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Micro-elasticity Of Porous Ceramic Baghdadite: A Combined Acoustic-nanoindentation Approach Supported By Homogenization Theory

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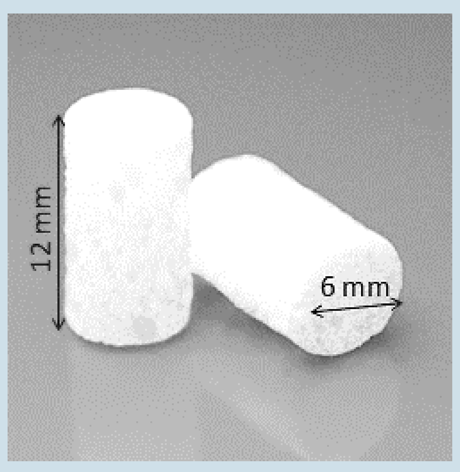
Motivation

Bone tissue engineering aims at repairing damaged bone and restoring its functions with the help of biocompatible materials cultivated with cells and corresponding growth factors [1]. Besides being osteoconductive and osteoinductive, the bone substitute or scaffold should exhibit sufficient porosity for good vascular and tissue ingrowth, while not overly compromising the overall mechanical properties of the implant, i.e. its stiffness and strength. The design process of such scaffolds requires a multitude of *in vitro* and *in vivo* experiments and has proven to be a challenging task, thus giving rise to the wish for rational, computer-aided design of biomaterials, regarding not only biological and cell transport aspects, but also mechanics.

Highly porous baghdadite ($Ca_3ZrSi_2O_9$) scaffolds have shown promising biological responses when used for the repair of critical size defects in rabbit radial bones [2]. However, the mechanical properties of these scaffolds require further investigation. Therefore, by using structure-property relations derived from **ultrasound and nanoindentation experiments**, and on the basis of theoretical and applied **micromechanics**, the current research aims at applying the state-of-the-art methods in computational biomechanics and biomaterials to this new material to investigate its **elastic properties**.

Material and Experimental Methods

Sample data

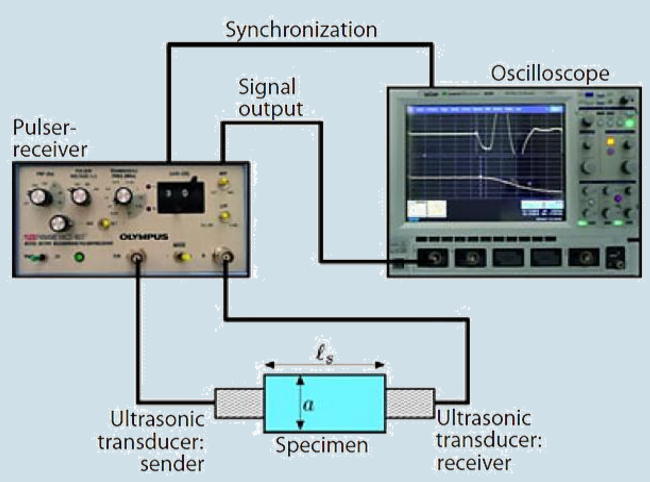


Density: $\rho = \frac{m}{V}$

Porosity: ϕ_i^{exp} = 66% - 94%

Pore diameter: d = 500 µm

Ultrasound tests



Wave velocity: $v = \frac{h}{t}$

Wavelength: $\lambda = \frac{v}{f}$

Separation of length scales

$$\frac{d}{\lambda} \leq 0.03$$

$$f = 0.1\ \text{MHz}$$

Normal stiffness component of the overall scaffolds

$$C_{1111}^{exp} = \rho v^2$$

Micromechanical model

$$\frac{E^{hom}}{E_s} \cong B_E (1-\Phi)^{C_E}$$

$$\nu^{hom}(\nu_s) \cong A_\nu(\nu_s) + B_\nu(\nu_s) \times (1-\phi)^{C_\nu(\nu_s)}$$

$$C_{1111}^{hom} = \frac{E^{hom}(1-\nu^{hom})}{(1+\nu^{hom})(1-2\nu^{hom})}$$

E_{hom} … homogenized Young's modulus

E_s… Young's modulus of a single crystal

ν_{hom}… homogenized Poisson's ratio

ν_s… Poisson's ratio of a single crystal

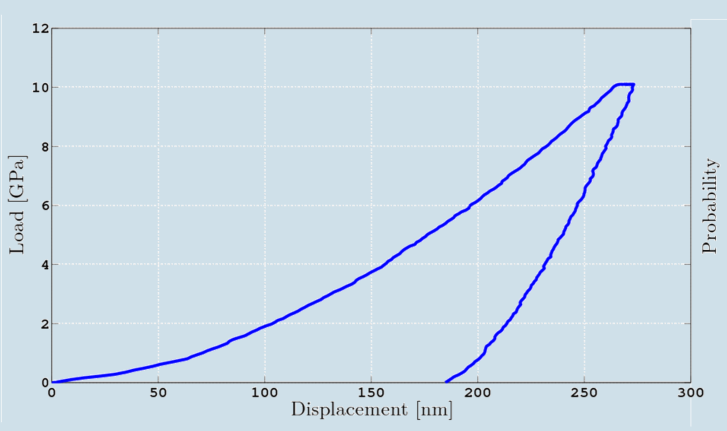
C_{1111}^{hom}…normal stiffness component of the homogenized stiffness tensor

$B_E, C_E, A_\nu, B_\nu, C_\nu$ as given in [4]

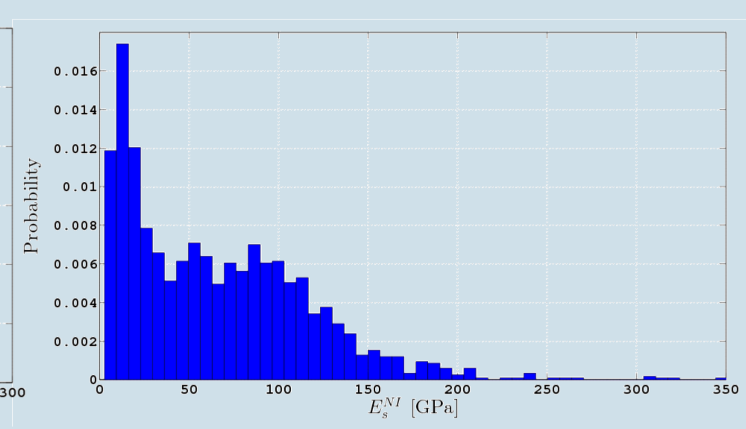
Minimizing the mean absolute error between the micromechanics-based stiffness evaluated for different experimentally determined porosities ϕ_i^{exp}, and the corresponding experimentally determined stiffness values, provides an estimate for the elastic properties of a single baghdadite crystal, E_s and ν_s:

$$\sum_i \left| C_{1111}^{hom}(\phi_i^{exp}, E_s, \nu_s) - C_{1111}^{exp,i} \right| \rightarrow min$$

Nanoindentation



Typical load-displacement curve



Distribution of all the 1750 measurements

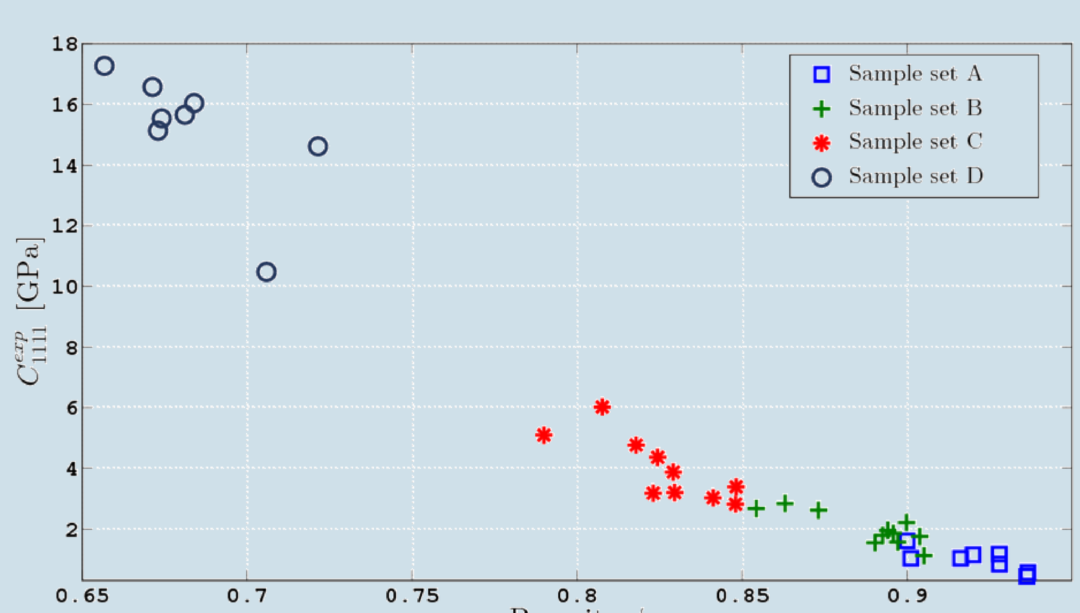
The nanoindentation tests were performed with a Berkovich tip with a loading- unloading rate of 30 mN/min, a holding time of 10 s, and four different maximum loads: 10, 15, 20 and 30 mN. The 1750 measurements were evaluated according to the method of Oliver and Pharr [3].

Results & Discussion

Experimentally determined stiffness

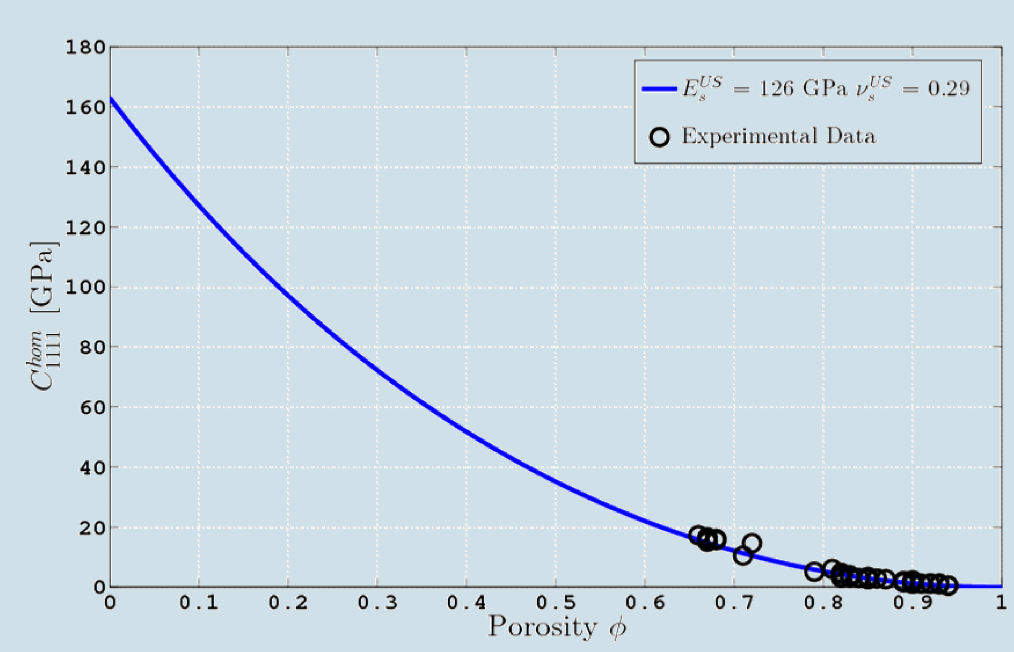
Sample set	$\bar{\phi}$[%]	$\bar{C}_{1111}^{exp}$ [GPa]
A	92 ± 1	0.98 ± 0.34
B	89 ± 2	1.99 ± 0.51
C	83 ± 2	3.97 ± 1.00
D	68 ± 2	15.16 ± 1.93

Mean value and standard deviation of the normal stiffness of baghdadite scaffolds obtained through ultrasonic testing, for the corresponding mean porosity of each sample group



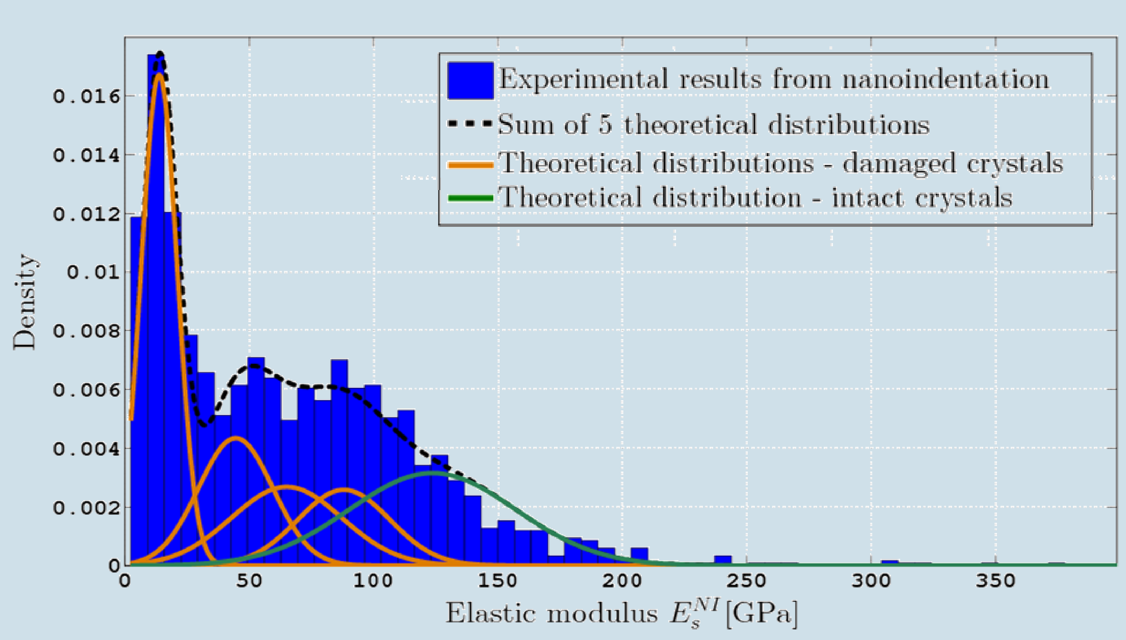
The normal stiffness increases with decreasing porosity.

Elastic properties of a baghdadite crystal



The minimization of the mean absolute error between the model-predicted and the experimentally determined stiffness delivered the elastic properties of pure (dense) baghdadite: E_s= 126 GPa and ν_s = 0.29.

Validation of the elastic properties by statistical evaluation of the nanoindentation tests



The 1750 nanoindentation results were optimally represented by the superposition of five Gaussian distributions: four corresponding to damaged or broken crystals, and the fifth corresponding to intact crystals. The mean value of the latter is $\overline{E_S^{NI}}$ = 124 GPa, which agrees almost perfectly with E_s = 126 GPa obtained from the micromechanical evaluation of the ultrasonic tests.

References

[1] Hutmacher DW. Scaffolds in tissue engineering bone and cartilage. *Biomaterials* 2000; 21(24): 2529 –43.

[2] Roohani-Esfahani SI, Dunstan CR, Davies B, Pearce S, Williams R, Zreiqat H. Repairing a critical-sized bone defect with highly porous modified and unmodified baghdadite scaffolds. *Acta Biomater* 2012; 8(11): 4162–72.

[3] Oliver W, Pharr G. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J Mater Res* 1992; 7: 1564–83.

[4] Fritsch A, Hellmich C, Young P. Micromechanics-derived scaling relations for poroelasticity and strength of brittle porous polycrystals. *J Appl Mech* 2013; 80(2): 020905.

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