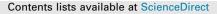
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# Macro-micro degradation process of fly ash concrete under alternation of freeze-thaw cycles subjected to sulfate and carbonation



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# HIGHLIGHTS

• The deterioration law of compressive strength was studied.

• The pore numbers, pore sizes, porosity and micro-pores distribution were considered.

• The alternation of freeze-thaw cycles subjected to sulfate & carbonation was included.

• The cracks after the freeze-thaw cycles under sulfate and carbonation were monitored.

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## ABSTRACT

Concrete is one of the most extensively used building materials. In the cold salty lake region, concrete structures generally undergo freeze-thaw cycles subjected to sulfate in the winter and early spring, and experience carbonation during the rest of the year. It is necessary to study the degradation process of concrete during the alternation of freeze-thaw cycles subjected to sulfate and carbonation. In this article, two types of experiments were implemented: freeze-thaw cycles subjected to sulfate (SF), and the alteration of freeze-thaw cycles subjected to sulfate and carbonation (SFC). Computed tomography (CT), as a non-destructive testing technology, was adopted to reveal the rules of pore changes and show generated cracks after the alternation of freeze-thaw cycles subjected to sulfate and carbonation. CT scanning results showed that pore numbers, pore areas, and porosity of each cross-section changed to some extent. Due to the different distances from the top, the changes of each cross-section were found to be dissimilar. From the 2D images, SFC specimens were identified to have corner erosion and internal cracks, and were damaged more severely than SF specimens. The compressive strength loss of SF specimens was very limited in the early state; after a certain period of erosion, the compressive strength decreased rapidly. The compressive strength loss of specimens under SFC was larger than the specimens under SF. Carbonation constantly consumed calcium ions, which negatively affected the stability of porosity system. Freeze-thaw cycles caused aperture degradation, coarsened the pore structure, and with the additional interaction of sulfate erosion, aggravated the deterioration of concrete.

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#### 1. Introduction

Concrete is one of the most extensively used building materials since the invention of Portland cement in 1824. However, as concrete durability problems rise, companies incur enormous economic losses. Environmental conditions, composition and properties of concrete influence the durability of concrete. In cold

https://doi.org/10.1016/j.conbuildmat.2018.06.037 0950-0618/© 2018 Published by Elsevier Ltd. regions of China, many concrete structures are damaged by freeze-thaw cycles. The content of  $CO_2$  in the atmosphere is about 0.03% [1], but it can reach about 0.1% without ventilation in the laboratory; the average  $CO_2$  concentration in an urban area can easily reach about 0.3%, with some regions as high as 1% [2].  $CO_2$  can invade into concrete, destroy the surface passivation film of steel and caused rust. Concrete carbonation is one of the main causes of reduced durability of reinforced concrete structures. Freeze-thaw cycles and carbonation are the important factors to be considered in the study of concrete durability; freeze-thaw action, carbonation, and other deterioration factors should be

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taken into account when concrete structures are designed based on durability.

Extensive research has been carried out and a large amount of achievements have been obtained for single or double factors of freeze-thaw cycles or carbonation [3–6]. The study on the alternation of freeze-thaw cycles subjected to sulfate and carbonation, however, is rarely reported. In the cold salty lake region, concrete structures generally undergo freeze-thaw cycles subjected to sulfate in the winter and early spring, and experience carbonation during the rest of the season. It is necessary to study the degradation process of concrete during the alternation of freeze-thaw cycles subjected to sulfate and carbonation.

Computed tomography (CT) scanning, a non-destructive testing technology, has been utilized in civil engineering [7–9]. It has been widely used to detect the structure damage, identify the internal microstructures or reconstruct 3D numerical models for civil engineering materials [10–15]. In recent years, material damage was studied with the aid of CT scanning to understand the damage process of single freeze-thaw cycles or sulfate attack and dryingwetting cycles [16,17]. However, only single freeze-thaw cycles or sulfate attack and drying-wetting cycles was investigated in such studies. In this study, CT scanning was utilized to reveal the internal structural changes (such as pores and cracks) of fly ash concrete after several alternations of freeze-thaw cycles subjected to sulfate and carbonation. The internal structural damage from the CT scanning, in addition to the strength change, were analyzed to evaluate the alternation damage of freeze-thaw cycles subjected to sulfate and carbonation.

Mass loss rate and relative dynamic elastic modulus (both of which are measurement criteria), and concrete performance test data obtained after quick freeze-thaw cycles, are used to determine antifreeze level of the concrete. In practice, mechanical properties of concrete are concerned; for instance, strength loss is directly related to the durability and safety of a building. In this article, the deterioration law of compressive strength was studied during the alternation of freeze-thaw cycles subjected to sulfate and carbonation.

#### 2. Materials and experiment design

#### 2.1. Raw materials

Ordinary Portland cement with a strength class of 42.5 was used for this experiment. It was produced by Ji-dong Cement Company in Shaanxi Province, China. The contents of main oxides of cement and fly ash were given in Table 1. The coarse aggregate was limestone, which had a continuous gradation of 5–16 mm, and an apparent density 2.818 g/cm<sup>3</sup>. River sand, with a 2.74 fineness modulus, and an apparent density of 2.630 g/cm<sup>3</sup>, was used. The concrete admixture was a polycarboxylic acid water-reducing agent, and was produced by a building scientific research institute. The aggregates, cement, and admixture were all mixed with tap water, and the mix proportion of concrete was given in Table 2.

#### 2.2. Experiment design

The experiments were divided into two groups, one being freeze-thaw cycles subjected to sulfate (SF), the other being the alteration of freeze-thaw cycles subjected to sulfate and carbonation (SFC). Concrete specimens with a size of 70.7 mm  $\times$  70.7 mm  $\times$  210 mm and 100 mm  $\times$  100 mm  $\times$  100 mm were cast. The former was used to CT scanning, and the latter was used to test

compressive strength after certain cycles. According to Chinese National Standard GB/T50082-2009 [18] and considering specific experimental conditions, the detailed experiment processes are listed below:

- (1) Specimens with sizes of 70.7 mm × 70.7 mm × 210 mm and 100 mm × 100 mm × 100 mm were cast and demolded after 24 h. All specimens were put in a standard curing room for 30 days (natural curing takes for 60 days).
- (2) All concrete specimens were soaked in 5% Na<sub>2</sub>SO<sub>4</sub> solution for 7 days before the age of specimens, to saturate the specimens.
- (3) Saturated specimens were placed in freeze-thaw box. After 25 freeze-thaw cycles, the samples were taken out, air dried for one day and dried in the oven at 60 °C for another day.
- (4) Specimens under SFC were put in carbonation tank for three days. The temperature of carbonation tank is  $(20 \pm 2)$  °C, the relative humidity is  $(70 \pm 2)$ % and the concentration of CO<sub>2</sub> is  $(20 \pm 1)$ %.
- (5) All specimens were taken out after carbonation and immersed in 5% Na<sub>2</sub>SO<sub>4</sub> solution for three days.

Experiment procedures of the alternation of freeze-thaw cycles subjected to sulfate and carbonization are listed above, afterwards repeating step (3)-(5). After several large cycles, the specimens were scanned by CT, and the compressive strength was tested.

As the SFC specimens carbonized, specimens under SF were comparative study while resting in the air. The experiment steps of group SF were identical as group SFC. Fig. 1 shows the main instruments used in this work. TYE-2000H compression testing machine (Fig. 1(d)) was used to test the compressive strength after the alternation of freeze-thaw cycles subjected to sulfate and carbonation.

#### 3. Compressive strength

#### 3.1. Shape change for compressive strength specimens

Fig. 2 shows the shape change of the specimen after damage. (a) is specimen after 450 freeze-thaw cycles subjected to sulfate (SF450), and (b) is specimen after the alternation of 450 freeze-thaw cycles subjected to sulfate and carbonation for 54 days (SF450C54).

It can be observed that the corner of specimen SF450 was lost, its surface appeared to peel off. The surface of specimen SF450C54 peeled off completely, interior cementitious materials pastes began to loosen, and coarse aggregate can be seen clearly. From the appearance, specimen SF450C54 degraded more severely than specimen SF450.

#### 3.2. Results of compressive strength

Experimental results of cubic compressive strength of fly ash concrete specimens were shown in Fig. 3.

As depicted in Fig. 3, the compressive strength of specimens under SF decreased gradually at first; however, for specimens under SFC, the compressive strength of fly ash concrete specimens initially increased. The compressive strength of specimens both decreased gradually as the experiments progressed. The specimen SF250 decreased to 75.7 MPa, with a strength loss of only 5.8%. The specimen SF250C30 decreased to 64.7 MPa, with a strength loss of 19.5%. After 450 freeze–thaw cycles subjected to sulfate, the compressive strength of specimens decreased to 38.6 MPa, indicating a strength loss of 52%, while the strength of specimen SF450C54 was only 15.2 MPa, with a strength loss as high as 81.1%. The compressive strength loss of specimens under SFC was larger than that of specimens under SF.

Table 1				
Contents of main	oxides i	in cement	and fl	y ash

Main oxide	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>
Cement	21.7	5.66	3.12	60.28	1.52	0.37	0.55	2.1
Fly ash	50.64	26.35	5.53	8.81	1.34	0.67	1.41	0.62

Note: The values in the table are mass percent content, unit %.

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