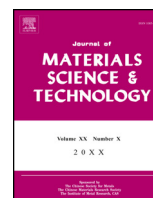




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Enhanced localized and uniform corrosion resistances of bulk nanocrystalline 304 stainless steel in high-concentration hydrochloric acid solutions at room temperature

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ABSTRACT

The localized and uniform corrosion resistances of bulk nanocrystalline 304 stainless steel (NC-304SS) produced by severe rolling technique, and its conventional polycrystalline 304 stainless steel (CC-304SS) counterpart, were investigated in high-concentration hydrochloric acid solutions at room temperature. NC-304SS can scarcely suffer from localized corrosion in 4 mol/L and 5 mol/L HCl solutions during 5-day immersion tests, and in 1–3 mol/L HCl solutions during thirty-five-day immersion tests. The corrosion rate of NC-304SS was also less than that of CC-304SS during these immersion tests. The improved localized and uniform corrosion resistances of NC-304SS were explained in terms of the adsorption and chemical activity of Cl^- on NC-304SS and CC-304SS characterized by X-ray photoelectron spectroscopy, and the valence electron configurations of NC-304SS and CC-304SS were characterized by ultra-violet photoelectron spectroscopy rather than conventional electrochemical results.

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Introduction

Stainless steels are very important metallic materials owing to their excellent general corrosion resistances. However, it is a well-known fact that they are particularly susceptible to localized corrosion in solutions containing Cl^- ions [1]. Localized corrosion can provide points of stress intensification and subsequent cracking via fatigue, stress corrosion cracking or corrosion fatigue during their service. Therefore, the localized corrosion of stainless steels can cause rapid failure, which can result in a sudden accident. Localized corrosion is one of the main causes of structural failure in industrial system for stainless steels. The material degradation through corrosion costs at least 2%–3% of gross national product per annum and a third of chemical plant failures are attributed to localized corrosion [2,3]. The properties and mechanism of localized corrosion of stainless steels and other metallic materials are usually studied by electrochemical measurement, and the parameters related to localized and uniform corrosion can be obtained [4–6]. The dynamical processes of localized and uniform corrosion also have been investigated with some in situ measurement technologies, such as atom probe tomography and in situ scanning tunneling

microscopy [7,8]. The localized corrosion of stainless steels was also characterized by other spectroscopic technologies, such as scanning vibrating electrode technique [9] and double loop electrochemical potentiodynamic reactivation [10]. Many methods for the improvement of localized and uniform corrosion resistances, have been proposed, such as the addition of some other metallic atoms to stainless steels [11], surface coating [12] and corrosion inhibitors [13–15]. The effects of temperature and Cl^- concentration [16], aging and annealing temperature [17] on the localized corrosion resistances of stainless steels have been also extensively investigated.

In 0.05 mol/L H_2SO_4 + 0.05 mol/L NaCl solution, the grain refinement of 304 L stainless steel induced by severe plastic deformation deteriorated the compactness of passive film, so the significant increase of grain boundaries provided more possibility for Cl^- diffusion along grain boundaries, and thus promoted pitting nucleation and growth [18]. The influence of surface mechanical attrition treatment on the corrosion behaviors of AISI 409 and 304 stainless steels in 0.6 M NaCl solution was studied. The results show that different technological parameters can decrease or increase corrosion resistances of 409 stainless steel [19]. Surface mechanical attrition treatment caused a deleterious influence on the corrosion resistance of 304 SS in 0.6 M NaCl. Increase in surface roughness, strain induced martensite and dislocations nullify the beneficial influence of surface nanocrystallization [20]. For 304 stainless steel in 3.5%

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NaCl solution, maximum pit lifetime, the average peak value of current transients and localized nucleation numbers increased with potential. The ohmic potential drop presented between the pit interior and bulk solution, but the growth controlling factor was metal cations diffusion [21]. For Al–Li alloy in 0.1 mol/L Na_2SO_4 + 100 ppm of Cl^- solution, the pitting potential, pit number, and stability of the passive layer underwent changes depending on the average grain size and the presence of precipitation or its lack, and corrosion resistance appeared to increase with decreasing average grain size [22]. For immersion test in 3.5 wt% NaCl solution and electrochemical tests in 0.01 mol/L NaCl solution, the improved corrosion resistance of ultrafine grained bulk pure Al resulted from the lower microgalvanic current and reduced susceptibility of localized corrosion due to the uniform distribution of fine Si containing impurities and the formation of a denser oxide film. The strain induced crystalline defects provided more nucleation sites for the formation of a denser and thicker oxide film, thus enhancing its corrosion resistance [23]. However, it is difficult to prevent stainless steels and other metallic materials from localized corrosion in solutions containing Cl^- ions, and the enhanced localized and uniform corrosion resistances of metallic materials are usually attributed to the compact oxide film on their corrosion surfaces. There were no the further understandings for the localized and uniform corrosion of metallic materials so far [1–23].

The localized and uniform corrosion of bulk nanocrystalline 304 stainless steel (NC-304SS) produced by severe rolling technique and its conventional polycrystalline 304 stainless steel (CC-304SS) counterpart were studied with immersion tests in 4 and 5 mol/L HCl solutions for 5 days and in 1, 2 and 3 mol/L HCl solutions for 35 days at room temperature. There are different types of localized corrosion including pitting and crevice corrosion. Here the pitting corrosion of NC-304SS and CC-304SS was investigated. The improved localized and uniform corrosion resistances of NC-304SS were explained in terms of the valence electron configurations of NC-304SS and CC-304SS characterized by ultra-violet photoelectron spectroscopy (UPS), and the adsorption and chemical activity of Cl^- on corroded NC-304SS and CC-304SS were characterized by X-ray photoelectron spectroscopy (XPS).

Material and methods

Materials and chemicals

NC-304SS was prepared by severe rolling technique with CC-304SS; severe rolling technique mainly consists of heat treatment, hot rolling and cold rolling; the details of this technique were described in our previous work [24]. CC-304SS is cylindrical bar with $\Phi 35 \text{ mm} \times 150 \text{ mm}$. NC-304SS did not undergo any other treatment after severe rolling process. The chemical compositions (at.%) of NC-304SS and CC-304SS (measured by X-ray spectrometer system, Magix Pro PW2440, PHILIPS) were identical, C 0.0020%, Si 0.85%, Mn 1.85%, P 0.045%, S 0.028%, Ni 9.03%, Cr 18.24% and Fe balance. The grain sizes of NC-304SS and CC-304SS are about 86–175 nm and 80–130 μm , respectively. The transmission electron microscope (TEM) images of NC-304SS and optical micrograph (OM) of CC-304SS, and their X-ray diffraction (XRD) are shown as Fig. 1(a)–(c), respectively [25,26]. According to Fig. 1(c), there was no martensitic phase in NC-304SS due to its cooling process from high final rolling temperature to room temperature. The dimensions of CC-304SS and NC-304SS specimens for immersion test and UPS measurement are 11 mm \times 11 mm \times 2.0 mm and 1 mm \times 11 mm \times 1.5 mm, respectively. The 11 mm \times 11 mm surface of NC-304SS sample is its rolled surface. The 11 mm \times 11 mm surface of CC-304SS specimen is parallel to its axis of cylindrical bar. Subsequently, they were polished using SiC papers of successive

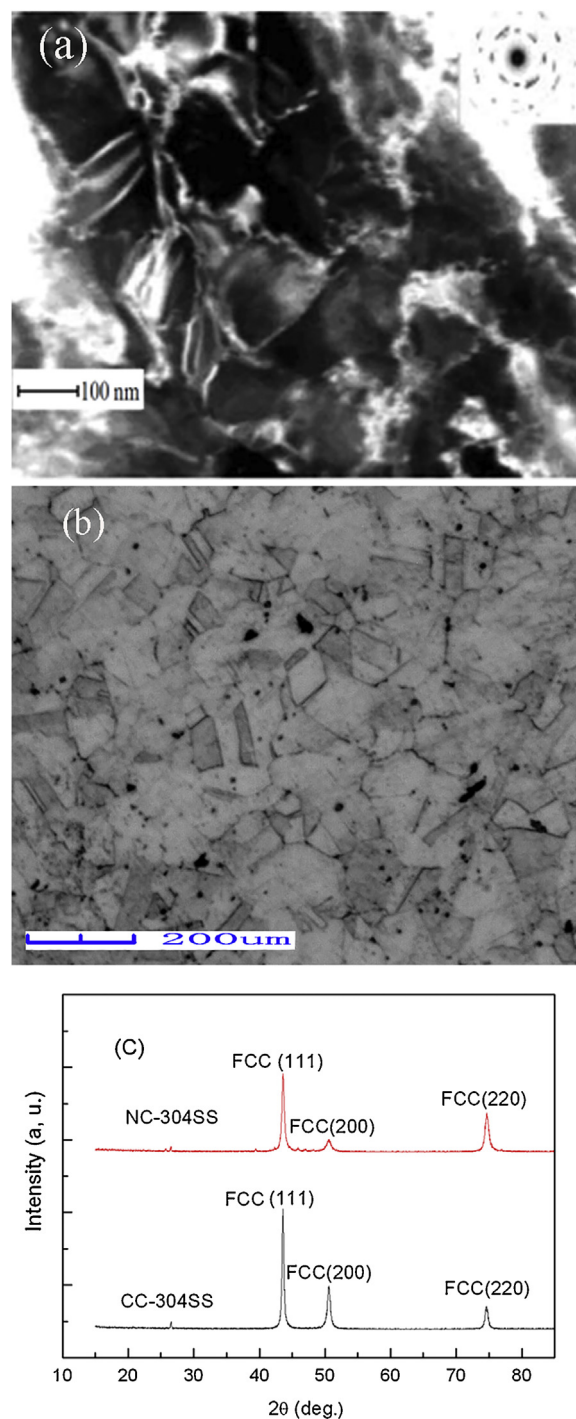


Fig. 1. TEM of NC-304SS (a), OM of CC-304SS (b) and their XRD (c) [25,26].

grades up to 2000#, cleaned by distilled water, degreased with acetone, and then dried with hair drier. The first weight measurement of NC-304SS and CC-304SS was carried out after above treatment was finished. The accuracy of analytical balance is 10^{-2} milligram during weight loss measurement.

Immersion test

During immersion test, NC-304SS and CC-304SS samples were placed on glass holder in 1–3 mol/L HCl solutions for 35 days, in 4 and 5 mol/L HCl solutions for 5 days in meter glass at $24 \pm 1^\circ\text{C}$ in order to eliminate the temperature sensitivity of localized and

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