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Indentation creep of cementitious materials: Experimental investigation from nano to micro length scales

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HIGHLIGHTS

- CSM can quantify the minimum indentation load and depth for homogeneous properties.
- Three characteristic lengths are used to quantify size of indentation affecting area.
- Gel porosity is suspected to play a significant rule in increasing creep of CSH.
- Microcreep is affected by microstructure of hardened paste.
- The age effect on the microcreep is manifested during the deviatoric creep stage.

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ABSTRACT

Many macroscopic properties of concrete originate from the underlying nano and microscale structures and phases. To explore the creep behavior across the nano to micrometer scales, the indentation technique is used in this study to determine the properties of hardened pastes by applying different magnitudes of load ranging from 2 mN to 5 N. Supplementary techniques, such as scanning electron microscopy (SEM) with backscattered electron (BSE) and continuous stiffness measurement (CSM) are also adopted to analyze the effects of load magnitude, microstructure, and age on the mechanical and creep properties. It is found that mechanical properties decreases with increasing applied load, the properties of composite instead of the individual phase in hardened cement paste can be measured by an indentation load of 1.5 N or greater. The projected area quantified by the radius (d) of interacting volume underneath the indent tip can be a better representation of the affecting area by indentation. Porous structure shows greater microcreep and shorter characteristic time, the contact creep function $[L(t) - L(0)]$ of outer C-S-H is 30% greater than that of inner C-S-H. The creep difference due to age is mainly manifested during the (long-term) deviatoric creep stage.

1. Introduction

As one of the most important properties of concrete, creep affects many aspects of structure performance, such as loss of prestress in concrete members and excessive deflection in bridge girders, etc. [1]. Establishing solid creep model is necessary not only for accurately predicting the time-dependent deformation and stress developments in concrete members during their service life, but also for material tailoring to achieve better concrete. The current creep theories and models are based on the macroscopic tests conducted on concrete specimens. The testing results rely greatly on the testing conditions, which might not reflect the true creep mechanism. As it is known, concrete is a highly heterogeneous composite containing randomly dispersed inclusions with length scale ranging from nano to micro meters. Many macroscopic properties of concrete originate from the underlying nano and microscale structures and phases. It has been found that C-S-H plays a significant role in concrete creep among all the unreacted and hydration products [2]. Jennings [3] proposed the existence of relationship between creep and the sliding and rearrange of C-S-H globules. Investigating from the microscale level is the fundamental requirement for understanding creep mechanism.

The micro mechanical and creep properties of materials can be obtained with the aid of nanoindentation technique [4]. Changes in load and indenter displacement over time are measured when an indenter tip is pressed into a sample in various loading modes, from which the elastic and creep properties are evaluated. This

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technique has been adopted for two decades to characterize homogeneous materials and thin films [5]. In the past decade, it has been increasingly used in characterizing the heterogeneous cementitious materials for modulus (E) and hardness (H) measurements [6–8]. At the same time, indentation technique was introduced to measure the creep behavior of cementitious materials due to its superior advantage of reducing significantly the measuring time to observe the creep behavior [7,9]. The indentation test is classified as either nanoindentation or microindentation depending on the magnitude of indentation depth [10,11]. Nanoindentation with peak load of 2 mN was normally used to experimentally prove the creep phenomenon of C-S-H and its logarithmic creep behavior. [7,12,13]. In contrast to nanoindentation test, microindentation does not probe individual phase, it is to characterize composite properties for hardened cement paste or other composite materials [14,15]. Nguyen et al. [9] measured microindentation creep of monophasic C-S-H under the maximum load of 1 N and 5 N, investigating the influence of porosity on indentation mechanical properties. Zhang et al. [16] measured the indentation creep of hardened cement paste under the load magnitude of 20 N, they concluded that microcreep behavior of hardened cement paste compares well to the macrocreep of concrete. The effect of peak load with magnitude ranging from 1 to 5 mN on mechanical properties of M and H has been investigated by Davydov et al. [17], they found that an increase of penetration depths leads to a general shift towards lower elastic modulus.

To further explore the creep mechanism and behavior across the nano to micrometer scale, the indentation technique is used in this study to determine the properties of hardened pastes by applying different magnitudes of peak loads at 2 mN, 0.1 N, 0.5 N, 1.5 N, and 5 N. Supplementary techniques, such as scanning electron microscopy (SEM) with backscattered electron (BSE) and continuous stiffness measurement (CSM) are also adopted to analyze the effects of microstructure, and magnitude of the applied load on mechanical and creep properties of hardened cement paste.

2. Methods

2.1. Materials and sample preparation

Ordinary Portland cement (OPC) was used as cementitious material. The chemical composition in terms of oxide mass percentage and the physical properties of OPC are listed in Table 1. The water/cement (w/c) ratio of pastes to be investigated is 0.3, 0.4, and 0.5. To refine the experimental work, the majority of the tests were conducted on the paste with w/c ratio = 0.3. After mixing, the fresh pastes were cast into plastic tubes with diameter of 2.5 cm, the tubes were then sealed and placed in a curing room with temperature controlled at 20 °C. The tubes were rotated manually during the setting period to prevent excessive bleeding.

For indentation and BSE tests, the 25-mm thick disc samples were cut from the tube sample at desired ages. The disc samples were then processed by grinding, polishing, and ultrasonic cleaning to obtain the final surface for testing. The grinding process was done by using silicon carbide papers following the sequence of 180, 400 and 1200 grit. The samples were then polished by using the polishing pastes containing diamond with particle size of 9, 3, and 1 μm. The samples were polished for 15 min under each diamond particle size. To avoid further hydration of cement, oil-based liquid was used as lubricant during the grinding and polishing processes, and the samples were cleaned in an ultrasonic bath filled with alcohol for removing the debris on sample surface.

2.2. Microindentation test

The Nano Indenter G200 with Berkovich tip was used to measure the micro-mechanical and creep properties of hardened pastes under the maximum loads of 0.1, 0.5, 1.5, and 5 N. For 0.1 N test, a smaller indent probe with load capacity up to 0.5 N and resolution of 0.05 mN was used, while for the greater load tests (0.5, 1.5, and 5 N), an indent probe with load capacity up to 10 N and resolution of 1 mN was used. Grid indentation was performed on the randomly selected areas. The indents were arranged at a certain spacing to ensure each indent to be an independent statistical event.

The typical indentation load (P)-depth (h) curve containing loading, holding, and unloading processes is shown in Fig. 1. The loading and unloading rates were set such that each process was done within 10 s. The holding period was 180 s. By analyzing the initial part of the unloading curve, the mechanical properties (indentation modulus M and hardness H) of the indented area were obtained based on Oliver-Pharr method [18]:

$$M = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A_c}} \tag{1}$$

$$H = \frac{P_{\max}}{A_c} \tag{2}$$

The indentation modulus M of material can be linked to its Young's modulus E and Possion's ratio ν through:

$$\frac{1}{M} = \frac{1-\upsilon^2}{E} + \frac{1-\upsilon_i^2}{E_i} \tag{3}$$

where, $S = \left(\frac{dP}{dh}\right)_{h=h_{\max}}$ is the contact stiffness and determined from the slope of the initial part of the unloading curve as shown in Fig. 1; h_{max} is the maximum indentation depth; β is geometrical correction factor, for the Berkovich tip used in this study, $\beta = 1.034$; P_{max} is the maximum or peak load; $E_i = 1141$ GPa and $\upsilon_i = 0.2$ are the elastic modulus and Poisson's ratio of diamond indent tip;A_c is the projected area of contact, it is a function of the half cone angel $\theta = 70.32°$ and the contact depth h_c:

$$A_c = \pi \cdot (\tan\theta \cdot h_c)^2 \tag{4}$$

h_c can be calculated from [18]:

$$\frac{h_c}{h_{\max}} = 1 - 0.75\frac{P_{\max}}{Sh_{\max}} \tag{5}$$

Eqs. (1) and (2) are derived based on the material homogeneity, they have been applied to and proven to be appropriate for the heterogeneous materials as well [4].

A contact creep compliance $L(t)$ has been introduced to describe the creep behavior of material under the indentation load. $L(t)$ is a material property which depends neither on the probe geometry nor on the load magnitude [16]. For conical indentation test, the indented material deforms plastically even at the lowest applied load. Base on the assumption that the plastic deformation appears only during the loading period instead of the holding and unload-

Table 1
Chemical composition and physical properties of Portland cement.

SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Na_2O (%)	K_2O (%)	Ignition loss (%)	Specific gravity	Blaine fineness (cm^2/g)
20.55	4.59	3.27	62.5	2.61	2.93	0.53	0.83	1.77	3.14	3500