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The Twisting of Dome-Like Metamaterial from Brittle to Ductile

Lizi Cheng, Tao Tang, Haokun Yang, Fengqian Hao, Ge Wu, Fucong Lyu, Yu Bu, Yilu Zhao, Yan Zhao, Guo Liu, Xuan Cheng, and Jian Lu*

Architected materials can exhibit mechanical properties that do not occur with ordinary solids. By integrating hierarchy and size effects, microarchitected metamaterials fabricated by two-photon lithography with a metallic or ceramic coating can be ultrastrong but lightweight. However, the attainment of both strength and ductility is generally mutually exclusive. Inspired by the Pantheon dome in Rome, which can withstand high load while keeping low density, microarchitected domes with a gradient helix are designed and deposited in a hierarchical nanostructured aluminum film with ultrahigh strength and considerable plasticity. Despite having a thick coating, which usually causes catastrophic collapse, the thick-walled metallic dome shows recoverability during compression. The compressive strength increases to 73 times that of current ductile-like microlattices, leading to the metamaterial occupying the domain of the material property space that is hitherto empty. Detailed in situ experimental and computational work reveals the graceful (noncatastrophic) failure due to the helical twisting and plastic flow in the supra-nanomaterial. It is a promising method of suppressing brittle failure via a combination of architectural and material design. It can be used to impart enhanced functionality, making programmable stiffness, and tailored energy absorption all possible.

1. Introduction

The field of mechanical metamaterials exploits the design freedom in geometry and material composition has recently opened the door to exotic mechanical properties previously inaccessible.

Typical examples are self-assembled 3D complex architecture in origami,[1,2] twisted auxetic structure in lattices,[3,4] and decoupling historically linked properties such as strength versus density.[5,6] The recent developments in two-photon lithography (TPL) and thin film deposition techniques have enabled the fabrication of mechanical metamaterials with several levels of hierarchical structuring, ranging from the micro- to the nanoscale.[7] The core–shell hierarchical composites with plastic polymer “core” and a layer of metallic or ceramic film “shell” have obtained superb stiffness and strength at a very low density.

The most common cause of the failure of metamaterials is the brittleness of the film. It has been reported that the ductile-like deformation and recoverability are attributed to the ratio between the thickness of the film (so-called “wall thickness,” t) and the semimajor axis (a) of the elliptical strut cross-section. When t/a ≤ 0.02, the nanolattices deform through shell buckling and recover when the force is removed; with t/a ≥ 0.03, referred to as “thick-walled,” the nanolattices fail catastrophically with little or no recovery.[6]

Much work so far has focused on alleviating catastrophic failures by creating hollow nanolattices via etching away the polymer substrates[8,9] or reducing the wall thickness below 100 nm to achieve the size-induced brittle-to-ductile transition (referred

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Figure 1. Schematic diagram of the geodesic dome-inspired hierarchical mechanical metamaterial. a) Classical dome architecture inspired by Pantheon, Rome. Intersecting ten rings (blue) and sixteen arches (red) hinged together to form a 3D compression system in (b). c) Illustration of the fabrication process for the core–shell dome architectures, with the characteristic sizes $2a$ (strut diameter of major axis) and $t$ (thickness of AlNiY coating). d) SEM image of microdome composites. e) Composite with FIB cut. f) TEM image of the hierarchical nanostructured Al alloy. g) HRTEM image of the Al film, showing nanograins are surrounded by amorphous phase. Inset: a fast Fourier transformation (FFT) image of the nearly pure fcc Al nanocrystal marked by the dashed line, oriented to the [011] zone axis.

to as “smaller/thinner is more ductile” size effect.

Nevertheless, the reduced wall-thickness contrarily limits its strength and stiffness. Thus, it is essential to explore more daring architecture, which can serve as a template for the strong but brittle layer to deform in a ductile manner.

The widely studied nano/microlattice is octet-truss, whose strength is highly dependent on the rigid beams, thereby sensitive to the flaw. However, when we look at some macroscopic architectures, we were inspired by the best-preserved monuments of ancient Rome—the Pantheon (Figure 1a). The structure, completed around 126–128 A.D. during the reign of Emperor Hadrian, features a rotunda with a massive domed ceiling that was the largest of its kind when it was built. The dome architectures (Figure 1b) have the merit of i) aesthetic and lightweight, which can enclose most amount of space with the least amount of material, ii) good deformability due to nonaxially oriented beams, iii) even distribution of the stress through a series of triangle elements, and iv) breaking the interconnected limit of building structures like lattices. In a pioneering work, auxetic cellular configurations (i.e., arrow-head, re-entrant, and trichiral) are successfully integrated into dome architectures as a new class of metamaterials, which prove to have an auxetic property (negative Poisson’s ratio) and its macroscopic strength is investigated. Due to the rotation of the chiral cells, auxetic
cellular domes, especially with re-entrant topology, show less snap-through behavior than conventional solid domes that have higher compressive strength. However, so far, very few works have been reported the integration of hierarchy. Here, to explore the new toughening mechanism, dome-shaped hierarchical microarchitected metamaterials are developed by introducing helix to arches and rings with structural chirality (Figure 1c; Figure S1, Supporting Information).

Compared with atomic layer deposition (ALD), magnetron sputtering is a high-speed and low-cost approach and is more suitable for thick film deposition. Recent studies have shown that metallic films deposited by magnetron sputtering have a 3D homogeneous structure and are prone to form nanograins with nanosized metallic glass shells (dual-phase nanostructuring), resulting in a theoretically “ideal” strength. Al$_{93.4}$Ni$_{2.2}$Y$_{4.4}$ alloy is chosen as the metallic film due to two reasons. This alloy has enhanced ductility by impeding dislocation gliding and flowing of the nanosized glass phase. On the other hand, it has a higher strength-to-weight ratio than other alloys such as high entropy alloy (HEA), which requires a thicker polymeric beam to support the heavy metallic film. Since the classical theory alone is insufficient to capture this novel architecture's mechanical behavior, we apply the in situ compression test and the finite element analysis (FEA) to develop a new principle to describe the chiral mechanical metamaterial.

2. Results

The thick-walled core–shell Al hierarchical structures with excellent strength and stiffness are synthesized through TPL and magnetron sputtering approach (Figure 1c,d). All struts in microdomes are entirely covered by the film (Figure 1e). The ratio of film thickness to semiaxis is measured by the effective area in the text given in the Supporting Information and is determined to be 0.67. Part of the metallic film on the polymer is then extracted by a focused ion beam (FIB) for transmission electron microscopy (TEM) analysis (Figure S2, Supporting Information). TEM results of the metallic film show a hierarchical structure that Al nanograins uniformly dispersed in the specimen and surrounded by nanosized interfaces (Figure 1f). The interface’s lengths between the nanograin ranging from ten to a hundred nanometers and the thickness are larger than the medium range clusters in metallic glasses. High-resolution transmission electron microscopy (HRTEM) image (Figure 1g) further confirms the interface is a secondary amorphous nanolayer rather than conventional grain boundaries, demonstrating the glass-crystal dual-phase an Al film. This hierarchical nanostructured aluminum film was proven to have ultra-high strength and large plasticity in our previous study because the nanosized metallic glass phase can impede dislocation gliding and exhibit a homogeneous plastic flow behavior. The film with an average grain size (almost pure Al) of ≈40 nm, indicating a hierarchical microstructure spans three orders of magnitude, from 40 nm (diameter in grain) to 50 µm (diameter in the whole sample). The energy-dispersive X-ray emission (EDX) element mapping and spectrum analysis are conducted in SEM-EDX with 10 kV acceleration voltage to minimize electron beam damage. The mapping result shows a uniform elemental distribution, implying the homogeneous nature of the coating. In order to avoid possible thickness variations, elemental composition is analyzed from the near-flat area in the top center of the dome (Figure S3, Supporting Information). Nanoindentation also shows the aluminum film exhibits an excellent mechanical property compare to its bulk counterpart (Figure S4, Supporting Information).

2.1. Mechanical Property of Microdome Polymers

Deformation behaviors of microdome polymers and composites are observed and compared with an octet structure via in situ uniaxial microcompression tests. For microdome polymers, all dome-shaped microarchitectures can almost fully recover to their original shapes under the large strain of 19.6% (Figure 2a–d). In sharp contrast, stretch-dominated octet architecture with the same height undergoes failure at 12.8% strain and demonstrates a lower specific load capacity (i.e., $F/\rho$) (see Figure S5 and Movie S1 in the Supporting Information). It confirms that dome structure enhances the damage resistance, as compared to the octet-truss in the same condition. The high strain elastic recovery is attributed to the physical mechanisms, a unique form of structural damping, whereby local elastic buckling of individual ribs releases energy upon loading. During the compression process, octet deforms by plastic hinging of the nodes, resulting in irrecoverable plastic deformation. In contrast, with the increased number of ribs, dome’s arches distribute strain energy efficiently to the ground via elastic buckling, accommodating large global deformation without failure.

The original dome undergoes a rapid transition between stable states (snap-through state, as indicated in Figure 2a), as the non-linear response always accompanies snapping. The arches initially bent hinges and then snap to their second stable configurations characterized by curvature inversion. The top part of the dome’s deformation behavior could be easily observed from FEA simulation results, although the indenter blocks the top area during the in situ compression test. The observed snap-through response in FEA from the front view is entirely consistent with the experiments and other solid or auxetic dome architectures. At around 2.5 µm of deformation, the original dome shows evident collapse at the top, and the dome with helical rings shows a slight depression, indicating snap-through instability raised from the soft nature of polymer (Figure 2e–g). By contrast, the helical-arches dome exhibits a more uniform stress distribution and excellent stability with a flat contact during compression without top collapse.

Moreover, helical arches allow the struts around the vertex to rotate as the depth of deformation increases. The overall structure with the top and bottom fixed. Dome architecture shows a pronounced chiral effect; hence it can be regarded as a mechanical metamaterial. The maximum rotation angle can be up to 25°, as shown in the twist angle–displacement curve (Figure 2h). Helical arches induce a rotating distortion meta-effect, thus release a portion of the total energy and lead to relatively lower stiffness. Meanwhile, it relieves the top collapse caused by shell snapping of soft constitute.

2.2. Mechanical Property of Microdome Composites

The density and curvature of the dome will affect the compressive resistance and the severity of the snap-through.
Here, we consider sputtering hierarchical nanostructured film and obtaining core–shell microdome composites to reinforce the top and prevent the top from collapse (Figure 3a–d). The stiffness in the linear elastic regime increases dramatically from 66.68–235.76 to 5168.64–8860.82 N m⁻¹ after coating. The configuration with helical arches exhibits a higher sensitivity to coating, whose increased stiffness is twice as much as the other two configurations. Moreover, two distinct deformation signatures are observed during the large deformation (Movies S2–S4, Supporting Information). The original dome behaves similarly to the design with helical rings, demonstrating linear elastic loading followed by a sudden catastrophic failure (Figure 3e,f,h,i), a typical brittle response in shell loading. The original dome experiences the failure at 7.31 ± 0.70% with a peak load of 12.32 ± 1.04 mN, which can be slightly increased to 7.98 ± 0.43% with the load of 12.70 ± 0.94 mN when helical rings are introduced, indicating that the helical rings can improve ductility by enhancing the yield strength and delaying fracture strain. Unfortunately, the modification for rings still fails to alleviate the sensitivity to defects in architected material. Surprisingly, the dome with helical arches effectively delays the fracture strain to 10.45 ± 0.49%, and shows no sudden decrease in the load after yielding, but a continuous flow behavior, indicating that the fracture occurred in a ductile manner (Figure 3g,j). The ductile dome undergoes plastic deformation during indentation, accounting for the observed nonlinear deformation in the F–D curve.

Chiral hinge lattice with auxeticity has been proven to have a more ductile behavior because of flexure of the ribs.27 Dome is composed of rings and arches hinged together. After introducing helical beams, the geometry with the cross-chiral topology is associated with a rolling-up mechanism, generating internal rotations and auxetic behavior.28 The metamaterials created in this work show an exceptional strength–ductility combination by coupling compressions with rotational deformation modes. We further
Figure 3. Compression experiments on the microdome composites. a) Force–displacement curve of microdome deposited AlNiY film (composite). Sample size, 9. b–d) SEM image of microdome composites with different helical architectures from the top view. e–j) SEM snapshots for the compression process corresponding to three different microdome composites, showing the catastrophic brittle failure and no post-compression recovery for domes without helical arches (black and red) and the reversible ductile-like deformation for domes with helical arches (blue). Scale bars: 10 µm.

compared with other reported ductile micro/nanoarchitectures made up of metals,[7,8,29] metallic glass,[10,11] or ceramics[11,24] (Figure 4a,b). In core–shell materials, stress concentrations will increase sharply when a significant difference in stiffness between the core and shell. Hence, localized bands of high stress emerge once loaded beyond the yield point, leading to a catastrophic collapse. Thus, unlike the hollow tube microlattice who combined the Euler buckling and shell buckling to give rise to a ductile deformation, the composite usually failed via brittle fracture. The formation of the failure criteria can be described as[16]

\[
\sigma_{\text{beam}} = \frac{E l^2}{(k l)^2 A} \approx \frac{3\pi^2 E}{4L^2} \frac{a b^2}{a + b}
\]

(1)

\[
\sigma_{\text{shell}} = \frac{E}{\sqrt{3} (1 - \nu^2)} \left( \frac{t}{a} \right)
\]

(2)

Here, \(a\) and \(b\) correspond to the semimajor and minor axis of the elliptical strut. \(E\) is Young’s modulus, and \(\nu\) is the Poisson’s ratio of the material. The film thickness is \(t\). To quantify the transition of two failure modes, the critical buckling transition value is defined as equalizing the two formulas. Previous works[6] have identified that when \(t/a\) is below the critical buckling transition value (≈0.03), the structure shows a ductile-like deformation. However, the ductile hollow microstructures have a detrimental effect on strength and stiffness as the load transfers only via shell wall bending. Besides elastic shell buckling, an ultrathin coating is also exploited for enhancing ductility. With the film thickness decrease, the failure mechanism eventually shifts from the film dominated brittle fracture to polymer dominated Euler buckling.[30] Notably, our helical-arched dome architecture enjoys a high value of wall thickness ratio without brittle failure, thus expand the domain of ductile microstructures by achieving a more abundant strain accommodation and higher compressive strength. Meanwhile, dome architectures can enclose more space, providing more flexibility and possible applications than classical truss, which become vulnerable when the local buckling is concentrated at the nodes (Figure S5 and Movie S5, Supporting Information). We attribute this ductile-like deformation primarily to the helix in structural integrity as geometric nonuniformity induced a twist (Figure 4c) that suppressed stress concentration. The rigidity of a dome mainly depends on the stiffness of junctions, which are more flexible in helical arched domes. Material property plots of the compressive strength versus density (Figure 4d) for existing materials include ductile and brittle microarchitected materials reported so far, such as graphene,[31] carbons,[32] ceramics,[33] or ceramic–polymer composites.[30] These plots reveal that our shape-optimized metamaterials exceed all existing technical foams with a density below 1000 kg m\(^{-3}\) and reach a new niche in the strong but ductile material parameter space.

Composite material exhibits better damping characteristics than conventional metals, as it will develop numerous microcracks under the action of an impact load. The formation of microcracks results in the absorption of energy impact and prevents catastrophic failure. Cyclic experiments are conducted on the helical-arched dome (Figure 5a–c). In the first cycle, damage occurs and the sample recovers to 65% of the original height after compression to 19.4% strain. The subsequent cycles show a decrease in the stiffness but a nearly identical unloading path. The peak force is absent during the second compression and shows the “pseudohardening” behavior, resulting from the flattening of the cross-sectional footprint area and the compaction of the structure under uniaxial compression. The strength and ability of recovery are higher after each cycle. The last curve showed 88% recovery to this initial deformed height after compression for five
times under 19.4% strain. The energy loss coefficient (Δu/u) reflects the hysteresis of the material during cyclic deformation. For the first loading cycle, the energy loss coefficient is 0.65, and the highest energy dissipation results from extensive node microcracking. After three cycles, the energy loss coefficient remains intact at ≈0.3. The stable cyclic behavior may account for the failure mode activated in the first cycle and can be reactivated in the following cycles, therefore minimizing the accumulation of additional damage. The graded topological design and nonhomogeneous microarchitecture result in a continuous redistribution of stress. A twist during compression (Figure 5d–g) triggers a tendency to return to its original state, naming the twist-back effect.[34] Our dual-phase Al film whose nanosized metallic glass phase impedes dislocation motion of nanograins during deformation result in high strength. Meanwhile, the flow behavior of the nanosized metallic glass phase,[22] together with the twist behavior in architecture which prevents beam buckling, further accommodates larger strains and facilitates the ductility.

2.3. Multiscale Hierarchical Metallic Domes

Dome-like metamaterials are intriguing for many reasons. Their low relative density and unique shape offer lightweight solutions for applications ranging from micro to macroscale. For example, it can be applied to the controlled steering of the catheter tip in interventional surgery (Figure 5h). The conductive dome-like blunt front will not hurt blood vessels during the insertion, ensuring the operation’s safety. When there is pressure in front of the vessel, the catheter tip requires bending and deflection.[35] The challenge is that current commercial catheters are not flexible enough and are all disposable. However, our tailored microarchitected metallic dome can be a perfect steerable catheter tip for its strength, flexibility, and reusability. In the aerospace field, dome metamaterial can be the damage-tolerant components or lightweight filling materials in the blade[14] or a full-wing structure[36] (Figure 5i). As a filling material inside the wings, the dome structure has a robust buffering ability, reducing the vibration caused by the airflow and absorbing energy through deformation. Face-centered cubic (fcc) units are widely used as damage-tolerant architected materials.[14] Here we compare the fcc unit with our architected dome under the same condition (initial height, strut diameter, material properties, boundary conditions, and load case) (Figure 5j). The uniaxial compression simulations are performed to measure large strain response and estimate the energy absorption capacity of different geometries. The dissipated energy is monotonic in the maximum strain amplitude, and here we define a cutoff-strain as 50%. Although stretching-dominated architecture (fcc or octet structure) shows superior stiffness and yield strength (see the orange point in Figure S7 in the Supporting Information), there is an abrupt decrease after the peak load at around 2% strain due to the buckling of the struts. In contrast, bending-dominated dome architecture maintains a monotonically increasing load–displacement
response and exhibits higher compliance as it distributes loads uniformly through bending moments at strut intersections.\(^\text{[17]}\) The local crack initiation will occur when the local equivalent plastic strain exceeds the critical strain of the base material, and deformed fcc configurations show all the joint nodes undergo failure at the strain of 30\%, whereas the dome only shows partial failure at the top (Figure S7, Supporting Information). The absorbed energy of dome metamaterial at 50\% strain is 0.13 J, whereas the fcc unit is only 0.055 J when using the Al7075 material. Thus, dome metamaterials with geometrical feature chirality have a higher energy absorption capacity than commonly used architected materials.

### 3. Conclusion

We propose an innovative structural design strategy to enhance ductility by introducing helical beams in dome architecture. By virtue of hierarchical nanostructured aluminum film, the stiffness are much improved compared to the microdome polymer, especially with the helical arches that increase 77 times. The metallic domes exhibit superior compressive specific strength of 156.09 ± 12.92 MPa cm\(^3\) g\(^{-1}\) with density well below the water. While the proposed architectures do not exhibit high strength and stiffness as stretching-dominated architectures, these architectures show higher compliance and higher energy absorption capacity. There is a brittle-to-ductile transition once helical arches introduced, expanding the domain of ductile microstructures in previous studies. It also shows a 73 times increase in strength than other ductile microstructures and reaches a new niche in material parameter space. We demonstrate experimentally and theoretically that the twist effect of helical-arched dome contributes to alleviate the snap-through instability in the soft materials via rotating distortion, and suppress the catastrophic collapse in the brittle composites via energy dispersion. This architecture design strategy is feasible to many other brittle material systems, thereby maximizing the advantages of the film and overcoming the strength–ductility trade-off.
4. Experimental Section

Fabrication: The fabrication process involved two stages: 1) fabrication of polymer dome, and 2) deposition of Al thin film. The dome architectures were fabricated layer-by-layer by 3D laser microprinting (Nanoscribe GmbH, Photonics Professional G.T.) using liquid photoresists (IP-L 780, Nanoscope GmbH, Germany). The optimum laser power for writing dome architecture was 25 mW. The printed structure was further processed by a bath in propylene glycol monomethyl ether acetate (PGMEA) for 15 min and then rinsed with isopropyl alcohol for 5 min. To fabricate the core–shell Al composites, Al$_{92}$Ni$_{2}$Y$_{6}$ (at%) alloy was chosen as the target. The film was deposited by magnetron sputtering in an argon environment with a pressure of 0.2 Pa, and an external substrate bias of $-50$V. The substrate temperature was maintained at room temperature.

Microstructural and Mechanical Characterization: Field-emission scanning electron microscopy (SEM, FEI) and EDX were used for microstructural characterization. FIB milling was used for strut dimension measurement and TEM sample preparation. The hardness (H) and Young’s modulus (E) of the aluminum film were obtained by nanoindentation (Hysitron, USA) using a Berkovich diamond tip. The indenter tip area function was calibrated using fused silica. The indentation depth was kept below 10% of the film thickness to avoid the substrate effect. The polymer constitute properties were measured via the nanoindentation test for 3D printed micropillars using a 10 µm flat diamond tip. The mechanical properties of microdomes were tested via the in situ quantitative Picoindentor (Hysitron TM P185) equipped with a 50 µm flat punch with a speed of 100 nm s$^{-1}$ inside the SEM.

FEM Modeling: The FEM simulation using Abaqus (Dassault Systemes, 2018) was introduced to model the compression process of the architectured domes. Geometrical models were based on different structure designs in Figure 1c. The static analysis was used to simulate the compression process due to the low loading speed. The material properties of polymer constitute and Al film constitute were obtained by the nanoindentation test. The contact algorithm was set as “general contact,” which means every face can contact each other based on a real situation. Furthermore, the tangential friction algorithm was set as 0.3 and the expected behavior as hard contact considering the real situation. The boundary was set as a fully rigid boundary condition in simulation according to the real condition. Considering the accuracy and simulation burden, structure element (C3D10) meshing technology was adopted with an element size of 2.0 nm. The contact area during compression could be obtained from the output variable CAREA of Abaqus computation result. As the contact area was increased with the displacement, the maximum yield displacement from the experiments was used to determine the contact area.

Statistical Analysis: All the force–displacement curves presented in this paper were the raw data obtained from in situ compression tests. The effective strain of the dome, defined as the percentage of applied displacement divided by the average height of the entire structure, was measured, as shown in Table S1 and Table S2 (Supporting information). The recoverability ($r$) is defined as the ratio of recovered strain ($\varepsilon_{rec}$) to applied strain ($\varepsilon_{ap}$), i.e., $r = \varepsilon_{rec}/\varepsilon_{ap}$. Compressive yield strength is defined as dividing the peak force before the plastic deformation by the nominal cross-sectional area. The structural stiffness can be measured by the slope of the load–displacement curve along with a linear regime. Deviations were measured through at least five tests and errors are shown in Figure S8 in the Supporting Information.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

J.L. designed the project. L.Z.C., H.K.Y., and F.Q.H. designed structural metamaterial and the experiments; L.Z.C. conducted the investigation, data analysis, and writing; T.T. and Y.Z. performed the finite element analysis, and T.T. wrote the simulation part; C.W. designed a PVD experiment of hierarchical dual-phase aluminum film, and assisted with TEM analysis; F.C.L., Y.B., and Y.L.Z. conducted TEM characterization; G.L. and X.C. revised the manuscript. All authors contributed to the discussion of the results.

Keywords

ductile-like deformation, hierarchical materials, mechanical metamaterials, microarchitecture

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