Microstructure of the Interface in Concrete Repairs

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ABSTRACT: A key parameter of the performance of concrete repairs is the bond strength between the repair material and the concrete substrate. Sufficient bond strength is required to withstand the stresses at the interface induced by the environmental and/or mechanical loads. The bond strength is determined by the microstructure of the repair-substrate interface. The microstructure development of the repair-substrate interface is influenced by the moisture exchange between the repair material and the concrete substrate and the “wall effect”. To understand the fundamental bond mechanisms, the microstructure development in concrete repairs are experimentally investigated using the scanning electron microscopy. The experimental results reveal that when the concrete substrate has lower water content, it absorbs more water from the repair material and results in a lower porosity both in the interface and in the repair material. Due to the “wall effect”, the cement particles have a poor packing at the repair-substrate interface and the w/c ratio locally increases. The increased w/c ratio results in a porous interfacial zone and poor mechanical properties.

1 INTRODUCTION

The microstructure of the repair-substrate interface determines its bond strength. In concrete repairs, the cement hydration and the microstructure development is often influenced by the moisture exchange between the repair material and the concrete substrate [Courard (2005)]. After the repair material is poured on the concrete substrate, the concrete substrate absorbs water from the repair material. This causes a water loss in the repair material. The water loss in the repair material results in a decrease in the degree of hydration and a decrease in the porosity. Simultaneously, the pore structure also influences the moisture exchange.

Apart from the moisture exchange, the so-called “wall effect” also has influence on the microstructure development. The “wall effect” is a well-known phenomenon at the interface between cement matrix and aggregate [Larbi (1992)]. Due to the “wall effect”, the interfacial zone between cement paste and aggregates has a loose packing of cement particles, and the w/c ratio of cement paste locally increases. Therefore, the cement matrix-aggregate interfacial zone shows a high porosity and poor microstructure. Pigeon and Saucier [Pigeon and Saucier (1992)] reported that the repair-substrate interface is very similar to the aggregate-cement matrix interface depending very much on the type of aggregates in the substrate. The “wall effect” also exists at the repair-substrate interface and causes a porous microstructure. Therefore, it can be expected that the interface is a weak point in concrete repairs.

This paper presents an experimental study on the microstructure development of the repair-substrate interface. In order to interpret the bond mechanism between the repair material and the concrete substrate, the cement hydration and the microstructure development in concrete repairs are investigated by the scanning electron microscopy (SEM) observation. Uniaxial tensile test was used to measure the tensile bond strength.
2 MATERIALS AND METHODS

2.1 Preparation of the concrete substrate

The concrete substrate was a 3-year-old concrete. The mix proportion of the concrete substrate is given in Table 1.

Table 1 Mix proportion of the concrete substrate

<table>
<thead>
<tr>
<th>Coarse aggregate [kg/m³]</th>
<th>Fine aggregate [kg/m³]</th>
<th>Cement [kg/m³]</th>
<th>Water [kg/m³]</th>
<th>w/c ratio</th>
<th>Maximum aggregate size [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1177.2</td>
<td>633.9</td>
<td>375</td>
<td>187.5</td>
<td>0.5</td>
<td>16</td>
</tr>
</tbody>
</table>

Two techniques were used to roughen the surface of the concrete substrates:
- Cutting (as shown in Figure 1 (a)), which was used to simulate the untreated surface of substrates.
- Grit-blasting (as shown in Figure 1 (b)). The injecting air pressure was 0.8 MPa, and the blasting speed on the surface of the concrete substrate was 10 cm²/minute.

![a. Cut surface](image1.png) ![b. Grit-blasted surface](image2.png)

Figure 1 Substrate specimens with (a) a cut surface and (b) a blasted surface.

After the surface treatment, the substrate specimens were placed in two comparable moisture conditions for 7 days, i.e. 50% relative humidity (RH) and 99.9% RH, to obtain the unsaturated and saturated concrete substrates, respectively. The preparations of the concrete substrates are summarized in Table 2.

Table 2. Preparation of the concrete substrates

<table>
<thead>
<tr>
<th>Concrete substrate</th>
<th>Surface treatment</th>
<th>Moisture condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>Cutting</td>
<td>50% RH</td>
</tr>
<tr>
<td>C2</td>
<td>Grit-blasting</td>
<td>50% RH</td>
</tr>
<tr>
<td>C3</td>
<td>Cutting</td>
<td>99.9% RH</td>
</tr>
</tbody>
</table>

2.2 Repair material

In this study, an ordinary Portland cement (CEM I 42.5N) paste with a w/c ratio of 0.4 was used as repair material.

2.3 Sample preparation

The substrate specimen was a cylinder with a diameter of 60 mm and thickness of 60 mm. Before casting the repair material, the top perimeter of the concrete substrate was encircled by
adhesive tape to avoid the leakage of the mixing water from the gap between the specimens and the molds as shown in Figure 2 (a and b). The specimens were then enclosed in two pieces of half PVC pipes as shown in Figure 2 (c). The PVC pipes were tightened by adhesive tape as shown in Figure 2 (d). The fresh repair material was cast on the prepared surface of the substrate specimen, and the thickness of the repair material was 60 mm. The specimen was then vibrated to remove air bubbles from the repair material. The specimens were cured at a temperature of 20°C and in a sealed condition for 28 days.

![Figure 2 Preparation for casting the repair material.](image)

2.4 **SEM observation**

After curing, the samples for SEM test were taken from the specimens at the interface. The samples were sawn into pieces of $4 \times 2 \times 1 \text{ cm}^3$. The freeze-drying was used to dry the samples, since this method causes less damage on the microstructure of cement paste compared with the other common drying methods, such as oven drying and vacuum drying [Ye (2003)]. The samples were quickly frozen by immersing in liquid nitrogen for 5 minutes. Then, they were placed in a freeze-dryer with a temperature of -24°C and a vacuum of 0.1 Pa. The samples were considered to be dry, until the water loss was less than 0.01%/day. This period lasted for 10-20 days depending on the w/c ratio and the curing age of the samples.

The dry specimens were firstly pre-ground in a grinding machine and freeze-dried. The dry specimens were then epoxy-impregnated. The dried specimens were placed in a chamber and the chamber was evacuated for 4 hours. Keeping under vacuum, the freshly mixed epoxy resin was poured on the prepared surfaces. The chamber was still evacuated for another 5 minutes. Then, air was slowly released into the chamber to force the epoxy resin impregnate into the specimens.

The penetration depth, varying with the pore structure of the samples and the viscosity of epoxy resin [Diamond (2007)], is often very small, particularly in the samples with a low w/c ratio. Kjellsen et al (2003) reported that the penetration depth in a 0.4-w/c-ratio cement paste was only 120 μm. Therefore, grinding and polishing should be done very carefully and not beyond the epoxy penetration depth. The samples were finely ground in a grinding machine, which can control the ground thickness by 7 μm. Each sample was ground for about 5 minutes until a 50 μm-thick surface was removed. The last grinding was done on a middle-speed lap wheel with a p4000 sand paper for 2 minutes. Then the samples were polished on lap wheels with 6, 3, 1 and 0.25 μm diamond pastes for 8 minutes (2 minutes for each step). Last, the samples were cleaned with a low-relief polishing cloth.

The images were taken on the prepared samples using the backscattered electron (BSE) detector with the vapor mode. The acceleration voltage of 20 kV was used in order to obtain high-contrast images. The spot size was 4.0 nm and the magnification was 500×. The working
distance between the final condenser lens and the specimen was 10 mm. The pressure inside the chamber was set at 0.5 Torr.

2.5 BSE image analysis

BSE observation can provide two-dimensional information of the microstructure. A typical BSE image of hardening Portland cement paste is shown in Figure 3 (a). The main phases in microstructure of the cement paste, i.e. pores, hydration products and unhydrated cement, can be distinguished by their grey levels as shown in Figure 3 (b). Since the pores are filled by epoxy and epoxy does not scatter electrons, the pore space appears black. Hydration products appear grey, and unhydrated cement particles appear bright. With a commercial image processing program, the area of each phase can be determined by counting the number of pixels. The number of pixels of the pore space and the unhydrated cement is first counted, since they can easily be distinguished from the other phases. Then, the number of pixels of the hydration products is calculated by subtracting the pixels of the former two phases from the total pixels. According to Ye (2003), more than 12 images are needed for BSE image analysis.

According to the stereology theory [Underwood (1970), Diamond and Leeman (1995)], the three-dimensional information of microstructure can be derived from the two-dimensional BSE images. The volume fraction of each phase is assumed to be equal to their area fraction in the BSE images. The porosity $\phi$ can be calculated by dividing the number of pixels of the pores $n_{\text{pore}}$ [-] by the number of total pixels $n_{\text{total}}$ [-]:

$$\phi = \frac{n_{\text{pore}}}{n_{\text{total}}}$$  \hspace{1cm} (1)

According to Powers’ model [Powers and Brownyard (1946)], for Portland cement the hydration products have a volume 2.1 times larger than that of the hydrated cement particles. Therefore, the number of pixels of the hydrated cement $n_{hc}$ [-] holds:

$$n_{hc} = \frac{n_{hp}}{2.1}$$  \hspace{1cm} (2)

where $n_{hp}$ [-] is the number of pixels of the hydration products. The degree of hydration can be calculated by dividing the pixel of the hydrated cement particles by the total number of the pixels of the cement particles before hydration:

$$\alpha = \frac{n_{hp}/2.1}{n_{hp}/2.1 + n_{uc}}$$  \hspace{1cm} (3)

where $n_{uc}$ [-] is the number of pixels of the unhydrated cement.

![Figure 3 Illustration of pore space, hydration products (HP) and unhydrated cement (UC) in (a) a typical BSE image and (b) its grey level histogram.](image)
The w/c ratio $\omega$ can also be calculated as follows:

$$
\omega = \frac{W_{w,\text{in}}}{W_{c,\text{in}}} = \frac{n_{\text{total}} - n_{\text{hp}} / 2.1 - n_{w}}{n_{hp} / 2.1 + n_{w}} \rho_w
$$

where $W_{w,\text{in}} [\text{g}]$ is the initial weight of water, $W_{c,\text{in}} [\text{g}]$ is the initial weight of Portland cement, $\rho_w$ is the density of water (1 g/cm$^3$) and $\rho_c$ is the density of Portland cement (3.15 g/cm$^3$).

2.6 Tensile bond strength test

The bond strength of the repair-substrate interface was measured by a direct tensile test according to European standard EN 14488-4 (2002). The tested specimens were layered cylinders with a diameter of 60 mm. The thicknesses of the repair material and the concrete substrate were both 60 mm. After 28-day curing, the specimens were then glued on the steel dollies, which were fixed on the loading device, as shown in Figure 4. After the glue hardened, an increasing tensile load was applied on the specimens, and the loading speed was controlled to be 0.05 MPa/s. The tensile load and the corresponding displacement were recorded by computer automatically. The tensile bond strength was calculated as the maximum load divided by the cross-sectional area. Three parallel measurements were done.

![Figure 4 Test set-up of direct tensile tests.](image)

3 EXPERIMENTAL RESULTS

3.1 Results of SEM observation

Figure 5 (a), (b) and (c) show the BSE images taken at the repair-substrate interface. From the BSE images, the repair material and the concrete substrate can be distinguished by their microstructures. The repair material, on the left hand side of the images, contains more unhydrated cement (white space) and more porosity (black space) than the concrete substrate, on the right hand side of the images. Adjacent to the concrete substrate, there is an interfacial transition zone with relatively more black space. This indicates that this interfacial transition zone has a higher porosity and probably lower strength.

Figure 5 (a) and (c) show that the cut concrete substrate results in a smooth surface. It can be expected that the smooth surface does not contribute to the mechanical interlocking between the repair material and the concrete substrate. For a blasted concrete substrate, the repair material can be embedded in the concrete substrate as shown in Figure 5 (b). Therefore, the repair material and the concrete substrate might have a strong mechanical interlocking.

Table 3 gives the degree of hydration ($\alpha$), porosities ($\phi$) and w/c ratios ($\omega$) at the repair-substrate interface and in the repair material obtained by BSE image analysis. The porosity in the repair material measured by the BSE image analysis is lower than that measured by the MIP test [Zhou (2011)].
The lower porosity obtained by BSE image analysis can be caused by the limitation of image resolution because the pores with a diameter smaller than 0.25 µm are not detectable in the image analysis. In the three specimens, the degree of hydration, porosity and w/c ratio at the repair-substrate interface are all higher than those in the repair material. Due to the “wall effect” [Bijen and Salet (1994)], cement particles have a poor packing on the surface of the concrete substrates. The poor packing causes a lower volume fraction of cement particles at the repair-substrate interface, and the repair-substrate interface, therefore, shows a w/c ratio around 48% (on average) higher than in the repair material as given in Table 4. The high w/c ratio results in a high porosity and a high degree of hydration.

No matter in the repair material or at the repair-substrate interface, the saturated concrete substrate results in a higher porosity, a higher degree of hydration and a higher w/c ratio compared with the unsaturated concrete substrate, while the surface treatment does not have a significant influence on these data. This is in a good agreement with the observation from non-evaporable water test and MIP test [Zhou (2011)].

Table 3 Degree of hydration $\alpha$, porosities $\phi$ and w/c ratios $\omega$ at the repair-substrate interface (20 µm wide) and the repair material at 28 days obtained by BSE image analysis.

<table>
<thead>
<tr>
<th>Concrete substrate</th>
<th>Repair-substrate interface</th>
<th>Repair material</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\alpha$ [%]</td>
<td>$\phi$ [%]</td>
</tr>
<tr>
<td>C1 (cut, unsaturated)</td>
<td>76.4 ± 2.3</td>
<td>24.0 ± 3.7</td>
</tr>
<tr>
<td>C2 (blasted, unsaturated)</td>
<td>78.0 ± 4.5</td>
<td>24.4 ± 3.0</td>
</tr>
<tr>
<td>C3 (cut, saturated)</td>
<td>80.8 ± 3.3</td>
<td>30.6 ± 2.3</td>
</tr>
</tbody>
</table>

Table 4 Proportions of the data at the repair-substrate interface to those in the repair material.

<table>
<thead>
<tr>
<th>Concrete substrate</th>
<th>$\alpha$</th>
<th>$\phi$</th>
<th>$\Omega$</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1 (cut, unsaturated)</td>
<td>1.17</td>
<td>1.82</td>
<td>1.46</td>
</tr>
<tr>
<td>C2 (blasted, unsaturated)</td>
<td>1.17</td>
<td>1.87</td>
<td>1.47</td>
</tr>
<tr>
<td>C3 (cut, saturated)</td>
<td>1.14</td>
<td>1.81</td>
<td>1.51</td>
</tr>
</tbody>
</table>
3.2 Bond strength of the repair-substrate interface

Table 5 gives the results of the bond strength between the repair material and the concrete substrate. Each value is the average result of three parallel measurements. For the specimens with the unsaturated substrate, the blasted substrate results in higher bond strength than the cut substrate. For the specimens with the cut substrate, the bond strength of the specimen with the unsaturated concrete substrate is higher than that with the saturated concrete substrate. Under uniaxial tensile load, the specimens with the cut substrates all failed at the repair-substrate interface as shown in Figure 6 (a) and (c), while those with blasted substrates failed in the repair material as shown in Figure 6 (b). It implies that the cut concrete substrate results in a poor interface, which is the weakest point in the specimens. For the specimens with the blasted substrate, the measured bond strength is the tensile strength of the repair material, and the real interfacial bond strength should be higher than this.

Table 5 Bond strength of the repair-substrate interface.

<table>
<thead>
<tr>
<th>Concrete substrate</th>
<th>Bond strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1 (cut, unsaturated)</td>
<td>1.13 ± 0.27</td>
</tr>
<tr>
<td>C2 (blasted, unsaturated)</td>
<td>1.64 ± 0.24</td>
</tr>
<tr>
<td>C3 (cut, saturated)</td>
<td>0.92 ± 0.23</td>
</tr>
</tbody>
</table>

Figure 6 Failure modes of the specimens under uniaxial tensile load.

4 DISCUSSION

4.1 Influence of the water content of the concrete substrate

When the concrete substrate has lower water content, it can absorb more water from the repair material, and the water absorption causes a bigger decrease in the w/c ratio of the repair material. The lower resultant w/c ratio has two effects on properties of the repair material. Firstly, it decelerates the cement hydration and lowers the degree of hydration. Secondly, a lower w/c ratio leads to a lower porosity and a denser pore structure.

The w/c ratio is a crucial parameter determining the ultimate strength of cementitious materials. At the same age, a lower w/c ratio leads to a higher tensile strength and, therefore, a higher bond strength. When the concrete substrate is dry, it absorbs more water from the repair material and causes a larger reduction in w/c ratio. As a result, the unsaturated substrate results in a higher bond strength, although repair material has a lower degree of hydration.
4.2 The “wall effect”

Due to the “wall effect”, cement particles have a poor packing on the surface of the concrete substrate. The w/c ratio of the repair material locally increases at the repair-substrate interface, which contains relatively more pore space. The main hydration product, calcium-silicate hydrate, forms on the original cement particles and slowly grows to the pore space [van Breugel (1991)]. Therefore, the pore space at the repair-substrate interface is more difficult to fill up with the hydration product. As a result, the repair-substrate interfacial zone has high porosity and low strength.

5 CONCLUSIONS

The microstructural development and the bond strength in concrete repair system were studied experimentally. Based on the experimental results and discussion, the following conclusions can be drawn:
- When the concrete substrate has a lower water content, it absorbs more water from the fresh repair material and causes a bigger decrease in w/c ratio of the repair material. This results in a decrease in degree of hydration and a decrease in porosity.
- When the concrete substrate is saturated, it can provide additional water for the hydration of the repair material. The degree of hydration in the repair material, therefore, increases.
- Due to the “wall effect”, the cement particles have a poor packing at the repair-substrate interface and the w/c ratio locally increases. The interface has relatively high porosity and high degree of hydration than the repair material.
- The surface preparation has a big influence on the bond strength. The grit-blasting results in a higher bond strength than the cutting.

6 REFERENCE

EN 14488-4 (2002), Testing Sprayed Concrete – Part 4: Bond Strength of Cores by Direct Tension.
Powers, T.C. and Brownyard, T.L. (1946), Studies of the physical properties of hardened Portland cement pastes, Journal of American Concrete Institute 18(2): 101-132;