



Electrochemical and passivation behavior investigation of ferritic stainless steel in alkaline environment



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HIGHLIGHTS

- Immersion time plays important roles in evolution of passive film composition and resistance.
- Passive film presenting a thinner n-type layer in simulated concrete pore solution.
- The primary constituents of passive film is (Cr, Fe)-oxides without chloride ions.

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ABSTRACT

The electrochemical behavior of 430 ferritic stainless steel in alkaline environment was evaluated by potentiodynamic curves, electrochemical impedance spectroscopy (EIS), Mott–Schottky, X-ray photoelectron spectroscopy (XPS) and Auger Electronic Spectrometer (AES). The results confirmed that n-type semiconducting behavior of the passive film is formed on the surface of 430 ferritic stainless steel after immersion in the alkaline solution. Based on the evaluations of the passive film by AES and XPS analysis, it can be concluded that the primary constituents of the passive film is (Cr, Fe)-oxides, like the Cr_2O_3 , $\text{Cr}(\text{OH})_3$, FeOOH and Fe_3O_4 . In addition, no chloride ions are detected in the passive film.

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1. Introduction

Concrete structures typically show high corrosion resistance, due to a protective oxide film formed on steel surface, created by concrete in strong highly alkaline environment. However, with the presence of concrete carbonation, chloride levels or mechanical stresses, reinforced concrete can also deteriorate [1,2]. Recent years, the applications of stainless steel are one of the most reliable solutions in concrete structures to reduce chloride-induced corrosion problems and, therefore, increase the structures service life [3]. In neutral or alkaline condition, the higher corrosion resistance of stainless steel is due to the presence of a very thin passive film on the surface. The structure of passive film is very complex, consisting in a chromium -oxide/hydroxide inner layer and an iron-oxide rich outer layer [4], sometimes, its composition can change with the applied potential or circumstances [5,6].

The corrosion resistance of stainless steel related to the composition, electrochemical or semiconductive behavior of its passive film [7]. Take the concrete environment for example, there are some interesting studies focused on the corrosion or passivation behavior of traditional austenitic or duplex stainless steel types—such as 304, 316 or 2205 stainless steel [8–10], besides, in many concrete environments, the new low Ni, high Mn type of stainless steels has also been considered for using [11,12]. However, currently due to high prices of nickel, especially in many projects, the austenitic stainless steel is replaced by the ferritic stainless steels. Compared with austenite stainless steel, ferritic stainless steel is a type of Fe–Cr series alloy, which retains its body centered cubic structure even after heat treatment in the normal temperature range. It is known to be extremely resistant of stress corrosion cracking in some chloride environments and other properties [13].

At present, a variety of experimental electrochemical or surface analysis techniques, including anodic polarization, electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), capacitance measurements, X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES), has been applied to study

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the chemical, electrochemical and electronic properties of the passive films formed on several types of stainless steel in simulated concrete environment. Moreover, the nature or structure of passive film formed in the concrete solution condition is also very interesting features to know [14–16]. What is more, combined electrochemical and analytical investigations to observe the behavior of ferritic stainless steels in alkaline concrete environment are scarce and somewhat controversial.

In present paper, it aims at the passivation behavior of 430 ferritic stainless steel in alkaline solution with the presence of chloride ions. The electrochemical and corrosion behavior is investigated by potentiodynamic measurements, EIS and Mott–Schottky approach. At the same time, the chemical composition of the passive film formed in the alkaline environment is studied by means of XPS and AES. The goal of this paper is to summarize, the thickness, composition and electronic structure of the passive film in alkaline condition for better understanding of the electrochemical behavior of ferritic stainless steel in the simulated concrete pore solution.

2. Experimental

2.1. Materials and solutions

All of the testing specimens were cut from a sheet of 430 ferritic stainless steel with the thickness of 5 mm, supplied by the Avesta Inc. The nominal chemical composition (wt.%) of the stainless steel were as follows: C 0.035, Si 0.50, Cr 17.30, Ni 0.14, Mn 0.35, S 0.004, P 0.029 and Fe balance. The specimens size were 1 cm × 1 cm, then ground sequentially from 400 to 2000 grit SiC paper, polished with 0.1 μm alumina powder, degreased with alcohol and deionised water, then dried in cooling air.

Aqueous sodium–calcium hydroxide solutions were prepared to simulate the electrolytes contained in the pores of concrete environment [17], at the same time, the 1% sodium chloride was added in to the solution, to observe the effect of chloride on its passive film. All the solutions were prepared with double distilled water, and the pH value of testing solution was 13, which was regularly checked using a Mettler pH meter. More important, the solutions were used immediately after preparation to avoid carbonation effects and all reagents were of at least ACS grade.

2.2. Electrochemical measurements

The electrochemical measurements were performed by using the PAR 2273 on a conventional three-electrode cell at ambient temperature (25 °C). A platinum foil and a saturated calomel electrode (SCE), which connected to the cell via a Luggin capillary, were used as the counter and reference electrodes, respectively. And all potentials measured in this paper refer to SCE. Before test, the solution was purged with pure nitrogen for 1 h, and the gas flow was maintained during the whole testing. Prior to electrochemical experiment, samples were initially reduced potentiostatically at -0.8 V for 20 min to remove air-formed oxides. The EIS measurement started after the stable of open-circuit potential (OCP), the frequency of EIS was swept from 100 kHz down to 10 mHz, at 10 data cycles/decade, with the applied AC amplitude of 10 mV. The impedance spectra were collected for increasing immersion times from 12 h to 480 h, and the impedance data were fitted by Zsimpwin software. The capacitance test was carried out at a fixed frequency of 1 kHz during a 50 mV/Step in the potential range from -1.0 to 1.2 V (vs. SCE). Potentiodynamic polarization tests were carried out using a scan rate of 1.0 mV s⁻¹ starting from -0.6 V vs. OCP to transpassive potential. Each experiment was repeated several times under the same conditions to control the reproducibility.

2.3. The AES and XPS characterization

Initially, a negative potential was applied during the constant-potential passivation treatment to remove the surface oxide layer, and then immersion in the solution for 3 h at OCP in order to form of a stationary passive film. The AES depth profiles were measured with a scanning Auger microprobe with a coaxial electron gun and a cylindrical mirror analyzer. The sputtering rate, as determined on a thermal oxidation SiO₂/Si standard, was approximately 2 nm/min. The chemical composition of the passive film was investigated by XPS with a monochromatic Al K α radiation source and a hemispherical electron analyzer operating at the pass energy of 25 eV. The curve fitting was performed by the commercial software Xpspeak version, which contained the Shirley background subtraction and Gaussian–Lorentzian tail function for better spectra fitting.

3. Results and discussion

3.1. Potentiodynamic polarization studies

The anodic potentiodynamic curves can provide some important features concern with the electrochemical behavior of stainless steel in certain environments. Fig. 1 shows the potentiodynamic polarization curves of 430 ferritic stainless steel in alkaline solution. It can be seen that ferritic stainless steel displays typical passive behavior, the curves pass through active region, active passive transition region, passive region and transpassive region with increasing potential from free corrosion potential to anode direction. As shown in Fig. 1, the passive region of 430 ferritic stainless steel in alkaline solution is from about the -0.52 V to 0 V. Moreover, the potential at which the current density exceeded 100 μA/cm² is defined as pitting potential, and the pitting potential of 430 ferritic stainless steel in alkaline solution is about 0 V. At the same time, an important feature revealed by the curves is an anodic oxidation peak at -0.2 V reveals that chromium oxide dissolution is taking place through the Cr³⁺ to the Cr⁶⁺ when the material suffers high anodic polarizations [18].

3.2. Electrochemical impedance spectroscopy studies

Nowadays, the EIS is widely applied for investigating the corrosion and passivation phenomena due to its non-destructive nature suitable for corrosion monitoring [19], especially in the electrochemical properties of passive film on the surface of stainless steel. Fig. 2 presents the open-circuit impedances spectra as a Nyquist diagram of 430 ferritic stainless steel at different immersion time into alkaline solution. It is clearly observed that all the electrochemical impedance plots with different immersion time were characterized by the presence of single unfinished semi-circle arc, which is attributed to charge transfer process occurring at the metal/electrolyte interface or related to the surface film property. It is showing that they have the similar corrosion mechanisms. As can be seen in Fig. 2, the evolution of the overall impedance shows higher values with the immersion time increases, revealing an enhancement of passive film protective behavior in this type of environment. The global impedance increases during first immersion time, and then slightly decreases, that is to say, with the immersion time from the first 12 h to 156 h, the overall impedance increase as the immersion increase, and then it started to decrease, revealing that the surfaces are more susceptible to pitting attack. The Bode plot of passive film in

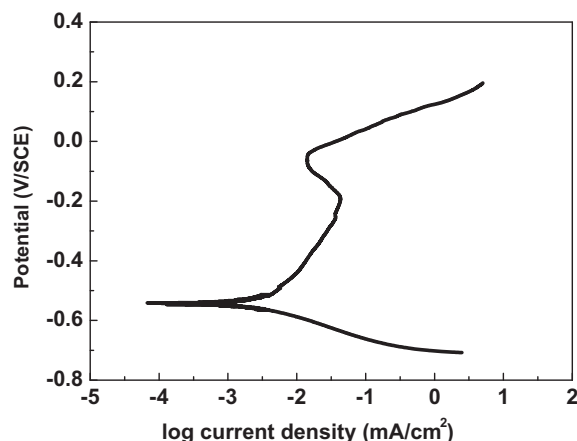


Fig. 1. Potentiodynamic polarization curve of 430 ferritic stainless steel in alkaline solution with the presence of chloride ions.

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