A damage-tolerant, dual-scale, single-crystalline microlattice in the knobby starfish, *Protoreaster nodosus*

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Abstract

Cellular solids (e.g., foams and honeycombs) are widely found in natural and engineering systems due to their high mechanical efficiency and tailorable properties. While these materials are often based on polycrystalline or amorphous constituents, here we report an unusual dual-scale single-crystalline microlattice found in the biomineralized skeleton of the knobby starfish, *Protoreaster nodosus*. This structure possesses a diamond-triply periodic minimal surface geometry (lattice constant, *ca.* 30 μ m), whose [111] direction is aligned with the *c*-axis of the constituent calcite at the atomic scale. This dual-scale crystallographically co-aligned microlattice, which exhibits lattice-level structural gradients and dislocations, combined with the atomic-level conchoidal fracture behavior of biogenic calcite, significantly enhances the damage tolerance of this hierarchical biological microlattice, thus providing important insights for designing synthetic architected cellular solids.

One-Sentence Summary

Knobby starfish skeletons exhibit a periodic porous lattice of single-crystalline calcite for enhanced protection.

Main Text

Weight reduction is often considered as a primary goal when developing structural materials, which, in turn, reduces material usage, energy consumption, and environmental impact. One effective solution is through the introduction of porosity to create cellular solids (1). Conventional cellular solids include honeycombs and open and closed-cell foams with 3D stochastic porosities (1). Recently, architected cellular materials, enabled by developments in computational design and additive manufacturing, further extend the mechanical property space of conventional cellular solids, while enabling unusual physical properties such as negative Poisson's ratio and negative stiffness (2). Current synthetic architected cellular structures are exclusively based on either polycrystalline or amorphous materials at the atomic scale (2). These synthetic architected lattice materials often exhibit catastrophic failure, especially for those with ceramic and glassy constituents. To address some of these performance limitations, and by leveraging the advantages of size effects and hierarchical design strategies, recent studies have demonstrated that ceramic nanolattices can exhibit high energy absorption capabilities (2-5). The strength of these materials, however, is usually limited by their feature sizes, such as the wall thickness in hollow nanolattices, which is maintained in the sub-100 nm range to achieve high relative strength and recoverability (2-5).

Widely found in the shallow waters of the tropical Indo-Pacific, the knobby starfish, *Protoreaster nodosus* is characterized by its hard pointy protrusions or "knobs" arranged radially along its aboral (dorsal) surface (Fig. 1A-C). Upon removal of the overlying soft tissue, a well-organized assembly of the calcareous skeletal elements (ossicles) are revealed with optical microscopy (Fig. 1B and Fig. S1) or micro-computed tomography (μ -CT, Fig. 1C, Fig. S2,3, Movie S1). Scanning electron microscopy (SEM) reveals that these millimeter-sized ossicles exhibit a porous lattice-like structure (Fig. 1D,E), which is so ordered that atomic terrace-like morphologies can be observed on the ossicle surfaces (yellow arrows, Fig. 1E). Furthermore, imaging of the fractured surfaces demonstrates that the periodic microlattice structure fully extends into the ossicle interiors (Fig. 1F, Fig. S4).

To determine its lattice structure and symmetry, we performed quantitative 3D analysis on a representative high-resolution μ -*CT* volume (243×243×243 µm³) extracted from a single ossicle (Fig. 1G, Movie S2). The skeletonized lattice network demonstrates the periodic arrangement of the tetrahedron units with a branch length (*l*) of 15.5 ± 2.5 µm (5,499 measurements in total) (Fig. 1H, Fig. S5, Movie S3, Methods). The corresponding 3D fast Fourier transform (3D-FFT) reveals that the most intense reflections result from the cubic "*Fd*3*m*" symmetry, such as the {111} and {220} families, confirming the diamond microlattice structure in these ossicles (Fig. 1I and Fig. S6-8, Movie S4) (*b*). Measurement of the reciprocal lattice constants from the 3D-FFT data reveals a lattice constant (*a*) of 34.0 ± 5.9 µm, which is in excellent correspondence with the real-space measurements ($a = 4l/\sqrt{3}$ for a diamond lattice). In addition to the standard "*Fd*3*m*" reflections, "symmetry-forbidden" intensity spots corresponding to the {*HKL*} planes with H + K + L = 4n + 2 (*n* is an integer) were also observed, for example, the {002} family (circled in green, Fig. 1I and Fig. S6,7) (*7*). In contrast to atomic diamond crystals, the presence of these symmetry-forbidden reflections results from the branches connecting neighboring nodes in the diamond lattice structure (Methods) (*7*, 8).

The surface morphology of the ossicles' diamond microlattice exhibits remarkable resemblance to a standard diamond-triply periodic minimal surface (diamond-TPMS) structure when viewed normal to low-index planes, such as the (111), (110), and (112) (Fig. 1J). From the synchrotron μ -*CT* data, we quantified the minimum and maximum principal curvatures (*i.e.*, κ_1 and κ_2 , respectively) of the microlattice surface and represented their distributions in an interfacial shape distribution plot (Fig. 1K). κ_1 and κ_2 are estimated as $-0.135 \pm 0.050 \,\mu\text{m}^{-1}$ and $0.137 \pm 0.043 \,\mu\text{m}^{-1}$, respectively. The corresponding mean curvature ($H = (\kappa_1 + \kappa_2)/2$) is $0.001 \pm 0.032 \,\mu\text{m}^{-1}$, which explains the single narrow spot on the line $\kappa_1 + \kappa_2 = 0$ in Fig. 1K. The contour plot of *H* mapped onto the original structure shown in Fig. 1L further reveals the uniform distribution of nearly zero *H* values. While the existence of periodic minimal surfaces in echinoderm skeletal elements has been broadly accepted in the literature (9), to the best of our knowledge, this current study represents the first quantitative confirmation of a mineralized biological diamond-TPMS structure in nature.

This diamond-TPMS microlattice fully comprises the entire volume of each ossicle, making it possible to "index" specific lattice planes of their outer surfaces (Fig. 1D and Fig. S9). As shown in Fig. 1D, the normal directions of the ossicles are primarily oriented along the [111] direction of the diamond microlattice. The μ -CT projection images and corresponding 3D-FFT patterns further demonstrate that the diamond microlattice structure is conserved in ossicles from both the aboral and oral sides (Fig. 1M, Fig. S10-11, Movie S5), and the lattice constant is 29.2-34.2 µm (corresponding to the branch length *l* of 12.6-14.8 µm) (Fig. S12 and Table S1). By using a customized cellular network analysis algorithm (*10, 11*), we further show that structural parameters, such as branch length and thickness, exhibit spatial variations within individual ossicles, as demonstrated in Fig. S13-S17 and Table S2,3.

Defects such as dislocations are ubiquitous in atomic crystals and play significant roles in controlling material properties (12). We investigated the structural defects at the lattice level within ossicles and compared them with known classes of defects in atomic crystals with the cubic diamond symmetry (e.g., diamond and silicon) (12). Careful examination of the μ -CT slices and SEM images of fractured ossicle

surfaces reveal the presence of dislocation-like lattice defects (Fig. 2A,B). In addition, we identified two prominent dislocation types in the ossicles, namely 60° dislocations and screw dislocations (Fig. 2C-H), which are also the primary dislocation types found in atomic diamond crystals (12-15). For the 60° dislocation, the Burgers vector (b_{60°) and the dislocation line direction (ξ_{60°) are oriented along different <110> directions (e.g., $b_{60^{\circ}} = 1/2[01\overline{1}], \xi_{60^{\circ}} = [\overline{1}0\overline{1}])$, making a 60° angle between $b_{60^{\circ}}$ and $\xi_{60^{\circ}}$ (13, 14). Similar to atomic diamond crystal structures, due to the presence of two inequivalent families of {111} planes, namely the glide set and shuffle set, two types of 60° dislocations are observed, i.e., G-type and Stype respectively, depending on where the inserted extra plane terminates (Fig. 2F,G) (14, 15). In addition, the branches on the "compressive" and "tensile" sides of the 60° dislocation cores have reduced and increased lengths, respectively, whereas the branch thickness remains relatively uniform (Fig. 2F,G). For the screw dislocations, the Burgers vector (b_s) and the dislocation line direction (ξ_s) are from the same <110> direction (e.g., $b_s = 1/2[011], \xi_s = [011]$), which form the classic zig-zag-like pattern of screw dislocations when viewed normal to a $(11\overline{2})$ plane (Fig. 2E). While the core structure of the screw dislocations exhibits multiple different configurations in atomic diamond crystals (13, 16), our analysis reveals that the dislocation core in the ossicles' diamond microlattice resembles an undissociated screw dislocation in the shuffle set with kinks (Fig. 2H) (17, 18). Further structural analyses of dislocations in ossicles are shown in Fig. S18-23.

At the whole ossicle level, the microlattice dislocation density is estimated in the range of 100-1200 cm^{-2} , corresponding to a normalized density of 0.001-0.011 (measurement based on eight ossicles randomly chosen from both aboral and oral sides, see also Table S4, Fig. S24, and Methods). This result is considerably higher than the dislocation density in natural and synthetic single-crystalline diamonds, which is typically less than 10^{-6} (19, 20). Intriguingly, beyond the ossicle example described here, lattice dislocations are frequently encountered in other highly periodic biological materials, ranging from the spiral-like comb geometries of stingless honey bees (21) to the damage-tolerant micro-laminate architecture of bivalve nacre (22, 23).

Previous studies have demonstrated that individual echinoderm ossicles largely diffract as singlecrystal calcite (9), despite the presence of intracrystalline organic occlusions, doping of the calcite with magnesium ions, and a mesocrystalline structure in which the calcite shows a nanoparticulate ultrastructure (24). Based on these observations, we further explored the potential relationship between the diamond-TPMS microlattice and the underlying atomic-level ordering of the constituent calcite. As expected, crystallographic mapping from electron backscatter diffraction (EBSD) data on ossicles from P. nodosus confirms its single-crystal-like nature at the atomic scale (Fig. 3A,B and Fig. S25). Moreover, to further investigate its multi-scale crystallographic relationship, we employed the epitaxial overgrowth strategy to induce the formation of synthetic calcite crystals on the ossicle surfaces (Methods) (25-27). As shown in Fig. 3C-E, the overgrown calcite crystals (shaded in light blue) still retain the rhombohedral symmetry of calcite, inheriting the crystallographic orientation of the underlying ossicles. When viewed along the [111] direction of the diamond-TPMS microlattice, the overgrown calcite crystals exhibit a three-fold symmetry, confirming that the *c*-axis of the calcite is oriented along the [111] direction of the diamond-TPMS microlattice (Fig. 3E). Moreover, the edges formed by two adjacent $\{10\overline{1}4\}$ planes of the calcite are aligned with the branches connected with the "higher nodes" in the (111) plane of the diamond microlattice (Fig. 3F,G). This observation demonstrates that the in-plane three-fold symmetry of calcite (space group, $R\bar{3}c$) is aligned with that of the diamond microlattice in the [111] direction. Lastly, the common edges formed by adjacent $\{10\overline{1}4\}$ planes of calcite crystals intersect with the *c*-axis at an angle of 116.3°, which is close to the measured inter-branch angle within the ossicles ($\alpha_m = 110.9^\circ \pm 10.2^\circ$) and the ideal tetrahedral angle in a diamond lattice (109.5°) (Fig. 3H, Fig. S26). Based on these observations, the dual-scale coordination observed in the diamond-TPMS structure is thus likely influenced by the crystal symmetry of the constituent calcite during microlattice formation, which may be further modulated by the mineral's interactions with macromolecules secreted by the bounding sclerocyte cells (25-27). While speculative at this point, this proposed mechanism is functionally analogous to the organic template-modulated,

directionally specific attachment of mineral particles and crystallization processes observed in other branched calcitic biomineralized structures, including sea urchin larval spicules and calcareous sponge spicules (25-28).

The diamond-TPMS microlattice also exhibits long-range variations of structural parameters at the ossicle level, such as branch length and thickness (Fig. S14-17 and Table S3). Fig. 4A represents an extreme example of this structural gradient where the branch thickness increases from 4.7 µm to 7.3 µm towards the ossicle surface over a distance of ca. 6 unit cells, despite a relatively uniform branch length. Such structural gradients give rise to mechanical property gradients, which were quantified by a micromechanical model based on the Timoshenko beam theory (Fig. S27,28, Table S5, Methods). From these studies, it was found that the normalized modulus $(E_{Ossicle}^{111,Iso}/E_{Calcite}^{Iso})$ and strength $(\sigma_{Ossicle}^{111,Iso}/\sigma_{Calcite}^{T,Iso})$ in the [111] direction of the diamond microlattice are strongly correlated with η (l_1/l_m) and λ (t_1/t_m) (Fig. 4B, Fig. S29-31, and Table S6). Here, $E_{Calcite}^{Iso}$ and $\sigma_{Calcite}^{T,Iso}$ represent the equivalent isotropic modulus and tensile strength of the solid calcite (Methods); l_1 and t_1 are the length and thickness of the branch in the [111] direction; l_m and t_m are the average length and thickness of the branches in non-[111] directions. It is conceivable that a larger η or a lower λ represent relatively shorter or thicker inclined branches (i.e., non-[111] branches) that possess higher bending stiffness, which results in a higher stiffness of the local diamond microlattice (Fig. S31). The local increase in mechanical performance in the upper portion of this ossicle (No. 7) may contribute to its resistance against external loads along the aboral to oral direction (Fig. 4B and Fig. S29).

While the theoretical analysis above permits the investigation of long-range mechanical heterogeneity within the ossicles, it ignores the important effects of the mechanical anisotropy of calcite and the crystallographic co-alignment between atomic and lattice scales. Calcite is a highly anisotropic material with a lower stiffness along the c-axis than along the a-axes (Table S7) (29). To accommodate these differences in crystal mechanical anisotropy, we developed a finite element (FE) model that incorporates the crystallographic co-alignment deduced above and the full stiffness matrix of calcite (Methods). This approach allows us to estimate the effective Young's moduli of the solid calcite along the diamond lattice directions of [111], [110], and [112] (denoted as $E_{calcite}^{111,Ani}$, $E_{calcite}^{110,Ani}$, and $E_{calcite}^{112,Ani}$, respectively) as 79.6, 126.8, and 130.8 GPa, respectively (Fig. 4C, Fig. S32, Table S6-7). This result again confirms the softness of calcite along the c-axis or the [111] lattice direction. However, the [111] direction is the stiffest for a diamond-TPMS lattice based on isotropic materials among other orientations, as evident from our modeling results and from previous studies (Fig. 4D, Fig. S33) (30). These results suggest that the alignment of the stiff [111] direction of the diamond-TPMS microlattice with the c-axis of calcite in the ossicles of P. *nodosus* represents an intriguing strategy to compensate for the material-level compliance. Indeed, when the anisotropic properties of calcite are employed in simulations, the increase in the normalized modulus of the diamond-TPMS structure is highest along the [111] direction (Fig. 4D,E and Fig. S33), which leads to a more uniform stress distribution compared to the case with isotropic material properties (Fig. 4F and Fig. S34).

Due to the inherent brittleness of ceramic materials, synthetic ceramic foams and the recently developed architected ceramic lattices often suffer from catastrophic failure when the applied load exceeds a critical value (1,3). As calcite is a brittle mineral, and biogenic calcite also fractures upon critical loading (31), we further investigated the ossicle's fracture behavior and energy dissipation mechanisms through a series of experimental approaches. Uniaxial compression tests on cube-shaped specimens cut from individual ossicles (edge length, ca. 2 mm) reveal the graceful failure behavior of ossicles as demonstrated by the large stress plateau after reaching the failure strength ($\sigma_p = 46.48 \pm 15.14$ MPa. n = 14) (Fig. 4G). This behavior leads to an energy absorption (W_v) of 14.25 ± 2.50 MJ/m³ and hence a remarkable specific energy absorption (W_v/ρ) of 9.76 ± 1.59 kJ/kg, outperforming many synthetic ceramic or even metallic foams (Fig. S35,36).

It is intriguing that the calcitic diamond-TPMS structure in these ossicles exhibits a large stress plateau upon compression, since this behavior is typically only observed in polymeric and metallic foams (1). We next utilized synchrotron in situ mechanical testing to track, visualize, and analyze the deformation processes in 3D. Upon loading, the ossicle samples developed slip-like damage bands at initial yielding (yellow arrows, Fig. 4H), and the sequential cross-sectional slices of the reconstructed data demonstrate that the co-parallel damage bands undergo local densification and increase in band width (white arrows, Fig. 4I and Fig. S37.38). In these representative data, the applied load is in the $[\overline{1}01]$ direction of the diamond microlattice, and the damage bands are in the $(11\overline{1})$ plane (Fig. 4I and Fig. S38). The preferred failure along the {111} planes is expected in the diamond-TPMS microlattice since the {111} planes contain the highest 2D cross-sectional porosity, and hence the lowest solid fraction, which leads to the highest local stress for crack initiation and the subsequent formation of damage bands along these planes (Fig. S39,40). However, unlike synthetic architected ceramic lattices in which damage bands fully propagate to the sample surface, the damage bands in ossicles are often deviated and constrained within the specimen (Fig. 4H.I). Furthermore, a correlative crystal lattice analysis of both undeformed and deformed volumes suggests that the damage bands do not propagate to the regions with a high density of dislocations (Fig. 4J). When encountering a lattice dislocation, the {111} damage band has to "jump" from one {111} to another {111} plane for further crack propagation (Fig. 4K). This mechanism is similar to the "pinning" behavior of slips by pre-existing dislocations in metals, which is responsible for their well-known strain hardening behavior (12).

The biogenic ossicle calcite, despite its single-crystalline nature, does not undergo cleavage fracture along the $\{10\overline{1}4\}$ planes as in its geological counterpart (24). Instead, it behaves as a glassy material, exhibiting a distinctive non-cleavage "conchoidal" fracture pattern (Fig. 4L) (24, 31). This behavior leads to continuous fragmentation of the calcitic lattices into micro- and nanoscopic pieces during the densification process within the damage bands (Fig. 4M), with continued loading leading to particle compaction, rotation, and friction, further contributing to enhanced energy absorption (31-33).

In the present study, we describe a natural ceramic architected microlattice structure in the calcitic skeletal system of the knobby starfish, *P. nodosus*, which exhibits exquisite crystallographic registration for both its atomic-scale calcite and its micro-scale diamond-TPMS lattice. This unique dual-scale microlattice offers multiple effective strategies to achieve high stiffness, strength, and damage tolerance, including crystallographic co-alignment, lattice geometric gradients, and suppression of cleavage fracture through microlattice dislocations. It should be noted, however, that some of the structural features described here, such as the crystallographic co-alignment or the microlattice-level defects, may not have necessarily evolved for increased mechanical performance, but may simply rather be a byproduct of the complex process of skeletal formation in echinoderms. Nevertheless, the engineering lessons learned here emphasize the importance of hierarchical structural and crystallographic design for single-crystalline materials to achieve improved mechanical performance. Lastly, future studies focused on investigating the in vivo formation mechanisms of these complex and highly periodic microstructures would be of great value to the materials science community, since these biological micro-lattices are produced through the synthesis of transient amorphous precursor phases under ambient conditions, instead of energy-intensive processes such as sintering and chemical vapor deposition, which are commonly employed for the production of engineering cellular solids.

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Supplementary Materials

Materials and Methods Supplementary Text Figs. S1 – S40 Tables S1 - S7 Captions for Movies S1 to S5 References (34 – 55)



Fig. 1| The diamond-triply periodic minimal surface (TPMS) microlattice in the ossicles of *P. nodosus*. A, Photo of live *P. nodosus*. B, A skeleton of *P. nodosus* with its superficial soft tissue removed (aboral view). C, μ -CT reconstruction of the skeleton showing the aboral and oral layers of ossicles along the normal direction (defined as the oral-aboral direction). The dashed line in (B) denotes the location of this cross-sectional cut. D,E, SEM images showing the ossicles' highly ordered lattice structure. The lattice directions of selected ossicles are labeled in (D). The yellow arrows in (E) highlight the terrace-like morphology formed by the $\{111\}$ lattice planes on the ossicle surface. F, SEM image of an ossicle's fracture surface. G,H, 3D rendering (G) and its corresponding skeletonized connectivity diagram (H) of a representative ossicle volume with the orthogonal edges along the [111], $[1\overline{1}0]$, and $[11\overline{2}]$ directions, respectively. The inset in (H) highlights the tetrahedral units of a diamond lattice. I, Corresponding 3D-FFT pattern with diffraction spots denoted by orange dots. Some spots are overlapping, representing multiple crystal planes, as designated by the blue, yellow, and red indices. J, A comparison of the crosssectional patterns between the ossicle (left) and a simulated diamond-TPMS structure (right) along the (111), (110), and (112) lattice planes. K, Interfacial shape distribution map (i.e., κ_2 v.s. κ_1) of an ossicle volume. The inset schematics illustrate the representative interface shapes in the different regions. S, solid; V, void. L, Contour map of the mean curvature H on the ossicle's diamond-TPMS structure. M, Projection image of the reconstructed volume in transparent mode and corresponding 3D-FFT pattern (inset) of an intact ossicle along the zone axis (ZA) of [111].



Fig. 2| Lattice dislocations in the ossicle's diamond-TPMS structure. A, A μ -CT reconstruction slice along the (111) plane, with dislocation-like defects highlighted. **B**, SEM image of a dislocation-like defect on the fracture surface of an ossicle. **C**, The reconstructed volume of an ossicle with representative 60° and screw dislocations highlighted. **D**,**E**, Magnified views of (**D**) 60° and (**E**) screw dislocations extracted from (**C**). **F**,**G**, 60° glide (**F**) and shuffle dislocations (**G**) in the ossicles: (1) 2D models; (2) μ -CT reconstructions of the dislocation core structures; and corresponding maps of (3) branch length, *l*; and (4) thickness, *t*. The 5-node ring in (**F**(2)) is shaded in green, while the 7-node ring in (**F**(2)) and the 8-node ring in (**G**(2)) are shaded in cyan. Burgers vector $b_{60^\circ} = 1/2[01\overline{1}]$. **H**, The shuffle screw dislocation with kinks: (1) 2D model; (2) μ -CT reconstruction of the observed dislocation core structure; (3) map of branch length, *l*; and (4) corresponding 3D connectivity diagram from an individual ossicle. The branches in the Burgers circuit are colored in red. Burgers vector $b_s = 1/2[011]$. N3, N4, and N5 nodes are colored in green, red, and blue for (**F**-**H**), respectively.



Fig. 3| The dual-scale single-crystalline diamond-TPMS lattice. A,B EBSD orientation map (A) and corresponding {0001} pole figure (B) of calcite for a polished ossicle surface. C,D, SEM images of the ossicle lattice with epitaxially overgrown calcite crystals (shaded in light blue) when viewed along the (111) plane of the diamond-TPMS lattice. The purple and cyan dots represent the lower and higher nodes on the (111) plane, characterized by their concave and convex surfaces, respectively. E, Co-alignment of a single overgrown calcite crystal and the branch orientations of the diamond lattice. F,G, 2D (F) and 3D (G) illustrations of the crystallographic co-alignment at atomic and lattice length scales. H, Comparison between the crystallographic angles of the { $10\overline{12}$ }, { $10\overline{14}$ }, and { $10\overline{18}$ } planes with respect to the *c*-axis of calcite, the ideal tetrahedral angle (109.5°), and the measured inter-branch angle distribution (α_m).



Fig. 4| Mechanical properties of the ossicle diamond-TPMS lattice. A, SEM image of a fractured ossicle showing the local increase in branch thickness towards the outer ossicle surface. **B**, Spatial variations of normalized stiffness ($E_{ossicle}^{111,Iso}/E_{calcite}^{Iso}$), strength ($\sigma_{ossicle}^{111,Iso}/\sigma_{calcite}^{T,Iso}$), and corresponding branch length ratio $(\eta = l_1/l_m)$ for a representative ossicle. **C**, Anisotropic moduli $(E_{Calcite}^{HKL,Ani})$ and equivalent isotropic modulus $(E_{Calcite}^{Iso})$ of the solid calcite. **D**,**E**, Moduli $(E_{Ossicle}^{HKL,Ani})$ (**D**) and corresponding normalized moduli $(E_{Calcite}^{HKL,Iso}/E_{Calcite}^{Iso})$ and $E_{Ossicle}^{HKL,Ani}$ (**D**) and corresponding normalized moduli $(E_{Ossicle}^{HKL,Iso}/E_{Calcite}^{Iso})$ (**E**) of the ossicle diamond-TPMS lattice calculated using the isotropic generation of calcite and $E_{Ossicle}^{HKL,Ani}/E_{Calcite}^{HKL,Ani}$ (**E**) of the ossicle diamond-TPMS lattice calculated using the isotropic generation of calcite. calculated using the isotropic and anisotropic properties of calcite. The values in (C-D) were calculated in three orthogonal directions of the diamond lattice ([111], $[1\overline{1}0]$, and $[11\overline{2}]$). F, von Mises stress contour plots (compression along the [111] direction) with isotropic and anisotropic material properties. G. Stress (σ) -strain (ε) curves of both ex-situ and in-situ compression tests. Inset: volumetric reconstruction of a representative in-situ test sample. H, A projection image of an ossicle sample under in-situ compression with damage bands denoted by yellow arrows ($\varepsilon = 0.11$). I, Sequential cross-sectional slices of the reconstructed data showing the deformation and fracture evolution within the sample. The vellow and red arrows denote the slip-like damage bands and the densified regions, respectively. J. 3D rendering of the sample with the damage bands colored in red and dislocations with Burgers vectors (yellow arrows). K, SEM image of a fracture surface deviated by a dislocation (denoted by the red arrow). The regions shaded in green and yellow represent two adjacent (111) fractured planes. L, Conchoidal fracture of the ossicle's biogenic calcite. M, SEM image of damage localization and densifications during indentation.