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Quantitative characterization of three-dimensional pore structure in hardened cement paste using X-ray microtomography combined with centrifuge driven metal alloy intrusion

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ABSTRACT

In this paper, a centrifuge device is proposed to facilitate the intrusion of a low-melting point metal alloy into the pore space of hardened cement paste. X-ray microtomography is combined with metal centrifugation porosimetry (MCP) to quantitatively investigate 3D pore structure. The low-melting-point metal alloy is melted and introduced into pore space in pastes with water cement ratio of 0.5 and 1.0 at a temperature of 65 °C. 3D pore structure is quantitatively analyzed by X-ray microtomography after the molten metal alloy has been consolidated. A new threshold value segmentation method for pore space was proposed using conversion coefficient on region of interest (ROI). Porosity and pore size distribution are tested by MCP and compared with the results based on mercury intrusion porosimetry (MIP). The results show that the contrast between pore space and solid phase in the X-ray microtomography device image is improved. The total porosity obtained by MCP was found to be consistent with the results obtained by MIP.

1. Introduction

Cement-based materials are considered as one of the most vital materials, as they play an important role in infrastructure development [1]. These materials consist of liquid, solid and gas phases whose properties are strongly correlated with the performance of modern concrete [2,3]. Porosity in cement-based materials is directly associated with the mechanical performance as well as transport properties [4]. Hence, quantitative characterization of pore structure of cement-based materials is important to assess the performance of concrete [5].

Several commonly used techniques are available for characterization of pore structure in cement-based materials, including small-angle X-ray scattering, Brunaures-Emmitt-Teller adsorption and mercury intrusion porosimetry (MIP) [6]. Among them, MIP technique is the most widely employed technique in research due to its advantages such as large dynamic range of pore size characterization (from a few nanometers to a few hundred micrometers), high performance and short testing durations. However, this method has limited applications due to the ink bottle effect [7] and it assumes cylindrical pore geometry [8]. Due to these assumptions, the estimation of pore parameters such as porosity, pore connectivity and surface-to-volume ratio by the MIP method are inaccurate. Moreover, the detailed topology of 3D pore structure has not be obtained until now, which is crucial to the performance in cement-based materials. Although, scanning electron microscopy (SEM) has been conventionally applied to analyze pore structure as it offers a high resolution, the low contrast between pore space and solid phase limits the information of pore structure to 2D only.

As a well-established technology, X-ray computed tomography (X-ray CT) can achieve the visualization of 3D pore structure [9]. However, current results mostly focused on pore structure of concrete with larger pore size, such as foamed concrete [10,11] and cracked concrete [12] due to high contrast between pore space and solid phase. The limited application of X-CT in common or high performance concrete are mainly due to the fact that the threshold value between pore space and solid phase in CT slice figure is not obvious arising from relatively low attenuation. Therefore the classification of threshold value is subjective [13]. In order to reduce subjectivity, contrast agents have been applied in porosity characterization of porous material, such as Wood's metal [14–18], polymethylmethacrylate (PPMA) [19] and mercury [20]. Pore structure [14–16] was analyzed only on 2D using Wood's metal combined with high pressure. Wood's metal was also used in clay

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Table 1
Chemical composition of cement.

Cement	Chemical composition (%)								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
Content	21.35	4.67	3.31	62.60	3.08	2.25	0.11	0.65	0.95

rock [17] to obtain 3D structure of materials combined with focused ion beam (FIB). PPMA [19] and mercury [20] were applied to research pore structure in crystalline rock with ordinary CT imaging. In addition, the resolution of ordinary X-ray CT and micro X-ray CT used are not enough for micro pore scale research [21]. Hence there is need for techniques that have a high contrast and a high precision to image 3D pore structures in cement-based materials.

In this study, a novel contrast enhanced X-ray microtomography technique has been employed for the first time to quantitatively characterize 3D pore structure in cement pastes. A new threshold value segmentation method for pore space was proposed based on ROI combined with volume compensation factor. The metal alloy is centrifuged into pore spaces to enhance their contrast in X-ray microtomography images.

2. Materials and Methods

2.1. Materials and Instrumentation

2.1.1. Preparations of Specimen

The cement used in this study is a Chinese standard Graded P.II52.5 type Portland cement with a density of 3150 kg/m^3 and specific surface area of 369.60 m^2/kg. Its chemical composition is listed in Table 1. It has an initial and final setting time of 132 min and 187 min, respectively. Its compression and flexural strength for a 28 day curing duration under standard conditions are 59.60 MPa and 9.20 MPa, respectively.

Cement pastes were prepared with water cement ratio (w/c) of 0.5 and 1.0. Cavity formation was facilitated in the prepared specimens to hold the molten metal using the proposed device, as shown in Fig. 1(a).

Table 2
Sample information.

Sample name	w/c	Age (days)	Centrifuge speed (RPM)	Intrusion pressure (MPa)
Untreated	0.5	28	4000	12.02
A	0.5	28	4000	12.02
B	0.5	14	4000	12.02
C	1.0	14	4000	12.02

Firstly, the fresh cement paste was poured into the tube and then pressed down the bar-pipe plug to produce the cavity. Then, the samples were put into a rotating device to constantly and slowly rotate up and down during the whole setting and hardening time of the fresh cement paste. After the setting and hardening of the paste, the bar-pipe plug was dialed out carefully and replaced with the pipe cap, curing paste at 20 ± 1 °C/95% RH for 28 d and 14 d (Table 2). Finally, the specimens were soaked in ethyl alcohol for 3 d to terminate hydration and dried at a temperature of 65 °C in the air oven until mass constancy to exclude gas in pores of the hardened cement paste.

2.1.2. Proposed Centrifuge Device Design

The experimental set-up used to intrude the metal into the sample consisted of an in-house developed thermal-insulation centrifuge tube (Fig. 1(b)) and a centrifuge machine (maximum speed: 4000 r/min) (Fig. 1(c)). Firstly, the molten metal alloy was injected into the cavity of sample. Then, the sample was tightly and hermetically coated with extruded polystyrene (heat conductivity coefficient 0.030 W/(m·K)) to maintain the experiment time (65 °C). Finally, the sample was placed in a centrifuge tube to be subjected to the centrifugal process at 4000 r/min for 30 min. Subsequently, the specimen was cooled below 47 °C, after which a cylinder with diameter of 1.0 mm was drilled for X-ray microtomography (Fig. 2). Three samples (A, B, C) were prepared under the conditions and one untreated sample was prepared under the same formation and curing conditions for control group, as listed in Table 2.

2.1.3. Metal Alloy Properties

The metal alloy used in this experiment has a chemical composition

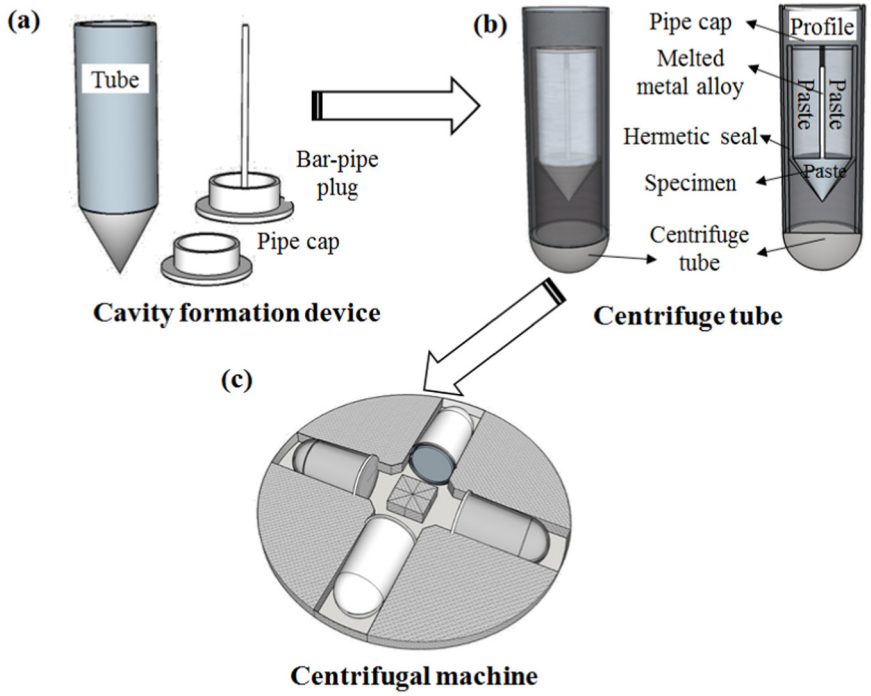

Fig. 1. Schematics of the proposed centrifugal device design. (a) Cavity formation device, (b) thermal-insulation centrifuge tube and (c) centrifuge machine.

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