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In situ 3D monitoring of corrosion on carbon steel and ferritic stainless steel embedded in cement paste

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ABSTRACT

In a X-ray microcomputed tomography study, active corrosion was induced by galvanostatically corroding steel embedded in cement paste. The results give insight into corrosion product build up, crack formation, leaching of products into the cracks and voids, and differences in morphology of corrosion attack in the case of carbon steel or stainless steel reinforcement. Carbon steel was homogeneously etched away with a homogeneous layer of corrosion products forming at the steel/cement paste interface. For ferritic stainless steel, pits were forming, concentrating the corrosion products locally, which led to more extensive damage on the cement paste cover.

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1. Introduction

The corrosion process of the reinforcing steel in concrete can be divided into two time periods: the initiation period and the propagation period [1]. During the initiation period the conditions for passive film break down and initiation of corrosion develop. During the propagation period, iron reacts with oxygen and water to form corrosion products, mainly iron oxide/hydroxides. Because the latter have larger specific volumes than metallic iron [2], corrosion products begin to inhabit the steel/concrete porous interfacial zone, generating radial pressure [3–5], and consequently causing cracking of concrete. Since predicting the time to concrete cracking could lead to significant decrease in repair costs and safety during structures' operation, numerous service life models are developed in order to monitor and predict the time to cracking due to the corrosion of reinforcement [3,4,6,7].

Predicting the stability and serviceability of structures requires crucial information about the propagation period, such as at what point do corrosion products begin accumulating at the steel/concrete interface, eventually leading to corrosion-induced cracking of the surrounding concrete. Although several models have been developed that predict the time-to-corrosion cracking, taking into account the nonlinear formation of corrosion products [4], and the residual mechanical properties of concrete and reinforcement during corrosion [3,8], differences between model predictions and experimental results remain. These difference are attributed to two factors: (1) a lack of understanding of the composition, mechanical properties, and transport of the corrosion products filling the porous zone and surrounding voids and pores [5,8,9]; and (2) most models assume a uniform layer of corrosion products around the steel/concrete interface, which is not accurate enough for characterizing the chloride-induced corrosion of steel reinforcement, as highlighted in the work by Zhao et al. [10].

The difference between uniform and localized corrosion becomes more pronounced in the case of stainless steel. With increasing demand of prolonging the service life of structures in aggressive environments, researchers have considered replacing carbon steel with stainless steels with lower concentrations of chromium and nickel [11–13]. These grades of steel can be used where the high cost/high corrosion resistance of austenitic stainless steel is not required e.g., for structures subjected to mild marine environments [14]. For stainless steel, the attack can be extremely localized with the formation of corrosion pits [15–18]. During localised corrosion a local loss of steel cross section takes place, which can induce dramatic consequences for reinforced and prestressed concrete structures under load. In addition, with

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the pressure from the corrosion products forming locally, it could lead to local increase of stresses at the steel/concrete interface.

Until now the formation of pits has been limited to investigations of stainless steel alone [15,18–20], but the kinetics of formation of these pits inside cement paste deserves increased attention. Most of the time, mechanisms of corrosion propagation were inferred from indirect observation methods: (1) a "destructive" approach, which requires cutting open the samples after evidence of extensive corrosion [21]; or (2) external observations of apparent crack propagation [5,6]. These indirect methods give valuable insight into the extent of corrosion; however, information about the corrosion products themselves is limited. When observing an open sample, cracks might have been induced in preparation of the sample, corrosion products may have been displaced, and additional oxidation, as a result of the preparation environment, might change their nature. Furthermore, information about morphology and volume of the studied phases is lost through visual observation [22]. Recently, initiatives for investigating the propagation period of reinforcement in concrete by non-destructive methods have been developed, such as X-ray attenuation measurement [23]; however, this work was limited by the low resolution of the X-ray camera—pixel size was 0.89 mm × 0.89 mm—and the lack of full three-dimensional (3D) information on the 2-cm-thick mortar samples.

The work presented herein demonstrates the unique advantages of X-ray microcomputed tomography (μCT) using synchrotron radiation to follow corrosion formation in cement paste. The technique allows for real time *in situ* 3D characterization of the following mechanisms: (1) corrosion of the steel reinforcement; (2) accumulation of corrosion products at the steel/concrete interface; and (3) development of cracks in the cement paste matrix. The main advantages of using μCT is that it is non-invasive, enables real-time monitoring without interfering with the chemistry and the morphology of the products and cracks, and offers the possibility of 3D-imaging of the morphology of the structure. Because the technique is sensitive to the density of materials, the different phases – steel, corrosion products, and cement matrix – are distinguishable. A similar μCT technique has already been successfully used to study concrete [24], steel corrosion [16,18,25,26], voids and defects in materials [27–29]. Monteiro et al. also highlighted the efficacy of μCT in quantifying issues concerned with the durability of concrete [30].

The aim of the experimental work was to use μCT technique for comparison of behaviour of carbon and ferritic stainless steels embedded in cement paste and exposed to accelerated corrosion by imposing high current density. Samples were galvanostatically corroded for 3 h with a constant anodic current of 5 mA cm^{-2} and were periodically analysed using μCT. The obtained 3D datasets, along with the polarisation curve, determined the mechanism of galvanostatic corrosion of steel inside concrete. A binary segmentation of the steel reinforcement helped to understand the kinetic of the corrosion pitting by computing its average and maximum penetrations, and comparing it with the penetration computed by the mass loss equation. Finally a detailed description of the corrosion product accumulation at the steel/cement paste interface and their further propagation through the corrosion-induced crack network was obtained. It shall be noted that corrosion process occurring due to the impressed current is very different from that occurring naturally by chloride-induced corrosion, as highlighted in details in the work by Poursaee and Hansson in [31]. Accelerating corrosion with impressed current can modify the corrosion mechanism, change the chemical conditions around reinforcing steel, alter the type of corrosion products formed, and induce high strain responses and crack widths [31–33]. Therefore, results presented in this paper and resulting conclusions should be limited to the comparison of corrosion behaviour of two grades of steel in cement paste under the same conditions of accelerated corrosion.

2. Materials and methods

2.1. Materials

The steel samples under investigation were composed of two different steels: carbon steel (ASTM A36), labelled CS, and low-nickel ferritic stainless steel (UNS S41000), labelled FSS. See Table 1 for a list of their elemental compositions.

Three samples for each combination of steel were prepared, 6 samples in total. #10 reinforcing bars were cut, thinned, and polished to create cylinders that were approximately 0.5 mm in diameter, and 5 mm in length. Isolated electrical wires were then connected to them to ensure the electrical connection. This connection between the steel and the wire was covered with impermeable paint to minimize the risk of corrosion of the wire and galvanic corrosion between the two (Fig. 1). The carbon steel specimen was 0.46 mm in diameter and had an exposed length of 3.5 mm; the ferritic stainless steel was 0.37 mm in diameter and had an exposed length of 3.6 mm. The specimens were placed into cylindrical moulds, 5 mm in diameter, and cement paste was poured into them. The cement paste was produced by mixing water with ordinary Portland cement type II at a water-to-cement ratio of 0.40. Once cement paste was poured in the mould, samples were placed for one day in humidity chamber (95% RH), to ensure sufficient humidity and minimize risk of shrinkage and consequent cracking. Next, the specimens were demoulded and subjected to additional curing in humidity chamber for 90 days.

It is important to highlight that the total size of samples was constrained by two things: the diameter of reinforcing rebar needed to be smaller than 0.5 mm in order to achieve optimal X-ray transmission through steel, and the thickness of concrete cover needed to be higher than 2 mm, in order to permit the pouring of the cement paste. For the purpose of this research numerous samples were prepared, and only not cracked samples, without gaps and pockets around reinforcement were used for further investigation.

2.2. Electrochemical cell

To conduct the electrochemical measurements, a three-electrode cell was used with the described reinforced cement paste sample used as working electrode, a Saturated Calomel Electrode (SCE) as reference electrode, and a stainless steel mesh surrounding the sample as counter electrode (Fig. 2). The samples were placed in a solution containing 3.5% per mass of sodium chloride to support the electron transfer. The end of the electrical wires was then electrically connected to a GAMRY PCI4/750 potentiostat/galvanostat. All potentials were measured with respect to the Saturated Calomel Electrode (SCE). The solution was considered saturated with respect to oxygen. Galvanostatic corrosion of the steel reinforcement was performed by imposing a constant anodic current density, i_G, of 5 mA cm^{-2} for similar lengths of time for the separate samples. The potential of the steels during galvanostatic corrosion, E_G, was measured with respect to the reference electrode to evaluate its corrosion evolution. The samples were taken out of solution at three different times, when a sudden decrease of potential was evident, and left at the open circuit potential during μCT imaging.

2.3. Micro X-ray computed tomography (μCT)

μCT experiments were conducted at beamline 8.3.2 of the Advanced Light Source at the Lawrence Berkeley National Laboratory [34]. The principle of X-ray tomography technique is to detect the intensity of the transmitted fraction of X-rays passing through the

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