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## Cement hydration and microstructure in concrete repairs with cementitious repair materials



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#### HIGHLIGHTS

- The moisture exchange has significant influence on the cement hydration and microstructure of interfaces in concrete repairs.
- The interface is of higher porosity than the repair material.
- The microstructure has a significant influence on the bond strength of the interfaces.

#### ARTICLE INFO

# Article history: Received 28 August 2014 Received in revised form 15 February 2016 Accepted 25 February 2016 Available online 5 March 2016

Keywords: Concrete repairs Interface Cement hydration Microstructure

#### ABSTRACT

One of the key parameters for the performance of concrete repairs is the quality of the interface between repair material and concrete substrate. To understand the properties of the interface in concrete repairs with cementitious repair materials, cement hydration and microstructure were experimentally studied using non-evaporable water test, mercury intrusion porosimetry, and scanning electron microscopy techniques. The experimental results reveal that the moisture exchange between repair material and concrete substrate results in a change of the moisture content in the repair material and thus affects the cement hydration process and pore structure development. Backscattered electron images demonstrate the existence of an interfacial transition zone of high porosity, since the cement particles of the repair material have a poor packing on the surface of concrete substrate.

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#### 1. Introduction

Civil infrastructure in developed countries is experiencing severe aging problem, which drives a rapid growth of repair, rehabilitation and retrofitting industry. In the United States, the annual cost for repair, maintenance and strengthening of the existing concrete bridges was estimated at 18–21 billion USD [1]. A statistical analysis estimated that around half of the European construction budget was spent on maintenance, rehabilitation and repair [2]. Due to the fast growth of concrete production since 1950s, the demand for repairs and associated costs may continue to grow [3]. However, the quality of concrete repairs is not satisfactory, and there is an urgent need for improving the performance of concrete repairs [4].

One of the key parameters for the performance of concrete repairs is the quality of the interface between repair material and concrete substrate [5]. After placing of a repair material on concrete substrate, the restrained shrinkage of repair material by concrete substrate often induces high tensile and shear stresses in repair systems, which may cause the debonding of interfaces [6]. Sufficient bond strength is, therefore, required to withstand the stresses at interfaces induced by environmental and/or mechanical loads. However, the interface is usually an inherently weak zone in repair systems, due to the incompatibility in physical, chemical and electrochemical properties between two materials [7].

The most previous studies focused on characterizing the bond strength between two materials using various test methods [8–14]. These studies revealed that the bond strength is mainly influence by the roughness and treatment method of substrate surface [8–11], the water content of concrete substrate [12], and the mix composition of repair materials [13,14]. The test methods used in the above studies have a limitation in providing information of failure zone and actual bond strength of interfaces. Since the failure often occurs in repair material or concrete substrate, the measured bond strength is actually the strength of the weakest point in repair systems, which is often not at interfaces.

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In order to investigate the mechanical properties of the interface itself, Lukovic et al. [15] have conducted an experimental study to characterize the local micromechanical properties within the interface zone by using nanoindentation technique. The experimental results of hardness and elastic modulus obtained from nanoindentation tests were used as input for simulating the fracture behavior of the interface by Delft Lattice model [16]. The simulation results give insight into fracture process and micromechanical response of the interface zone. This study revealed that the interface cannot be considered as a layer with uniform mechanical properties and consists of discrete zones with different properties.

For cementitious repair materials, cement hydration and microstructure play an important role in the mechanical properties of interfaces [17]. Courard [18,19] observed that the water content of concrete substrate has an influence on the bond strength of interfaces and attributed this phenomena to the changed cement hydration and microstructure development in repair systems due to the moisture exchange between repair material and concrete substrate. For concrete, cement particles have a poor packing on the surface of aggregates, resulting in an interfacial transition zone of high porosity [20]. Pigeon and Saucier [21] reported that the interface between repair material and concrete substrate is very similar to the interface between cement matrix and aggregates, and porous zone may also exist in the interface between repair material and concrete substrate, resulting in the interface having higher porosity than the repair material. However, the phenomena and its effect on the quality of the interface between repair material and concrete substrate have not been proven yet.

Understanding the cement hydration and microstructure of interfaces is critical to modifying the bond strength and to making durable concrete repairs. However, the knowledge of the cement hydration and microstructure of interfaces is lacking, which can be attributed to the limitations of proper test methods and difficulties in characterizing constitutive parameters in microscale. This paper presents an experimental study on investigating the cement hydration and microstructure in concrete repairs with cementitious repair materials. Although many polymer-modified repair materials have been developed, this group of repair materials is beyond the scope of this research. Hereinafter, "repair material" refers to cementitious repair materials, and "interface" refers to the interface between cementitious repair material and concrete substrate.

#### 2. Materials and methods

In the experimental program, non-evaporable water analysis and mercury intrusion porosimetry (MIP) were used to measure degree of hydration and porosity, respectively. Scanning electron microscopy (SEM) study was used to characterize the microstructure in the interface. Backscattered electron (BSE) image analysis was also conducted to evaluate the degree of hydration and the porosity in the interface and in the repair material.

**Table 1**Chemical composition of Portland cement CEM I 42.5N.

Composition	CaO	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>
Weight [%]	64.1	20.1	4.8	3.2	1.2	0.5	0.3	2.7

#### 2.1. Repair material

Cement paste is the essential ingredient in cementitious repair materials, and its properties govern the cement hydration and microstructure in the interface and the repair material and also the bond strength. In order to simplify the tests and to avoid the uncertainties and difficulties in experimental result analysis due to the presence of aggregates, Portland cement paste with a w/c ratio of 0.40 was used as a repair material. The chemical composition of Portland cement CEM I 42.5N is given in Table 1.

#### 2.2. Concrete substrate

The substrate concrete was a 3-year-old concrete, and its mix composition is given in Table 2. The concrete substrate specimen was a cylinder, 60 mm in diameter and 60 mm in thickness, with a flat surface for casting the repair material. To get concrete substrate with different moisture content, before casting the repair material two groups of substrate specimens were placed in rooms with 50% relative humidity (RH) and 99.9% RH for 7 days, respectively.

The degree of saturation of the substrate specimens was determined. The weight of specimens was first measured and recorded as  $W_i$  [g]. The specimens were vacuum-saturated for 6 h and the weight of the saturated specimen  $W_s$  [g] was measured. The specimens were then dried in an oven with a temperature of  $105\,^{\circ}\mathrm{C}$  for 24 h. The weight of dried specimens was recorded as  $W_d$  [g]. The degree of saturation  $S_w$  [–] was calculated as follows:

$$S_w = \frac{W_i - W_d}{W_s - W_d} \tag{1}$$

The substrate specimens placed in a room with 99.9% RH become almost fully saturated and had a degree of saturation of 99.0%, while the substrate specimens placed in a room with 50% RH had a degree of saturation of 14.4%.

#### 2.3. Casting and curing of the repair material

Before casting the repair material, the top perimeter of the concrete substrate was encircled by adhesive tape as shown in Fig. 1(a) and (b), in order to avoid the leakage of the repair material from the gap between the substrate and the mold. The specimens were then enclosed with two pieces of PVC pipes as shown in Fig. 1 (c), and the PVC pipes were tightened as shown in Fig. 1(d).

The repair material was mixed with a HOBART® mixer at low speed for 2 min and at high speed for another 2 min. The fresh repair material was cast on the prepared substrate specimens, reaching a thickness of 60 mm. The specimens were then vibrated to remove air bubbles from the repair material. A plain Portland cement paste with a thickness of 60 mm was cast as a reference for non-evaporable water test and MIP. All specimens were cured at a temperature of 20 °C and in a sealed condition for 28 days.

#### 2.4. Sample preparation for non-evaporable water test, MIP and SEM

The sampling for non-evaporable water test, MIP and SEM is shown in Fig. 2. The samples for MIP and SEM were sawn into the size of about  $5\times10\times10~\text{mm}^3$  and  $10\times10\times50~\text{mm}^3$ , respectively. Freeze-drying was used to dry the samples. The samples were quickly frozen by immersion in liquid nitrogen for 5 min and were placed in a freeze-dryer with a temperature of  $-24\,^\circ\text{C}$  and a pressure of 0.1 Pa. Until the water loss decreased to 0.01% per day, the samples were considered to be fully dried. For non-evaporable water test, the samples were further ground into powder.

For SEM, the freeze-dried samples were further prepared in the following procedures: epoxy impregnation, grinding and polishing. The dried samples were placed in a chamber, evacuated for 4 h. Keeping vacuum, the freshly mixed epoxy resin was poured on the samples. The chamber was evacuated for another 5 min. Air was then slowly released into the chamber to force the epoxy resin to impregnate the samples. After the epoxy resin hardened, the samples were finely ground in a grinding machine, which can control the ground thickness by 7  $\mu m$ . Each sample was ground for about 5 min until a 50  $\mu m$ -thick surface was removed. The last grinding was done on a middle-speed lap wheel with a p4000 sand paper for 2 min. Then, the samples were polished on lap wheels with 6, 3, 1 and 0.25  $\mu m$  diamond pastes for 8 min (2 min for each step). Last, the samples were cleaned with a low-relief polishing cloth.

**Table 2**Mix composition of the substrate concrete.

Coarse aggregate [kg/m <sup>3</sup> ]	Fine aggregate [kg/m³]	CEM III/B 42.5 [kg/m <sup>3</sup> ]	Water [kg/m³]	w/c ratio [-]	Maximum aggregate size [mm]
1177.2	633.9	375	187.5	0.5	16

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