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Characterization of dental cement paste: grid nanoindentation and ultrasound

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Motivation

Dental cement pastes are used for many clinical applications in order to restore the functionality of teeth. They need to be workable for a few minutes after mixing, and to harden quickly thereafter. Moreover, their biocompatibility with enamel, dentin, and pulp is required, while their mechanical strength needs to allow for biting. Accordingly, they typically exhibit strength values exceeding those of chemically similar construction cements, with Biodentine (Septodont, Saint-Maur-des-Fosses, France) reaching some 300 MPa uniaxial compressive strength. However, the micromechanical performance of dental cements, i.e. the contribution of the material's individual constituents to its overall mechanical behavior, has hardly been studied so far.

Investigated material

Composition of Biodentine™ dental cement

Dry binder powder:

- $3CaO{\cdot}SiO_2$ = main hydraulic phase
- $CaCO_3$ = finely ground filler
- ZrO_2 = radiopacifier

Mixing liquid:

- Water
- Superplasticizer
- Accelerator

Setting in 12 minutes; close-to-final properties after 24 hours

Source: https://www.septodont.com/

septodont

Methods

Grid nanoindentation

The 5748 indentation tests were performed under force control with max P = 1 mN [1]. The maximum indentation depths h_{max} amount to some 100 nm. The experimentally determined histograms were approximated by the superposition of three log-normal distributions each. This was realized by minimizing the square root of the sum of squared errors [1]. This optimization problem was solved iteratively, based on progressively refined and shifted search intervals.

Ultrasonic contact pulse transmission

Longitudinal transducers were used with excitation frequencies ranging from 50 kHz to 20 MHz [1]. Wave velocities were found to amount to some 5000 m/s, independent of the frequency. The ultrasonics tests probed "macroscopic" elastic properties of RVEs with a characteristic size in the order of tens of micrometers [1]. Overall, 325 tests were performed at material ages from 7 to 28 days.

Micromechanical modeling

Methods of continuum micromechanics were used to compute estimates and bounds of the homogenized stiffness of RVEs of hardened Biodentine [1]. The upper bound is the volume average of the stiffness tensors of the constituents. The lower bound is the inverse of the volume average of the compliance tensors of the constituents. As for the self-consistent stiffness estimate, spherical phase shapes were assigned to all microstructural constituents.

Results

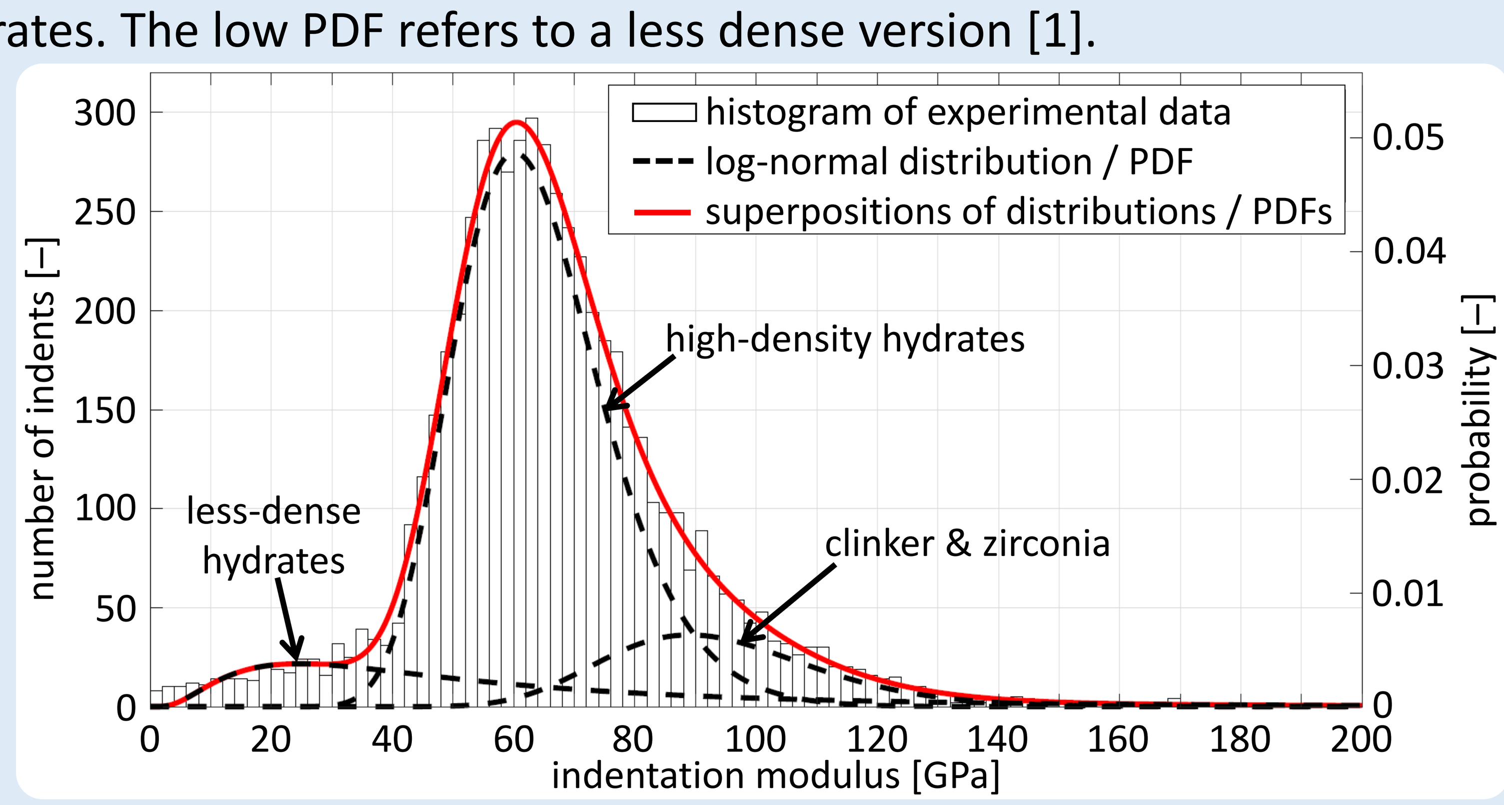
Grid nanoindentation

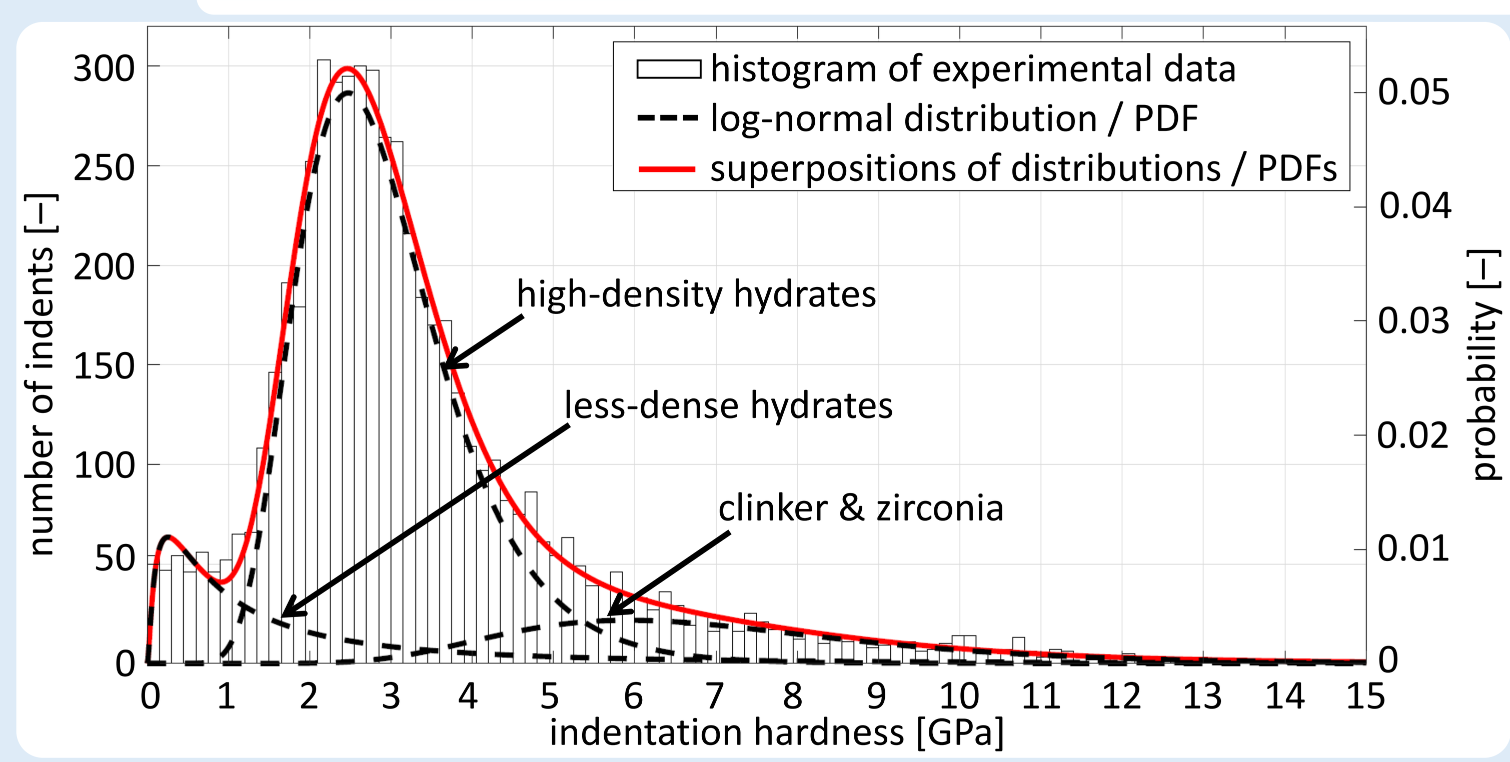
Histograms of indentation modulus and hardness were represented by means of three log-normal probability density functions (PDFs) each [1]. They are referred to as the "low PDF", the "medium PDF", and the "high PDF".

The high PDF refers to indents probing unhydrated clinker ($3CaO{\cdot}SiO_2$) and zirconia (ZrO_2) inclusions embedded in a softer matrix made of hydration products. The medium PDF and the low PDF refer to two types of hydrates reinforced by calcite ($CaCO_3$) particles of single-to-sub micrometric size. The medium PDF corresponds to a high-density version of the calcite reinforced hydrates. The low PDF refers to a less dense version [1].

Table 1: Results of the nanoindentation tests evaluation of based on log-normal distributions; median Md(X) and mode Mo(X) values of the indentation hardness (H) and modulus (M), after [1]

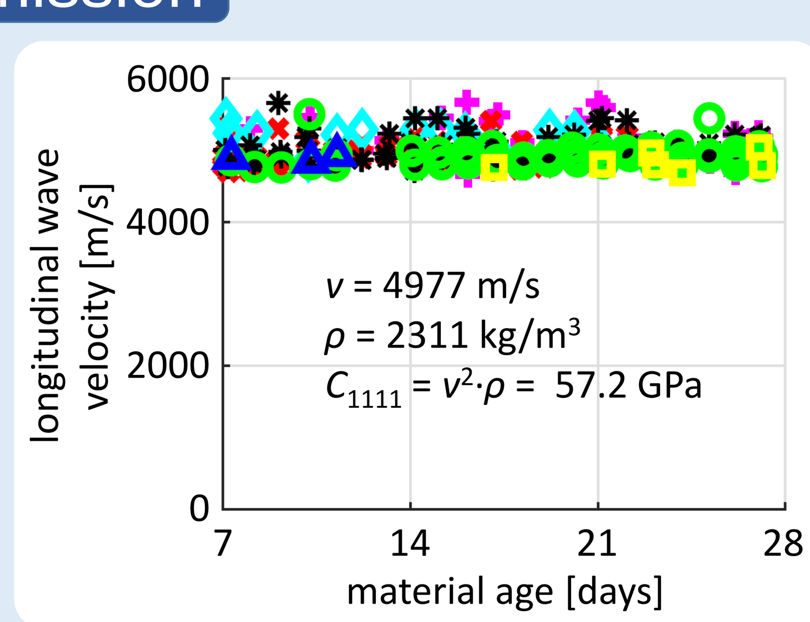
statistical sub-sample	Md(M) [GPa]	Md(H) [GPa]	Mo(M) [GPa]	Mo(H) [GPa]
low PDF	45.1	1.15	24.5	0.26
medium PDF	62.6	2.78	60.2	2.47
high PDF	92.2	6.66	89.0	5.93





Ultrasonic contact pulse transmission

The ultrasonic tests delivered age- and frequency-invariant longitudinal wave velocities amounting to v = 4977±191 m/s [1]. Considering the mass density of hardened Biodentine, ρ = 2311 kg/m^3, the theory of wave propagation through linear elastic media provides access to the expected value of the macroscopic stiffness tensor component C_{1111}, amounting to C_{1111} = 57.2 GPa [1].



Micromechanical modeling

The median values of indentation stiffnesses were translated to elastic moduli considering a diamond Berkovich tip and values of Poisson's ratio of 0.24 for the low and medium PDF, and 0.30 for the high PDF. The ultrasonic-derived stiffness is smaller than the stiffness estimates and bounds, evaluated based on the results of nanoindentation testing. This indicates the presence of micro-defects weakening the macroscopic stiffness [1].

Table 2: Estimates and bounds of the homogenized stiffness of hardened Biodentine, after [1]

upper bound	C_{1111} = 77.6 GPa
self-consistent estimate	C_{1111} = 75.3 GPa
lower bound	C_{1111} = 73.7 GPa

Conclusions

- Biodentine contains cementitious hydrates significantly reinforced by calcium carbonate. At a higher scale, clinker and zirconia act as additional reinforcements of the microstructure.
- Grid nanoindentation, with several thousands of individual indents, allows for identifying properties of asymmetric PDFs of the material constituents. They can be well represented by log-normal distributions.
- Countermeasures against defects could further improve the mechanical properties of hardened Biodentine.

Acknowledgement

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Reference

[1] Dohnalík, P., Pichler, B., Zelaya-Lainez, L., Lahayne, O., Richard, G., Hellmich, C. Micromechanics of dental cement paste. Under minor revision at: *Journal of the Mechanical Behavior of Biomedical Materials*, 2021.