

Ionic bombardment of stainless steel by nitrogen and nickel ions immersion

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Abstract: A new nitriding process was used to carry out the ionic bombardment, in which nickel ion was introduced. The microstructure, composition and properties of the treated stainless steel were studied by means of scanning electron microscopy(SEM), micro-hardness test and electrochemistry method. The results show that the hardness of the stainless steel is greatly increased after ionic bombardment under nitrogen and nickel ions immersion. Vickers' hardness as high as Hv1268 is obtained. The bombarded stainless steel is of a little reduction in corrosion resistance, as compared with the original stainless steel. However, as compared with the traditional ion-nitriding stainless steel, the corrosion resistance is greatly improved.

Key words: stainless steel; nitriding; ionic bombardment; corrosion resistance

1 Introduction

Stainless steel is widely used in various fields such as petroleum, chemical, textile, nuclear energy and aviation industries due to its excellent corrosion resistance[1]. However, its low hardness and poor wear resistance have limited its application in many other areas. Plasma nitriding is the most successfully and widely used method to improve the wear resistance[2–4]. In order to accelerate the diffusion of nitrogen into the stainless steel matrix, the nitriding process is normally carried out at a temperature higher than 500 °C, which causes the precipitation of chromium nitrides. The precipitation of chromium nitrides leads to the depletion of chromium content in the stainless steel and a significant reduction in the corrosion resistance of the nitrided layer[5–6].

Under some working conditions, good corrosion and wear resistant properties are required for stainless steel. Attempts have been made to improve the corrosion resistance of nitrided stainless steel. In Refs.[7–9], it is reported that the corrosion and wear resistance of stainless steel could be achieved by low temperature

plasma nitriding to produce a single *S* phase layer which has high hardness and good corrosion resistance. However, the nitrided layer at low temperature of about 450 °C was thinner than that at high temperature of above 500 °C. Furthermore, it is difficult to maintain a uniform furnace temperature distribution when the nitriding temperature is low[10–14]. Recently, an active screen plasma nitriding technology has been developed to solve the problem. Yet, the process time needs to be extended and the efficiency is low[15–21].

In the present work, a new nitriding process was reported, in which nickel ion was introduced. The effects of the nickel ion immersion on the composition and structure of the stainless steel by ionic bombardment were studied. The phase content and the hardness of the bombarded samples were analyzed.

2 Experimental

The stainless steel used in the present work is 17-4PH, with chemical compositions (in mass fraction) of 0.05%C-16.50%Cr-4.25%Ni-3.30%Cu-0.60%Mn-0.35%Nb-Fe. The specimens with a dimension of 10 mm × 10 mm × 10 mm were mechanically polished with

abrasive papers from 180 grit to 900 grit. They were subsequently cleaned in soapy water and then acetone.

Ionic bombardment process was carried out in a conventional DC plasma nitriding furnace (power: 30 kW) equipped with nickel ion source. The samples were prepared under nitrogen-nickel binary ion immersion. Details of the process control are discussed in the paper and other parameters are summarized in Table 1.

Table 1 Processing parameters of ionic bombardment of stainless steel

Voltage/V	Current density/ (mA·cm ⁻²)	Temperature/ °C	Pressure/ Pa	Processing time/h
350–550	0.5–10	580	650–700	9

The cross-sectional morphologies investigation and composition measurement were carried out by using a scanning electron microscope(SEM) equipped with an energy dispersive spectrometer(EDS). The crystallographic structure was determined using an X-ray diffractometer (Philips X'pert PRO MRD system) with a Cu K_α radiation (wavelength $\lambda=0.154\ 2\ \text{nm}$). Corrosion tests were performed using an Autolab electrochemistry workstation (AUTOLAB PGSTAT30) in 5% H₂SO₄ solution. The corrosion resistance was then evaluated from the potentiodynamic curves of anodic polarization. Micro-hardness measurements were carried out by a micro-hardness tester (HVS-1000) using a load of 5 N. The hardness values are average values calculated from 5 measured points.

3 Results and discussion

Fig.1 shows the schematic diagram of ionic bombardment process. The samples were placed on a table in the vacuum chamber. By introducing nickel ion source, the samples could be immersed with nitrogen-

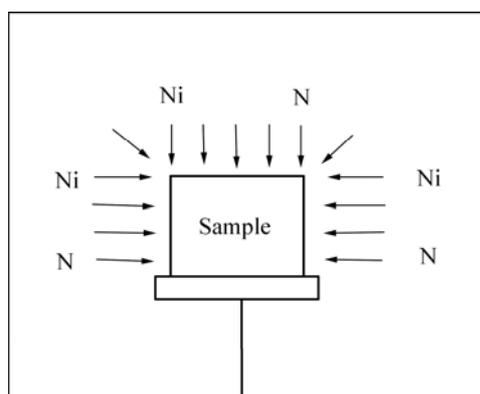


Fig.1 Schematic diagram of binary ion immersion with nitrogen and nickel ions

nickel ions.

Fig.2 shows the SEM cross-sectional micrograph and the line scanning analysis profiles of the sample after the treatment. On the top of the sample surface there exists a layer with different pattern as compared to the 17-4PH matrix. The layer is smooth with a thickness of about 15 μm . A clear interface was observed between the layer and matrix.

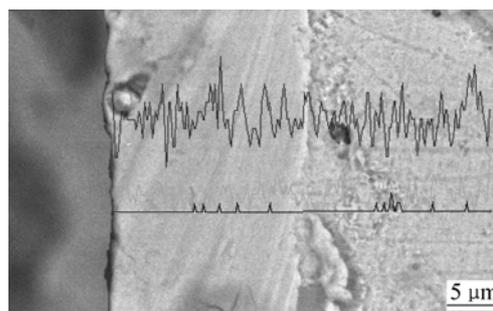


Fig.2 SEM cross-sectional micrograph and line scanning profile of 17-4PH steel ionic bombarded by nickel and nitrogen ion immersion

There is no remarkable change of the composition from the nitrided layer to the matrix. The concentration of nitrogen is very low. No evidence shows that the content of nitrogen and nickel could be increased by ion bombardment process by nickel and nitrogen ion immersion. However, the surface hardness is increased to Hv1268. This value is as high as that obtained by conventional plasma nitriding process.

Fig.3 shows the X-ray diffraction patterns for the untreated 17-4PH steel and the one treated with ion bombardment by nitrogen and nickel ion immersion at 580 °C for 9 h. Peaks of γ and α phase are detected for both samples, which attribute to the stainless steel matrix; whereas two weak peaks of CrN and γ' phases are found for the treated sample. The presence of CrN and γ' phases indicates the penetration of the nitrogen, and leads to the surface hardening. The low nitrogen content is due to the existence of nickel ion, which could hinder the reaction between nitrogen and chromium.

The anodic polarization curves are shown in Fig.4. The ion bombardment by nickel and nitrogen ion immersion causes an increase in anodic current density in the active-passive range. A broadening of the peak corresponding to active dissolution and a shift of the corrosion potential towards lower values are also observed. From 0 to 0.200 V, the corrosion current density is stable, indicating a passive layer formed; then the passive layer dissolves. From 0.300 to 0.750 V, another passive layer forms because there exist multiple phases. From the above observation, it is concluded that

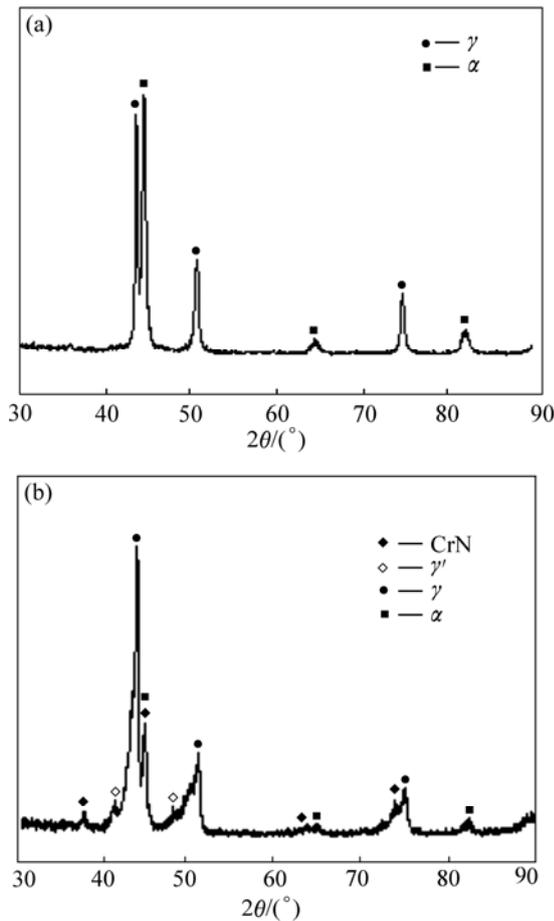


Fig.3 XRD patterns of 17-4PH steel samples untreated and treated with ion bombardment by nickel and nitrogen ion immersion at 580 °C for 9 h: (a) Untreated; (b) Treated

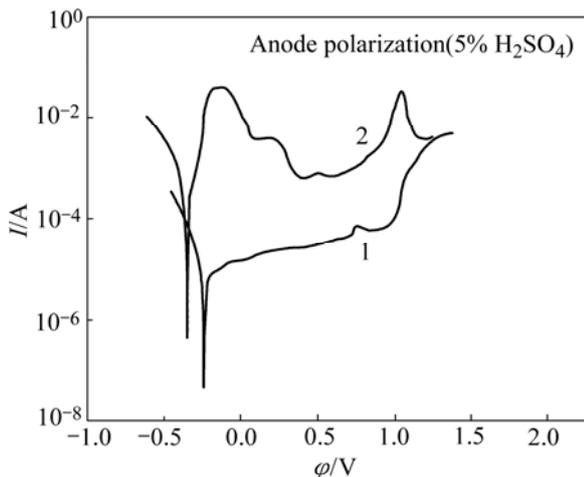


Fig.4 Polarization curves of 17-4PH steel in 5% H_2SO_4 solution: 1 Untreated; 2 Treated with ion bombardment by nickel and nitrogen ion immersion at 580 °C for 9 h

the corrosion resistance of stainless steel after ionic bombardment under nitrogen and nickel ion immersion is slightly reduced compared with stainless steel, but is significantly improved compared with traditional ion

nitriding stainless steel. Table 2 lists the average values of the corrosion potential (φ_{corr}), the corrosion current density (J_{corr}) and the polarization resistance (R_p) for 17-4PH steel untreated and treated with ion bombardment by nickel and nitrogen ion immersion at 580 °C for 9 h. The φ_{corr} of 17-4PH steel treated with ion bombardment is similar to that of the 304 stainless steel untreated. After the passive layer appears, the J_{corr} of 17-4PH steel treated with ion bombardment is similar to that of the 304 stainless steel untreated. It should be noted that, in spite of a decrease in corrosion resistance caused by multiple phases, the stainless steel treated with ion bombardment retains its ability to passivate and can satisfy general operational requirement.

Table 2 φ_{corr} , J_{corr} and R_p of 17-4PH steel in 5% H_2SO_4 solution for untreated and treated with ion bombardment by nickel and nitrogen ion immersion at 580 °C for 9 h

Sample	φ_{corr}/V	$J_{corr}/A \cdot cm^{-2}$	$R_p/k\Omega$
Untreated	-0.240	9.717×10^{-6}	235.6
Treated	-0.348	2.834×10^{-4}	93.35

4 Conclusions

1) Ionic bombardment on 17-4PH stainless steel was carried out by nitrogen and nickel ions immersion at 580 °C for 9 h. The content of nickel on the stainless steel surface does not increase after the treatment. The formation of CrN is also hindered by the nickel ion. However, the hardness of the stainless steel is significantly increased. Vickers' hardness as high as Hv1268 is obtained.

2) The treated stainless steel is of a little reduction in corrosion resistance, as compared with the original stainless steel. However, as compared with the traditional ion-nitriding stainless steel, the corrosion resistance is greatly improved. Therefore, the treatment is an effective method to prevent the reduction in corrosion resistance of stainless steel with the traditional ion-nitriding process.

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