

Obtaining auxetic and isotropic metamaterials in counterintuitive design spaces: An automated optimization approach and experimental characterization

Timon Meier^{†,1}, Runxuan Li^{†,1}, Stefanos Mavrikos¹, Brian Blankenship¹ Zacharias Vangelatos¹, M. Erden Yildizdag², Costas P. Grigoropoulos^{1,*}

¹ Department of Mechanical Engineering, University of California, Berkeley, CA 94720, USA.

² Faculty of Naval Architecture and Ocean Engineering, Istanbul Technical University, Istanbul, 34469, Turkey.

[†] The authors contributed equally to this work.

* To whom correspondence should be addressed: cgrigoro@berkeley.edu

Supplementary Information

Methods:

Finite element analysis

All FEA simulations were performed with the multipurpose finite element software ANSYS® Mechanical™ version 2021 R1 using the ANSYS® Parametric Design Language (APDL). To enhance our analysis capabilities, we utilized PyMAPDL, a Python Application Programming Interface (API), which granted us access to the local MAPDL solver and the simulation data it generated. PyAnsys has transformed into a comprehensive collection of Python packages that enable seamless integration with ANSYS®. The python code will be made available upon reasonable request to the corresponding author. The following beam lattice properties were used in the FEA: 1.281 GPa elastic modulus, 0.4999 Poisson's ratio. Only elastic material properties were used. The structures were meshed with 3D 2-node, beam finite elements of type BEAM188 with circular cross section. The average element size was selected by conducting a mesh convergence study and set to an element length of 0.5 μm . Thus, each 5×5 structure roughly contained between 50,000 and 200,000 nodes.

The stiffness tensor $\mathbf{C}$ of the $5 \times 5 \times 5$ microlattice that exhibits cubic symmetry is of the form:

$$\mathbf{C} = \begin{bmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{bmatrix} \quad (1)$$

The three components stiffness tensor were obtained by simulating compression and shear loading of the structure and then using the measured average stress and strain components. We employed the methodology used in reference: (Xu, S., Shen, J., Zhou, S., Huang, X. and Xie,

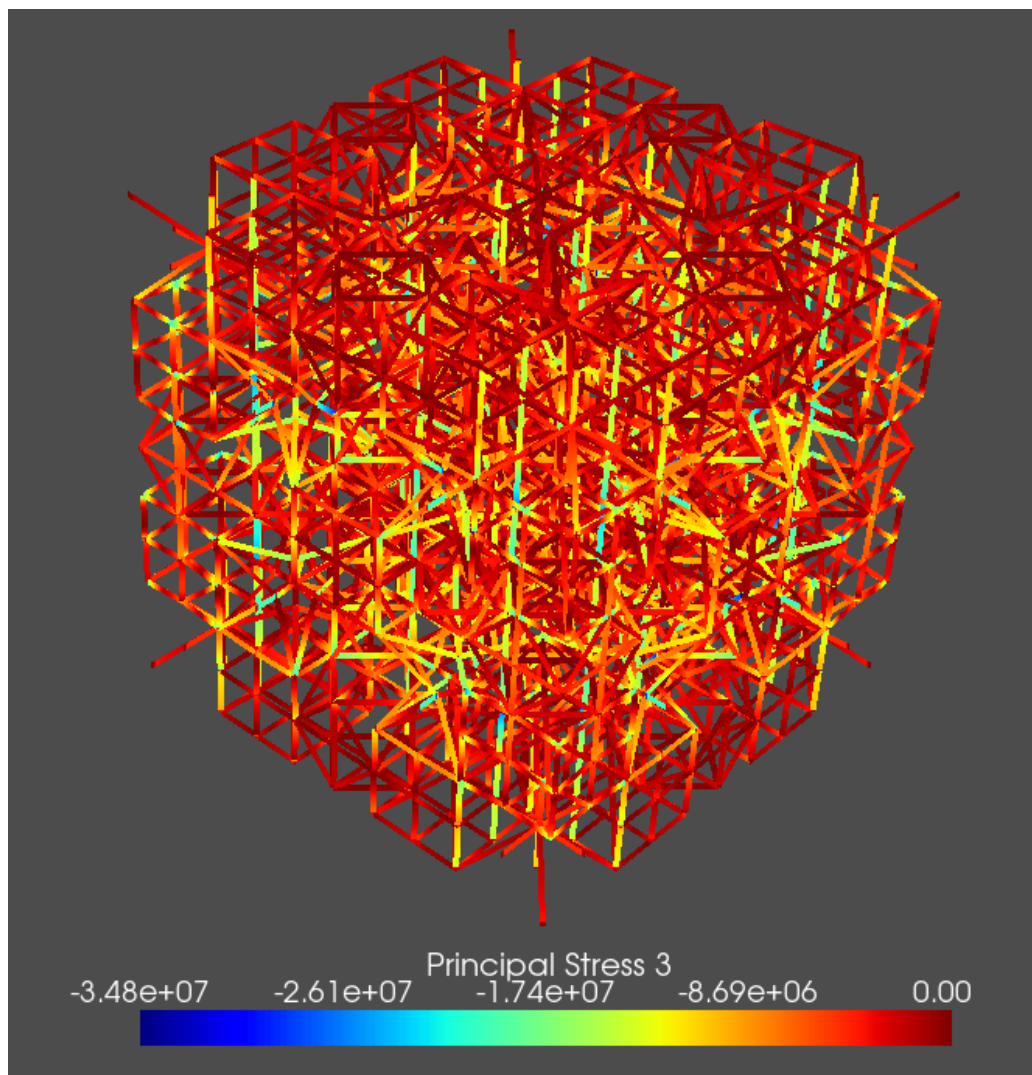
Y.M., 2016. Design of lattice structures with controlled anisotropy. Materials & Design, 93, pp.443-447.).

The boundary conditions for compression (2) and shear testing (3) are defined by:

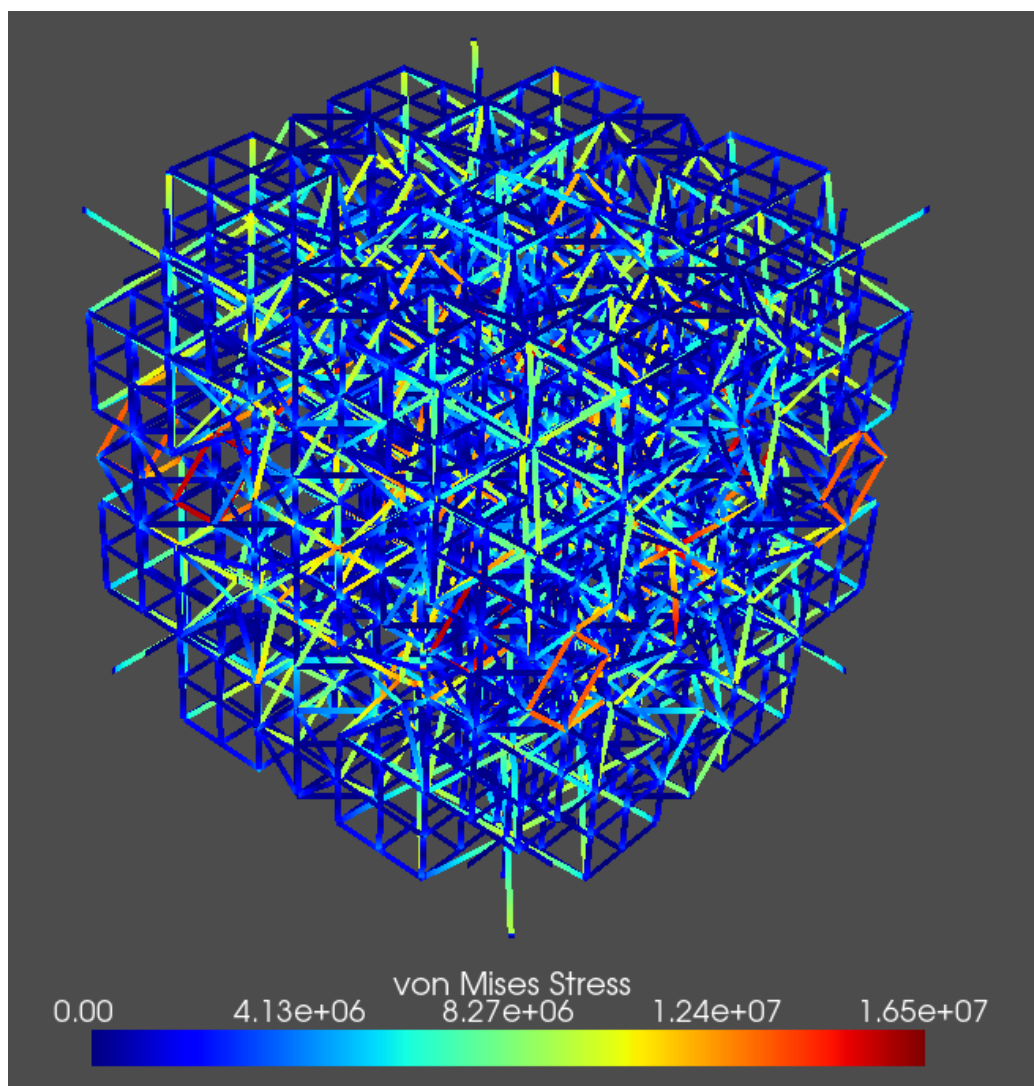
$$\begin{aligned} \Delta l_x|_{x=l_x} &= 0.01 * l_x \\ \Delta l_x|_{x=0} &= \Delta l_y|_{y=l_y} = \Delta l_y|_{y=0} = \Delta l_z|_{z=l_z} = \Delta l_z|_{z=0} = 0 \end{aligned} \quad (2)$$

$$\begin{aligned} \Delta l_x|_{z=l_z} &= 0.005 * l_z, \quad \Delta l_z|_{x=l_x} = 0.005 * l_x \\ \Delta l_z|_{x=0} &= \Delta l_y|_{y=l_y} = \Delta l_y|_{y=0} = \Delta l_z|_{z=l_z} = \Delta l_x|_{z=0} = 0 \end{aligned} \quad (3)$$

Supplementary Figure 1 and 2 show FEA simulation results for compression and shear testing for structure $\omega = [H G B E D G D B D A]$ illustrative:



Supplementary Figure 1: Compression Testing – Principal nodal stress [Pa]



Supplementary Figure 2: Shear Testing – Von mises stress [Pa]

Fabrication

The microlattice structures are fabricated using a hybrid organic-inorganic resin, which was produced mixing methacryloxypropyl trimethoxysilane (MAPTMS, 99%), which had been first hydrolyzed using HCl solution (0.1M) at 1:0.1 ratio, and zirconium propoxide (ZPO, 70% in propanol). After 15 min stirring, 2-(dimethylamino) ethyl methacrylate (DMAEMA, 99%) was added at a DMAEMA/MAPTMS molar ratio 5:5. Finally, 4,4-bis(diethylamino) benzophenone (BIS), which is used as photoinitiator, was added at 1% w/w concentration with respect to MAPTMS and DMAEMA, and the final composite was filtered using a 0.22 μm syringe filter. All chemicals were obtained from Sigma-Aldrich and were used as received unless otherwise stated. The fabrication of the structures for mechanical testing is achieved through sub-micron resolution direct femtosecond laser writing, utilizing a FemtoFiber pro NIR laser with a wavelength of 780 nm, pulse width of 100 fs, and repetition rate of 80 MHz. The photopolymerization of the photosensitive material was performed using a 100x microscope objective lens (Plan-Apochromat 100x/1.40 Oil M27, Zeiss).

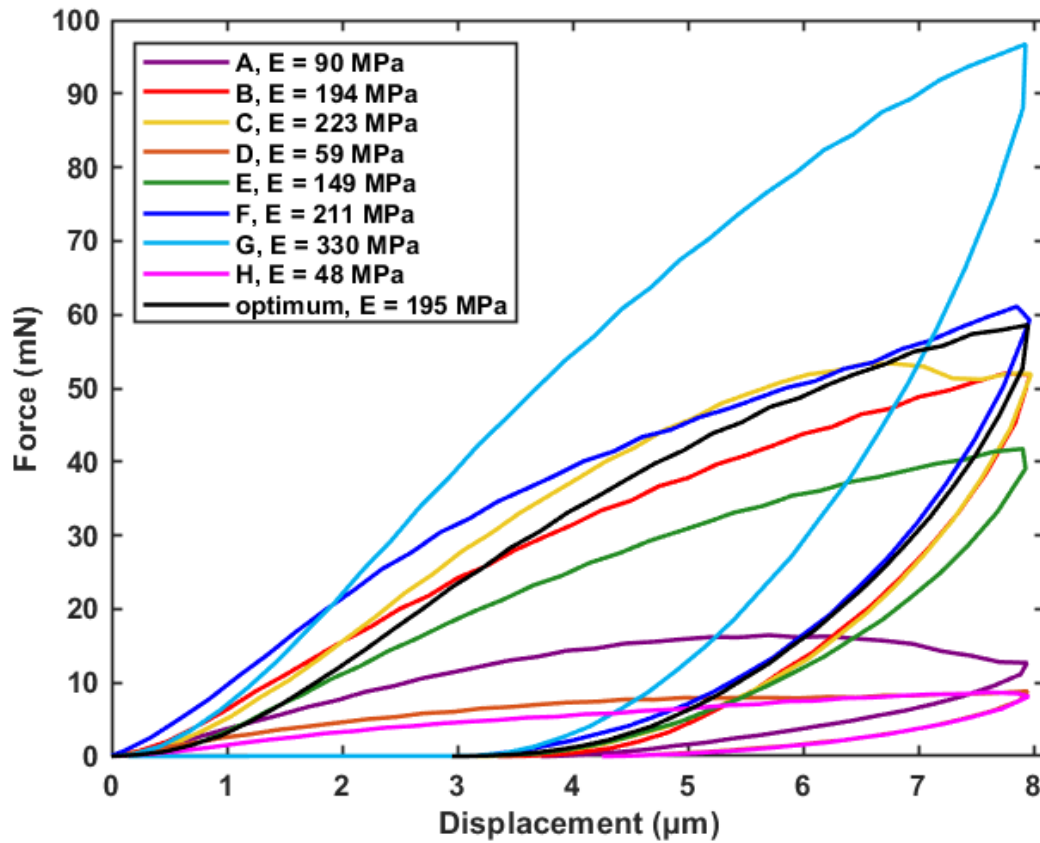
Power control is achieved using an electro-optical amplitude modulator that enables rapid and precise adjustments to the laser beam power. Additionally, an automatic calibration system is implemented to ensure accurate and consistent calibration of the laser power. This system includes a power meter mounted on a motorized flipper mount, allowing precise positioning of the power meter. By employing this setup, the correlation between different experiments is enhanced, providing a safer and more reliable basis for comparison. A parametric study is conducted to determine the optimal laser power and writing speed for achieving the best printing results of the microlattices. The laser output energy during the fabrication process is measured at 6 mW before the objective lens, while the scanning speed is set to 35 $\mu\text{m/s}$. The resin sample was secured on a stage equipped with piezo and servo elements, enabling movements in the XYZ directions.

A camera is utilized to locate the substrate surface, specifically defined as the bottom of the fabricated structure. By accurately identifying the substrate surface, the camera assists in ensuring the alignment and positioning of the laser writing process. This step is essential for achieving the desired structural integrity and dimensional accuracy of the microlattices. Furthermore, the camera enables in situ monitoring of the polymerization process. It captures real-time videos of the fabrication procedure, allowing researchers to observe the formation of the microlattice structure as it progresses. This monitoring capability is valuable for assessing the quality of the fabrication process and detecting any potential complications or irregularities that may arise during the polymerization. In addition to its primary functions, the camera serves as a tool for early detection of issues such as laser misalignment, material defects, or other factors that could affect the quality of the fabricated microlattices. By promptly identifying and addressing these problems, researchers can take corrective measures and optimize the fabrication process to ensure consistent and reliable results. For a more visual demonstration of the in situ camera imaging during the fabrication of a microlattice, a representative video has been included in the Supplementary Video 10. This video provides a detailed insight into the fabrication process, showcasing the dynamic formation of the microlattice structure and illustrating the real-time monitoring capabilities facilitated by the camera.

Testing

The mechanical testing apparatus, it consisted of a nanoindentation apparatus (PI 87 SEM PicoIndenter, Hysitron), inside a Scanning Electron Microscope (FEI Scios 2, Thermo Fisher Scientific) which enables high-precision nanomechanical testing and in situ recording of the deformation. A molybdenum flat tip indenter with a diameter of 70 μm , obtained from Probing Solutions Inc., was utilized for all mechanical tests. All of the fabricated arrays were designed such that the surface area of the periodic structure would be less than the contact area of the sample for uniform loading. Quasi static experiments were performed with loading rate 250 nm/s triangular loading profile to hamper creep during the indentation experiments.

The force-displacement curves for all monolithic structures A - H and the optimum are presented in Supplementary Figure 3. Youngs modulus and Poisson's ratios can be determined based on the force displacement curve and the in-situ measured ratio between lateral and transversal deformation. Videos showing the compression testing of the 8 monolithic structures A – H and the optimum configuration are attached (Videos 1 – 9).



Supplementary Figure 3: Force displacement response of monolithic structures A – H and optimum

Other Supplementary Material for this manuscript includes the following:

Supplementary Video 1: Deformation of monolithic structure A during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 2: Deformation of monolithic structure B during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 3: Deformation of monolithic structure C during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 4: Deformation of monolithic structure D during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 5: Deformation of monolithic structure E during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 6: Deformation of monolithic structure F during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 7: Deformation of monolithic structure G during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 8: Deformation of monolithic structure H during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 9: Deformation of optimum structure during the first load/unload cycle (magnification = 3500X, voltage = 20 kV, beam current = 0.80 nA).

Supplementary Video 10: In-situ real time captured screen video showing the dynamic formation of the first 5×5 layer of the optimum microlattice using the MPL setup.