



Time-dependent variation of the electrochemical impedance for thermo-mechanically treated versus plain low alloy steel rebars in contact with simulated concrete pore solution



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HIGHLIGHTS

- EIS for protective properties of oxide films on reinforcement steel.
- Passive films grown in chloride contaminated SPS.
- Thermo-mechanically treated mild steel and low-alloy steel.

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ABSTRACT

Electrochemical studies (AC impedance and DC polarization) have been used to study the protective properties of passive films that form on reinforced steel embedded in concrete as a function of time and space. Experiments have been performed on passive/oxide films grown on the surface of steel reinforcement bars exposed to chloride-contaminated simulated concrete pore solution (SPS) prepared by dissolving a representative amount of sodium hydroxide and potassium hydroxide in distilled water and saturated with lime. A common type of locally available concrete reinforcement TMT (thermo-mechanically treated) mild steel with TM (tempered martensitic) and PF (pearlite ferrite) rings (source “A”) was investigated in comparison to a type of hot rolled low-alloy steel without thermo-mechanical treatment (source “B”). The results of the elemental analysis and metallographic microstructure coupled with electrochemical studies indicate that the coupled surface and sub-surface composition of TMT rebar has a time-dependent reversing effect on the protective properties of the passive oxide films that provide durability and protection to the steel rebars against corrosion in concrete. Steel with TM–PF rings has a better initial corrosion resistance in comparison to low alloyed steel. However, with the passage of time, its corrosion resistance decreases, and alloyed steel finally dominates over the TMT rebar.

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1. Introduction

Concrete experiences various environmental stressors throughout its service life span. For example, the reinforced concrete structures exposed to an on shore/off shore sea environment suffer from corrosion of the steel bars due to chloride ingress. The corrosion of the reinforcing steel in concrete occurs via an electro-chemical process at the concrete/steel interface, where the steel is oxidized in the concrete pore solution. Corrosion causes

loss of metal in the reinforcing steel; however, due to the high alkalinity provided by concrete which helps in strengthening of the passive film, the corrosive effect of the environment is countered. Under service conditions, the steel can de-passivate, i.e., the protective oxide film gets deteriorated leading to high rates of metal loss. The major factors affecting the de-passivation and corrosion of reinforcing steel include the presence of chloride ions, elevated temperature, the pH of the pore solution, the surface characteristics and chemical composition of the steel, and the presence of mechanical stresses [1]. In addition, the de-passivation of rebar and the successive corrosion of steel in concrete also depend on the rebar surface condition, the elemental composition and the properties of the rebar-concrete interfacial transition zone [2–5].

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Substantial researches in the last several years had been performed on the effect of the surface conditions and material composition of rebar for specialized type of steels, such as stainless steel SS316LN, 316L clad, 2102 LDX [6], 304, 2205 [7,8] micro-composite, SS304, SS316LN [9,10], and low carbon chromium steel [11]. However, the literature review indicated that only limited research has been performed on the effect of the surface condition and the material composition on the corrosion of the conventional type of black steel commonly used in the construction industries [12–17]. The development of a passive layer and the successive corrosion of black steel in reinforced concrete are also significantly affected by the rebar type, elemental composition, surface condition and material inclusions [18,19]. Some other authors [20] also indicated that the corrosion may not always follow a linear relation with the various factors and that the specific combination of steel type and surface condition play an important role in determining the overall magnitude of corrosion [21,22]. They found that the same type of surface condition may be more resistant to corrosion with a different type and chemical composition of rebar, and vice versa. In particular, the past research referenced above indicated that there is no comprehensive research available for the time-dependent behavior of the TMT and normal hot rolled steel rebars produced without quenching and tempering treatment (Q&T). Additionally, a difference of opinion existed among the various researchers in the past with respect to the time and space constraints involved in the surface corrosion. Therefore, in this research, time-dependent electrochemical studies have been performed for the TMT steel rebars and compared with a low alloy steel rebar produced without Q&T treatment.

2. Experimentation

2.1. Sample preparation

Two types of concrete reinforcement steel specimens, TMT mild steel (abbreviated as source “A”) having TM (tempered martensite) rim and PF (pearlite ferrite) core and low alloy weathering type steel (designated as source “B”) were prepared for this study. The chemical composition of the steels is recorded in Table 1. Both types of specimens were cut to 30-mm length from the deformed steel rebars of 10-mm nominal diameter. The steel specimens were mounted in cold resin leaving the cross sectional area open to act as the working electrode. This open cross sectional area of both the steels were polished on metallurgical polishing machine starting from rough emery paper and finally on alumina paste of 1 μm size. To get electrical contact, the back side of the mold was drilled to touch the rebars' opposite side cross sectional area. A copper wire was inserted in this drilled hole and molten soldering wire (lead–tin alloy) was poured in the hole to fix the copper wire in electrical contact with the rebar specimens.

2.2. Simulated concrete pore solution

The synthetic pore solution was prepared by dissolving 7.4 g of NaOH and 36.6 g of KOH per liter of distilled water. The solution was saturated with $\text{Ca}(\text{OH})_2$ to simulate conditions in ordinary Portland cement concrete. Prior to its use, the solution was kept for 24 h under continuous stirring on a magnetic stirrer and then filtered using Whatman paper of No. 15 grade. This filtering was necessary to remove insoluble CaO from the solution. 3.5% NaCl was there after added to this solution to simulate the aggressive conditions that cause the steel to be prone to corrosion. The pH of the final solution was 12.6 at 27 °C. Such a simulated concrete pore solution is widely used to understand the mechanism of nucleation, growth

and deterioration of passive film formed on steel rebars. Since the pore solution in the concrete has the same composition and pH as in simulated synthetic pore solution, the mechanism of the nucleation and growth of the passive film on steel reinforcement bars determined in synthetic pore solution remains the same as that for the rebars embedded in solid concrete. However, the kinetics of formation of the passive film and its deterioration may substantially differ in the two cases.

2.3. Experimental program

Electrochemical techniques were used to investigate the above described two types of the rebars. For this purpose, EIS (Electro-chemical Impedance Spectroscopic) measurements, DC polarization and open circuit corrosion potential measurement techniques were used. These measurements helped to determine the polarization resistance, pitting tendency and nature of the film formed on the surface of steel rebars in contact of the test solution.

The cross section area of rebar specimens were passivated in two beakers containing the SPS solution mixed with 3.5% NaCl. The immersion time was not optimized but was selected based on the results of previous tests that indicated at least 8 days was required to achieve stable passive films on the rebar surfaces [22]. Since there were no significant changes in the parameters during the tests; the averages of these measurements are presented in this paper. Further details of all of the tests performed in the study are presented in the following sections.

The EIS tests were performed on replicate rebar specimens that were immersed in the SPS solution with 3.5% NaCl using a GamryPC4/Potentiostat/EIS Potentiostat (Fig. 1). To avoid extensive exposure to air, the rebar specimens were transferred within seconds from the breakers to the co-axial corrosion cell, which contained the same solution. The photograph showing the electrochemical cell used for the study is presented in Fig. 1. The EIS measurements were performed at the free corrosion potential of the steel specimens imposing an AC voltage of 10 mV RMS, between 10^5 and 10^{-3} Hz at 10 equally spaced frequencies per decade.

Cyclic polarization tests were performed on replicate rebars specimens using the same instrument as used for the EIS studies. The potential scan of each cyclic polarization tests were carried out after completion of the EIS tests, beginning at the corrosion potential (E_{corr}), and the scan was performed in the more noble direction at a scan rate of 0.6 V/h. The current was continuously recorded with the change in potential. The onset of localized corrosion is usually indicated by a rapid increase of the anodic current at potentials below the oxygen-evolution potential. When the potential reached 1.5 V above the open circuit potential, the direction of the scanning was reversed (toward more active potentials); the reverse scan was continued until the hysteresis loop closed or until the corrosion potential was reached.

3. Results and discussion

3.1. Microstructure and material composition

The surface and sub-surface conditions of the TMT steel rebars used most commonly as reinforcement in the local concrete industry were investigated. In steel source ‘A’, one separate ring of TM was observed to be formed on the outer periphery of the mild steel with an inner core of PF, as shown in Fig. 2(a). In contrast, the structures of other steel from source ‘B’ did not exhibit the presence of any rim on its cross sectional area, as shown in Fig. 2(b). These two different types of surface development for the reinforcement types may cause a nonlinear time-dependent effect on the behavior of the protective properties of the steel reinforcement. The elemental composition of the steel bars used in this study is

Table 1
Elemental composition of the steels in this study.

Element (%)	Source A	Source B
C	0.22	0.12
Mn	0.59	0.85
Si	0.17	0.21
Cu	–	0.26
Cr	–	0.07
Ni	–	0.10
Ti	–	0.02

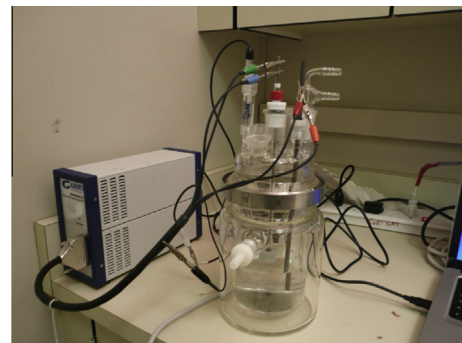


Fig. 1. Gamry Potentiostat for the EIS testing.

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