

Visco-elastic behavior of calcium-silicate-hydrates

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Materials Science and Technology

Introduction

Hardened concrete shows pronounced visco-elastic behavior under sustained loads (creep) / deformations (relaxation), see Fig.1. This visco-elastic response has a significant impact on the evolution of restraint stresses (manifesting as stress relaxation) and therefore cracking, in particular at early ages [1], and may lead to substantial deformations of concrete members in the long term [2]. Indeed, it plays an important role on the durability and service life time of concrete structures.

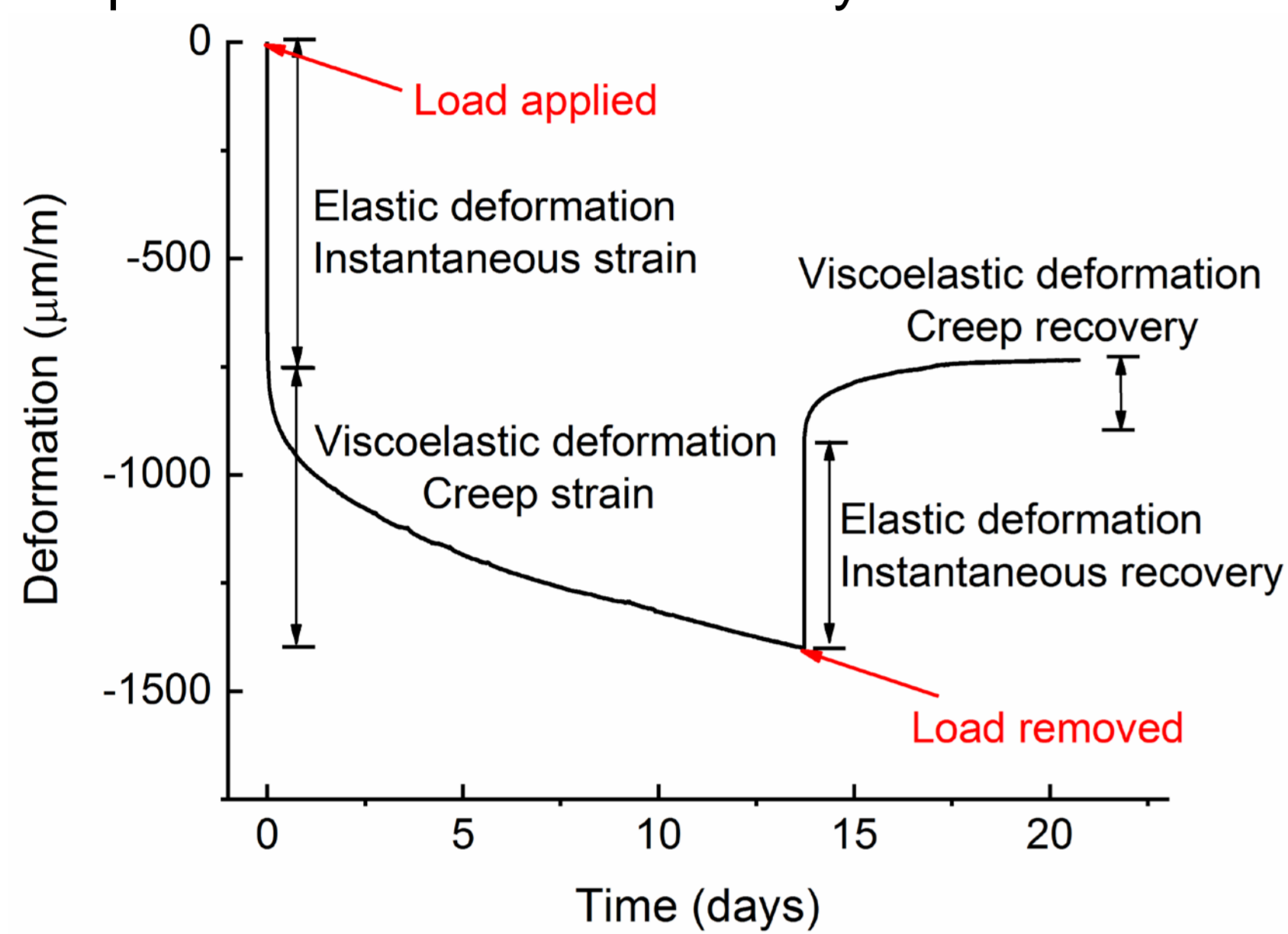


Figure 1 Deformation of normal Portland cement paste under an external load and when the load is removed

With decades' research, **calcium silicate hydrate (C-S-H)**, the main hydration products, with **water** has been dedicated to be the main phase for creep in concretes [3]. Different mechanisms associated with C-S-H and water of the visco-elastic behavior of cement-based materials were proposed in the literature. However, due to lack of studies on the C-S-H level, the real mechanism is unclear.

Objectives

The final goal of this project is to elucidate the mechanisms of the visco-elastic response of C-S-H and establish a feasible model of creep. In order to achieve that, some sub-objectives need to be accomplished:

- To study the effects of structure (e.g., C-S-H polymerization), Ca/Si and porosity of synthetic C-S-H gel on mechanical properties;
- To investigate the water movement (distribution) in C-S-H under stress;
- To clarify the interaction forces between C-S-H in different water environment to describe later the physical and chemical evolution of C-S-H.

Materials and methods

C-S-H is synthesized using the precipitation method, following the protocol in [4]. Studied Ca/Si: 0.8 - 2.0

Foil-like morphology of synthetic C-S-H is observed under microscope and similar structures with different Ca/Si are characterized with Fourier transform infrared spectroscopy (FTIR), see Fig.2.

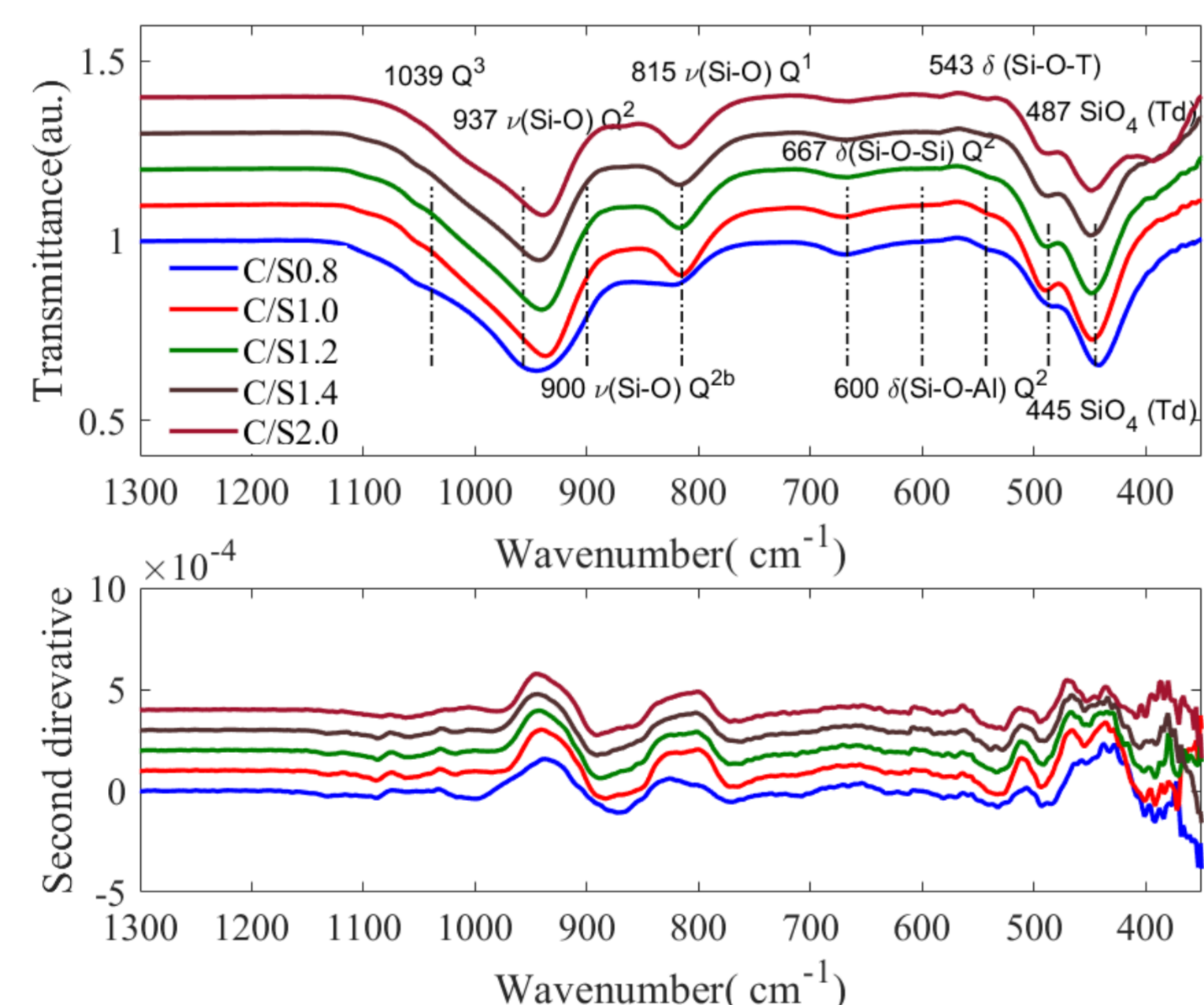
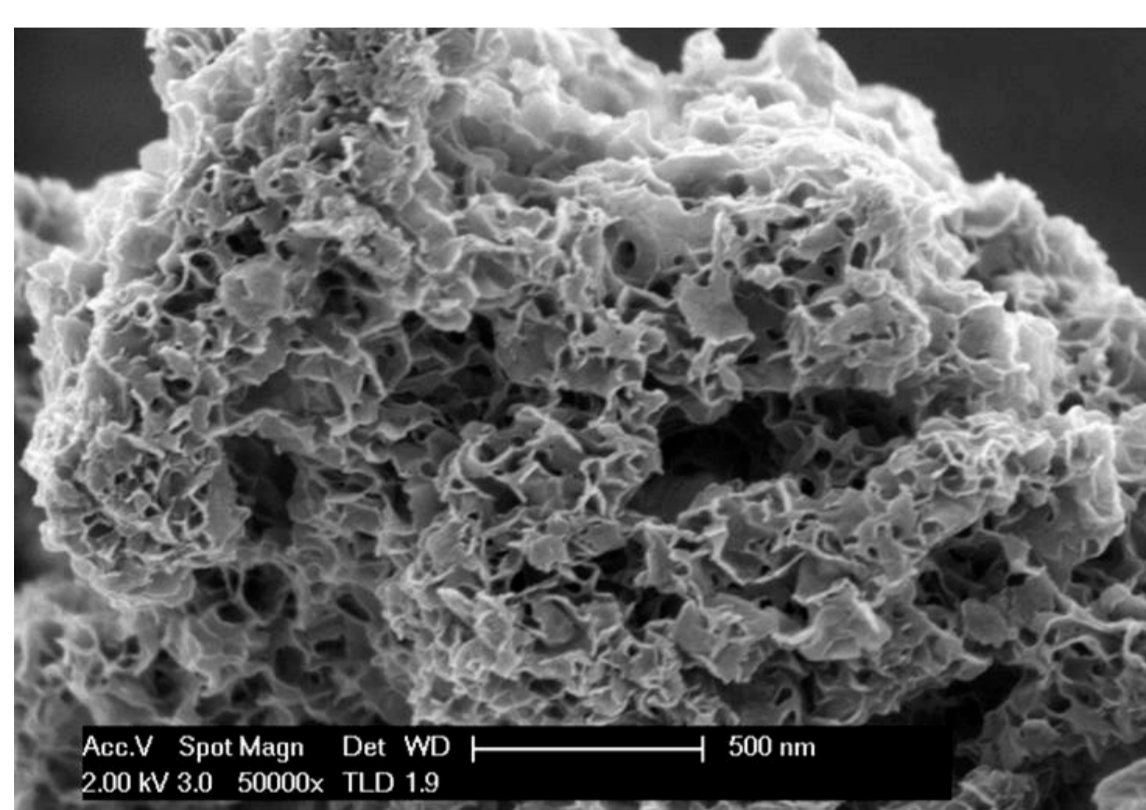


Figure 2 Left: Morphology of synthetic C-S-H (Ca/Si of 1.2); Right: FTIR spectra and band assignments

Following the framework of the entire project in Fig.3, at the macro and meso-scale levels, the water distributions will be studied. Mechanical properties will be determined with uniform C-S-H powder compacts. At the microscopic level, the interaction forces between C-S-H in different solutions will be investigated.

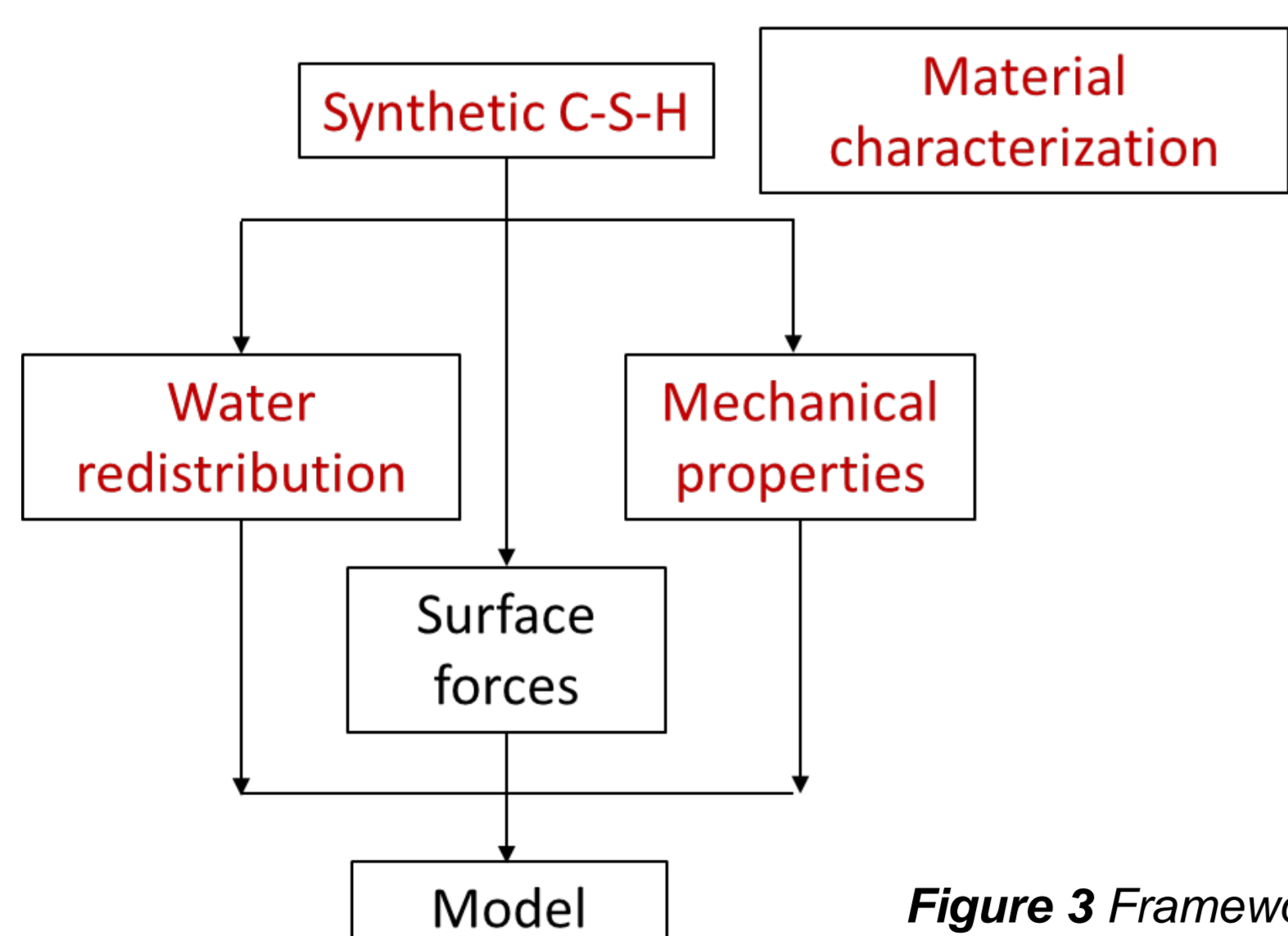


Figure 3 Framework of the current project.

- The water redistribution was studied with **¹H nuclear magnetic resonance (NMR)** *
- Mechanical properties are studied with both **micro-indentation** and **macroscopic creep tests**
- The interaction forces are studied with **extended surface force apparatus (eSFA)** **

References

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 [2] P. Z.P. Bažant, M.H. Hubler, Q. Yu, ACI Struct. J. 108 (2011) 766–774.
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 [5] M. Wyrzykowski, P. Lura, Cem. Concr. Res. 65 (2014) 58–63
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* The new 20MHz frequency NMR setup at Empa will be mainly used.

** Collaboration with laboratory of advanced fibers at Empa. eSFA at ETH Zurich will be used

Water redistribution under stress

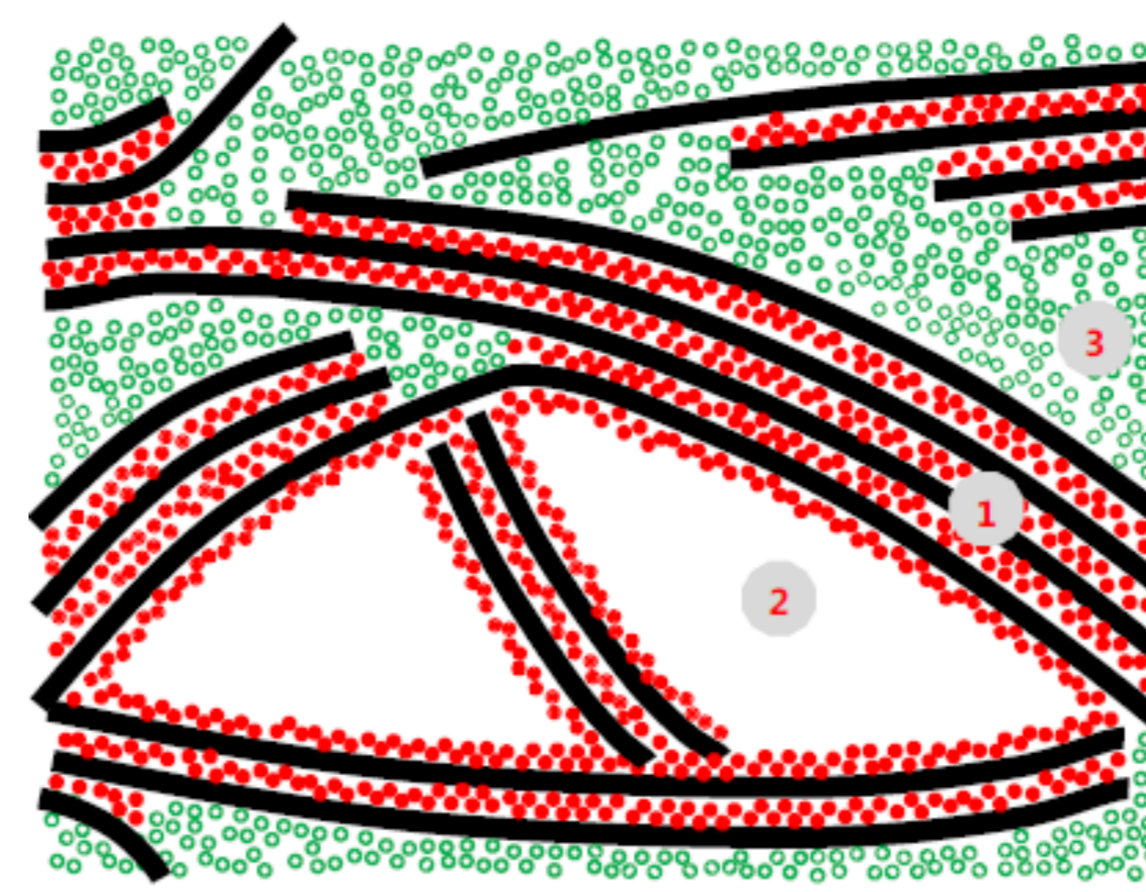


Figure 4 Schematic figure of C-S-H layers (black lines), interlayer water (○), gel water (⊙) and emptied gel pores (⊖) in cementitious materials [5]

Different water populations in cementitious materials: **capillary water**, **gel water** and **interlayer water** (Fig.4) are detected under ¹H NMR setup as separated peak signals (Fig.5).

Under thermal stress, the fraction of interlayer water decreased and the fraction of gel water and of capillary water increased (Fig.6). Contrary to what occurs in cement pastes, the water migration between gel water and interlayer water may not be fully reversible. It may be due to influence of the presence of elastic phases in cement pastes.

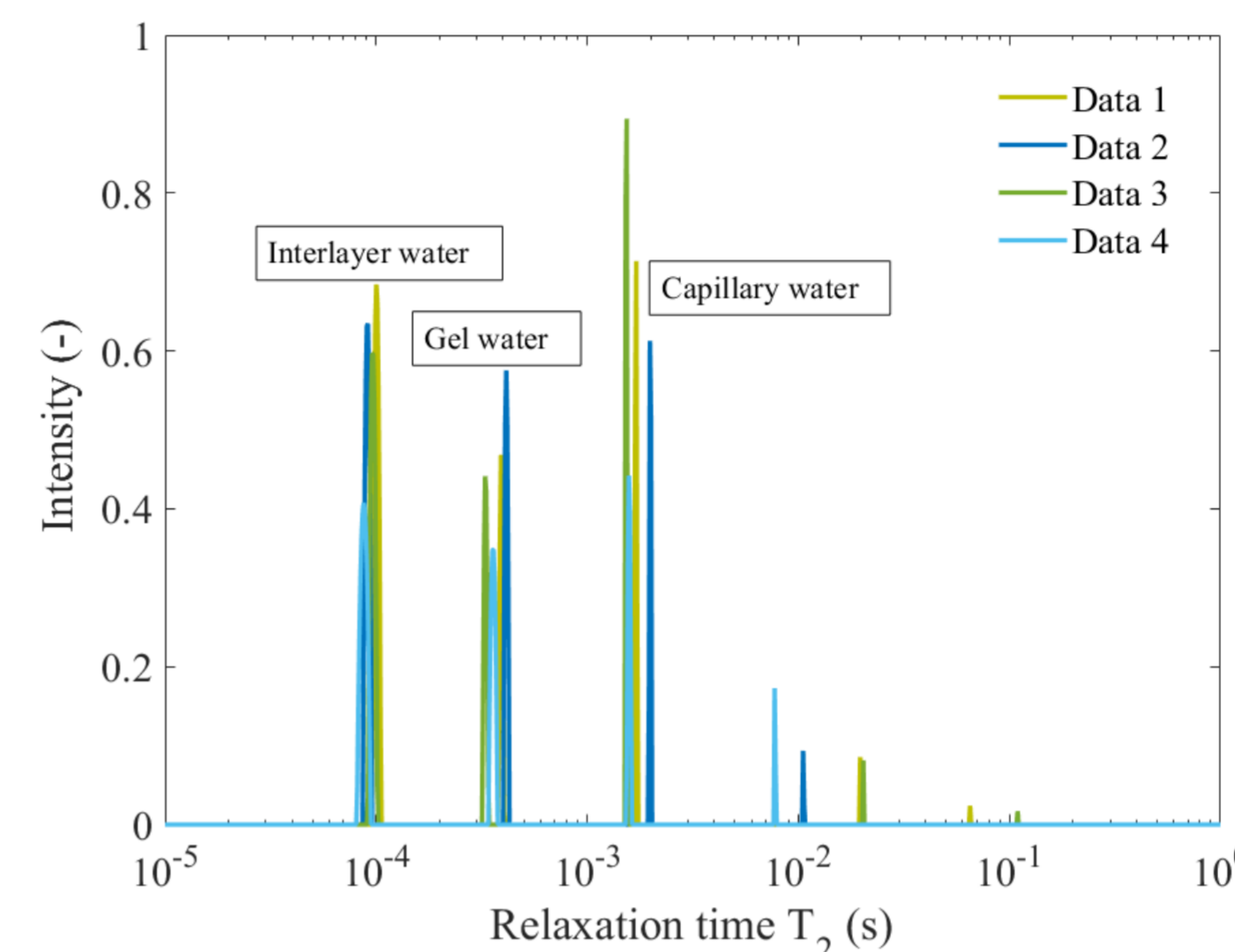


Figure 5 Detected signals from ¹H NMR for different water populations

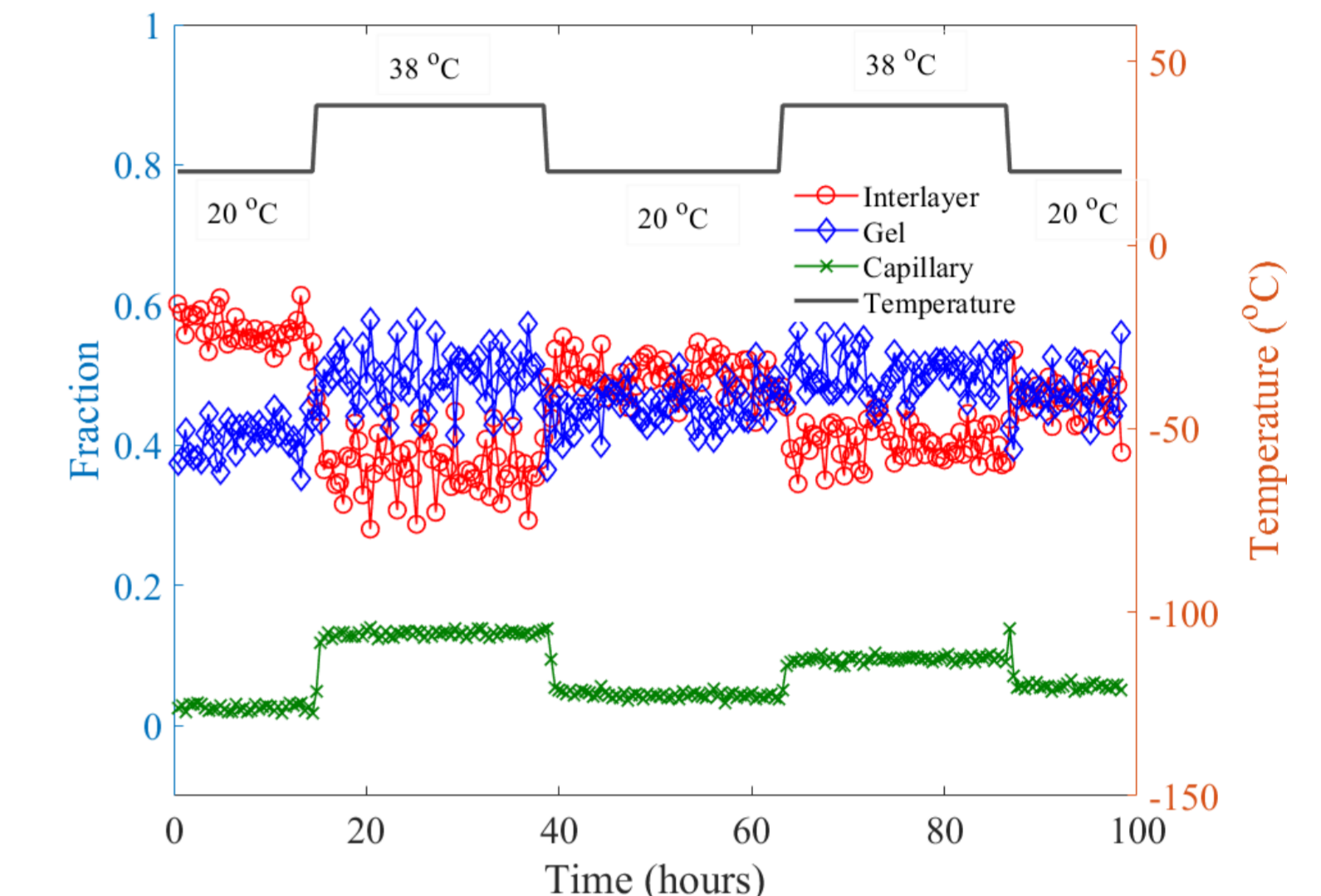


Figure 6 Water redistribution in synthetic C-S-H with Ca/Si of 1.2 under thermal stress

Micro-indentation results

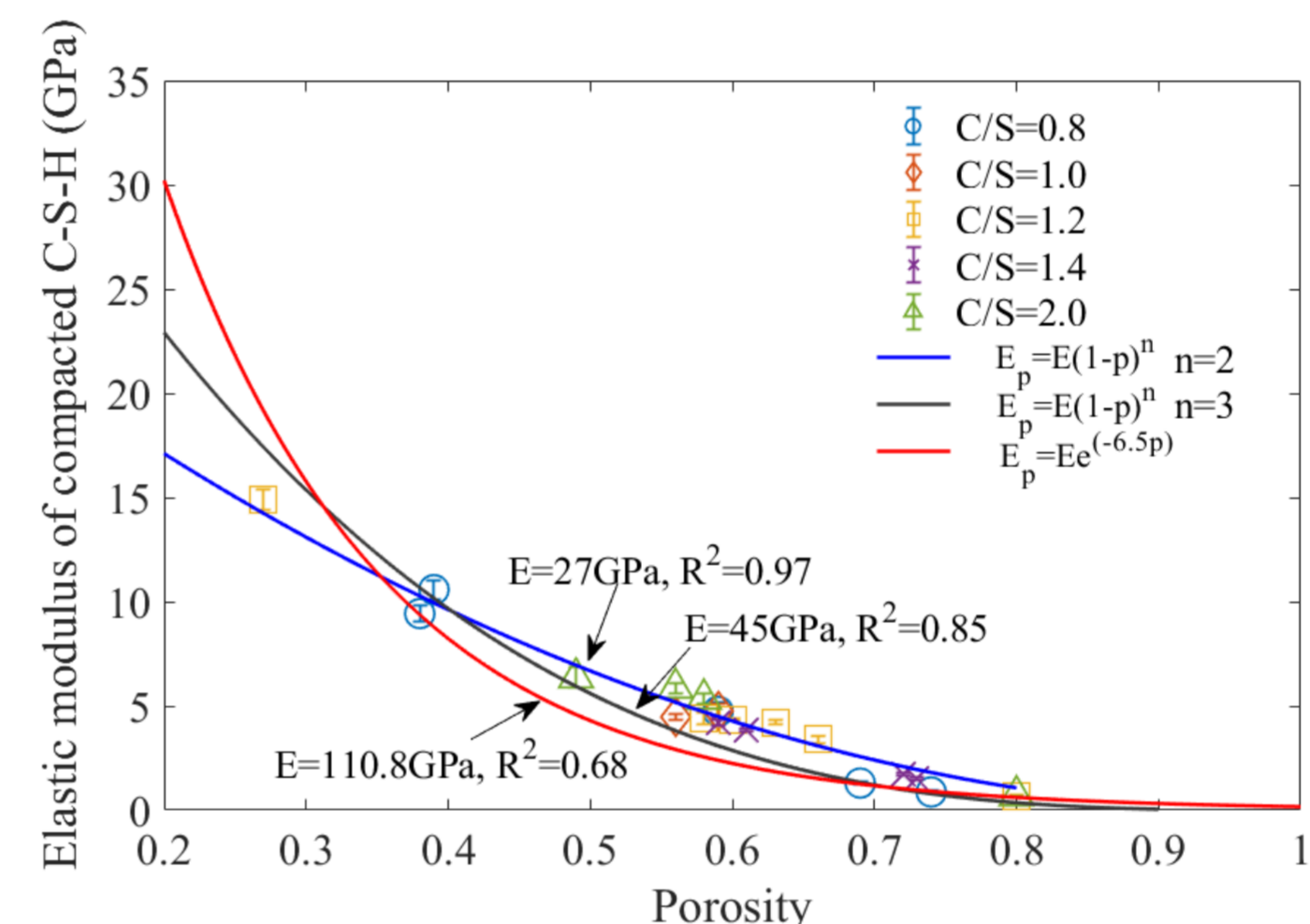


Figure 7 Elastic modulus of compacted C-S-H

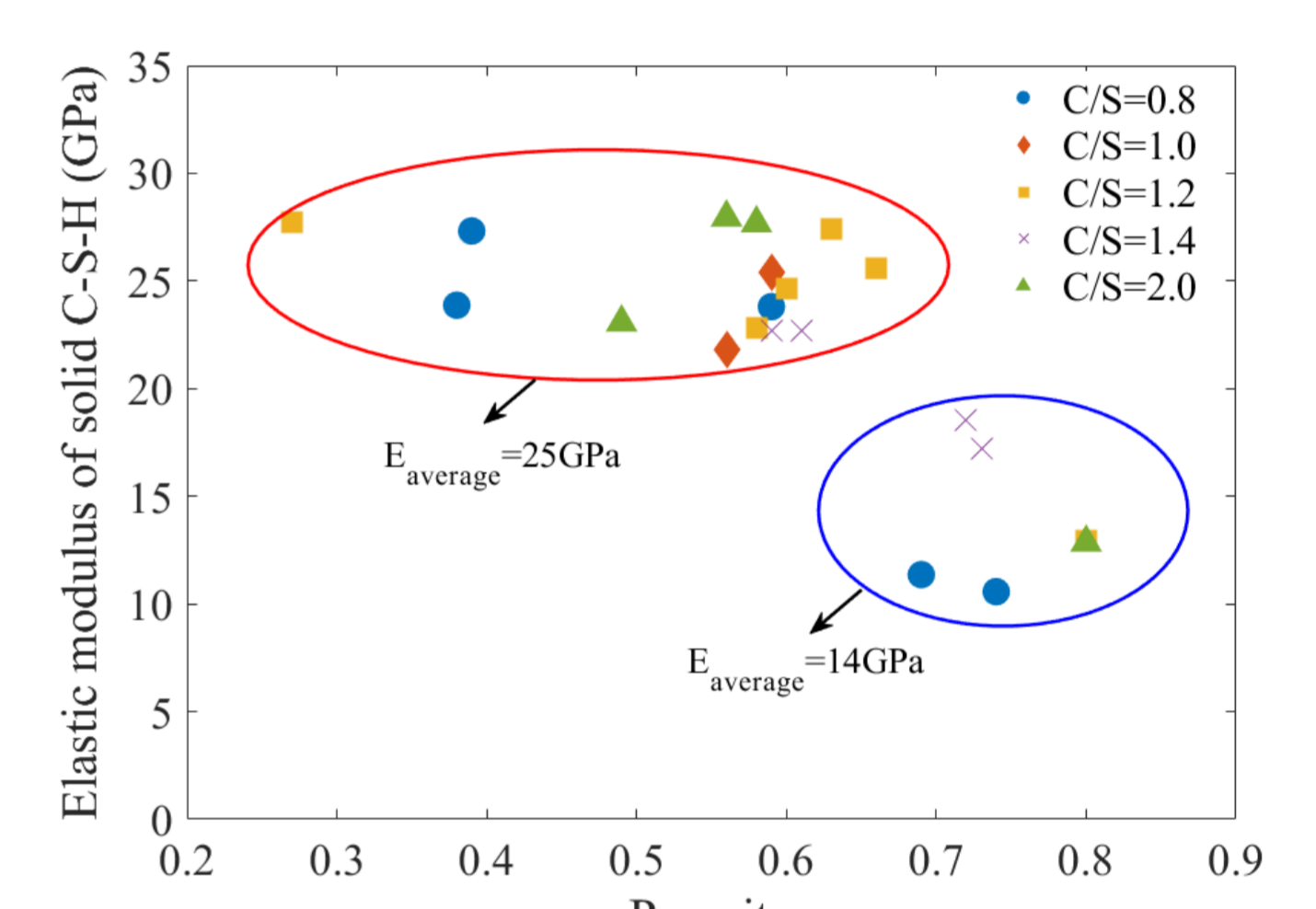


Figure 8 Calculated elastic modulus of solid C-S-H

Measured elastic modulus of compacted C-S-H decrease with increasing porosity. No difference is found within C-S-H of different Ca/Si (Fig.7). With proposed models, the elastic modulus of C-S-H (zero porosity of C-S-H compacts) can be determined.

Assuming as a two-phase porous body, the elastic modulus of the solid C-S-H is around **25 Gpa** (data group in the red ellipse in Fig.8).

Calculated contact creep modulus of C-S-H compacts using creep results decrease with increasing porosity in compacts, indicating higher creep of C-S-H compacts (Fig.9).

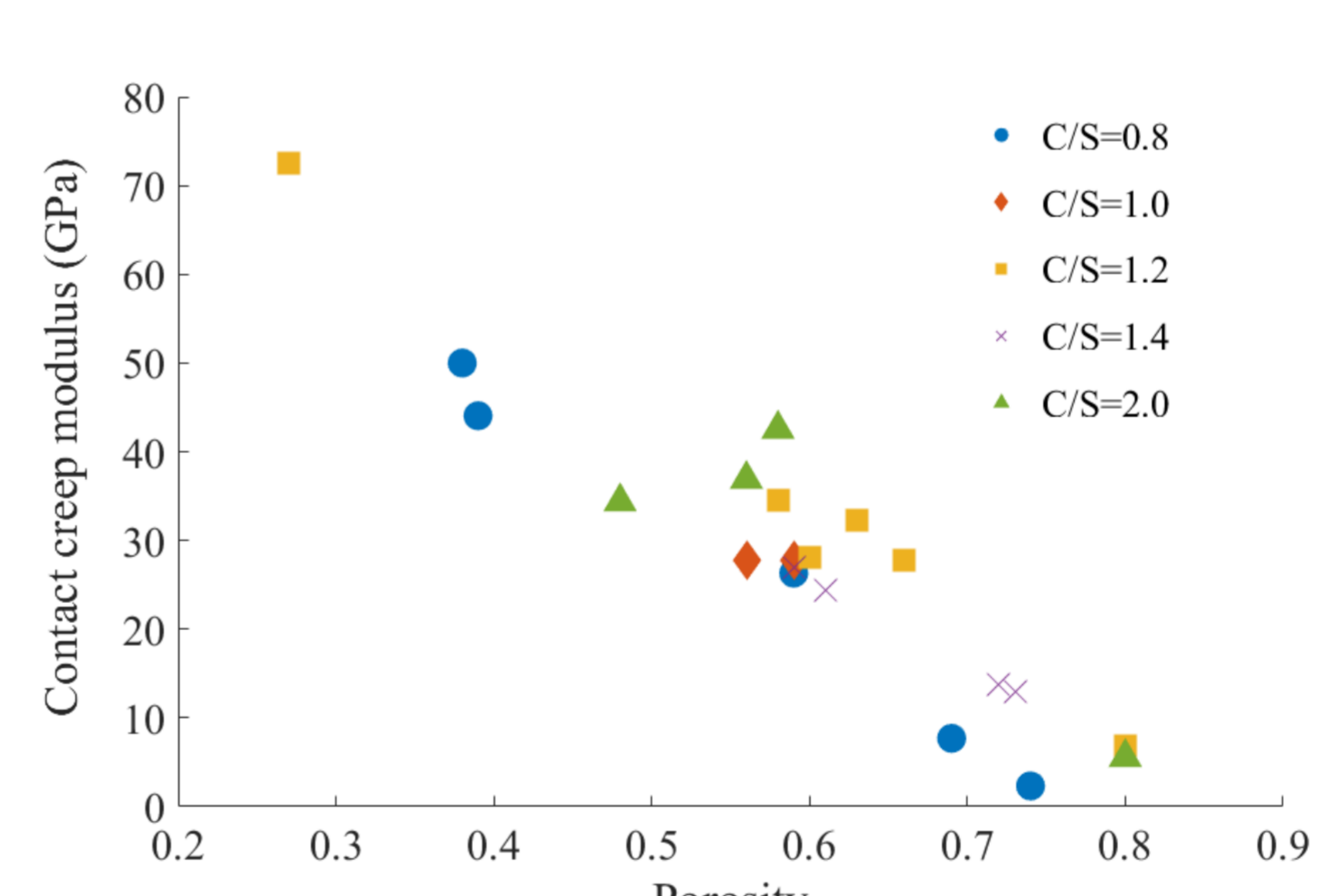


Figure 9 Contact creep modulus of C-S-H compacts

Further work

To measure surface force, e.g. van der Waals forces, repulsive or attractive forces on C-S-H with eSFA, nanoscale C-S-H samples need to be prepared. Plasma sputtering will be used to prepare the required sample. C-S-H sputtering targets are made with CuBe₂ molds (Fig.10). Next step will be to prepare nano-layer C-S-H for surface force measurements

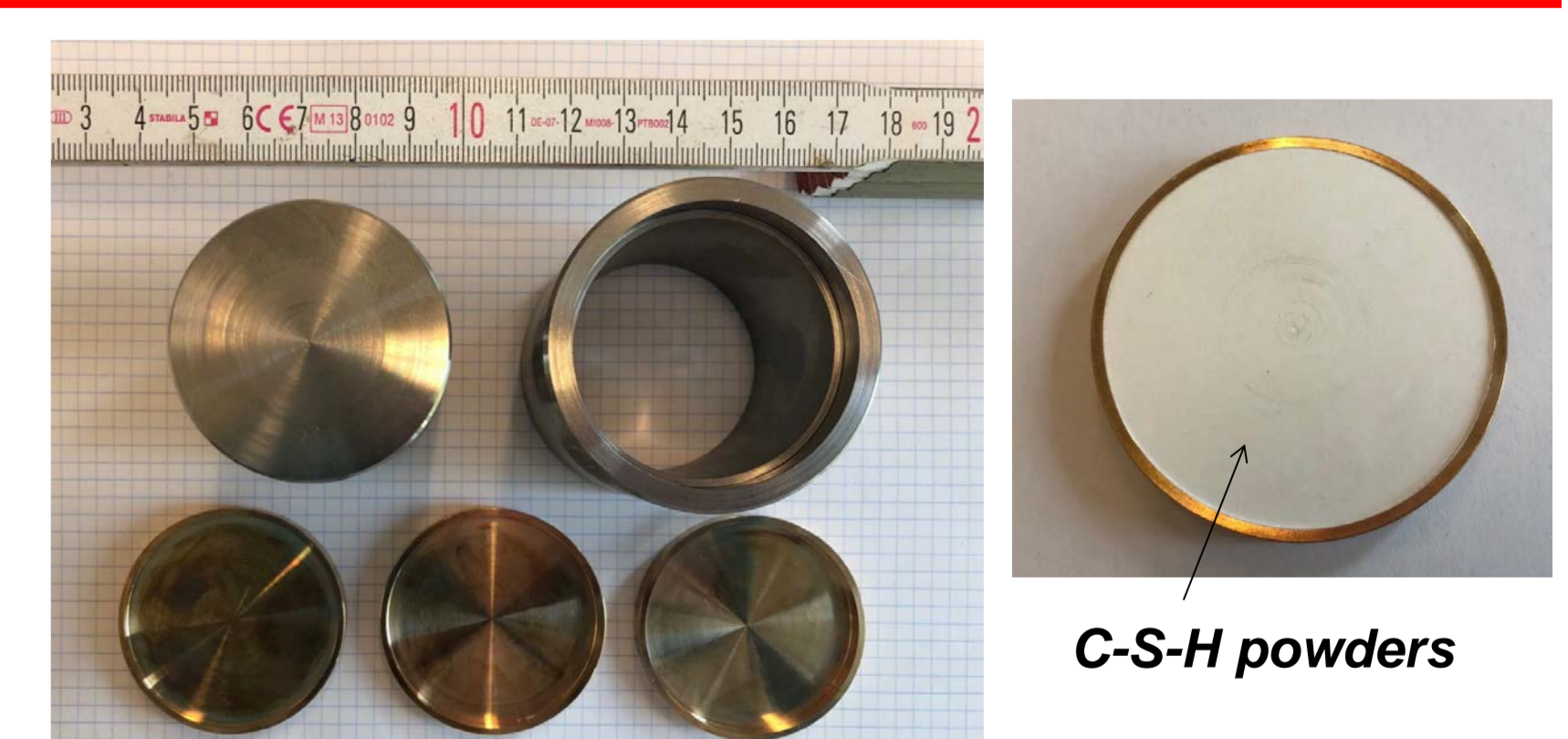


Figure 10 C-S-H sputtering target, preparation molds and compacting die

Macro-level creep measurement using creep frames will be done on C-S-H compacts. The information will be collected and combined with micro-indentation results to compare the microscale and macroscale mechanical properties of C-S-H. The results will be compared with simulated viscoelastic behavior of C-S-H in previous study [6].

Investigations on water distribution and interaction forces can provide valuable information to clarify the mechanism of creep at C-S-H level.