



## Microindentation creep of monophasic calcium–silicate–hydrates



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### ABSTRACT

Microindentation creep results for monophasic synthetic C–S–H ( $C/S = 0.6–1.5$ ), 1.4 nm tobermorite, jennite and calcium hydroxide at 11%RH are reported. Creep results for well hydrated cement paste and  $C_3S$  ‘composite’ systems are also described. The significance of the co-linear behavior of creep modulus functions of indentation modulus and indentation hardness for C–S–H obtained by microindentation and nanoindentation methods is discussed. The porosity dependence of creep modulus and the general equivalence of density values determined by helium pycnometry and by calculations employing unit cell dimensions (obtained using X-ray crystallography techniques) are also discussed in terms of postulates for the existence of two types of C–S–H. Comment on the compatibility of the creep modulus data for 1.4 nm tobermorite and jennite with models of C–S–H present in cement paste is provided.

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### 1. Introduction

There has been a plethora of publications over several decades dealing with the creep behavior of cement-based materials [1]. These have included numerous contributions on creep mechanisms. Indentation techniques for measuring creep of cement systems have been introduced recently [2]. They have the advantage of significantly reducing the time scale for observing creep phenomena. Recent studies utilizing nanoindentation techniques conclude that creep is likely due to the rearrangement of nanoscale particles associated with theories of granular physics [3,4]. Creep is then said to originate from a rearrangement of nanoscale C–S–H particles around limited packing densities of 3 compositionally similar but structurally distinct C–S–H phases: low density (LD), high density (HD) and ultra-high density (UHD). The separation of these phases is based on deconvolution methods applied to indentation modulus and indentation hardness frequency distributions obtained from nanoindentation tests performed on hydrated cement paste. It was stated that creep properties of C–S–H have never been measured directly [2]. Further the role of interlayer water in the creep process was not delineated. Stress relaxation measurements of C–S–H having varied  $C/S$  ratios were subsequently reported by the current authors [5]. It was observed that removal of water from interlayer spaces modified the viscoelastic behavior of C–S–H. A sliding mechanism involving the translation

of C–S–H layers themselves taking into account the interaction of silica tetrahedral and cations in the interlayer region at various moisture contents was proposed. This mechanism is in contradistinction to that proposed by Vandamme and Ulm [2].

A study was designed to determine the indentation creep behavior of pure C–S–H phases prepared from compacted specimens including synthetic C–S–H, 1.4 nm tobermorite and jennite. Microindentation techniques were used as these systems are monophasic and interference from other phases is avoided. Issues with the relative size of homogenous domains and the interaction volume beneath the indenter that may arise in an indentation experiment, as well as surface roughness influences, are avoided. Creep measurements on calcium hydroxide, hydrated  $C_3S$  and hydrated Portland cement were also included in the study. Hydrated  $C_3S$  and hydrated Portland cement paste are, of course, multicomponent systems containing phases other than hydrated silicates. The creep measurements for these materials represent ‘composite’ behavior and are included for comparison purposes. Attempts at separation of frequency distributions of microindentation parameters, in this context, would of course be meaningless.

The microindentation creep, indentation modulus and indentation hardness results are reported. Insights on the mechanism of creep in cement-based materials are provided. The merits of interpreting creep as a nanogranular-centered phenomenon and the existence of two types of C–S–H based on nanoindentation measurements are debated.

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## 2. Experimental

### 2.1. Materials

Six cementing systems were prepared including four monophasic materials: synthetic calcium-silicate-hydrate (C–S–H with C/S = 0.6, 0.8, 1.0, 1.2 and 1.5); 1.4 nm tobermorite; jennite; hydrated Portland cement paste (water/cement ratio = 0.40); hydrated C<sub>3</sub>S (water/solid ratio = 0.40); reagent grade calcium hydroxide (obtained from Anachemia).

C–S–H: synthetic C–S–H was produced from the pozzolanic reaction between CaO and amorphous silica in excess water (water/solids = 11). Calcium oxide was obtained by calcining reagent grade calcium carbonate at 900 °C. Reactive silica (CAB-O-SIL, grade M-5 from Cabot Corporation, USA) was heated at 110 °C to remove any surface adsorbed water. Distilled water was de-aired and used for the reactions. All materials were kept sealed in N<sub>2</sub> purged bottles until they were used. Variation in C/S ratio was achieved by adjusting the stoichiometric amounts of the reactants. The reaction period was 6 months. The material was then filtered and dried under vacuum for 4 days at room temperature. The dried C–S–H was stored in nitrogen purged glass vials before the experiments. Characterization of these materials by X-ray diffraction (XRD) and thermal methods gave results directly comparable to C–S–H (I) as reported by Taylor [6].

1.4 nm tobermorite: the reactants (CaO and SiO<sub>2</sub>) were prepared as described above. The C/S ratio was 0.9. The reactants were placed in a high density polyethylene bottle mixed in excess deionized water (water/solids = 11) and maintained at 80 °C using a heating wrap. The mixture was continuously agitated with a magnetic stirrer for a period of 4 months. The material was then filtered and dried under vacuum for 4 days at room temperature. The XRD spectrum and TGA curve were similar to those obtained by Yu and Kirkpatrick [7].

Jennite: the reactants (CaO and SiO<sub>2</sub>) were prepared as described above. The C/S ratio was 1.4. The reactants were placed in a high density polyethylene bottle mixed in excess deionized water (water/solids = 11) and maintained at 80 °C using a heating wrap. The mixture was continuously agitated with a magnetic stirrer for a period of 4 months. The material was then filtered and dried under vacuum for 4 days at room temperature. The XRD pattern was similar to that obtained by Yu and Kirkpatrick [7], Gard and Taylor [8] and Hara and Inoue [9]. The TGA curve matched that published by Yu and Kirkpatrick [7].

Portland Cement Paste: The Portland cement paste (made with Type I Portland cement) was prepared using a water/cement ratio of 0.40. Rectangular prisms (250 × 100 × 12 mm) were cast. The samples were vibrated and stored in a moist curing room for 24 h. They were then demoulded and curing was continued for 3 years in a saturated lime solution. Thin slices (1 × 12 × 60 mm) were cut from the paste prism using an Isomet diamond saw. Selected slices were also ground into a fine powder and passed through sieve No. 100 (nominal opening size of 0.149 mm) for fabrication into compacted specimens.

Hydrated C<sub>3</sub>S Paste: Tricalcium silicate paste was prepared using a water/solid ratio of 0.40. Specimens with circular cross-section (25 mm in diameter) were cast in glass cylinders. The specimens were demolded after 24 h and sheathed in a rubber membrane containing a few drops of lime saturated water. They were then stored in stoppered glass tubes for 32 years. The use of 32 year old samples assures that the degree of hydration approaches 100%. Slices 1 mm thick were then cut and ground into a fine powder for fabrication into compacted specimens.

### 2.2. Humidity conditioning and environmental control

Specimens for all six systems were conditioned for several weeks at 11%RH in vacuum desiccators containing saturated lithium chloride solution. The powders were conditioned at 11%RH before compaction and for one week days after compaction. Theoretically there is a monolayer of water on the surfaces of the particles in addition to interlayer water at this humidity. The choice of 11%RH is a particularly significant datum point in the study of porous calcium silicate hydrate systems as the influence of capillary water on mechanical performance can be negated. The effects of 'structural' water can be isolated and controlled by incremental removal of interlayer water from this moisture condition if required. Mechanical properties e.g. modulus of elasticity and hardness are humidity dependent [10]. Modulus of elasticity on first drying is at a maximum value as opposed to hardness which reaches a minimum value at this humidity [11]. The associated mechanisms are discussed in a detailed description of the Feldman-Sereda microstructural model for cement paste.

Precautions were taken to minimize any contamination of the samples due to carbonation. Desiccators were purged with nitrogen gas (in a glove box) prior to conditioning of the samples. The microindentation tester was located within an environmental chamber kept at 11%RH. Carbonation, in general, is minimized at low humidities. The chamber also contained CO<sub>2</sub> absorbers. Transfer of samples prior to indentation testing took place from the desiccators placed in the chamber. Finally the samples were periodically checked for any carbonation after testing using TGA methods. Generally TGA methods indicated that the precautions were satisfactory with little or no carbonation detected.

### 2.3. Preparation of compacted specimens

Solid circular disc samples for all the powdered materials (from the six cementing systems) were prepared by pressure compaction in steel moulds with a cross-section of 25 mm. The thickness of most of the disc samples was nominally 1 mm. Numerous studies on the use and validity of compacts as models for hydrated cement systems have been published [10,12–15]. It has been shown that compacted specimens of powdered hydrated Portland cement have similar mechanical property-porosity relationships to that of the original hardened paste of the same material [10]. The porosity of compacted samples was determined using helium pycnometry or in the case of the phase pure minerals by calculation using published density values. The calculation is made knowing the apparent volume and the solid volume of the sample. Porosity is varied by controlling the compaction pressure.

### 2.4. Microindentation measurements

All the microindentation tests were performed using a CSM Instruments Instrumented Indentation Tester. The apparatus is housed in an environmental chamber. All tests were conducted at 11% RH on specimens equilibrated at 11%RH. Tests were conducted using a Berkovich indenter. The CSM microindentation instrument has a load range of 0.03–30 N with a resolution of 0.3 mN. The rate of loading in the current experiments was 2000 mN/min. The specimens were tested over a wide range of porosity values varying from about 10% to as high as 60%. There were approximately 25 indents on each sample at each porosity level for a total of approximately 125 indents for each material system. The composition of the solid phase for monophasic systems remains constant. Pure materials do not require as many

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