Experimental Investigation of the Variation of Concrete Pores under the Action of Freeze-Thaw Cycles by Using X-Ray CT

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The variation of concrete pores under the action of freeze-thaw cycles was investigated experimentally by using the X-ray CT. Firstly, the statistical characteristics of pores of concrete specimens were obtained by using the X-ray image analysis. Secondly, the variation of porosity and pore volume of concrete pores were analyzed and discussed by comparing with above characteristics. Thirdly, the failure process of the concrete specimens acted by the freeze-thaw cycles was investigated by scanning the interior of concrete specimens. The results showed that the pore volumes of concrete pores whose volumes were located at the interval $[0.5 \text{ mm}^3, 20 \text{ mm}^3]$ have no big variation in both the amounts and volume of concrete pores, while others were found to have huge change during the process of experiment. The extent of damage acted by the repeated freezing and thawing gradually ranged from surface to complete disintegration of the interior of concrete specimens after 30 cycles of freeze-thaw acting.

1. Introduction

Improvement of the durability of concrete material [1–4] is a significant and scientific issue for the design of civil structures. In cold climate regions, the durability of concrete structures decreases owing to the effect of some environmental factors, especially one of which is well known as the freeze-thaw cycles [5–7]. Under the repeated action of freeze-thaw, the frost damage, which means that the concrete material gradually loses its stiffness and strength, is a major concern for the design and maintenance of concrete structures. Therefore, it is necessary to deeply investigate and reasonably evaluate the deterioration of concrete structures in order to improve the durability and safety of concrete structures.

The characteristics of plain concrete under the action of compression and freeze-thaw cycles were investigated by Qin [8], and the effects of stress ratio and number of freeze-thaw cycles on strength and stiffness of concrete were analyzed experimentally. A modified freeze-thaw experimental procedure was proposed [9] by investigating the durability of cellular concrete specimens. Mulheron and O’Mahony [10] found out that the lean concrete combined with recycled aggregates has the similar or better performance as the regular concrete material subjected to the freeze-thaw environment. When the chert gravel was taken as the aggregate, Buck [11] found out that the capacity of concrete structures to resist the action of freezing and thawing could go up as the number of cycles of freeze-thaw increased. Compared with above research, the performance of the high-strength concrete under the action of freeze-thaw cycles was also investigated gradually. The durability of internal cracking on ice formation for high-strength concrete was studied [12]. Sun et al. [13] investigated the capacity of high-strength concrete to resist the action of flexural load and freeze-thaw cycles. Marzouk and Jiang [3] investigated the tension properties of high-strength concrete after freezing and thawing cycles. Henry et al. [14] examined the internal microstructure of high-strength concrete in order to clarify the effects of heating and recuring on microstructure characteristics.

As discussed above, the damage of concrete structures caused by the action of freeze-thaw cycles was, in most cases, evaluated by investigating the failure process of concrete
structures. For the effective maintenance of concrete structures under freeze-thaw condition, it did not focus on the damage of structures themselves but focused on the failure mechanism of concrete material. Considering this point, the X-ray microtomography (X-ray CT), which is a powerful tool for nondestructively investigating the three-dimensional microstructure of a material [15, 16], was applied to investigate the failure process of concrete material under the action of freeze-thaw cycles in this study. Through X-ray CT and image analysis techniques, the variation of the internal porosity and pore volume of concrete pores were qualitatively and quantitatively analyzed in three dimensions.

To this end, the remainder of this paper is organized as follows. The details of the experimental material and procedure are introduced in the next section. The experimental results are analyzed and discussed in Section 3. At last, conclusions are drawn.

2. Materials and Experiments

2.1. Materials and Mix Proportions. The local materials were utilized in order to implement this study. A Chinese Standard (GB175-2007) 42.5 Ordinary Portland cement was applied for the cementitious material during the experiment. The fine aggregate was natural river sand with the fineness modulus of 2.9, and the coarse aggregate was single-grading crushed stone with diameter from 5 mm to 10 mm. The water reducer took the polycarboxylate superplasticizer, and the designed mix proportions of concrete for experiment are listed in Table 1.

2.2. Experiments

2.2.1. Apparatus and Testing Specimens. A desktop microfocus CT system (mode: Phoenix v|tome|xs) was applied to obtain the slice images of each specimen. The set-up of this experimental system consists of a microfocus X-ray emitter, a five-axis rotation table, an image intensifier detector, and a software subsystem (VGStudio MAX 2.0). Some photos of this experimental system are shown as Figure 1.

To simulate the effect of freeze-thaw cycles on concrete specimen, slow-freezing method and quick-freezing method are two common options. The latter was chosen in this study. The procedure of quick-freezing method is that one cycle of freeze-thaw should be completed in 4 hours, and the melt time is less than one quarter of one cycle of freeze-thaw. The highest temperature during the freeze-thaw cycles is 8°C and the lowest one goes to −17°C.

Each specimen takes Φ75 mm × 150 mm cylinder owing to the space limit of the X-ray CT system. The compressive strength of each specimen is 30 Mpa. After 28 days' curing under the standard condition, the concrete specimens were soaked in water for 3 days. Before the action of freeze-thaw cycles, we measured the initial mass and the propagation time of ultrasonic wave going through the specimen. The propagation wave acting on the specimens was generated by using the ultrasonic detector. During the process of experiment, the mass and the propagation time of each specimen were measured every 6 cycles of freeze-thaw. The whole experiment was terminated until the loss of mass of each specimen was bigger than 5 percent of the initial total mass or the relative dynamic modulus of each specimen decreased to lower than 60 percent. The dynamic modulus of each specimen was obtained by using the propagation time of ultrasonic wave going through each specimen.

To investigate the distribution of concrete pore, each specimen is divided into four parts. The rule for this partition is described as follows. As shown in Figure 2, firstly, the diameter of the specimen is deemed as the biggest one. A square could be determined, and this square is the biggest one belonging to the circle whose diameter is the biggest diameter. Secondly, the side length of this square is deemed as the diameter of a circle, and then another circle is determined. Repeating this partition, four different parts are obtained, and the diameters of three circles are Φ75 mm, Φ60 mm, and Φ40 mm, respectively.

2.2.2. Initial Statistical Results of Concrete Pore before the Action of Freeze-Thaw Cycles. Before the performing of the freeze-thaw cycling test, we investigated the statistical feature of concrete pores of all specimens without the action of freeze-thaw cycles. As shown in Figure 3, comparing with large volume pore, the small volume pore takes a big percentage of the total number of concrete pores of concrete specimens. On one hand, the number of concrete pores whose volumes are smaller than 0.01 mm³ is about 1.8 × 10⁵,
Figure 2: Sketch map of the partition of each specimen.

Figure 3: Initial statistical results of concrete pore.
Figure 4: Variation ratio of concrete porosity along with the number of freeze-thaw cycles.

Figure 5: Ratio of A to B under the action of freeze-thaw cycles (A: volume of connected pores; B: volume of all pores).

Figure 6: Variation ratio of RDM under the action of freeze-thaw cycles (RDM: relative dynamic modulus).
Table 1: Mix proportions of concrete used for experiment.

<table>
<thead>
<tr>
<th>Number</th>
<th>Amount of concrete per cubic meter (kg/m³)</th>
<th>Water-cement ratio</th>
<th>Sand (%)</th>
<th>28-day compressive strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>380</td>
<td>0.5</td>
<td>37</td>
<td>35.5</td>
</tr>
<tr>
<td>B</td>
<td>430</td>
<td>0.4</td>
<td>38</td>
<td>46.4</td>
</tr>
<tr>
<td>C</td>
<td>477</td>
<td>0.29</td>
<td>33</td>
<td>60.5</td>
</tr>
</tbody>
</table>

Figure 7: Changing process of the connected pores under the action of freeze-thaw cycles.

and the number of concrete pores whose volumes belong to the interval \([0.1 \text{ mm}^3, 0.5 \text{ mm}^3]\) is close to 1700. On the other hand, there are about 170 concrete pores whose volumes are located at the interval \([0.5 \text{ mm}^3, 1.0 \text{ mm}^3]\), and only 6 concrete pores whose volumes are bigger than \(20 \text{ mm}^3\).

As shown in Figures 3(b) and 3(c), considering the volume interval \([0.01 \text{ mm}^3, 20 \text{ mm}^3]\), there are more concrete pores whose volumes belong to the intervals \([0.1 \text{ mm}^3, 0.5 \text{ mm}^3]\) and \([1 \text{ mm}^3, 5 \text{ mm}^3]\), respectively, and the total volume of these concrete pores is about \(400 \text{ mm}^3\). For the other concrete pores, the average total volume of concrete pores is close to \(180 \text{ mm}^3\) such as the concrete pores whose volumes are located in the following intervals: \([0.01 \text{ mm}^3, 0.05 \text{ mm}^3]\), \([0.05 \text{ mm}^3, 0.1 \text{ mm}^3]\), \([0.5 \text{ mm}^3, 1 \text{ mm}^3]\), \([5 \text{ mm}^3, 10 \text{ mm}^3]\), and \([10 \text{ mm}^3, 20 \text{ mm}^3]\). Another phenomenon is that the concrete pore whose volume is bigger than \(20 \text{ mm}^3\) takes the largest percentage among all the concrete pores. The reason for this lies in the connection and combination of some concrete pores caused by the concrete vibration and the missing of releasing agent for concrete blinding. From Figure 3(d), the porosity does not change a lot for the concrete pores whose diameter is about \(60 \text{ mm}\), while the porosity has a huge change for the concrete pores whose diameter is bigger than \(60 \text{ mm}\). The reason for this huge change also goes to the connection and combination of couples of concrete pores.

3. Results and Discussions

In this section, the variation of concrete pores and damage situation of concrete specimens under the action of freeze-thaw cycles were investigated by using the X-ray CT technique.
3.1. Variation of Concrete Pores Acted by the Free-Thawing Cycles

3.1.1. Variation of Porosity of Concrete Pores. During the action of freeze-thaw cycling, we found out that the variation of concrete pores was uneven along with the height of concrete samples. Herein, we use D, ZK, LZ to represent the diameter of concrete samples, the whole porosity of concrete pores, and the connected porosity of concrete pores, respectively. As shown in Figure 4, with the processing of freeze-thaw cycles, the variation of porosity of concrete pores goes up as the increase of diameter of concrete samples, and the biggest change of the porosity of concrete pores lies in the Φ75 mm samples. Comparing with the initial situation of all samples shown in the last section, the porosity of concrete pores of Φ75 mm samples increases from 1.79% to 3.2% and this rate arrives at 7.2% after 30 cycles of freeze-thawing action. For the samples with other diameters, the porosity of concrete pores goes to 1.23% after 12 cycles of freeze-thawing action, and this porosity reaches the stable state after 30 cycles of freeze-thawing action. The porosity of concrete pores of all the samples changes dramatically when the number of cycles is over 30. The similar changing rule of connected pores is found out as shown in Figure 4. It was concluded that the variation of porosity of concrete pores was induced mainly by the generation of connected pores at the initial phrase of freeze-thawing cycles.

If the connected pores are deemed as one whole pore, the porosity of concrete pores reaches 34% at the initial state without freezing and thawing according to Figure 5. During the process of freeze-thawing cycles, the water could easily enter into the concrete pores. The water staying at the concrete pores transforms from solid state into liquid state when the temperature goes down. Owing to this state transforming, the whole volume of concrete pore expands, and then the expansion force is generated. As the expansion force exceeds the limit tension strength, some cracks occur and more pores also generate. Therefore, as the number
of freeze-thaw cycles increases, the whole volumes of the concrete pores expand gradually.

As shown in Figure 5, for the Φ20 mm circle region, Φ40 mm circle region, and Φ60 mm circle region, the porosity of concrete pores is close to zero at the start of freeze-thaw cycles. This porosity of Φ60 mm samples begins to change at the 18th freeze-thaw cycle, and this rate reaches 75% after 35 cycles. The porosity of the other two types of samples starts to change at the 25th freeze-thaw cycle, and the changing rate reaches 60% after 35 cycles. According to Figure 7, the volume of connected pores starts to expand after 12 cycles, and this volume expands dramatically as the number of freeze-thaw cycles is over 30. From these phenomena, it is concluded that (i) the connected pores generate at the region near the surface of samples and (ii) the volumes of connected pores expand deeply as the number of freeze-thaw cycles increases; that is, more pores start to connect with each other gradually, so the number of concrete pores goes down.

The variation of relative dynamic modulus of concrete is shown in Figure 6. From Figures 5 to 7, the relative dynamic modulus is sensitive to the change of porosity of concrete pores. As shown in Figure 6, the relative dynamic modulus of concrete starts to change dramatically after 12 cycles of freeze-thaw action.

### 3.1.2. Variation of the Volumes of Concrete Pores

(1) **Experimental Results of Φ75 mm Circle Region of Specimen.** As described in Section 3.1.1, more and more microcracks generate inside of the concrete specimens during the process of freeze-thawing cycles. On one hand, some big pores generate since many small pores connect with each other. On the other hand, the number of small pores increases such that some microcracks exist independently. It is shown in Figure 8 that the big pores generate among the pores whose size locates at the interval [10 mm$^3$, 20 mm$^3$] and small pores generate from the pores ranging from 0.01 mm$^3$ to 0.05 mm$^3$. The porosity of concrete pores ranging from 0.01 mm$^3$ to 0.05 mm$^3$ does not change a lot since the expansion of this size of pores which destroy the bigger size of pores eliminates the pores generating from smaller size of pores. The volumes of concrete pores ranging from 0.5 mm$^3$ to 10 mm$^3$ go down under the action of freeze-thaw cycles. This shows that

**Figure 9:** Variation ratio of pore volume of Φ60 mm circle region of specimen.
the freeze-thaw action fastens the destroying of the pores ranging from 0.5 mm$^3$ to 10 mm$^3$. For the concrete pores whose volumes are bigger than 10 mm$^3$, more pores connect with each other with the processing of freeze-thaw cycles. Therefore, it is easy to explain the trend drawn in Figure 8(d).

(2) Experimental Results of Φ60 mm Circle Region of Specimen. As shown in Figure 9, the volumes of concrete pores have obvious change for the concrete pores whose initial volumes are smaller than 0.05 mm$^3$ and bigger than 20 mm$^3$, and the volumes of the other concrete pores have no big change. With the continuous action of freeze-thaw cycles, the interior of concrete specimens generates more cracks so that it is easier for the water to enter into the pores. After 30 cycles, the water has completely entered into the whole specimens. In this situation, the freeze expansion starts to destroy the whole specimen as the number of freeze-thaw cycles increases, and then more pores combine into the connected pores. This phenomenon occurs obviously at the concrete pores whose initial volumes range from 0.05 mm$^3$ to 20 mm$^3$. This is the main reason for explaining the results shown in Figure 9.

(3) Experimental Results of Φ40 mm Circle Region of Specimen. As shown in Figure 10, comparing with the Φ60 mm specimens, the similar changing rule of the volume of concrete pores of the Φ40 mm specimens is found except the concrete pores whose initial volumes are bigger than 20 mm$^3$. Although the changing volume of concrete pores between two types of specimens is different, the changing amplitude of volume of concrete pores is similar for two types of specimens. This means that all the water completely enters into the interior of the whole specimen after enough cycles of freeze-thaw action. The uneven expansion of the central part of the specimen does not appear during the process of the freeze-thaw cycles.

3.2. Damage Analysis of Concrete Specimens Acted by the Freeze-Thaw Cycles. Through the X-ray CT image analysis, we investigated the damage situation of concrete specimens under the action of freeze-thaw cycles. Three sections of
Figure 11: Damage process of the upper section under different cycles of freeze-thaw.

Figure 12: Damage process of the middle section under different cycles of freeze-thaw.
each specimen were defined from top to bottom in order to compare the damage extent along the height of each specimen. As shown from Figures 11, 12, and 13, three figures represent the damage situation of each specimen from top to bottom, that is, the upper part, the middle part, and the lower part. Each figure has six items which represent the damage situation of each part after 0, 12, 18, 24, 30, and 36 cycles of freeze-thaw, respectively. 

As shown in Figures 11, 12, and 13, the upper part of each specimen was damaged firstly among the three parts of each specimen. We found out that the amounts of concrete desquamated from the upper part were bigger than the other two parts. The reason for that phenomenon lies in the existence of some angular surface in the upper region of concrete specimen. From this phenomenon, it was concluded that the angular parts and the crack between the stone and set cement were easier to be damaged under the action of freeze-thaw cycles. Otherwise, the different damage extent of three parts of each specimen showed that the effect of freeze-thaw cycles was gradually serious from the surface of specimens to interior of specimens. A threshold of the numbers of freeze-thaw cycles has existed.

4. Conclusions

The variation of concrete pores and damage process of concrete under the action of freeze-thaw cycles were investigated experimentally by using the X-ray image analysis technique. The following conclusions are drawn.

(i) The pore volumes of relative small pores and big pores are sensitive to the action of freeze-thaw cycles. For the concrete specimens used in this study, the small pores and big pores are defined as the concrete pores whose volumes are smaller than 0.05 mm$^3$ and bigger than 20 mm$^3$.

(ii) A threshold of freeze-thaw cycles was found out during the experiment, that is, 30 cycles. After 30 cycles, the freeze expansion starts to destroy the whole specimen, and more pores start to combine with each other.

(iii) The failure process of concrete specimens caused by the repeated freezing and thawing gradually ranged from surface to complete disintegration of the interior of concrete specimens as the number of freeze-thaw cycles increases.

(iv) The relative dynamic modulus is sensitive to the change of porosity of concrete pores. According to the experimental results, the relative dynamic modulus of concrete specimens starts to change dramatically after 12 cycles of freeze-thaw action.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.
References


