Construction and Building Materials 186 (2018) 182–190

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Nondestructive monitoring the deterioration process of cement paste exposed to sodium sulfate solution by X-ray computed tomography

Yonggan Yang [a,b], Yunsheng Zhang [a,b,*], Wei She [a,b,*], Zhitao Wu [a,b], Zhiyong Liu [b,c], Yajiu Ding [d]

[a] School of Materials Science and Engineering, Southeast University, Nanjing 211189, China
[b] Jiangsu Key Laboratory of Construction Materials, Southeast University, Nanjing 211189, China
[c] State Key Laboratory of Geomechanics & Deep Underground Engineering, China University of Mining and Technology, Xuzhou 221116, China
[d] Tianjin Branch, China Construction Eighth Engineering Division Co., Ltd., Tianjin 300452, China

HIGHLIGHTS

- X-ray CT method is proposed to in situ monitoring the damage process of cement paste.
- The information of cracks, gray value and pore structure can be obtained by X-ray CT.
- The evolution of corrosion depth can be tracked by X-ray CT.

ARTICLE INFO

Article history:
Received 12 March 2018
Received in revised form 27 June 2018
Accepted 19 July 2018

Keywords:
Nondestructive monitoring
X-ray computed tomography
Cement paste
Damage process
Sulfate attack

ABSTRACT

X-ray computed tomography (X-ray CT) method is proposed to in situ monitoring the damage process of cement paste containing different water-to-cement ratio (0.55, 0.40) exposed to sodium sulfate solution. In addition, the corrosion products were analyzed by X-ray diffraction (XRD). The results show that X-ray CT can track the time-dependent development of cement paste exposed to sodium sulfate solution. Furthermore, the information of cracks spatial distribution, gray values, corrosion depth and pore volume distribution can be clearly obtained by this method. Additionally, XRD results show that the main corrosion products of the cement paste with high water-to-cement ratio are ettringite and gypsum in the later stage.

1. Introduction

Sulfate attack on the cement-based materials, sometimes called "sulfate corrosion", the deterioration of cement-based materials structures caused by sulfate attack has become one of the serious problems in many countries [1–3]. The hydration products react with sulfate ions in the pore solution to form gypsum and ettringite when the external sulfate ions into the interior of the cement-based materials. With the increase of gypsum and ettringite content, it can cause the overall expansion and extensive deterioration from the out surface into core of the cement-based materials [4–8]. Meanwhile, the erosion of the sulfate ions to cement-based materials has caused great loss of property at home and abroad. Such as about 40% of total resources of civil engineering are being applied to repair due to sulfate attack. Therefore, a good understanding of sulfate attack on concrete is very important to establish the life prediction model of concrete.

In the literature, the process of sulfate attack on concrete has been reported many times and many studies have achieved significant results in this field. For instance, Ma Xu et al. [9] studied the expansion and degradation of cement paste in sodium sulfate solutions by using MIP and EDS elemental mapping and found that after the immersion time of 420d, the generated stresses is as high as 13.1 MPa in high sulfate solution and 8.3 MPa in low sulfate solution. Gao Jianming [10] studied the durability of concrete exposed to sulfate attack under flexural loading and drying-wetting cycles. The research results showed that drying-wetting cycles and flexural loading could accelerate the sulfate accumulation in concrete and aggravated the damage degree of concrete subject to sulfate attack. Sotiriadis [11] determined that sulfate resistance of limestone cement concrete exposed to combined chloride and sulfate environment at low temperature through the mass changes and compressive strength changes of concrete.

* Corresponding authors at: School of Materials Science and Engineering, Southeast University, Nanjing 211189, China.
E-mail addresses: zhangys279@163.com (Y. Zhang), weishe@seu.edu.cn (W. She).

https://doi.org/10.1016/j.conbuildmat.2018.07.145
0950-0618/

The research results indicated that chlorides have a mitigating effect on sodium sulfate attack. However, most of the researches have mainly focused on the sulfate resistance of concrete by using traditional methods. That is, the measured response using these traditional methods does not reveal the information of cracks spatial distribution, gray values, corrosion depth and pore volume distribution in the samples. X-ray computed tomography (X-ray CT) can add insight into the continuously visual tracking the internal damage process of sulfate attack concrete [12–13]. Furthermore, X-ray CT is a nondestructive detection method without damaging the internal structure of the specimen [14–16].

This paper deals with the deterioration process of cement paste exposed to sodium sulfate solution. In this study, regardless of how the specimens are destroyed, the result is the specimens appeared cracks and then spalling. Therefore, a method of quantitative observation of sulfate attack of cement paste by X-CT combined with image processing technology was proposed. The failure process of the continuous in situ crack was studied by using the whole process of its formation, development and destruction. To study the corrosion products, XRD-analyses was performed [16].

2. Experimental

2.1. Materials

To study the experiments, a Chinese standard P.Ⅱ type Portland cement was used. The composition is given in Table 1. The water-to-cement ratio was maintained at 0.55 and 0.40 by weight.

2.2. Specimen preparation

Cylindrical specimens were cast in plastic molds with dimensions of Φ 35 × 50 mm. After 48 h, the specimens removal from the molds and were cured in the condition of 20 ± 3 °C and 95% of relative humidity for 60 days. After that, the specimens were scanned by X-ray CT, and then put them in 5 wt% sodium sulfate solution. Furthermore, the progress of sulfate attack on specimens was monitored visually by X-ray CT, after they were immersed for 3 months, 6 months, 9 months and 12 months, respectively.

3. Test methods

3.1. X-ray CT

Y.CT Precision S X-ray CT scanner (YXLON, Germany) was employed as shown in Fig. 1. The sample platform can rotate for 360°. The working voltage and current of X-ray tube were 195 kV and 0.34 mA. The X-ray CT raw data are collected by 1530 projection images that recorded by a CCD camera with an array of 1024 × 1024 pixels.

3.2. Mercury intrusion porosimetry (MIP)

MIP test was conducted by Autopore IV 9500 (applied pressure ranging from 0.5 to 16,370 psi), mercury surface tension and the contact angle were 485 dynes/cm and 130°, respectively.

3.3. X-ray diffraction (XRD)

The surface powder of specimens was mixed with approximately 10 wt% corundum (α-$Al_2O_3 \geq 99.99\%$) by mass as an internal standard in the glove box (nitrogen protection). The X-ray diffraction (XRD) was carried out using the D8-ADVANCED with Cu Kα. The working voltage was 40 kV and the current was 40 mA. Furthermore, the angle scan range was chosen from 5° to 70°.

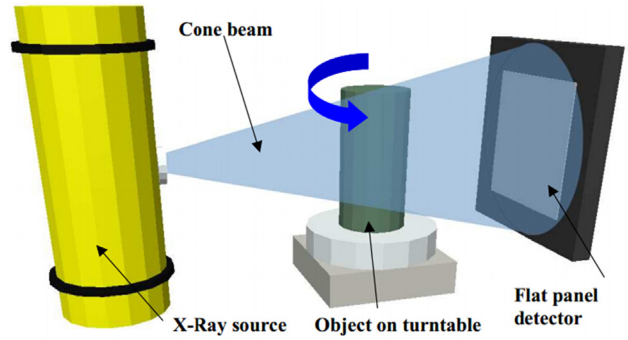

Fig. 1. The working principle of the X-ray CT system.

4. Results and discussion

4.1. Expansion

The influence of water-to-cement on expansion of specimens under sulfate attack was investigated by measuring function of VG Studio MAX software. The biggest advantage of this method is to avoid the error caused by human measurement and increase the accuracy of the test. Before immersion, the initial length of specimens was recorded [17]. The expansion of specimens was calculated according to Eq. (1).

$$\text{Expansion}\,(\%) = \left(\frac{l_t - l_i}{l_i}\right) \times 100 \quad (1)$$

Where l_t is the length at immersion time, l_i is the initial length before immersed in the 5 wt% sodium sulfate solution.

The expansion values of specimens until an immersion time of 12 months are shown in Fig. 2. It can be see that the specimen with water-to-cement of 0.55 expanded more rapidly as compared to the specimen with water-to-cement of 0.40. After 12 months of immersion, the expansion for specimen with water-to-cement of 0.55 was 3.0%, while the specimen with water-to-cement of 0.40 expansion was 0.87%. The relationship between the expansion (y) of the specimens and immersion time (x) under sulfate attack can be described by Eqs. (2) and (3), respectively.

$$y_{0.55} = 0.01032x^2 + 0.12619x + 0.02571 \; (R^2 = 0.98) \quad (2)$$

$$y_{0.40} = 0.005x^2 + 0.01167x + 0.004 \; (R^2 = 0.99) \quad (3)$$

4.2. Visual inspection

Fig. 3 shows the morphology of the cement paste with the water-to-cement ratio of 0.55 for different immersion times. It can be seen that small cracks appeared on the surface of the specimen after 3 months. As the immersion time increases, the width of the cracks increases and cracks become more numerous on the sur-

Table 1
Chemical compositions of cement.

Material	Chemical composition /wt%								Specific surface area /($m^2 \cdot kg^{-1}$)	Density/($kg \cdot m^{-3}$)
	SiO_2	MgO	Al_2O_3	Fe_2O_3	CaO	Na_2O	K_2O	LOI		
Cement	20.87	2.13	4.87	3.59	64.47	0.11	0.65	0.77	309	3115