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Technical note

Characterization of internal damage of concrete subjected to freeze-thaw cycles by electrochemical impedance spectroscopy



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HIGHLIGHTS

• Internal damage of concrete under F-T cycle was investigated by EIS technique.

• A equivalent circuit model was applied to explain EIS results of concrete.

• Decrease of resistance of continuous path was due to the propagation of cracks.

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ABSTRACT

In this work, internal damage condition of concrete subjected to freeze-thaw (F-T) cycles was investigated by means of electrochemical impedance spectroscopy (EIS) technique. The equivalent circuit model was adopted to explain the EIS results according to the establishment of physical model simulating the internal micro structure of concrete. As a result, continuous decrease of resistance of continuous path was found out in the process of F-T cycle because of the expansion and propagation of internal cracking of concrete. This was also demonstrated by microscopic analysis obtained from environmental scanning electron microscope (ESEM) observation. Besides, damage degree calculated based on the decrease of resistance was introduced to evaluate the internal degradation condition of concrete suffering from F-T cycles.

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1. Introduction

Freeze-thaw cycle usually caused surface scaling as well as internal damage of concrete, the latter one played the dominate role in the deterioration of concrete since a large amount of macro and micro cracks were produced in matrix and interfaces leading to the degradation of stress-stain relationship in compression and tension, compression and tensile strengths and so on [1]. Therefore, it had significant senses in evaluation of the internal degradation degree of concrete suffering from freeze-thaw cycle by quantitative techniques. Though relative dynamic modulus [2,3] and electrical resistivity [4,5] methods have been widely used to characterize the damage condition of concrete exposed to freeze-thaw cycle, surface condition produced significant interference on the accuracy of measurement meanwhile evolution of

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internal damage of concrete due to cracking in the process of F-T cycle through these techniques was scarcely impossible [6–9].

Electrochemical impedance spectroscopy (EIS) technique was commonly applied to study the corrosion condition of steel [10,11], pore structure of cement-based materials [12,13] and hydration process of cement [14]. The high frequency part represented the electrochemical reaction happened in the internal structure of concrete [15]. Therefore, the variation of internal structure of concrete under the effect of F-T cycle could be evaluated through analysis of high frequency region of EIS plot by means of the equivalent circuit model derived from the establishment of physical model simulating the internal structure. In this investigation, internal damage condition of concrete after different F-T cycles were investigated and observed by EIS and ESEM techniques, respectively. Damage degree was also introduced to perform the quantitative assessment of internal degradation situation of concrete.



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2. Materials and methods

P.II 42.5 (GB175-2007) Ordinary Portland Cement (OPC) was used. S95 grade of ground granulated blast-furnace slag (GGBS) and gradeIIfly ash (FAH) were also used as the supplementary cementitious materials. The oxide compositions of OPC, GGBS and FAH were listed in Table 1. Local clean river sand (fineness modulus of medium sand equal to 2.6) and locally available well graded aggregate of normal size ranging between 5 mm and 20 mm were chosen as fine and coarse aggregates respectively. The compositions of concrete were presented in Table 2. Concrete specimen with the size of $10 \times 10 \times 10$ cm was fabricated and the number of every type of concrete produced is 15. These specimens were stored in a 95% humidity chamber at $20 \pm 2 \,^{\circ}$ C for 28 days. After the end of curing, 3 concrete specimens of every type were chosen to detect the 28 d compressive strength. It was noted that measurements of compressive strength and air content were referred to the Chinese Standard GB/T 50081–2002 and GB/T 50080–2016, respectively.

Then concrete specimens were subjected to freeze-thaw (F-T) tests according to the "Test Method for Rapid Freezing and Thawing" described in GB/T 50082–2009. The instrument was HDK Rapid Freezing and Thawing Machine (Suzhou Donghua Examination Appartus Co.,Ltd). Specifically speaking, the temperature decreased from 7.5 to $-17.5 \,^{\circ}$ C in 150 min at an average cooling rate of 0.166 °C/min. After suffering from 0, 50, 100 and 150 F-T cycles respectively, two opposite sides of concrete specimens were closely pasted with stainless steel meshes in order to conduct electrochemical impedance spectroscopy (EIS) measurement by application of a sinusoidal potential perturbation of 10 mV at the open circuit potentials with the frequency ranged from 10 mHz to 2 MHz by means of a Princeton Applied Research (PAR) START 2273 Potentiostat. It should be noted that the concrete specimen was immersed in water for a period of 24 h before conducting the EIS measurement. Schematic diagram of the EIS measurement is described in Fig. 1. Furthermore,

Table 1

Chemical composition of OPC, GGBS and FAH used in this work (%).

S	biO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	K ₂ O	Na ₂ O	SO ₃	Ignition loss
OPC 2	22.55	9.35	61.30	3.10	1.35	1.03	0.15	0.99	0.16
GGBS 0	0.33.60	20.30	27.40	1.00	13.50	0.41	0.33	0.20	0.48
FAH 5	53.93	31.31	2.99	3.56	0.0.58	0.0.29	1.82	0.94	2.82

Table 2Mixing proportions of concrete.

Mix	Mixture p	proportio	n of conci	rete (kg/m ³)						28d Compressive Strength	Air Content
	Cement	FAH	GGBS	Fine Aggregate	Coarse Aggregate	Water	W/B	Water Reducer	Air Entraining Agent $(\times 10^{-2})$	(MPa)	(%)
5a	336	-	-	657	1221	168	0.5	0.840	-	44.3	2.1
5b	202	135	-	657	1221	168	0.5	0.840	-	42.4	1.7
5c	202	-	135	657	1221	168	0.5	0.840	-	46.8	2.5
5d	336	-	-	657	1221	168	0.5	0.840	0.168	38.5	4.6



Fig. 1. Schematic diagram of EIS measurement for concrete specimen.

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