X-RAY COMPUTER TOMOGRAPHY STUDIES OF MESO-DEFECTS EVOLUTION OF UHPCC DUE TO FREEZING-THAWING ACTION

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Abstract

X-ray computed tomography (X-ray CT), as a non-destructive test, was used to analyze the evolution of three dimensional (3D) meso-defects in ultra-high performance cementitious composite (UHPCC) due to freezing-thawing action. It can be found that the amount of meso-defects almost doubled in UHPCC after 800 freezing-thawing cycles. The smaller meso-defects increased more significantly than the bigger ones. The mass loss and relative dynamic modulus of elasticity versus numbers of freezing-thawing cycles were also tested. It is indicated that the freezing-thawing resistance of UHPCC with steel fibers is better than that of UHPCC without fibers.

1. INTRODUCTION

UHPCC is one novel type of composite material which was invented in the early 1990s by Bouvgues’ laboratory in France [1]. UHPCC possesses ultra-high strength and excellent durability [2, 3], which make it have great prospects in hydraulic structures, offshore structures, explosion and penetration resistant structures [4].

UHPCC can also be used in some cold region because of its great resistance to freezing-thawing damage. For normal concrete structures in cold region, the freezing-thawing action is one of the most significant causes of deterioration, and has shortened the service life of many structures like railways and bridges.

Concrete that is not resistant to cyclic freezing-thawing action manifests two principal types of damage, namely surface scaling and internal cracking. Several methods have been developed to observe and quantify these types of damage caused by freezing-thawing action. Normally, the scaling can be evaluated by the mass loss, and the internal cracking can be reflected by the change of relative dynamic modulus of elasticity. Scanning electron microscopy(SEM) and backscattered electron (BSE) are also widely used to characterize the internal cracks and pore structure in the mortar or concrete [5]. However, it is extremely complicated to get the 3D structure information from the 2D images. And specimen preparation for SEM-BSE test may lead to possible artifacts.
In this paper, X-ray computed tomography was used to investigate the 3D meso-defects of UHPCC exposed to freezing-thawing action. X-ray computed tomography (X-ray CT) is a noninvasive and nondestructive method which can get 3D microstructure information of cementitious materials without any prior specimen preparation. Recently X-ray computed tomography has been applied by various researchers in cement based materials [6-8]. However, little is known about application of this technique on UHPCC exposed to freezing-thawing action. The purpose of this study is to reveal the meso-defects evolution of UHPCC under freezing-thawing environments with the help of X-ray CT.

2. MATERIALS AND METHODS

2.1 Sample preparation

Two types of materials were prepared for this research, named as UHPCC-1 and UHPCC-2. They had the same matrix, while UHPCC-2 was reinforced with steel fibers.

Three cementitious materials were used in this study, including Portland cement with 28 days of compressive strength of 64.5 MPa, Class F fly ash and silica fume. The mass fraction of FA and SF is 40% and 10% of total binder mass, respectively. The superplasticizer we used is a kind of liquid agent with 28% solid content. Its dosage used was about 3.5% of total binder mass. The maximum particle size of natural sands was 2.36 mm. The water-binder and sand-binder ratio were 0.16 and 1.2, respectively. The volume fraction of steel fibers in UHPCC-2 was 2%, and the diameter, length and tensile strength of the fine steel fibers were 0.2 mm, 13 mm and 1800 MPa, respectively.

The specimens were cased in steel molds with a size of 40mm×40mm×160m. After 24 hours, they were demoded and cured in a standard condition (20°C, RH>95%) for 90 days before tested.

2.2 Mechanical test

Specimens for mechanical tests were 40 mm×40mm×160mm prisms. Flexural strength and compressive strength were tested according to Chinese standard GB177-85. At first, the three-point bending test was performed to obtain flexural strength. After bending test, the broken specimens with sizes of approximately 40mm×40mm×80mm were used to conduct compressive test. Three samples of each batch were tested. The average values were served as the final flexural strength and compressive strength.

2.3 Freezing-thawing test

The freezing-thawing test was performed according to the Chinese standard GB/T 50082-2009. The temperature of a freezing-thawing cycle was between -20°C ~20°C, and one cycle took about 4 hours. After a specified number of freezing-thawing cycles, the specimens were moved out from the test box. Then the mass and relative dynamic modulus of elasticity were tested.

2.4 X-ray computed tomography

In the present work, all samples were examined visually using Y.CT Precision System (YXLON, Germany), and the voltage and current of the X-ray tube were 195 kV and 0.41 mA, respectively. The detector type was flat panel Y.XRD 0820, and the numbers of detector elements was 1024. The number of projections was 1080. The object rotation angles were 360
degree. The 2D pixel size was 86µm×86µm, pixel numbers were 1024×1024. The 3D voxel size was 86µm×86µm×86µm, and the density resolution was 0.3%.

3. RESULTS AND DISCUSSION

3.1 Mechanical properties

The compressive strength and flexural strength of UHPCC-1 and UHPCC-2 after 90d standard curing are given in table 1.

Table 1: Compressive strength and flexural strength of UHPCC-1 and UHPCC-2

<table>
<thead>
<tr>
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<th>Compressive strength (MPa)</th>
<th>Flexural strength (MPa)</th>
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<tbody>
<tr>
<td>UHPCC-1</td>
<td>108.2</td>
<td>15.0</td>
</tr>
<tr>
<td>UHPCC-2</td>
<td>156.1</td>
<td>30.7</td>
</tr>
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</table>

It can be observed that the compressive strength and flexural strength of UHPCC-2 are much higher than that of UHPCC-1, the small-sized steel fibers evidently improved the mechanical properties of UHPCC.

When curing age increases, the pozzolanic activity of mineral admixture develops completely and the interfacial zone is improved continuously. As the bond strength between matrix and fiber increases, the mechanical performance of UHPCC improves.

3.2 Freezing-thawing performance

The results of the freezing-thawing test show that well-prepared UHPCC exhibit great resistance to freezing-thawing action.

Figure 1 shows the mass loss of UHPCC-1 and UHPCC-2 as a function of freezing-thawing cycles. Early in the freezing-thawing cycles, the mass loss of UHPCC-1 and UHPCC-2 had negative growth because of water absorption. Then the mass loss increased slightly along with the increase of freezing-thawing cycles. After enduring 800 freezing-thawing cycles, the mass loss of UHPCC is less than 1%, as well as that of UHPCC-2 is controlled within 0.5%. The spalling of the surface is the main course of mass loss.

![Figure 1: Mass loss of UHPCC at different freezing-thawing cycles](image1)

![Figure 2: Relative dynamic modulus of elastic of UHPCC at different freezing-thawing cycles](image2)
Figure 2 is a plot of the variation of relative dynamic modulus of elasticity during the process of freezing-thawing tests. The relative dynamic modulus of elasticity of both UHPCC-1 and UHPCC-2 were nearly constant with the increasing of the cycles until it reached 350 cycles. It suggests that the materials did not show any significant internal deterioration then. After 800 freezing-thawing cycles, the relative dynamic modulus of elasticity are approximately 10% and 5% for UHPCC-1 and UHPCC-2 respectively. The presence of steel fibers can obviously improve the freezing-thawing performance of UHPCC.

3.3 X-ray computed tomography

Meso-defects of UHPCC before freezing-thawing action

Figure 3 shows the original meso-defects in UHPCC-1 and UHPCC-2 before freezing-thawing action. Due to the resolution limitation, the minimum defects volume is 0.002mm$^3$. The meso-defects volume fraction of UHPCC-1 and UHPCC-2 are 1.67% and 1.94%, respectively. They are close to each other, but the maximum value of meso-defects volume of UHPCC-2, which is 49 mm$^3$, is much higher than that of UHPCC-1 (18 mm$^3$). Since the presence of steel fibers caused an obvious loss of workability, more and bigger entrapped air voids were captured in the matrix of UHPCC-2 during the casting process.

![3D meso-defects of UHPCC-1 and UHPCC-2 before freezing-thawing action](image)

(a) UHPCC-1  (b) UHPCC-2

Figure 3: 3D meso-defects of UHPCC-1 and UHPCC-2 before freezing-thawing action

Meso-defects of UHPCC after freezing-thawing cycles

Figure 4 shows the 3D meso-defects of UHPCC-1 and UHPCC-2 after 800 freezing-thawing cycles. Compared with Figure 1, it is obviously that more defects occurred in the materials, and the size of original defects increased.
Figure 4: 3D meso-defects of UHPCC-1 and UHPCC-2 after 800 freezing-thawing cycles

Figure 5: Volume distribution of meso-defects in UHPCC-1 and UHPCC-2 before and after freezing-thawing action

The volume distribution of meso-defects in UHPCC-1 and UHPCC-2 before and after freezing-thawing action is illustrated in Figure 5. The point at 0.01 means the counts of defects between 0.002mm$^3$ and 0.01mm$^3$; the point at 0.02 means the counts of defects between 0.01mm$^3$ and 0.02mm$^3$; and so forth for the other points. It can be seen that, before freezing-thawing action, the amount of defects in UHPCC-2 which can be seen by X-ray CT is a little higher than that in UHPCC-1 due to the presence of steel fibers. And the total amount of defects of UHPCC-1 and UHPCC-2 were both almost doubled after 800 freezing-thawing cycles. The volume of the defects, which had most significant increase, is between 0.002mm$^3$ and 0.1mm$^3$. This is because some smaller defects (such as small air pores), which could not be detected by X-ray CT at first, became larger due to the freezing-thawing action. Meanwhile, it is also can be observed from Figure 5 that the defects in UHPCC-1 increased more than that in UHPCC-2. The presence of steel fibers improves the resistance of UHPCC to freezing-thawing damage, because of the fibers’ toughening effect.

Some comparative parameters on defects evolution of UHPCC-1 and UHPCC-2 before and after 800 freezing-thawing cycles are listed in Table 2. Both the size and the amount of meso-defects in UHPCC-1 and UHPCC-2 increased because of the freezing-thawing damage.
defects volume fraction of UHPCC-1 and UHPCC-2 increased by 43.1% and 32% respectively after 800 freezing-thawing cycles. The maximum value of defects volume of UHPCC-1 raised by 111.1%, while that of UHPCC-2 was only 6.1%.

Table 2: Parameters on defects evolution of UHPCC before and after freezing-thawing action

<table>
<thead>
<tr>
<th></th>
<th>Defects volume fraction</th>
<th>Maximum value of defects volume</th>
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<tbody>
<tr>
<td></td>
<td>UHPCC-1</td>
<td>UHPCC-2</td>
</tr>
<tr>
<td>Before freezing-thawing action</td>
<td>1.67%</td>
<td>1.94%</td>
</tr>
<tr>
<td>After freezing-thawing action</td>
<td>2.39%</td>
<td>2.56%</td>
</tr>
<tr>
<td>Increment</td>
<td>43.1%</td>
<td>32.0%</td>
</tr>
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Influence of large entrained air voids on freezing-thawing performance of UHPCC

Due to the low water to binder ratio, UHPCC is very viscous when it is fresh. Large air voids can be easily entrained in the matrix if it is not prepared well. Figure 4 shows the 2D X-ray CT Scanning images of the only one specimen of UHPCC-1 with large entrained air voids at different numbers of freezing-thawing cycles.

![Figure 4: 2D X-ray CT Scanning images of UHPCC-1 with large entrained air voids](image)

(a) 250 cycles (b) 800 cycles

After 250 freezing-thawing cycles, cracks began to occur around the large entrained air void because of the hydraulic pressure. The irregular shape of entrained air voids will cause local stress concentration, which may result in crack initiation in the matrix at a low stress state, and cracks can propagate rapidly since the matrix of UHPCC is very brittle. The surface near the large entrained air void totally expanded after 800 freezing-thawing cycles. Cracks and large entrained air voids were not found by X-ray CT in any other UHPCC-1 specimens after 800 freezing-thawing cycles. Specimens of UHPCC-2 did not crack either because of anti-crack effect contributed by steel fibers. Hence, large entrained air voids are harmful to the freezing-thawing performance of UHPCC without fibers, and careful preparation is essential for UHPCC to fulfill its excellent properties.
4. CONCLUSIONS

Based on the results of analysis and freezing-thawing performance tests, the following conclusions are drawn:

- Both the size and amount of meso-defects in UHPCC increased after freezing-thawing action. The smaller defects increased more significantly than the bigger ones, and the volume of the defects which had most significant increase is between 0.002mm³ and 0.1mm³.
- Test results of the X-ray CT results and freezing-thawing tests show that the presence of steel fibers improves the resistance of UHPCC to freezing-thawing action.
- Large entrained air voids, which come from inappropriate preparation, are harmful to the freezing-thawing performance of UHPCC without steel fibers.

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REFERENCES