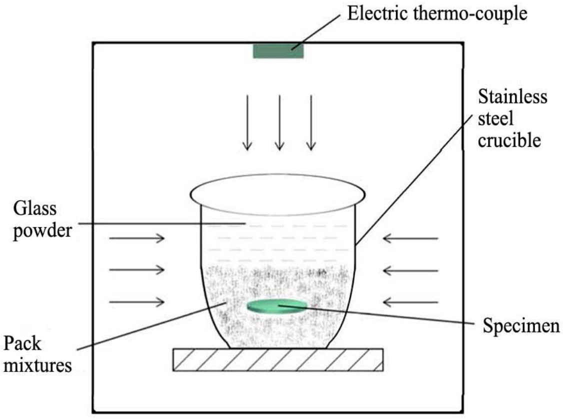

Fig. 1 Schematic diagram of pack cementation device

2 Results and discussion

2.1 Microstructural characterization

To investigate the thickness of the deposition coatings, the samples were cut by a wire-electrode cutting machine along the cross section. The specimens were etched in hydrochloric acid solution with ferric chloride for the observation.

Fig. 2 shows surface SEM micrographs of the substrate and deposited coatings after pack cementation. As demonstrated in Fig. 2(b), a compact and continuous Ti coating was fabricated on the surface of 304 stainless steel, which is shown in Fig. 2(a). After CeO_2 modified Cr treatment, in Fig. 2(c), the surface was covered with reticulate structure. Unfortunately, some cracks distribute through the settled layer resulting from the pack cementation temperature or the ratio of the reagents which affected the process of pack cementation. In Fig. 3(a), SEM cross-sectional micrographs of the coatings are presented, apparently, a smooth and continuous Ti coating was obtained at the experimental process and a second CeO_2 modified Cr coating was fabricated on the Ti coating. Fig. 3(b) depicts the line scanning results of Cr, Ce, Ti, Fe and Ni over the coatings by EDS along the white arrow in Fig. 3(a). It can be noticed that Ti coating and CeO_2 modified Cr coating verified by the composition profile. Cr and Ce distribute across 0 to 10 μm from the surface, then decrease in gradient. Ti distributes across 40 to 90 μm and reaches the highest intensity at 80 μm from the surface. There is a diffusion region between CeO_2 modified Cr coating and Ti coating from 10 to 40 μm from the surface. In this transition layer, to Cr, Ti and Fe, several waves can be observed, which illustrate that some stable compounds are formed. The compounds can significantly improve bonding strength of CeO_2 modified Cr coating and Ti coating. From 90 to 130 μm, the dis-

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