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Reexamining the mechanical property space of three-dimensional lattice architectures

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ABSTRACT

Lightweight materials that are simultaneously strong and stiff are desirable for a range of applications from transportation to energy storage to defense. Micro- and nanolattices represent some of the lightest fabricated materials to date, but studies of their mechanical properties have produced inconsistent results that are not well captured by existing lattice models. We performed systematic nanomechanical experiments on four distinct geometries of solid polymer and hollow ceramic (Al_2O_3) nanolattices. All samples tested had a nearly identical scaling of strength (σ_y) and Young's modulus (E) with relative density ($\overline{\rho}$), ranging from $\sigma_y \propto \overline{\rho}^{1.45}$ to $\overline{\rho}^{1.92}$ and $E \propto \overline{\rho}^{1.41}$ to $\overline{\rho}^{1.83}$, revealing that changing topology alone does not necessarily have a significant impact on nanolattice mechanical properties. Finite element analysis was performed on solid and hollow lattices with structural parameters beyond those realized experimentally, enabling the identification of transition regimes where solid-beam lattices diverge from existing analytical theories and revealing the complex parameter space of hollow-beam lattices. We propose a simplified analytical model for solid-beam lattices that provides insight into the mechanisms behind their observed stiffness, and we investigate different hollow-beam lattice parameters that give rise to their aberrant properties. These experimental, computational and theoretical results uncover how architecture can be used to access unique lattice mechanical property spaces while demonstrating the practical limits of existing beam-based models in characterizing their behavior.

1. Introduction

Incorporating three-dimensional architecture into materials design across multiple length scales has led to the creation of advanced materials with novel mechanical properties, such as ultralight weight [1–3], negative Poisson's ratios [4,5], and near infinite bulk-to-shear modulus ratios [6,7]. The versatility of current fabrication methods and processing techniques engenders a virtually unbounded potential design space by which new materials can be created [8–17]. Despite many proof-of-concept demonstrations, very few guiding principles exist for designing architectures that efficiently integrate structural and microstructural deformation mechanisms. Understanding the complex interplay between constituent materials and architecture is crucial to creating and optimizing new materials with tunable properties.

One of the more prominent recent successes of architected materials has been the creation of simultaneously lightweight, strong and stiff three-dimensional micro- and nanolattices. These materials are 3D assemblies of beams with micro- and nanoscale constituent dimensions, and it is the confluence of nanometer-sized dimensions and architecture that gives rise to their unique properties [1–3,15,18–26]. The theoretical maximum Young's modulus (E) and yield strength (σ_y) of a lightweight porous material are set by the Voigt bound, which are functions of the relative density ($\overline{\rho}$) that scale as $E = E_s\overline{\rho}$ and $\sigma_y = \sigma_{ys}\overline{\rho}$. E_s and σ_{ys} are the constituent material's Young's modulus and yield strength, respectively. This means that if a material is 10% dense, its highest possible stiffness and strength are 10% of those of the fully dense solid [27]. For isotropic solids, the Young's modulus limit is defined by the Hashin-Shtrikman bound [28], and for isotropic beam-based lattices the modulus limit is defined by the tighter Gurtner-Durand bound [29]. This scaling of stiffness and strength with relative

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density becomes particularly conspicuous for light and ultralight materials, where poor scaling relations can have orders-of-magnitude effects on the overall mechanical properties.

A large body of theoretical and experimental work has been dedicated to creating new lattice architectures and investigating their properties [1,15,18,19,23,30–34]. Most analytical models for the mechanical behavior of both 2D and 3D lattices are derived using beam theory, and these models generally predict that strength and modulus follow a power law scaling with relative density as

$$E = BE_s\overline{\rho}^{\,m}, \tag{1}$$

$$\sigma_y = C\sigma_{ys}\overline{\rho}^{\,n}. \tag{2}$$

The proportionality constants B and C and the scaling coefficients m and n change depending on whether lattice deformation is dominated by stretching or bending of the beams. To predict if a lattice will be stretching- or bending-dominated, pin-jointed versions of the lattices have to be analyzed to assess their rigidity. A pin-jointed structure is defined to be *rigid* if any shape change requires a corresponding increase in strain energy [30,35]. Structures can also be periodically rigid if they are constructed from rigid periodic subunits. An in-depth discussion of rigidity can be found in the Supplementary Materials. According to classical formulations, lattices with a rigid topology have properties governed by stretching of the beams, with $E \propto \overline{\rho}$ and $\sigma_y \propto \overline{\rho}$ $(m = n = 1)$, and lattices with a non-rigid topology are governed by bending of the beams, with $E \propto \overline{\rho}^{\,2}$ and $\sigma_y \propto \overline{\rho}^{\,1.5}$ $(m = 2,\ n = 1.5)$ [36]. Topology here refers to structures as defined by their connectivity, and it is invariant to changes in structural parameters like beam diameter or shell thickness.

These theories provide a simple framework to predict the mechanical performance of lattices, but their utility as analysis tools for physically realizable systems has yet to be fully quantified. Experimental and theoretical work on lattices have shown mixed results on the exact role of topology in governing strength and modulus scaling; a wide range of reported strength and stiffness power law scaling relationships exists, even for topologically identical systems, and no experimentally realized lightweight lattice matches the performance predicted by the Gurtner-Durand bound [1,2,18,21–23,37,38].

We conducted systematic nanomechanical experiments and finite element analysis on nanolattices made from two different material systems, with four different topologies each. We found that the mechanical properties of nanolattices in a currently experimentally realizable property space are nearly independent of architecture, and that the strength and stiffness of rigid and non-rigid topologies at the same relative density are nearly identical. This result represents a significant point of departure from theories relating mechanical properties to the rigidity of the lattice topology [19,22,29,30,36,39]. Uniaxial compression experiments reveal a non-linear scaling of strength and stiffness with relative density, with exponents between $m = 1.41 - 1.83$ for stiffness and $n = 1.45 - 1.92$ for strength for all nanolattice topologies and material systems. Finite element simulations (Abaqus FEA) reproduce the observed nonlinear scaling within the range of relative densities tested experimentally for both solid and hollow-beam nanolattices. They further reveal that for solid lattices with relative densities of $\overline{\rho} < 5\%$, the stiffnesses of rigid and non-rigid topologies deviate from one another to show good agreement with existing bending- and stretching-dominated scaling laws [39]. For hollow lattices, finite element simulations reveal a highly complex parameter space with orders-of-magnitude deviations in stiffness arising from small variations in parameters. We propose a simple analytic framework that provides insight into the stiffness scaling of solid-beam lattices, and we investigate some of the mechanisms for the large variances in hollow-beam lattice properties.

2. Methods

2.1. Fabrication

Polymer nanolattices were fabricated using a two-photon lithography direct laser writing process in IP-Dip photoresist using the Photonic Professional lithography system (Nanoscribe GmbH). Structures were written using laser powers of $6 - 14mW$ and writing speeds of $\sim 50\mu m\, s^{-1}$. Laser power is used to control the beam diameters. As a byproduct of the fabrication process, all beams were elliptical, with an aspect ratio of ~3:1. Beams can be made to be circular by writing structures using a layer-by-layer process, but this writing method results in structures with larger dimensions. The smallest beam dimensions that can be written using this process are on the order of ~200 nm, and in this work the beam dimensions range from 400 nm to 2 μm. Unit cell sizes of fabricated nanolattices ranged from 3 to 15 μm, and overall sample dimensions were between 25 and 85 μm.

Hollow structures were written using the polymer nanolattices as a base scaffold; polymer surfaces were conformally coated in alumina (Al_2O_3) using atomic layer deposition (ALD). Deposition was done at 150 °C in a Cambridge Nanotech S200 ALD system using the following steps: H_2O is pulsed for 15*ms*, the system is purged for 20*s*, trimethyl aluminum (TMA) is pulsed for 15*ms*, the system is purged for 20*s*, and the process is repeated. The carrier gas is nitrogen, flown at a rate of 20*sccm* (standard cubic centimeters per minute). The process was cycled for between 50 and 1200 cycles to obtain the desired thickness coatings on the nanolattices, which ranged from 5 to 120 nm. The thickness of the coatings was verified using spectroscopic ellipsometry with an alpha-SE Ellipsometer (J.A. Wollam Co., Inc.). After deposition, two outer edges of the coated nanolattices were removed using focused ion beam (FIB) milling in an FEI Nova 200 Nanolab system to expose the polymer to air. After this exposure, the samples were placed into an O_2 plasma barrel asher for a time period between 50 and 75 hours with a 300*sccm* flow rate of O_2 under 100*W* of power to fully remove the polymer. This process is nearly identical to that reported in Ref. [21].

2.2. Nano-mechanical experiments

Monotonic and cyclic uniaxial compression experiments were performed on nanolattices in a G200 XP Nanoindenter (Agilent Technologies). Structures were compressed uniaxially to $\sim 50\%$ strain at a rate of $10^{-3}s^{-1}$ to determine their yield stress (σ_y), Young's modulus (E) and overall deformation characteristics. The data obtained from nanolattice compression experiments performed in this work had a wide range of stress-strain responses, which required the formulation of a consistent method to measure meaningful Young's modulus and yield strength. In every sample tested, the stress-strain data was comprised of a toe region, a linear region, and a failure region (Fig. S1). The toe region is a non-linear segment of data at the beginning of loading, and generally corresponds to slight misalignments and imperfections between the sample and the indenter tip. For each sample, a subset of stress-strain data was taken starting at the beginning of loading and going to the onset of failure (shown in blue in Fig. S1). The maximum slope of this data subset was defined as the Young's modulus, E. This is done to mitigate the effect of the toe region on the stiffness measurement. In polymer samples, or any sample with