Effect of Nano-sio$_2$ on Crack Width in ITZ (SEM Observation)

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ABSTRACT

The traditional picture of the interfacial transition zone (ITZ) in concrete involves an approximately 30µm zone around each aggregate, within which the porosity increases as the aggregate interface is approached. Effect of nano-sio$_2$ on cracks width in ITZ was studied in this research. For investigation of crack width, SE signals of SEM were used. The samples were cut from edges and middle of 5 × 5 × 5 cm cubic specimens. It was seen that nano-sio$_2$ could less the crack width. It is for the first time that SE use for ITZ imaging.

KEYWORDS: ITZ; crack width; nano-sio$_2$; SEM observation.

INTRODUCTION

The concept that an interfacial transition zone (ITZ) or "aureole de transition" exist around sand and coarse aggregate particles in concrete has been one of the accepted tenets of concrete technology for many years. This generally accepted picture was based originally on optical microscopy and on experimental observations made on "model systems" rather than on concrete perse. In essence, the argument is that a region, extending about 30 µm or more from the aggregate, is deficient in content of cement particles due to the so-called wall effect, and therefore has a substantially higher porosity than the bulk paste [1]. A number of models of the ITZ exist. While some models assume that the ITZ can be viewed as a uniform shell, it is generally assumed that there is a gradation of porosity and other characteristics within the aureole. A very high porosity, ca 30% or more, is assumed to be characteristic of the innermost portion of the aureole, i.e., within 5 µm of the aggregate surface [2]. At the outer boundary, porosity and other characteristics are considered to merge into those of "bulk" cement paste. The content of CH and of ettringite are usually considered to be higher within the aureole than in the bulk cement paste, and much of the CH is considered to be preferentially oriented [3].

For concrete in which adjacent aureoles overlap significantly, it has been claimed that a continuous easy percolation path can be established across the section, leading to high permeability and rapid diffusion of ions and other dissolved species [4]. The aureoles are perceived as weakening zones which limit the strength that would otherwise be developed in concrete. One of the reasons is that the amount of unhydrated cement is less than other part of bulk matrix. High performance concretes are thought to derive augmented strength levels partly from a filler effect, in which fine particles such as silica fume fill up the extra ITZ porosity that would otherwise exist, and by doing so eliminate the weakening effect [5]. In most previous researches ITZ images were carried out by back scattered electron (BSE) signals, but here secondary electron signal (SE) used for investigation. It was because of better crack shown in SE images.

Diagram 1: Average area percent of unhydrated cement vs distance from the aggregate for a three-day-old well-mixed concrete made with dolomite sand and coarse aggregate [1].

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We have carried out an extensive series of investigations to attempt to establish the degree to which this generally accepted picture of the ITZ is valid for ordinary concrete. Furthermore especially cracks width in ITZ has been considered and effects of nano-sio2 on cracks were investigated. It has been thought that nano particles can increase cohesion and fill the very small cracks.

**MATERIALS AND EXPERIMENTAL PROCEDUES**

In this study the concrete mixtures were prepared with two type of ordinary and nano-sio2 contained. The standard grain distributed sand was used. mix proportion of the concrete is given in table 1. 5X5X5cm specimens were prepared. For more precision 3 specimens for each case were made. The samples for SEM observation were cut from middle and edge of specimens. The microcrack initiation and propagation were analyzed by interpreting the 7-days compressive strength.

**Table 1: Mix proportion of concrete**

<table>
<thead>
<tr>
<th>Ingredients</th>
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<tr>
<td>Sand</td>
<td>1800</td>
</tr>
<tr>
<td>Cement</td>
<td>600</td>
</tr>
<tr>
<td>water</td>
<td>300</td>
</tr>
<tr>
<td>Nano-sio2</td>
<td>60</td>
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</tbody>
</table>

Briefly our procedure involved preparation of the specimens as ordinary done for secondary SEM investigations. In such procedures thin slices are cut from bulk concrete. The slices are dried at low temperature and covered with gold. As the ITZ is an aureole has 30µm width around the aggregate, the images were getting from the aureole. The goal was investigation of effect of nano-sio2 to reduction of cracks width in ITZ.

**Test results and discussion**

The effect of nano-sio2 on microcrack initiation and width can be explained by investigation of ITZ of the samples. Figure 1 shows a crack in middle sample with 1000X magnification. The crack is occurred in ITZ of cylindrical sand grain. The specimen had not nano-sio2. It is seen that matrix is separated from sand grain completely and leads to great crack in ITZ (9.5µm width – about one third of ITZ width). In 5000X magnification of previous sand grain (fig 2), a hole is seeable in ITZ which can be filled with nano materials.

Fig 1: Secondary electron image of ordinary specimens without nano-sio2. The crack width in ITZ region of a sand grain is 9.5µm. (1000X)
Figure 2 shows an image of a previous sand grain. The hole is visible at the end of the crack. (5000×)

Figure 3 shows an image from ITZ cracks in a sample which contained nano-sio2 and separated from the middle of the specimen. A flat surface of sand grain and ITZ crack is visible in this 1000× image. Nano materials could decrease the crack width. There is only one crack occurred in the sample which is in ITZ. Thus ITZ is the most important region for cracks.

As predicted before nano material could fill the crack in ITZ. Figure 4 shows the previous crack (showed in fig 3) in 5000× magnification. The crack width is 1µm. it means that nano-sio2 could less the crack width in ITZ about ten times.
Fig 4: Secondary electron image of crack around previous sand grain. Nano material could decrease the crack width about 10 times. (5000×)

Comparing figure 2 and 4 shows the better cohesion between matrix and sand is in samples which contain nano-sio₂. For consideration of the effect of sampling, samples were cut from edges of specimen. For SEM observation these sample were simpler to imaging Because one dimension of the sample has flat surface.

Fig 5: Secondary electron image of crack around sand grain. The sample was edge cut without Nano-sio₂. (1000×)

It is shown that the crack diameter is about 18µm. By zooming in the ITZ crack of figure 5, interesting details of an ITZ crack will be visible. Figure 6 shows these details of the crack of previous sample with higher magnification. The sample has not nano-sio₂.

Fig 6: Zoom in previous crack around sand grain. The sample was edge cut without Nano-sio₂. (5000×)
Figure 7 shows an ITZ crack in edge cut sample which contains nano-sio$_2$. Comparing this crack with figure 6, shows that crack width has been decreased. The crack width is about 1.4µm as shown in figure7. Therefore using nano-sio$_2$ leads to decreasing the crack width about 10 times.

![Image of ITZ crack in edge cut sample](image)

**Conclusions**

1- Interfacial transition zone (ITZ) can be observed with SE images of SEM as a volumetric 30µm aureole around the sand grains. This is the most sensitive region for failure. All less 30µm cracks around sand grains are in ITZ.

2- Most of microcracks occurred in ITZ whether the sample cut from the edge of specimen or middle, except edge sample observation is simpler because they have at least one flat surface.

3- By using nano-sio$_2$ crack width in ITZ will be decreased about 10 times. This reduction will lead to prevent or delay greater cracks. Furthermore specimen with smaller ITZ cracks shows more compressive strength.

4- Nano-sio$_2$ is so small to play role of filler in micro cracks, but it can increase cohesion of matrix and grain sand. On the other hand by using nano-sio$_2$ more cohesive and stronger concrete will be produced.

**REFERENCES**

1- Diamond, S and Huang, J. "The ITZ in concrete – A different view based on image analysis and SEM observation" Cement & Concrete Composites 23 (2001) 179-188.


