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Strain-rate sensitivity of cement paste by microindentation continuous stiffness measurement: Implication to isotache approach for creep modeling

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ABSTRACT

Strain rate ($\dot{\varepsilon}$) of concrete is crucial for structures due to its significant effect on the mechanical properties of concrete. Understanding strain rate effect can ensure safe design of concrete structures with enhanced performance. In this study, strain rate effect on time-dependent deformation and mechanical properties of hardened cement pastes is investigated by performing continuous stiffness measurement (CSM). The w/c ratios of hardened cement pastes are 0.3, 0.4, and 0.5, the applied strain rates are 0.01 s^{-1}, 0.05 s^{-1}, 0.1 s^{-1}, and 0.5 s^{-1}. Experimental results show that the contact hardness (H) increases with increasing strain rate, and their relationship can be well captured by an empirical power law equation. However, strain rate has a negligible effect on the elastic modulus (E). The viscoplastic depth (h_{vp}, shown in Fig. 2d)-force (P) curve, which is controlled by the micro creep behavior of cement pastes, is also affected by the strain rate. Similar to the long-term creep behavior of soils, a unique isotache-type h_{vp}-P-$\dot{\varepsilon}$ relationship is found for hardened cement pastes at micro level. The results preliminarily confirm the applicability of isotache approach to characterize the time-dependent behavior of cement pastes at micro level.

1. Introduction

Creep is one of the most important mechanical properties of concrete, which affects the durability and serviceability of concrete structure profoundly [1]. Although extensive investigations have been conducted on this topic, concrete creep and its origin are still not fully understood [2]. One possible reason might be that most experimental research efforts were focused on the macroscopic creep behavior of concrete, which is time consuming and impossible to reveal the true mechanism due to the complex heterogeneous microstructure of concrete. It is well acknowledged that the complex creep behavior of concrete depends significantly on its microstructure. Therefore, to understand the creep mechanism of concrete fully and to predict its creep behavior correctly, one could think of investigating the creep behavior of cement paste on the microscopic scale [3].

Recently, instrumented indentation has been increasingly used in measuring the mechanical properties (i.e. elastic modulus and hardness) of cement-based materials at micro level because of its high resolution in recording load and depth data [4–6]. Inspired by the successful application of this technique on studying the elastic properties, some researchers attempted to utilize such technique to investigate the micro creep behavior of cement-based materials [7–10].

The previous studies have shown that there are analogies between the creep property of cement paste and that of soils [8], thus it might be a promising means to possibly uncover the creep mechanism of cement-based materials based on the findings in soil creep. In the light of the success in isotache approach for modeling soil creep, Vandamme [11] suggested that concrete creep might be characterized by the isotache-type model.

It is widely accepted that the volume changes of soil materials under a constant effective stress are caused by the viscoplastic behavior (creep) of the soil skeleton. Such a viscoplastic deformation not only takes place during the second consolidation, but also during the primary consolidation. The isotache approach [12], a strain rate-dependent model, is very useful and highly effective for describing the viscoplastic behavior of soils. In this approach, the viscoplastic strain is not only determined by the effective stress, but also by the strain rate. In other word, the effective stress and the viscoplastic strain relationships of soils can be expressed as a series of strain rate lines (isotache lines), these isotache lines can be normalized into one unique line by a corresponding reference stress. Then the consolidation settlement of soils in engineering practice can be estimated based on the isotache model directly. The validity of this model for soils has been experimentally confirmed by previous researches [13–14].

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It has also been reported that cementitious materials at macro level show a significant strain rate sensitive behavior, and strain rate is a fundamental issue for the safe service of concrete structures due to its significant effect on the mechanical properties and deformation of concrete [15]. This topic has received considerable attentions over the past several decades [16–18], however, most researches about the strain rate effect were focused on elastic modulus, strength, Poisson's ratio, etc. at macro level [17]. Acker et al. [18] proposed an incremental model (also called the equivalent time method) considering the strain rate effect to evaluate the viscoelastic property of concrete in the 1990s, which is similar to the isotache approach for soils. Although increasing researches on the micro creep behavior of cementitious materials have been conducted by instrumented indentation recently [7–10], to the best of the authors' knowledge, there has been no experimental study on the strain rate effect at the micro scale.

The objectives of this work are to verify the strain rate effect on the time-dependent deformation and mechanical properties of cement pastes at micro level, and possibly to improve the general understanding of creep mechanism. Continuous stiffness measurement in the instrumented indentation system is used to investigate the effect of strain rate on the elastic modulus, hardness, and time-dependent deformation of hardened cement pastes with w/c ratios of 0.3, 0.4, and 0.5. The indentation strain rate defined as $2\frac{1}{h}\frac{dh}{dt}$($h$ is the indentation depth), is applied at 0.01 s^{-1}, 0.05 s^{-1}, 0.1 s^{-1}, and 0.5 s^{-1}, respectively. The microstructure of the cement pastes is characterized by the backscattered electron images analysis (BSE-IA). Applicability of the isotache approach to characterize time-dependent deformation of cement paste at micro level is discussed.

2. Methodology

2.1. Isotache approach in soil

There have existed many approaches for practical and theoretical evaluation of long-term creep behavior of soils [13]. Among these approaches, the isotache approach [12–14] is particularly successful and has attracted a lot of attentions. However, it should be kept in mind that this approach doesn't show the creep behavior (i.e., time-dependent strain under constant stress) directly. Instead, the original isotache approach suggests that the time-dependent deformation of soils is controlled by a unique stress (σ)-strain (ε)-strain rate ($\dot{\varepsilon}$) relationship. As shown in Fig. 1, σ-ε relations of soils vary with $\dot{\varepsilon}$, however, the σ-ε relations under different $\dot{\varepsilon}$ can be normalized into one unique σ-ε curve by a reference stress (σ_p) which is a function of $\dot{\varepsilon}$. The unique σ-ε-$\dot{\varepsilon}$ relationship can be simply described by two curves as expressed by Eqs. (1) and (2), respectively.

$$\sigma_p = f(\dot{\varepsilon}) \tag{1}$$

$$\frac{\sigma}{\sigma_p} = g(\varepsilon) \tag{2}$$

where, ε is the total strain; $\dot{\varepsilon}$ is strain rate; σ is the vertical effective stress; σ_p is the reference stress for normalization, in geotechnical engineering, the preconsolidation pressure is usually selected as the reference stress.

The isotache approach implies that the time-dependent deformation of soils is independent of previous history, which depends only on the present stress and strain rate [13]. This approach has a specific physical meaning: Eq. (1) characterizes the relationship between the reference stress and strain rate, which represents the capability of the soil skeleton to creep. Eq. (2) is the representative of the reaction of soil structure and their mineralogy and geological histories, which varies with the soil types. To predict the time-dependent deformation of soils using the isotache approach, Eqs. (1) and (2) can be determined by one long-term consolidation test and one constant rate of strain consolidation test using an oedometric cell, respectively. Once these two relationships are known, they can be used directly to analyze the consolidation problems of soils [14].

2.2. Method of verifying isotache approach for cement paste at micro level

Since there are analogies between the creep of cement paste and that of soils, the simple but useful isotache model might be capable of characterizing creep of cement paste. In order to verify the applicability of isotache approach to cement paste at micro level, it is necessary to demonstrate that the strain rate has an effect on time-dependent deformation of cement paste by illustrating that the σ-ε curves vary with $\dot{\varepsilon}$. Then, it is essential to demonstrate that the σ-ε curves can be normalized with respect to a reference stress so that all the σ-ε curves can fall on one unique curve.

Continuous stiffness measurement (CSM) technique is a well-established technique for obtaining elastic modulus and hardness data continuously during the loading stage. It also involves pressing an indenter into contact with a test material while continuously measuring contact force and penetration. But in addition, a small oscillation is superimposed on the semistatic force [19]. Moreover, CSM is capable of recording the force (P)-indentation depth (h) developments under various strain rates [19]. The indentation strain rate in a force-controlled nanoindenter can be defined as follow [20]:

$$\dot{\varepsilon} = \frac{1}{P}\frac{dP}{dt} \tag{3}$$

It is noted that it does matter whether one defines strain rate as $\frac{1}{P}\frac{dP}{dt}$ or $\frac{1}{h}\frac{dh}{dt}$ because $\frac{1}{P}\frac{dP}{dt}$ is directly proportional to $\frac{1}{h}\frac{dh}{dt}$ [20]. Since $P = ch^2$ holds during the holding stage for a cone indenter [21], one can obtain $\dot{\varepsilon} = \frac{1}{P}\frac{dP}{dt} = 2\frac{1}{h}\frac{dh}{dt}$ by differentiating both sides of $P = ch^2$ with respect to time and then dividing both sides of the differential equation ($\frac{dP}{dt} = 2ch\frac{dh}{dt}$) by $P = ch^2$ accordingly.

CSM is a promising technique to illustrate strain rate effect on the time-dependent deformation of cement paste. Indeed, this technique has been successfully used for investigating the strain rate effect on the mechanical property of composites and multilayered materials (e.g. carbon-fiber composite and magnetic tapes) [19]. The schematic measuring system of CSM is shown in Fig. 2b. During the measurement, a harmonic force $F(t) = F_0e^{iwt}$ (F_0 is the amplitude of the applied sinusoidal force) is imposed to the nominally increasing load (P) to the indenter. The depth response of the indenter $z(t) = z_0e^{(wit - \Phi)}$ (z_0 is the amplitude of the sinusoidal depth) at the excitation frequency (ω) and the phase angle (Φ) between the force and the depth are measured continuously as a function of depth. A typical P-h curve obtained from the CSM measurement is shown in Fig. 2c. The second-order differential equation describing the relationship between the harmonic force and response of indenter during the loading stage is [20]:

$$M\ddot{z}(t) + D\dot{z}(t) + Kz(t) = F(t) \tag{4}$$

where, M is the moving mass of the transducer; $D = D_i + D_c$ is the effective damping coefficient, D_i is the damping coefficient of the indenter, D_c is the damping coefficient of the contact; $K = K_s + S$ is the effective stiffness, K_s is the stiffness of the transducer, S is the stiffness of the contact.

Combining the relevant equations above, the contact stiffness can be expressed as follow [20]:

$$S = \left[\frac{1}{(F_0/z_0)\cos\Phi - (K_s - Mw^2)} - \frac{1}{K_f}\right]^{-1} \tag{5}$$

where, K_f is the stiffness of the frame.

Once the contact stiffness is determined, the contact depth (h_c) for a conical indenter can be expressed as Eq. (6) based on Sneddon's P-h relation [21].

$$hc = h - 0.75\frac{P}{S} \tag{6}$$

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