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MECHANICAL CHARACTERIZATION OF NANOPOROUS SILICON MEMBRANES USING ELASTIC GUIDED WAVES

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ABSTRACT

Nanoporous solids play an increasingly important role for the study of matter in extreme spatial confinement and for designing functional materials in areas ranging from nanofluidics via bio-sensing to energy harvesting, transformation and storage. A detailed knowledge of their mechanics and pore geometry is of utmost importance both for studying confinement effects on matter [1] and for their application as robust nanomaterials in macroscopic devices [2]. However, small sample sizes, mechanical fragility and thin-membrane geometries make a reliable application of traditional mechanical characterization methods, such as conventional ultrasound [3] or indentation [4] techniques, on porous media often challenging, if not impossible. For example, the impacts of self-organized porosity on the mechanics of the main stream semiconductor silicon is still elusive, despite a very broad scientific interest in this material caused by its wafer-scale, single-crystalline availability, multiphysics properties along with manifold functionalization schemes on the single-nanopore scale.

Here, we present experiments on elastic guided waves (propagating modes) and zero group velocity (ZGV) resonances (standing waves) detected contactless and non-destructively in a dry and liquid-infused nanoporous silicon (pSi) membrane using a laser ultrasonics (LUS) setup [5]. The pSi membrane was obtained from single-crystalline silicon by electrochemical etching, resulting in a complex heterogeneous and anisotropic material (with a thickness $d = 105 \mu\text{m}$), which presents typical patterns of self-organized tubular pores (diameter of 7 nm), perpendicular to the membrane surface. A pulsed laser source (355 nm, 350 ps, 25 μJ) generated elastic waves by thermo-elastic conversion and broadband displacement signals were detected by stabilized Michelson interferom-

etry. Spatio-temporal displacement fields were measured by varying the distance between the source and detection points. Then, by applying a two-dimensional Fourier transform [6], the dispersion curves of several guided modes were obtained for frequencies up to 100 MHz (see Fig. 1). First, a direct comparison with measurements on non-porous bulk silicon showed that the high porosity of $\sim 50\%$ and irregular pore shapes make the material effective stiffness properties nearly elastically isotropic in the membrane plane, and lead to significant deviations of the usual Lamb modes behavior detected in bulk silicon (red ellipses). Most prominently, for pSi, we observed distinct Rayleigh waves on the membrane surfaces. Second, these measurements were further analyzed by means of numerical waveguide modeling (continuous white lines) and inverse identification, from which the effective anisotropic stiffness tensor and a surrogate for the nanopore geometry (conicity) could be retrieved at the mesoscale.

Altogether, these findings highlight the complex mechanics of pSi, as dictated by an interplay of asymmetric pore shape, anisotropic elasticity and liquid's nature within the pores. In particular, (1) our technique allows measuring multiple guided modes and sharp ZGV resonances in thin nanoporous membranes and it is sensitive to the presence of liquid inside the pores; (2) our measurements evidenced that the synthesis of pSi results in an asymmetric membrane, whose impact manifests itself as a repulsion of the guided modes. Moreover, this repulsion effect could be adequately modeled by considering a linearly varying density profile across the membrane's thickness, which is traced back to the cone-shaped geometry of the pores; (3) the porous network prevails over the crystalline nature of the pristine bulk silicon membrane (*i.e.*, cubic symmetry), thus leading to a transversely isotropic behavior at the mesostructural scale; and (4) the effective stiffness coefficients estimated with our model-based approach are much

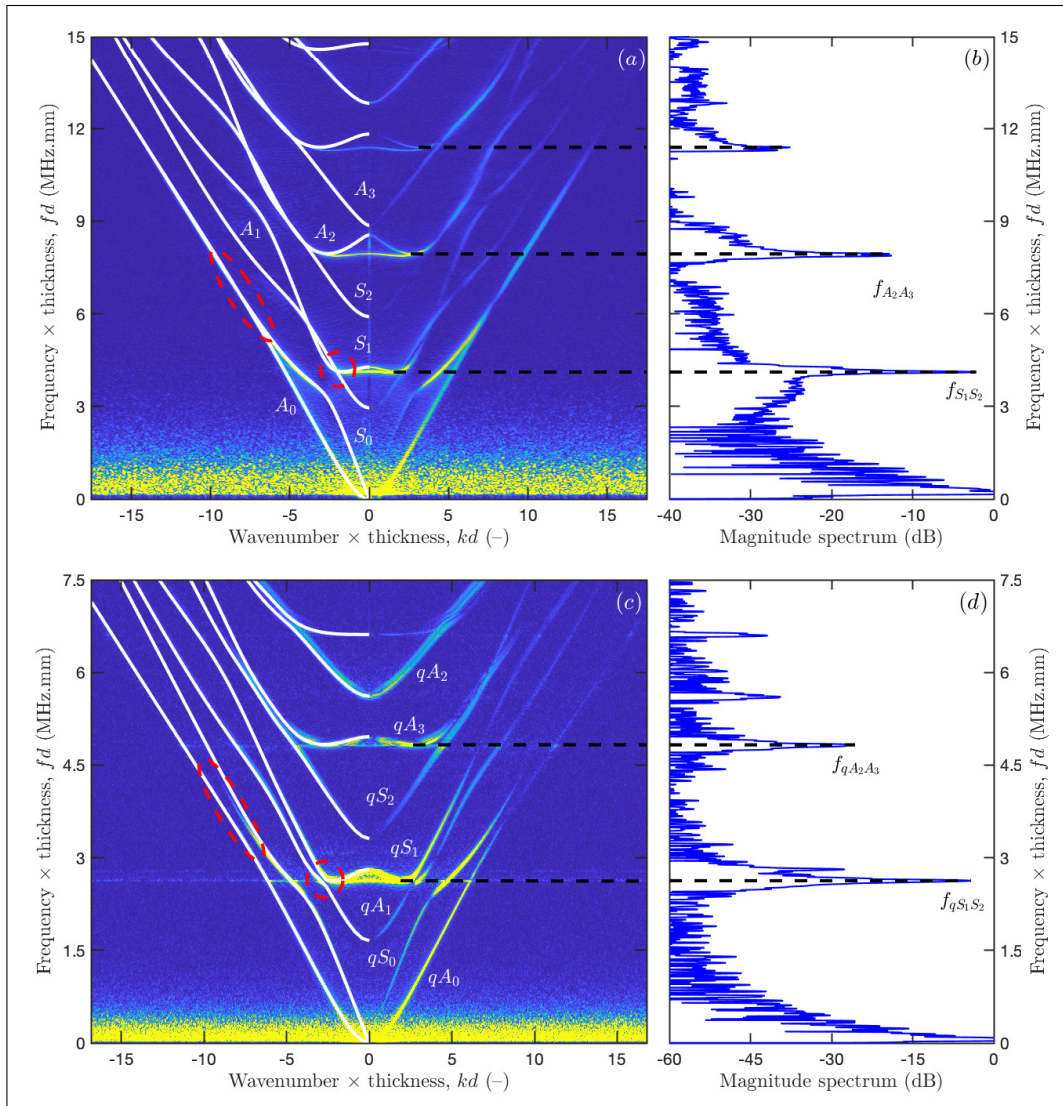


Figure 1. LUS measurements for bulk silicon (upper panels) and the dry pSi membrane (lower panels): (a)–(c) Dispersion curves in the normalized wavenumber-frequency plane, obtained by 2D Fourier transform of the spatio-temporal signals (modeled guided modes are displayed in white continuous lines for comparison, whereas red ellipses highlight the main differences between both media); and (b)–(d) ZGV resonances obtained by temporal Fourier transform of the signal measured with the laser source and probe on epicenter.

lower and amount to around 20% of those of bulk silicon.

Therefore, our experiments provide the base for a predictive application of pSi in mechanically robust on-chip devices and open new frontiers for in-operando non-destructive mechanical and geometrical characterization of functional porous materials as well as for the fundamental study of nanopore-confined matter in general.

1. REFERENCES

- [1] P. Huber, “Soft matter in hard confinement: phase transition thermodynamics, structure, texture, diffusion and flow in nanoporous media,” *J. Phys. Condens. Matter*, vol. 27, no. 10, p. 103102, 2015.
- [2] M. R. Begley, D. S. Gianola, and T. R. Ray, “Bridging functional nanocomposites to robust macroscale devices,” *Science*, vol. 364, no. 6447, p. eaav4299, 2019.
- [3] J. Bustillo, J. Fortineau, G. Gautier, and M. Lethiecq, “Ultrasonic characterization of porous silicon using a genetic algorithm to solve the inverse problem,” *NDT & E Int.*, vol. 62, pp. 93–98, 2014.
- [4] D. Bellet, P. Lamagnere, A. Vincent, and Y. Brechet, “Nanoindentation investigation of the Young’s modulus of porous silicon,” *J. Appl. Phys.*, vol. 80, no. 7, pp. 3772–3776, 1996.
- [5] C. Prada, D. Clorennec, T. W. Murray, and D. Royer, “Influence of the anisotropy on zero-group velocity Lamb modes,” *J. Acoust. Soc. Am.*, vol. 126, no. 2, pp. 620–625, 2009.
- [6] D. Alleyne and P. Cawley, “A two-dimensional Fourier transform method for the measurement of propagating multimode signals,” *J. Acoust. Soc. Am.*, vol. 89, no. 3, pp. 1159–1168, 1991.