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Effect of carbonation on the electrochemical behavior of corrosion resistance low alloy steel rebars in cement extract solution





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HIGHLIGHTS

• New low Cr alloy steels resistant to corrosion are developed for steel rebar.

• Cr can promote the passivation of steel in carbonated cement extract solution.

• With pH reducing, the effect of Cr becomes more obvious.

• The carrier density of 5Cr steel is merely 1/3 of that of CS at pH 10.5.

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ABSTRACT

In this paper, cyclic voltammetry technique, potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and Mott-Schottky measurement combined with surface morphology observation were applied to investigate the electrochemical behavior of carbon steel (CS) and three Cr modified low alloy steels in the carbonated cement extract solution. Results show that pH plays an important role in the formation of passive films on steel rebars. In non carbonated cement extract solution, corrosion behaviors of CS and Cr modified steel vary slightly, and Cr addition brings down the amount of defects in passive film. In carbonated cement extract solution, pitting potential shifts negatively as pH value decreases. Polarization resistance declines and the density of the current carrier in the passive film increases. With Cr content increasing, corrosion current density declines; pitting potential, polarization resistance and passivation range are magnified. Besides, the effect of Cr is more significant in lower pH. The good-to-poor sequence of corrosion resistance is: 5Cr > 3Cr > 1.5Cr > CS, and 5Cr steel possesses the best corrosion resistance.

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1. Introduction

Reinforcing bars in concrete are not prone to corrosion within a short time due to the high resistivity [1,2] and alkalinity [3–5] of the concrete cover. However, the erosion of Cl⁻ and carbonation of concrete tend to lead to the failure of reinforced concrete structures [6–10]. In high alkaline environment, passive film of carbon steel rebars is stable, whereas it tends to dissolve and degrade on condition that alkalinity is lowered, causing the rebar out of protection. What makes the decline of alkalinity of concrete is CO₂ in neutral atmospheric environment or SO₂ and NO₂ in acid rain, which may react with Ca(OH)₂ and C-S-H gel in concrete; carbonation caused by CO₂ is the most common. It was reported that the

* Corresponding author. *E-mail address:* chengxuequn@ustb.edu.cn (X. Cheng). passive film of carbon steel rebars in concrete pore solution with pH ranging from 9.8 to 11.5 is in a non-stable state and cannot protect rebars from corrosion; while pH is less than 9.8, passive film fails to form and the rebar is in active corrosion [11]. However, for stainless steel, the addition of Cr participates in forming more protective passive film; as a result, stainless steel rebars presents excellent corrosion resistance in carbonated Cl⁻ – containing environment [12–15]. Freire et al. [13] proposed that Fe^{2+}/Fe^{3+} value is lowered in passive film of 316 stainless steel in simulated concrete pore solution containing 10wt.% NaCl when pH = 9 and the proportion of Cr increases in passive film, which prevents steel from corrosion at relatively lower pH.

Simulated concrete pore solution is frequently used to study corrosion of rebars in concrete for short test period and elimination of experimental errors caused by multiphase and heterogeneity in the actual concrete. At present, the commonly used simulated concrete pore solution consists of KOH, NaOH [8,9], saturated Ca (OH)₂ [9,16–19] and cement extract solution [19–21]. Huet et al. [22] suggested that after hydration of cement, alkali, sulfate and silicate are crucial in the formation of passive film of rebars in carbonated environment. Valcarce et al. [23] found that carbonation leads to uniform corrosion of rebars and passivation is closely related to CO_3^{2-}/HCO_3^{-} at a fixed pH value. Moreno et al. [24] declared that in high carbonate/bicarbonate concentration solution, rebars can remain a passive state and present excellent pitting corrosion resistance; while in low carbonate/bicarbonate concentration solution, rebars are incapable of passivation. Liu et al. [9] studied the critical chloride concentration of carbon steel rebars in saturated Ca(OH)₂ + KOH + NaOH solution and cement extract solution at various degrees of carbonation, and concluded that the adoption of $Ca(OH)_2$ + KOH + NaOH solution is unreasonable to simulate the carbonated concrete solution attributed to the facilitation of high HCO_{3}^{-} concentration for stability of passive film. On the contrary, HCO_3^- can combine with $Ca(OH)_2$ and C-S-H, forming solid products, which lowers the concentration of dissolved HCO_{3}^{-} ; therefore, cement extract solution is more practical in the simulation of actual concrete pore solution. Veleva et al. [21] compared the electrochemical behavior of AISI 316 stainless steel in cement extract solution and saturated Ca(OH)₂ solution through cyclic voltammetry technique and surface observation, and results showed that passive film formed in cement extract solution is more compact and more uniform.

Nowadays, low alloy steel rebars have become the research hotspot. According to our previous work [16], low Cr alloyed steels exhibit high critical chloride concentration and low corrosion rate in saturated Ca(OH)₂ solution. The carbonation of concrete has a strong impact on the corrosion behavior of rebars. Hence, for making improvements to provious research, cyclic voltammetry, potentiodynamic polarization, EIS and Mott-Schottky measurement were employed to investigate the effect of carbonation on the electrochemical behavior of Cr modified steel rebars in cement extract solution. The findings are expected to offer guidance for the development and manufacturing of low alloy corrosion resistant rebars.

2. Experimental

2.1. Materials and solution

In this work, HRB400 carbon steel rebar and three Cr-modified low alloy steel rebars are used for experiments [16]. Table 1 shows the chemical composition (in% by mass) of the four kind of steel rebars determined by analytical analysis. The specimens for electrochemical tests with dimensions of $10 \times 10 \times 5$ mm, the exposed surface of the specimens was $10 \text{ mm} \times 10 \text{ mm}$, with an area of 1 cm^2 . A copper wire was welded to one end surface of the steel specimen and the other end surface was used to test in the electrochemical methods. The flank and the end surface with a thick copper wire were sealed by means of epoxy resin (Fig. 1 (a)). Prior to the experiment, the samples were polished with silicon carbide (SiC) water polishing papers down to 2000#, and then ultrasonically cleaned in acetone and rinsed in distilled water.

The test solution was supernate of the mixture of PO 42.5R portland cement and deionized water [9,19]. The cement and deionized water were mixed according to the proportion of 1:10, and the supernate was taken out after being stirred for 15 min and placed for over 5 h. The initial pH of cement was 12.5, and the other two kinds of carbonated simulated concrete pore solution (pH = 11.5 and 10.5) were acquired by adding different amount of NaHCO₃ [9] and calibrating with a pH meter.

2.2. Electrochemical measurements

Electrochemical tests were performed with PAR multi-channel workstation. A three-electrode system was applied, with the steel rebar as working electrode, a saturated calomel electrode (SCE) as reference electrode and platinum gauge as counter electrode (Fig. 1(b)). The electrochemical tests were conducted after 30 min immersion of the specimens. The potentiodynamic polarization measurement was carried out from -0.25V (vs. OCP), and ended when the anodic current reached 1 mA, the scanning rate of which was 1 mV/s. Electrochemical impedance spectroscopy (EIS) tests were performed at OCP with a scanning range of 100 kHz \sim 10 mHz at the amplitude of 10 mV and the results were interpreted based on an equivalent electrical circuit by using a suitable fitting procedure of ZSimpWin. Mott-Schottky measurements were carried out at a frequency of 1 kHz, an amplitude of 10 mV with a scanning potential range of -1 V to 1 V, and the interval was 50 mV. Cyclic voltammetry (CV) tests were performed between -1.5 V and 1.0 V at scanning rate of 50 mV/s for five scanning cycles.

2.3. Surface morphology observation

The macro morphology was observed with scanning electron spectroscopy (SEM, FEI Quanta 250). Besides, the composition of corrosion products was analyzed with energy disperse spectroscopy (EDS).

3. Results and discussion

3.1. Cyclic voltammetry

Cyclic voltammetry has been widely used in studies of passive film of rebars on account of effectiveness of characterizing redox reactions of rebars in alkaline environment and reproducibility. Fig. 2 shows the fifth cycle of cyclic voltammetry of rebars tested in carbonated cement extract. As shown in Fig. 2(a), in non-carbonated solution (pH = 12.5), the curves varies slightly in patterns, and two anodic peaks, A_1 , A_2 as well as two cathodic peaks, C_1 , C_2 can be seen. The first anodic peak, A_1 arises at the potential of -1.1 V, corresponding to the following reaction [17,25,26]:

$$Fe + 2OH^- \leftrightarrow Fe(OH)2 + 2e^-$$
 (1)

When the potential reaches -0.6 V, the second anodic peak, A₂ occurs, which proves the formation of passive film and the transformation from Fe²⁺ to insoluble Fe³⁺ [17,25,26]:

$$3Fe(OH)_2 + 2OH^- \leftrightarrow Fe_3O_4 + 4H_2O + 2e^- \tag{2}$$

$$3FeO + 2OH^- \leftrightarrow Fe_3O_4 + 2e^- \tag{3}$$

Table	1
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Chemical composition (wt%) of the tested HRB400 carbon steel and Cr-modified ste	els.
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Steel	С	Si	Mn	Р	S	Cr	Fe
CS	0.196	0.57	1.57	0.024	0.017	0.08	Residual
1.5Cr	0.171	0.66	1.30	0.014	0.008	1.50	Residual
3Cr	0.184	0.65	1.23	0.007	0.012	3.02	Residual
5Cr	0.157	0.45	1.57	0.010	0.004	5.06	Residual

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