



# Loading history dependence of retardation time of calcium-silicate-hydrate



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## HIGHLIGHTS

- The dependence of retardation time of C-S-H on loading history was found by nanoindentation test.
- The relation between the retardation time and loading rate follows a negative power-law.
- The relation between the retardation time and creep load follows a power-law.

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## ABSTRACT

Calcium-silicate-hydrate (C-S-H) exhibits a viscous effect, which significantly affects long-term deformation of concrete. In this study, treated cement pastes are examined to investigate the viscous effect of C-S-H. Creep experiments are performed by the nanoindentation technique, and the results indicate that creep deformation in the dwelling period is dependent on loading history. This phenomenon can be explained by the motion activation of different kinematic elements of C-S-H as well as micro-damage evolution. Further analysis indicates that the effect of loading rate and magnitude of creep load on retardation time approximately follows a power-law.

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## 1. Introduction

Concrete is a time-dependent material because the stress in concrete depends on the strain as well as the strain rate (or loading rate) [1,2]. This character of time-dependency is also considered a viscous effect. Previous studies experimentally and theoretically investigated creep in concrete to study this type of viscous effect (e.g., [3,4]). Based on experimental investigations, Gopalakrishnan et al. [5] and Chuang et al. [6] pointed out that concrete creep under a uniaxial condition exceeded that under multiaxial compression of the same magnitude in a given direction. Brooks et al. [7] reported that the creep strain is related to the compressive strength of concrete as well as ambient temperature. Dussere et al. suggested that the Drucker–Prager creep model could perfectly characterize the creep behaviour of concrete subjected to high temperature [8]. Their analysis revealed that the creep flow

is dilatant and is associated with the stress state and loading conditions. Specifically, the creep character of concrete differs with respect to time and mix design. Huo et al. [9] studied the creep of high performance concrete and suggested that it was characterized by a more rapid development at an early age when compared with that of traditional concrete. An investigation carried by Day et al. [10] indicated that the creep in concrete with fly ash was less than that of concrete without fly ash in the same condition. Geng et al. [11] proposed a creep model to accurately predict the creep of recycled aggregate concrete.

Creep mechanism is related to a time-dependent property. A few studies focused on the mechanism of different creep kinetics. A new creep experimental method was implemented by Bernard et al. [12]. Wittmann et al. [13] improved this method and studied the creep kinetics of concrete. In the study, an experiment was performed to show that, there are at least two distinct creep kinetics, namely a long-term deviator creep followed by a short-term volume creep. The kinetics of long-term deviator creep and short-term volume creep were investigated by Ulm et al. [14] and Bazant

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et al. [15]. Chen et al. [16] and Feldman [17] developed another approach that considered the effect of density and porosity on concrete creep.

Creep implies that concrete possesses a viscous effect [18,19]. However, until 2004, a limited number of studies explored the mechanism of this kind of viscous effect [20]. Recently, researchers noted that the viscous effect could be caused by C-S-H. For instance, Nguyen et al. [21] found that the creep of pure C-S-H powder exceeds that of hydrated cement paste. Alizadeh et al. [22] further proposed that the viscoelastic performance of C-S-H is caused by the presence of interlayer water. Although creep tests were performed by using macroscopic tests, they were unable to completely assess long-term creep deformation. Vandamme et al. [23] employed nanoindentation techniques to measure the creep properties of C-S-H in cement paste, and found that the relation between creep displacement and time could be expressed by a log-arithmetic or a power function. Additionally, Šmilauer et al. [24] presented a FFT-based homogenization method to study the viscoelastic behaviour of C-S-H in cement paste. The results revealed that the decay rate of C-S-H creep is lower than that of cement paste.

Generally, the viscous effect of C-S-H also could be demonstrated by its relaxation time or retardation time. However, Nguyen et al. [21] noted that the mechanical behaviour of pure C-S-H powder is different from that of C-S-H gel in hydrated cement paste. Hence, the method proposed by Hu et al. [25] was adopted in the present study. That is, the creep deformation of C-S-H was investigated by performing a nanoindentation technique on samples of cement paste. The experiment involved investigating the effect of different loading rates as well as creep loads on the creep properties of C-S-H. Based on the experimental results, an elastic-viscoelastic-viscous (EVEV) model was proposed to describe the creep character of C-S-H. The results obtained in the study indicated that the retardation time of the C-S-H gel is related to the loading history and that the relation between retardation time and loading rate and relation between retardation time and creep load approximately obey a power law.

**2. Experimental**

**2.1. Materials**

In this study, Portland cement paste (composed of Type I Portland cement) was used to produce the specimens of concrete. The chemical components of the cement are listed in Table 1.

The water-to-cement ratio of the specimens was fixed to be 0.45. All specimens are in cubic shape with the dimensions of 30 mm × 30 mm × 30 mm. The specimens were cured initially for 24 h in an ambient condition. After then, they were demoulded and placed in standard curing conditions (23 ± 1 °C and 95% relative humidity) curing for 28 days.

After curing and before testing, the specimens were placed in solutions of alcohol for at least 48 h to prevent continued hydration. The specimens were then cut with an Isomet diamond saw into thin slices (3 mm × 15 mm × 30 mm). Based on the experiment method suggested by Miller et al. [26], the exposed surfaces of slices were ground and polished such that the test requirements were satisfied. Following this, the slices were cleaned in an ultrasonic bath with n-decane for 5 min.

**2.2. Method**

The nanoindentation test was performed by MTS G200 with a diamond Berkovich indenter, and the diameter of the indentation head is 20 nm (Fig. 1). In order to investigate the effect of loading history on the viscosity of C-S-H, the loading path was designed as follows: 1) Loading from 0 to 10 mN (creep load) at different load-

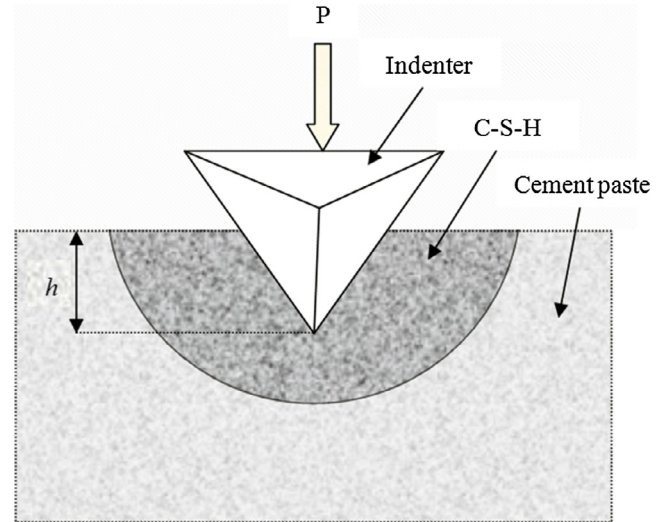


Fig. 1. Schematic of the nano-indentation test.

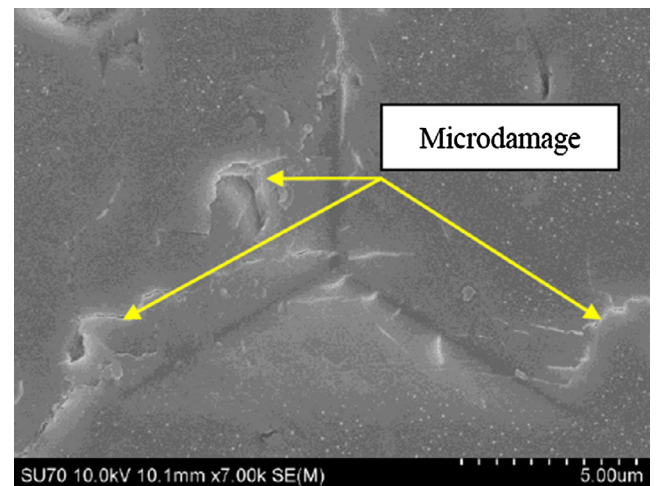


Fig. 2. A SEM image of residual impression of the C-S-H gel.

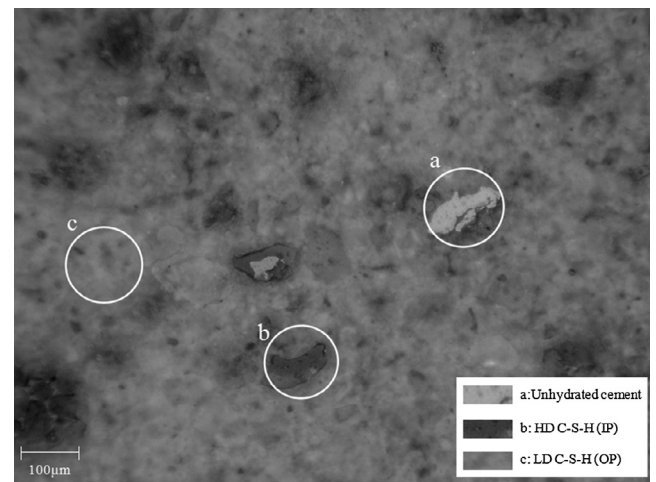


Fig. 3. A SEM-BSE photograph of the cement paste.

**Table 1**

The chemical composition of Type I Portland cement (wt%).

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	SO <sub>3</sub>	Na <sub>2</sub> O	LOSS	f-CaO	C <sub>3</sub> S	C <sub>2</sub> A
22.35	4.03	2.72	1.96	63.73	2.40	0.54	1.65	0.43	53.45	6.23

ing rates of 1 mN/s, 5 mN/s and 10 mN/s, and this was followed by keeping the load constant for 50 s (this time interval is referred to as dwelling time in the study) and finally linearly unloading back to zero in 10 s, 5 s, and 1 s, respectively; 2) Creep

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