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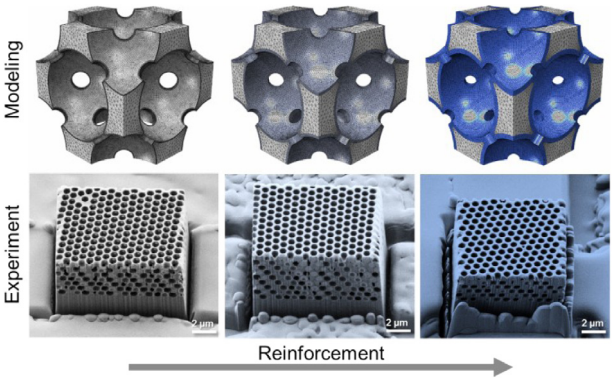
The stiffness and strength of metamaterials based on the inverse opal architecture

Jefferson J. do Rosário [a,*], Jonathan B. Berger [b], Erica T. Lilleodden [a,c], Robert M. McMeeking [b,d], Gerold A. Schneider [a]

[a] Institute of Advanced Ceramics, Hamburg University of Technology, Denickestrasse 15, D-21073 Hamburg, Germany
[b] Materials Department and Mechanical Engineering Department, University of California, Santa Barbara, CA 93106, USA
[c] Institute of Materials Research, Materials Mechanics, Helmholtz-Zentrum Geesthacht, Max-Planck-Strasse 1, D-21502 Geesthacht, Germany
[d] School of Engineering, University of Aberdeen, King's College, Aberdeen AB24 3UE, Scotland

GRAPHICAL ABSTRACT



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ABSTRACT

The inverse opal architecture, a class of mechanical metamaterials recently shown to exhibit high specific strength and modulus, is further investigated here using carefully coupled experiments and finite element modeling. We demonstrate that this architecture can be exploited to achieve optimized specific strength and modulus, while simultaneously offering tunable optical bandgaps and large-area fabrication. Starting with a silica inverse opal structure and adding different thicknesses of titania (10–34 nm) the strength was gradually increased from 41 to 410 MPa and the elastic modulus from 1.7 to 8.3 GPa, within densities of 300–1000 kg m^{-3}. Simulations confirmed that the inverse opal structure can outperform the state-of-the-art octet- and isotropic-truss designs in terms of Young's, shear and bulk modulus, as well as in structural efficiency (total stiffness). Simulations also predict stresses in the titania coating and in the silica that are on the order of the theoretical tensile yield stresses at failure, indicating that size effects controlling defect population are responsible for the high strengths.

1. Introduction

The inverse opal (IO) architecture – a design based on the inverse FCC (face-centered cubic) geometry – has recently been

* Corresponding author.
E-mail addresses: jeffersonjean.dorosario@tuhh.de (J.J. do Rosário), g.schneider@tuhh.de (G.A. Schneider).

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presented as a class of mechanical metamaterials that opens a new design space for the development of microscaled lightweight structures [1]. The IO is a known class of photonic crystal, with its characteristic photonic-bandgap controlled by the size of its pores. This combination of classical photonics and high mechanical performance makes this architecture interesting for multi-functional applications.

In the last few years, much effort has been put into developing microscaled lightweight structures with high strength and modulus [2–12]. Novel combinations of design and fabrication techniques have allowed many metamaterials to populate unexplored areas of design space with high specific properties (desired property per density). These metamaterials often rely on a size effect, i.e. the "smaller is stronger" paradigm, to achieve high specific strength at the micrometer and nanometer scale. Many of these experimental investigations of metamaterials are based on the work of Deshpande et al. [13,14] and the octet-truss [3,4,7–10]. Other lattice designs [2,4,6,12] and honeycomb structures [4,12] have also been shown to have interesting properties. Nevertheless, the IO architecture structures have only been outperformed by carbon-based microscaled metamaterials [2,12].

Our previous work showed that the silica based IO structure has substantial stiffness and strength, but the addition of a stiffer and stronger coating (titania) by atomic layer deposition (ALD) is responsible for a significant increase in performance. These materials, uncoated and coated silica IO, possess exceptionally high specific stiffness and strength, and up until recently, are only rivaled by highly anisotropic honeycomb structures [4,12].

In this work, experiments and modeling are used to further understand the properties of IO, and how the addition of a stiff, strong coating affects mechanical properties. Experimentally, we show that the strength and modulus of silica IO can be gradually increased by adding different thicknesses of a titania coating (namely 10, 20 and 34 nm). Average strengths from 41 MPa up to 410 MPa and average elastic moduli from 1.7 GPa up to 8.3 GPa were achieved while varying the density from 330 kg m^{-3} up to 890 kg m^{-3}. Simulations show that the addition of this stiffer, stronger coating can decrease stress concentrations in the silica by 60%. Using simulations, we are able to quantify the structural efficiency and performance of the IO geometry, based on theoretical bounds as metrics. We calculate the Young's, shear and bulk moduli, as well as structural efficiency and isotropy, of the IO geometry. As practical benchmarks for performance, we compare the properties of IO to two high-performance truss geometries, the octet-truss and a truss with maximum stiffness for an isotropic lattice material, recently identified by Gurtner and Durand [15]. The IO is found to have higher structural performance than optimized lattice designs due to its quasi-closed cell geometry which helps to support membrane stresses, while the addition of a robust titania coating increases the absolute performance greatly.

2. Materials and methods

2.1. Opal fabrication

Silica IO films were fabricated by vertical co-assembly based on the procedure described by Hatton et al. [16] 0.6 ml of a 10 w/v% stock suspension of monodispersed colloidal polystyrene (PS) spheres with a diameter of 756 ± 20 nm (Microparticles GmbH) was mixed with 0.450 ml of a hydrolyzed TEOS (tetraethylorthosilicate) solution in deionized water to a total volume of 50 ml. The TEOS solution was composed of 1:1:1.5 ratio by weight of TEOS, 0.10 M HCl and EtOH stirred for 1 h. Soda-lime silica glass substrates were vertically positioned in PTFE beakers containing the prepared suspension. The beakers were placed in a humidity chamber at 60 °C for 3–5 days resulting in a growth rate of 0.5 cm/day of the FCC structure. The substrates were cleaned by soaking in and brushing with an alkaline detergent solution and rinsing with hot tap water and deionized water. Finally, the substrates were dried with filtered nitrogen, and subsequently O_2-plasma cleaned for 20 min. After the co-assembly was carried out the films were calcined at 500 °C for 30 min in order to burn-out the PS template resulting in an inverse opaline structure of amorphous silica. The final IO structure is an inverted FCC elliptical pore arrangement with pore size of 725 nm parallel to the substrate and 670 nm perpendicular to the substrate. The elliptical shape arises from an anisotropic shrinkage upon calcination. The pores are interconnected by holes of approx. 170 nm in diameter. Subsequently, some samples were ALD-coated at 95 °C with an amorphous TiO_2-layer with different thicknesses (10, 20 and 34 nm). The ALD-coating thicknesses were measured in a spectroscopic ellipsometer from a thin film deposited on a silicon wafer during the infiltration with an error of ± 1 nm.

2.2. Opal density determination

The density of the silica IO was estimated by four independent methods: gravimetric, pycnometric and two optical methods. The density of the silica IO coated with TiO_2 was measured by two independent methods: gravimetric and optical. In summary, the densities of pure silica IO and 10, 20 and 34 nm-TiO_2 ALD-coated silica IO are 330, 500, 660 and 890 kg m^{-3}, respectively. The average among different methods lies within a maximum of 10% uncertainty. For more details, see Appendix A (supplementary data).

2.3. Micropillar preparation and microcompression tests

Focused ion beam (FIB) milling was employed to fabricate pillars with square and rectangular cross section using a Nanolab 200 DualBeam microscope (FEI, Co.). Minimal exposure of the fabricated pillars to the Ga ion beam was achieved since no direct imaging of the structure with the ion beam was required. Cross sectional milling was used to fabricate the four planar sides, with the final milling steps imposing beam currents of 300 pA, at an accelerating voltage of 30 kV. A series of microcolumns, all milled into the underlying soda-lime glass substrate, were prepared. This design is advantageous to the analysis of the compression data, since the underlying glass substrate serves as a stiff and hard platen; no filleting at the bottom of the pillar is present, in contrast to pillars that are FIB-milled from the bulk. Here, the deformation is strongly limited to the IO film, with a single deformation volume and in turn a straightforward analysis of the uniaxial stress–strain response.

The compression tests were conducted using a Nanoindenter XP outfitted with a flat ended 60° conical diamond indenter at a constant displacement rate of 10 nm s^{-1} in the (111)-direction to a specified target depth. In all tests a target of 700 nm was chosen, which was in all cases greatly surpassed, as a mechanical instability at the critical failure stress occurred; a large displacement burst up to a few microns typically resulted at failure. In some cases a partial unloading segment prior to failure was included to assess whether an elastic regime is truly present. The stress, σ, and strain, ε, were computed using the load, P, and displacement, h, data along with the geometric parameters, namely the height, H, and the top cross sectional area, A, of the pillars, as measured from SEM micrographs. The stress and strain were computed as $\sigma = P/A$ and $\varepsilon = h/H$, respectively. The elastic modulus was assessed with the continuous stiffness measurement (CSM) method, using a dynamic displacement amplitude of 2 nm at a frequency of 45 Hz superimposed