

## DNA-Silica Nanolattices as Mechanical Metamaterials

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### Summary

1 Mechanical metamaterials consist of periodic structures with enhanced material properties. The  
2 best additive manufacturing techniques have resolutions of 100s of nanometers which cannot  
3 fully realize material size effects. Further, they cannot easily combine disparate materials (e.g.,  
4 soft, biological polymers with hard ceramics). Here, DNA origami is used to construct  
5 octahedral-based isotropic and anisotropic nanolattices which are coated with silica. These DNA  
6 nanolattices have features two orders of magnitude smaller than additively manufactured lattices  
7 and obtain material properties comparable to the best nanolattices, due to material size effects.  
8 Atom probe tomography confirms the nanoscale distribution of DNA and silica in the octahedral  
9 lattice. Finite element modeling (FEM) reveals two dominate failure modes: buckling at lower  
10 coating thicknesses and tensile fracture at higher thicknesses. Molecular dynamics (MD)  
11 simulations reveal that the DNA suppresses global buckling modes in favor of surface buckling  
12 which delays failure and contributes to increased strength at large strains.

**Keywords:** Self-assembly; Nanomaterials; Mechanical; Strength; Energy absorption; DNA origami; Composites; Architected Materials; Molecular dynamics; Finite element; Atom probe tomography

## Introduction

Architected metamaterials with nano and microscale features have record strength, energy absorption<sup>1</sup>, and stiffness per weight, as well as properties such as high recoverability under large strains<sup>2,3</sup> and negative Poisson's ratio<sup>4</sup>. These extraordinary properties arise from the combination of an optimized 3D architecture, material properties and sample dimensions<sup>5,6</sup>. Strut-based lattices have properties such as high strength per weight,<sup>5,7</sup> but tend to buckle and undergo localized deformation. Plate-based structures<sup>8</sup>, triply periodic minimal surfaces<sup>9</sup>, and tensegrity lattices<sup>10</sup> overcome these shortcomings by minimizing stress concentrations and effectively transferring load. Enhancements in strength, stiffness and fracture toughness have been observed in nanoscale metals, ceramics and polymers<sup>11–16</sup>. Thus, reducing the feature size of architected metamaterials to the nanoscale can improve mechanical performance without adding weight<sup>5</sup>.

Scalable fabrication of nano-architected metamaterials remains challenging. Stereolithography and two-photon lithography are high-resolution additive manufacturing techniques for printing polymeric lattices with features down to  $\sim 20\text{ }\mu\text{m}$ , and  $\sim 300\text{ nm}$ , respectively<sup>17,18</sup>. Resins have been developed to directly print nanocomposite<sup>1</sup>, semiconductor<sup>19</sup> and piezoelectric<sup>20</sup> lattices. Glassy carbon and metallic lattices can be achieved through the pyrolysis<sup>21</sup> or chemical reduction of two-photon lithographed lattices<sup>22</sup>. Metal, ceramic and hybrid nanolattices have also been fabricated by printing a polymeric scaffold and then depositing an inorganic coating via sputtering<sup>2,3</sup>, electroless plating<sup>23</sup>, or atomic layer deposition<sup>24</sup>. This produces core-shell<sup>2,3</sup> or hollow lattices<sup>23,24</sup> with shell thicknesses down to tens of nanometers, and unit cells of a few microns in size. These lattices exhibit size effects in strength but are prone to buckling due to the high aspect ratio of the thin shell coatings.

These additive manufacturing techniques print structures point-by-point or layer by layer, with volumetric throughputs of  $0.02\text{ mm}^3/\text{h} - 6000\text{ mm}^3/\text{h}$ , such that a  $3\times 3\times 3$  lattice takes minutes to hours to print. There is a tradeoff between resolution and speed<sup>25</sup>, which means that the ultrasmall feature sizes in mechanical nano-lattices are slow to print. Colloidal self-assembly, dealloying of metals and spinodal decomposition are alternative methods for the rapid fabrication of centimeter scale samples, in which nano and microscale features are formed in parallel. These methods are limited to randomly porous or simple periodic structures<sup>26,27</sup>. For instance, self-assembled polymer spheres can be used as templates for silica, face-centered cubic inverse opal structures<sup>28</sup>, but alternative geometries with superior mechanical properties cannot be achieved.

Recent advances in DNA self-assembly allow for the fabrication of arbitrary 3D shapes with resolution of a few nanometers<sup>29–31</sup>. Programmable control of geometry is achieved through DNA nucleotide pairing. Complex 3D lattice structures with features on the order of  $6\text{ nm}$  can be fabricated, which is at least 2 orders of magnitude smaller than typical additive manufacturing techniques. Small DNA objects can be further assembled into larger structures. Towards this end, researchers have successfully assembled DNA origami<sup>30,32</sup> monomers into microscale 3D

49 superlattices with tetrahedron<sup>33</sup>, tensegrity triangle<sup>34</sup>, cube, and octahedron geometries<sup>35</sup>.  
50 Different superlattice geometries can be achieved through control of the building blocks<sup>34–36</sup>,  
51 connection valence strength<sup>37</sup>, and the surrounding environment<sup>38</sup>.

52 However, DNA is a soft material, which limits the load bearing ability of DNA lattices, despite  
53 their programmable, intricate nanoscale structures. Post-assembly modifications have been  
54 developed to enhance the rigidity of DNA structures. DNA structures have been used as  
55 templates to grow or incorporate inorganic materials, including silica<sup>29,36</sup>, silicon<sup>39</sup>, and metals<sup>40</sup>.  
56 Individual DNA tetrahedra that are coated with silica<sup>36</sup> or stained with uranyl acetate<sup>41</sup> have been  
57 shown to have much higher elastic modulus than the bare DNA tetrahedra. Silica coated, face  
58 centered cubic DNA-nanoparticle lattices have previously been compressed under high  
59 hydrostatic pressure in a diamond anvil cell<sup>42</sup>. Li et al. assembled DNA-metal nanoparticle  
60 nanolattices with high strength and elastic modulus, but high densities compared to other  
61 lightweight nanolattices<sup>43</sup>. Michelson et al. used DNA origami to fabricate octahedral DNA  
62 templates that were coated with silica, heat treated to solidify the SiO<sub>2</sub> and remove the DNA, and  
63 then tested in compression for a single silica shell thickness (relative density)<sup>44</sup>. Here, we use  
64 similar methods to fabricate DNA core-silica shell nanolattices with isotropic and anisotropic  
65 unit cells, and a variety of silica shell thicknesses, in order to investigate the influence of 3D  
66 architecture and nanoscale material interactions on mechanical behavior. In our work, we show  
67 that DNA-silica lattices have specific strength and energy absorption on par with the best 3D  
68 printed mechanical metamaterials and determine plasticity and failure mechanisms. Experiments  
69 and computational simulations show that the nanoscale silica coating thickness is found to have a  
70 dramatic effect on mechanical properties due to material size effects and leads to highly resilient  
71 lattices when combined with DNA. The DNA core is found to improve the mechanical  
72 performance at low silica coating thicknesses.

## Results and Discussion

73 DNA origami is used to create the lattice structure. Using a previously published design<sup>29</sup>, a  
74 symmetric octahedral DNA origami is used as a building block, and corresponding DNA staple  
75 sequences were generated with caDNAno<sup>45</sup>. DNA origami monomers were prepared by mixing  
76 the staples strands with a M13mp18 viral scaffold DNA strand in aqueous solution, which was  
77 then subjected to a thermal annealing process<sup>30</sup>. The octahedral monomer contains 12 structurally  
78 identical struts. Each strut is composed of a six-helix bundle with a dimension of 28.6 nm in  
79 length and 6 nm in diameter (Figure S1A). To form a 3D lattice, two octahedron monomers were  
80 prepared separately and connected vertex-to-vertex via hybridization between complementary  
81 DNA linkers (Figure S1B). Each octahedron vertex consists of four single-stranded linkers.  
82 Every linker contains three domains: a 42-nucleotide (nt) staple domain that binds stably to the  
83 scaffold DNA, a 22-nt non-binding polythymine (polyT) in the middle to provide sufficient  
84 flexibility, and an 8-nt sticky end (a sequence that is complementary to its counterpart on the  
85 other DNA octahedron). The length of polyT domains and sticky ends were carefully screened in  
86 a previous study for optimal lattice growth<sup>30</sup>.

87 After assembly of the 3D DNA lattices, the structures were reinforced by silicification with a  
88 modified Stöber reaction<sup>46</sup>. Briefly, the negative-charged surface of the DNA lattice adsorbs the  
89 positive-charged precursor molecule N-trimethoxysilylpropyl-N,N,N-trimethylammonium

chloride (TMAPS). Then, tetraethyl orthosilicate (TEOS) is added and reacts with the TMAPS to form a silica coating over the DNA lattice via hydrolysis and co-condensation (Figure S1C; more details can be found in Supplementary Information). This forms nanometer-thick coatings, which retain the shape of the DNA lattice, and allows direct characterization with TEM and SEM (Figures S2-S6). The final product of DNA-silica lattices are multiscale structures (Figure 1)<sup>29</sup>. Each unit cell (a DNA octahedron and its surrounding space) is approximately 50 nm×50 nm×50 nm in size. The lattice structures grow into uniform cubes consisting of thousands of unit cells that are on the order of 10 μm in size. This lattice differs subtly from the octahedral lattice<sup>47</sup> due to the short DNA linkers that connect the vertices of octahedral unit cells.

The thickness of the silica coating can be modulated by tuning reactant concentrations and reaction times. Samples were fabricated with coating thicknesses from ~1.65 nm to 5.1 nm (Figure 2). These thicknesses were estimated from high resolution SEM images (Figure S12). A near space filling coating was fabricated as well. These samples correspond to relative densities from 9% to 91% when only considering the silica. The DNA itself takes up an additional ~9% of the volume. These relative densities are higher than other nano-lattices with ~20-50 nm coatings<sup>23,48</sup>, because the DNA lattice unit cell is ~50 nm in size, while nano-lattices made through lithographic methods have unit cells of ~10 μm in size<sup>23,24</sup>. Approximately 1 out of 30 lattice struts is a vacancy defect that occurs randomly during the self-assembly process (details in Supplementary Information).

Atom probe tomography (APT) was performed on the space filling DNA-silica lattice to provide nanoscale elemental mapping. APT succeeded in identifying molecules containing C, N, and P, which originate from DNA. Additionally, a small amount of Mg was found adsorbed to the DNA. This is expected because Mg is used in the buffer solution used in self-assembly. Figure 2F shows the concentration of each ionic species as a function of the distance from C. This shows that the concentrations of the organic species (N, Mg, CN, PO) are elevated in the presence of C whereas inorganic species (Si, SiO, SiO<sub>2</sub>) are depleted. The detected C is assumed to be in the DNA backbone. A heatmap of the organic-to-inorganic ratio from a 6 nm-thick slice through the lattice shows that organic-rich volumes appear at the expected distances for the octahedral lattice struts (Figure 2G). This analysis suggests that the silica coating is in fact evenly and conformally coated across the DNA structure without damaging or distorting the underlining lattice structure. Further, it suggests that the lattice with the space filling coating is a solid or nearly solid structure because inorganic markers are found throughout the DNA depleted regions.

Anisotropic octahedral DNA lattices were also assembled (details in Supplementary Information). As shown in Figure 3A-C, the standard octahedral lattice has a unit cell with an aspect ratio of 1:1:1, while the elongated lattice has a unit cell that is a rectangular prism with an expected aspect ratio of ~1.4:1:1 (this ratio corresponds to the x:y:z coordinates, where y is the out of plane direction in figure 3A-C). The flattened lattice has a unit cell that is a rectangular prism with an expected aspect ratio of ~1.7:1.7:1. From the top view, the standard lattice unit cell and the flattened lattice unit cell appear isotropic, while the elongated lattice appears anisotropic. From the side view, both the elongated and flattened lattice appear anisotropic. The strut length

in the standard lattice is 28.6 nm. In the elongated lattice, the strut lengths are increased to 35.7 nm along two axes, and remains 28.6 nm along one axis. In the flat lattice, the strut length is increased to 35.7 nm along one axis, and remains 28.6 nm along two axes. The elongated lattice has a silica coating of 2.29 nm while the flat lattice has a silica coating of 1.33 nm. SEM imaging shows that the actual aspect ratios are 1.5:1.2:1 and 2.4:2.3:1 for the elongated and flattened lattice, respectively. These results demonstrate the range of DNA crystals that can be achieved by designing the corresponding building blocks.

In-situ SEM mechanical testing is used to compress the lattices with 1.75, 3.1, and 5.1 nm silica thickness with a diamond flat punch tip. Michelson et al. showed that DNA-templated silica lattices with larger sizes tend to have a higher concentration of defects, leading to worse mechanical performance<sup>44</sup>. For this reason, cubes of 2-4  $\mu\text{m}$  in side length were chosen for mechanically testing in our study. The mechanical properties of the DNA-silica samples are plotted against cube size in Supplementary Figure S16.

Compression of lattices with a 1.75 nm coating thickness resulted in two types of stress-strain curves. The first curve, which is shown in Figure 3D in blue, has an elastic modulus of 232 MPa up to ~60% strain, at which point the modulus increases due to densification. The stress-strain curve has a smooth shape without sudden displacement events. This stress-strain curve corresponds to the lattice in Figure 3E, which formed vertical cracks during compression. The second curve (green) has an elastic modulus of 812 MPa, and initial fracture stress of 200 MPa. The initial fracture strength is defined as the onset of the first zero or negative stiffness regime. This stress-strain curve corresponds to the formation of diagonal (~45°) cracks (Figure 3F), which can be observed on in-situ deformation videos (see supplementary videos, S1-S5).

The 3.1 nm and 5.1 nm lattices tend to form vertical cracks while the space filling lattice tends to form horizontal cracks (Figure S10). Smooth stress-strain curves were observed for the 3.1 nm lattice. The 5.1 nm and space filling lattices have stress-strain curves with sudden drops in stress at high strains. The 3.1 nm and 5.1 nm lattices have failure stresses of  $1.6 \pm 0.8$  GPa and  $1.0 \pm 0.2$  GPa, respectively, and elastic moduli of  $3.2 \pm 2.2$  GPa and  $4.4 \pm 1.0$  GPa, respectively. Lattices with 1.65 nm and 2.1 nm silica coating were compressed in a benchtop nanoindenter (see Supplementary Figure S15). These samples show similar mechanical performance to the 1.75 nm sample (Supplementary Table 1 lists the measured yield strength of each sample). Without the video capabilities of in-situ testing, an exact fracture point cannot be determined. Instead, a 1% offset yield<sup>44</sup> stress is used to characterize the strength of the structures.

Anisotropic lattices were compressed using a benchtop nanoindenter in the geometry shown in Figure 3B-C. The elongated and flat octahedral lattices display similar stress-strain behavior before yield, but distinct post-yield behavior. Elongated octahedral structures have an elastic modulus of  $810 \pm 400$  MPa and a 1% yield stress of  $95 \pm 67$  MPa while the flattened octahedral structures have an elastic modulus of  $630 \pm 400$  MPa and 1% yield stress of  $126 \pm 52$  MPa. The elongated octahedral structure exhibits dips in the stress-strain plot consistent with layer-by-layer failure or cracking, which is corroborated by post-compression imaging (Figure 3H). The flattened octahedral structure instead has an elastic region followed by a subtle yield point and no evidence of fracture, which is consistent with post compression imaging (Figure 3I). This may be due to the pancake-like aspect ratio of the flattened samples.

The 1% offset yield stress of the DNA-silica lattices is compared to other nano-lattices in Figure 4A. The DNA-silica lattices are also highly deformable, reaching strains of 30% or more without significant fracture events. This leads to extremely high energy absorption before fracture of ~50 MJ/m<sup>3</sup>, 340 MJ/m<sup>3</sup>, 180 MJ/m<sup>3</sup>, and 270 MJ/m<sup>3</sup> for the 1.75 nm, 3.1 nm, 5.1 nm, and space filling standard lattices, respectively (Figure 4B). Energy absorption is an important property of impact resistant materials. The specific strength and energy absorption of the DNA-silica lattices are similar to glassy carbon plate-lattices<sup>8</sup> and spinodal structures<sup>49</sup>, and are well above the performance of conventional engineering materials and other strut-based nanolattices. The elastic modulus of the DNA-silica lattices is in the range of 0.5-5 GPa, which is comparable to other ceramic nano-lattices (Figure 4C). The observed compressive strength values are similar to that of bulk silica (1-1.6 GPa). The elastic moduli of the tested sample, however, are significantly lower than that of bulk silica (~72 GPa). This results in a significantly higher strain at failure and energy absorption when compared to bulk silica. The energy absorption of bulk silica is estimated to be ~18 MJ/m<sup>3</sup> from a linear elastic strain energy density function.

Surprisingly, the elastic modulus and strength of the lattices do not monotonically increase with increasing density. Instead, the elastic modulus and strength of the 3.1 nm and 5.1 nm lattices are higher than lattices with lower or higher coating thickness. To understand this, the 3D architecture of the isotropic DNA-silica lattice is considered. Traditionally, a 3D lattice can be considered stretching dominated if the relative density is below ~10% and the number of nodes and struts in a unit cell satisfies the following<sup>7</sup>:

$$s - 3n + 6 \geq 0 \quad (1)$$

where  $s$  is the number of struts and  $n$  is the number of nodes. The DNA-silica lattice unit cell has 15 struts and 6 nodes so it is stretching dominated, which means that the dominant deformation of each strut will be either simple tension or compression along its long axis. A simple pin-joint analysis can be used to predict elastic modulus and strength. We assume a homogeneous and isotropic material, cylindrical struts, and frictionless pins at the nodes. This analysis (see Supplementary equations S.1-S.17) results in the following first order scaling laws:

$$E_{lattice} = 0.12 E_{bulk} \bar{\rho} \quad (2)$$

$$\sigma_{lattice} = 0.26 \sigma_{bulk} \bar{\rho} \quad (3)$$

where  $E_{lattice}$  and  $\sigma_{lattice}$  are the elastic modulus and yield strength of the lattice,  $E_{bulk}$  and  $\sigma_{bulk}$  are the elastic modulus and yield strength of the bulk material, and  $\bar{\rho}$  is the relative density of the lattice. The linear scaling of elastic modulus and strength with relative density is the same as other stretching dominated lattices<sup>5</sup>.

Equation 2 and 3 are applicable to relative densities up to ~10%<sup>5,54</sup>. For relative densities up to ~30%, structures can still be considered as open cell foams, but will have scaling coefficients larger than one due to the influence of bending. For higher relative density lattices, the 1<sup>st</sup> order approximations no longer hold; instead, the nonlinear Hashin-Shtrikman bound and Suquet bound can be used to predict the maximum elastic modulus and failure strength for a porous

material<sup>55</sup>. The Hashin-Shtrikman upper bound on modulus ( $E_{HS}$ ) and Suquet bound for strength ( $\sigma_{Su}$ ) can be calculated as the following<sup>5,8</sup>:

$$E_{HS} = \frac{2\bar{\rho}(5\nu-7)}{13\bar{\rho}+12\nu-2\bar{\rho}\nu-15\bar{\rho}\nu^2+15\nu^2-27} E_{bulk} \quad (4)$$

$$\sigma_{Su} = \frac{2\bar{\rho}}{\sqrt{4+\frac{11}{3}(1-\bar{\rho})}} \sigma_{bulk} \quad (5)$$

where  $\nu$  is the Poisson's ratio of the material. For the DNA-silica lattices, we observe strengths above the Suquet prediction (Figure 4) for bulk material. Typical lattice materials do not approach either bound for strength or elastic modulus, and if they do, they are meticulously designed structures<sup>8,56</sup>. However, the experimental strength values for some of the 1.75 nm and 3.1 nm silica coatings samples lay above the Suquet bound. This indicates that size effects must occur within the nanoscale silica coating to enhance the material properties compared to the bulk material.

At ultrasmall sizes, mechanical strength can be significantly improved compared to their macroscale counterparts<sup>5</sup>. For brittle materials, the effect of the nanoscale sample size on strength can be obtained using Griffith's law<sup>5</sup>. Griffith's law states the relationship between fracture stress ( $\sigma_f$ ) and critical crack size ( $a_c$ ) to be:

$$\sigma_f = Y \frac{K_{IC}}{\sqrt{\pi a_c}} \quad (6)$$

where  $Y$  is a geometric parameter and  $K_{IC}$  is the fracture toughness.  $a_c$  is taken to be equal to half the thickness of the silica coating. Due to the inverse relationship between  $\sigma_f$  and  $a_c$ , the effect becomes more pronounced at the nanometer scale. The theoretical strength for silica is ~22 GPa when approximated as  $\frac{E}{\pi}$ <sup>57</sup>. Based on equation 4, this strength is reached for a silica coating thickness of ~1 nm (the thinnest silica coating is ~1.65 nm). The gradient theoretical bound shown in Figure 4A uses the bulk properties of silica for the lower bound and replaces  $\sigma_{bulk}$  in equation 5 with the theoretical strength of silica for the upper bound. We ignore the properties of DNA in this analysis because silica has 2-3 orders of magnitude higher modulus and strength than DNA. For instance, DNA has an elastic modulus of ~300 MPa – 1 GPa<sup>58,59</sup>, which is considerably lower than that of bulk silica (~72 GPa). Yet, the addition of DNA may impact the failure mode and therefore the failure stress. The DNA should not affect the elastic regime, but may affect the mechanical behavior of the lattice at large strains<sup>60</sup>.

Pin-joint analysis indicates that uniaxial compression of the lattice leads to compression in struts that are aligned with the loading direction (vertical struts), and tension in struts that are perpendicular to the loading direction (horizontal struts) (see equations S.2 and S.4), which is corroborated by finite element modeling (FEM) (see Supplemental Figures S24-26). Buckling is an expected failure mode of slender rods or hollow struts in compression. The shell buckling criterion for a hollow strut with a thin shell is:

$$\sigma_{buckle} = \frac{E_{bulk}}{\sqrt{3(1-\nu^2)}} \left( \frac{t}{R} \right) \quad (7)$$

where  $t$  and  $R$  are the thickness of the shell and radius of the strut, respectively. This suggests a critical buckling load of  $\sim 19$  GPa for a hollow strut with a silica coating thickness of 1.65 nm. If it is assumed that tensile (horizontal) struts approach the theoretical strength for silica, then compressive (vertical) struts should buckle before the tensile struts fail. For larger coatings, equation 7 predicts values significantly higher than the theoretical strength of silica, which indicates that buckling is unlikely in these samples.

FEM simulations are implemented to understand the deformation and failure modes of the lattice structure. Simulations are performed on a hollow silica shell lattice consisting of  $3 \times 3 \times 3$  unit cells with different silica shell thickness. Simulations are also performed on a DNA lattice and a DNA-silica lattice to small strains (see Supplemental S28). Once the hollow silica shell thickness is large enough to prevent buckling, the FEM stress-strain response agrees with experiment. This is the case for the stress-strain curves shown in Figure 5A. The thinner coating ( $\sim 1.4$  nm) shown in Figure 5B fails via a collapsing of the structure at the vertically aligned joints while the thicker coating ( $\sim 4.4$  nm) shown in Figure 5C fails via tensile fracture at the horizontally aligned joints. These results indicate that the high stiffness and strength observed in the experimental thin silica shells is likely due to the presence of the DNA core. This is further explored in molecular dynamics simulations.

Molecular dynamics (MD) simulations are used to investigate the role of the DNA on mechanical behavior in the post-buckling regime. Individual struts, rather than an entire unit cell, are studied. The compressive stress-strain curves from MD simulations are shown in Figure 5D. Two samples are simulated: a hollow silica strut, and a DNA core-silica shell strut. The shell thickness is 1 nm for both samples, and the strut diameter is 6 nm, which corresponds to the experimental lattices. To explore the effect of the DNA core, each strut was subjected to a set of boundary conditions on the inner surface. The first set of boundary conditions simulated the presence of a stiff DNA core by specifying a reflective wall at the inner diameter. This prevents inward radial deformation of the silica wall and off-axis bending of the silica wall. The second set of boundary conditions removed these restrictions and allowed for full displacement of the silica wall, to simulate a hollow silica strut. For both samples, stress-strain data indicate an initial linear elastic loading segment with an average slope of 76 GPa. Stress begins to diverge from this linear segment at  $\sim 4\%$  strain. For the hollow silica strut, stress reaches a maximum of 3.2 GPa after which the stress decreases. For the DNA core-silica shell strut, stress continues to increase for the remainder of loading, after diverging from the initial linear segment.

The final atomic configurations are shown in Figure 5E and 5F for the two samples. Without the internal boundary condition, the hollow silica strut undergoes local buckling and forms an off-axis kink. With a rigid boundary condition, the DNA-silica strut undergoes local buckling, but wrinkles along the loading axis. The presence of the DNA interior modifies the buckling behavior of the compressive struts, resulting in a wrinkling type buckling mode as opposed to a kinking mode and prevents the appearance of a negative stiffness regime in the stress-strain plot.



The pre-buckling behavior is largely unaffected by the presence of DNA. The DNA increases the sustained stress at high strains and suppresses the negative stiffness regimes in the compressive struts. Globally, this results in a smoother stress-strain curve without sudden, progressive failure events like those observed in other ceramic nano-lattices<sup>49</sup>. It is therefore likely that the peaks observed in the experimental stress-strain plots correspond to local tensile failures leading to global crack formation. Figure S31B shows the compressive stress-strain data for struts with 2 nm silica shell thickness, which qualitatively agree with the results on 1 nm shell thickness shown in Figure 5.

MD simulations of hollow silica and DNA-silica struts in tension show linear elastic behavior, followed by sudden failure at high stresses ( $>20$  GPa) (see Figure S31D-E). The two separate regimes observed in the 1.75 nm samples may be explained by two separate buckling modes causing a difference in the modulus and fracture behavior at higher strains or by earlier local tensile failures due to preexisting flaws in some samples. These MD results complement the FEM results and show that the silica shell only is not sufficient to model the behavior of the thinner shell thicknesses in the post buckling regime. Indeed, the change in buckling mode due to the presence of the DNA core leading to sustained load bearing likely accounts for the difference between the experimental and FEM simulated structures with low shell thicknesses.

## Conclusion

This work demonstrates that DNA self-assembly can produce high performance mechanical metamaterials. We find that octahedral DNA lattices that are coated with silica have specific strength and energy absorption as high as  $2.5 \text{ GPa cm}^3/\text{g}$  and  $540 \text{ J/g}$ , respectively, which is on par with the best lithographed nanolattices. The DNA-silica lattices are made of ultrasmall—and therefore ultra-strong—materials. The  $\sim 50$  nm sized unit cell is 2-3 orders of magnitude lower than what is achievable using lithography. This, combined with the nanoscale silica coating thickness results in strong material size effects that lead to strength above the Suquet upper bound for bulk silica. The DNA interior likely suppresses global buckling modes in favor of surface modes which suggests that the global failure events observed experimentally correspond to an accumulation of tensile failures.

3D architectures such as the octet<sup>7</sup>, tensegrity<sup>10</sup>, plate lattices<sup>56</sup>, and triply periodic minimal surfaces<sup>9</sup> have better mechanical performance compared to the simple octahedron unit cell in this study. Despite this, the DNA-silica lattices outperform nanolattices with these optimized architectures. This indicates that further improvements can be achieved by expanding the geometries of DNA lattices in the future. Additional DNA staple strands can be added to strengthen stress concentrations, such as the nodes between lattice struts. Different materials such as metals could be deposited on the DNA lattices<sup>61</sup>, to create highly recoverable structures that still benefit from nanoscale size effects<sup>5</sup>. In this way, additional properties such as high electrical conductivity, thermal conductivity, and optical reflectivity could also be introduced. The use of DNA self-assembly demonstrates a pathway for the scalability challenges of architected metamaterials, in which bottom-up self-assembly methods are used for massively parallel nanofabrication<sup>62</sup>.

## Resource Availability

*Lead Contact:* Further information and requests for resources should be directed to and will be fulfilled by the lead contact, Wendy Gu ([xwgu@stanford.edu](mailto:xwgu@stanford.edu))

*Materials Availability:* This study did not generate new unique reagents. DNA-silica lattices presented can be reproduced following the procedure described in the experimental procedures below.

*Data and Code Availability:* Data needed to reproduce the results can be found in the Dryad repository: <https://doi.org/10.5061/dryad.g4f4qrfxz>

## Experimental Procedures

*Design and synthesis of octahedron unit cell:* Octahedron framework is designed by caDNAno software (<http://cadnano.org/>) and synthesized by mixing P7249 scaffold strand (extracted from M13 bacteriophage) and unpurified DNA staple strands (see Supplementary tables) in 1×TAE buffer (40 mM Tris, 1 mM EDTA, pH 8.3) containing 12.5 mM MgCl<sub>2</sub> and thermal annealing ramps from 90°C to 20°C for 24 h in a PCR thermos cycler (S1000 Thermal Cycler, Bio-Rad). Each edge of the frame is composed of a six-helix bundle with 28.6 nm (84 base pairs) in length and 6 nm in diameter. Two single strands (sticky ends) stretch out from two ends of one 6HB respectively, and four sticky ends extend from one vertex.

*Design and assembly of DNA lattice:* The DNA lattice was assembled by combining two kinds of octahedron frames with complementary sticky ends in an equal molar concentration and carrying out a slow annealing ramp from 50°C to 20°C in 150 h. The base pairing interaction from vertex-to-vertex hybridization and slow annealing lead to formation of crystal habits with minimal surface energy.

*SiO<sub>2</sub> growth protocols:* The silicification is based on the modified Stöber reaction by adding two silica precursors TMAPS and TEOS in sequence. Briefly, the quaternary ammonium group in TMAPS with positive charge binds with the anionic DNA phosphate backbone by electrostatic attraction. The siloxane group on TMAPS and TEOS go through hydrolysis and co-condensation, forming connective siloxane bridges (Si-O-Si). Considering the competitive effect between TMAPS and magnesium ions, we wash the sample with 1×TAE buffer containing 7 mM MgCl<sub>2</sub> several times to lower the magnesium concentration. Then, TMAPS is added into the DNA lattice sample at room temperature and shaken on a vortex mixer at a rate of 400 rpm for 20 min. The absorption of TMAPS on the DNA double-strand backbone determines the condensation sites and distribution of the silica coating. Later, TEOS is added into the solution and shaken for another 30 min at same rate. After completing the addition of the two silica precursors, the mixture is kept static for 12 h at room temperature. A cloudy precipitate will appear at the bottom of tube, which indicates the completion of the DNA lattice silicification and formation of silica clusters in solution. After washing with deionized water several times, some isolated silica clusters are removed.

*SiO<sub>2</sub> thickness control:* The silica thickness is related to several parameters including the concentration of the DNA lattice (all the nucleotides) and the two silanes (TMAPS and TEOS).

We mix the DNA lattice sample and the two silanes (nucleotide: TMAPS: TEOS) in different molar ratio. According to the nucleotide quantities of DNA lattice sample in the solution, we add different amounts of silanes. Here, we add TMAPS and TEOS according to the reported ratio of 1:10:20, which forms  $\sim 1.75$  nm silica coating. As mentioned earlier, TMAPS absorption on the crystal produces the nucleation sites for silicification, and is vitally important for the uniform-distribution of silica on the microstructure. We modified this silica precursors ratio by changing the TEOS amount to modulate the thickness of silica on the DNA lattice.

*Liftout and sharpening:* For atom probe tomography (APT), it is necessary to shape a sample into a needle with a tip diameter  $< \sim 100$  nm. This was achieved using a dual-beam plasma focused ion beam scanning electron microscope (FIB-SEM, Hydra, Thermo Fisher) equipped with a nanomanipulator (EZLift, Thermo Fisher). A  $0.5\ \mu\text{m}$ -thick protective capping of Pt was deposited on the (100) surface of a space filling DNA nanolattice using a 30 kV 0.1 nA Xe ion beam. While keeping the (100) surface normal to the ion beam, rectangular patterns were then used with the 30 kV 0.1 nA ion beam to remove the excess material on either side of the Pt cap. Cleaning cross sections were then used with 30 kV 0.1 nA ion beam to thin the lamella and polish the sides. The stage was tilted back to  $0^\circ$  (i.e. (100) surface normal to electron beam), then a small deposition of Pt was deposited with a 30 kV 30 pA ion beam to attach the lamella to the nanomanipulator. A cleaning cross section pattern was then used to make an undercut with 30 kV 30 pA ion beam, separating the lamella from the substrate and causing the bottom face of the lamella to be at a  $52^\circ$  angle (the same angle between the ion beam and electron beam). Silicon microposts (m22 array, CAMECA) were similarly milled to allow for a flush contact between the micropost and sample lamella, then Pt depositions used to secure the sample (Figure S8).

Annular ion beam milling patterns at 30 kV 0.1—0.03 nA with progressively decreasing outer and inner diameters ( $4\text{--}3.5\ \mu\text{m}$  and  $1.2\text{--}0.3\ \mu\text{m}$ , respectively) were used to shape the liftout into a needle, then finally sharpened to a final tip diameter  $< 100$  nm with a 5 kV 10 pA ion beam “shower” with no pattern (Figure S8).

*Atom Probe Tomography:* APT experiments were conducted on a local electrode atom probe 6000 (LEAP 6000 XR, CAMECA). The new simultaneous voltage pulse mode—a capability of the LEAP 6000 not seen in previous generations of atom probe wherein the  $\sim 1$  ns pulse of the  $\lambda = 257.5$  nm wavelength laser used to stimulate ion evaporation is timed to overlap with a brief increase in the applied voltage—was used for this experiment. This new approach is purported to decrease the noise floor in the collected mass-to-charge spectra by decreasing the standing electric field between pulses, reducing the chance for unintended/mistimed evaporation events. A 5% voltage pulse fraction was used in conjunction with a 3.5-10 pJ laser pulse energy at a pulse frequency of 100 kHz. A 1% target detection rate was maintained using the “rapid” voltage control algorithm. The specimen base temperature was maintained at 40 K.

Data processing, three-dimensional reconstructions, and APT analysis were conducted in the integrated visualization and analysis software module of APSuite (version 6.3, CAMECA). The mass-to-charge spectrum contained many ( $> 100$ ) peaks. For this study, only those peaks with signal exceeding double the surrounding background when using a bin size of 0.005 Da were ranged and identified (Figure S9). To help ensure the accuracy of the mass-to-charge spectra,

only select prominent peaks with a clear identity were used for the peak calibration stage of processing and are listed in the Supplemental Information. The tip profile was measured from a high-resolution SEM micrograph (Figure S8) of the final sample shape to guide the radius evolution during reconstruction, in addition to setting Si as the primary element with an evaporation field of 33 V/nm.

In the reconstruction analysis, custom regions of interest can be imported as .STL files. The DNA origami octahedra unit cell was reproduced in AutoDesk Inventor 2023 at 106 scale (i.e. 1 mm = 1 nm) and exported as a .STL file. Upon importing as ROI objects into APSuite, the scale is automatically adjusted such that the struts are 28.6 nm long with a diameter of 6 nm. Maneuvering an assembly of these ROI octahedra proved useful in validating the distribution and periodicity of the organic phase in the reconstruction (Supplementary Figure S9).

*Mechanical characterization:* Silica coated DNA lattices were drop cast on silicon wafer pieces. Mechanical characterization was performed using the NanoFlip nanoindenter (KLA Corporation) in a FEI Helios Nanolab 600i DualBeam SEM or with a iMicro nanoindenter (KLA Corporation) benchtop system. Lattices were compressed to ~50-70% strain at a displacement rate of 10 nm/s using a 20  $\mu$ m diamond flat punch tip. Each silica thickness is tested for at least 3 samples. SEM images were taken using a FEI Helios Nanolab 600i DualBeam or Thermo Fisher Scientific Apreo S LoVac SEM.

*FEM simulations:* Models are created in Solidworks 2022 and meshed in Hypermesh 2022 with the described geometry for the DNA-silica lattice. The octahedral unit cell is 47 nm in size, with DNA struts of 6 nm in diameter, and 28.6 nm in length. Numerical simulations are performed in Abaqus 2023 with dynamic explicit for quasi-static loading analysis (the rate of kinetic energy and internal energy is within 5%) and linear perturbation for buckling mode analysis. Figure 5 uses mesh assignments as provided in Supplementary Figure S30. For structures which consider the DNA core (Supplementary Figures S25-29), the silica coatings and DNA cores are meshed by shell elements and solid elements, respectively. The perfect contact interfaces are adopted between the DNA core and the silica shell. Hence, the coatings and cores are tied to ensure a coincident deformation for the DNA-silica lattice. For loading conditions, one side of the lattice is placed under a fixed boundary condition and the opposite side is given a prescribed displacement.

*MD simulations:* We employ molecular dynamics (MD) simulations to understand the deformation response of the amorphous DNA-silica nanolattices. The simulated sample matches the geometry of a single strut of the experimental lattice. The length of the strut is 29.5 nm, and the inner diameter of the strut is 6 nm. We consider samples with silica thicknesses of 1 nm and 2 nm, which leads to an outer diameter of 8 nm and 10 nm. To construct an amorphous microstructure, we follow a melt and quench methodology. We specify a mold using reflective walls following the geometries described above and populate the volume with Si and O atoms in a stoichiometry of 1:2 and with a density that approximates the experimentally known density of 2.2 g/cm<sup>3</sup> for amorphous silica<sup>63</sup>. These atoms were brought to 6000 K to the liquid phase and held there for 200 ps. The samples were then quenched to 300 K at a rate of 2 K/ps. The resulting geometry is shown in Figure S17. Before applying deformation, all samples were thermalized at

300 K for 20 ps. Because various struts throughout the lattice experience stress states of tension and compression, all sample geometries were subjected to tensile (Figure S31D-F) and compressive loading by changing simulation box size at a strain rate of  $10^9 \text{ s}^{-1}$  up to a final engineering strain of 20%. Interactions among Si and O pairs were modelled using a Tersoff potential developed by Munetoh, et al.<sup>64</sup> All simulations were performed using the LAMMPS molecular dynamics software<sup>65</sup> and all steps of the simulation process were time integrated using the NVT ensemble with an integration timestep of 1 fs. OVITO<sup>66</sup> was used for common neighbor analysis (CNA), calculation of local atomic strain, and visualization of atomic configurations. Stress and strain are calculated with reference to the initial sample geometry and only considering the solid silica wall material.

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### Author Contributions

JK, MMW, DD, and ACL performed mechanical testing. SW designed and provided samples. JK and SW performed imaging. JK analyzed and curated data. ZA performed MD simulations. WH and BSG performed FEM simulations. JK, SW, ZA, and XWG wrote the manuscript. YK and XWG provided conceptualization and supervision. WH, YWZ, YK, and XWG provided expertise. JG and AD conducted the APT sample preparation and analysis and contributed to the manuscript drafting.

### Declaration of Interests

The authors declare no competing financial interest.

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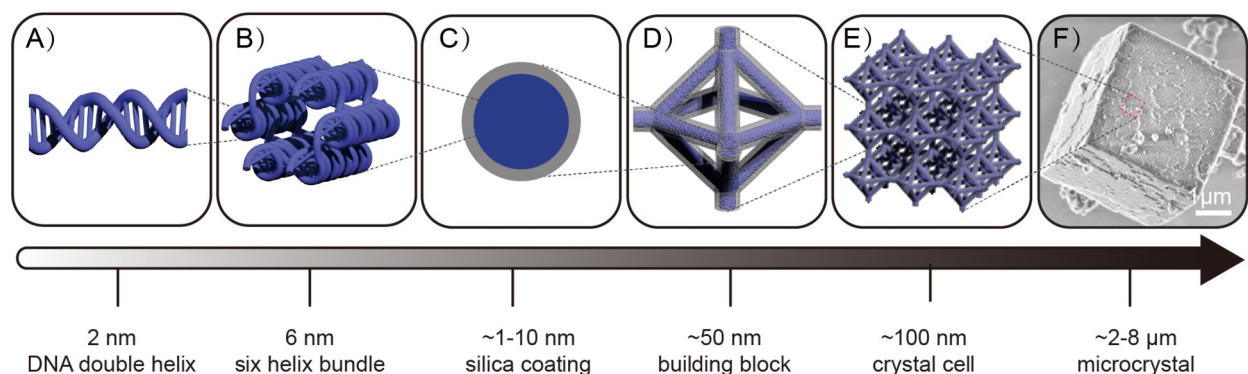
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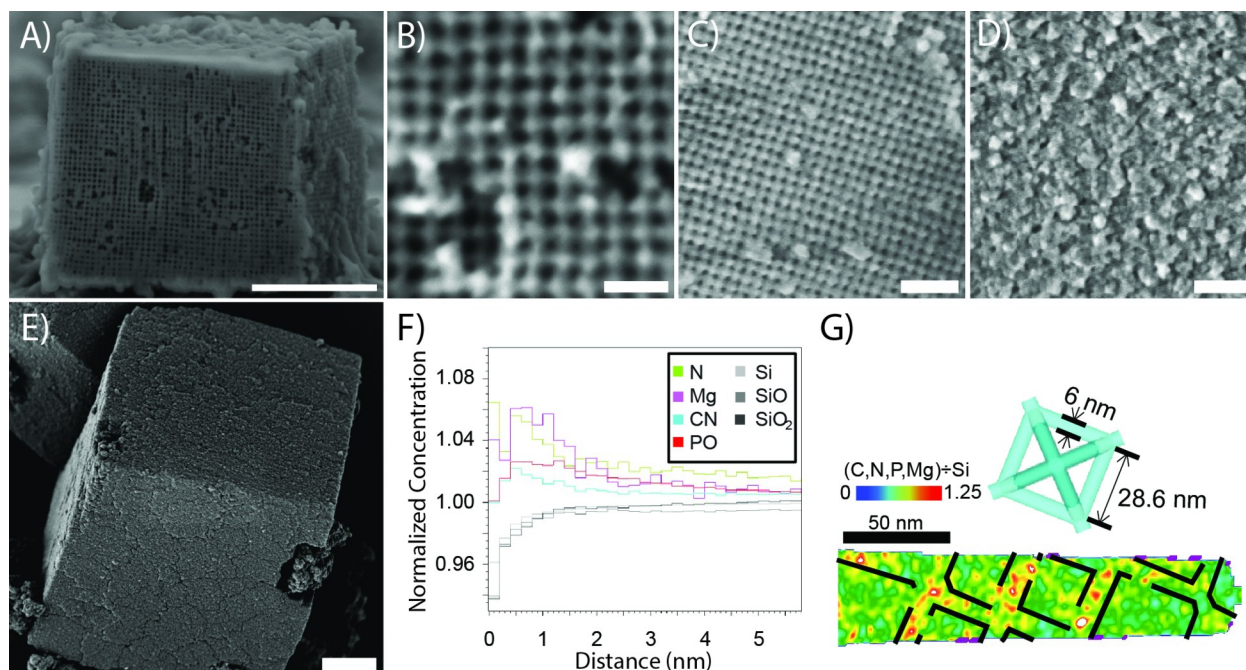
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## Figure Titles

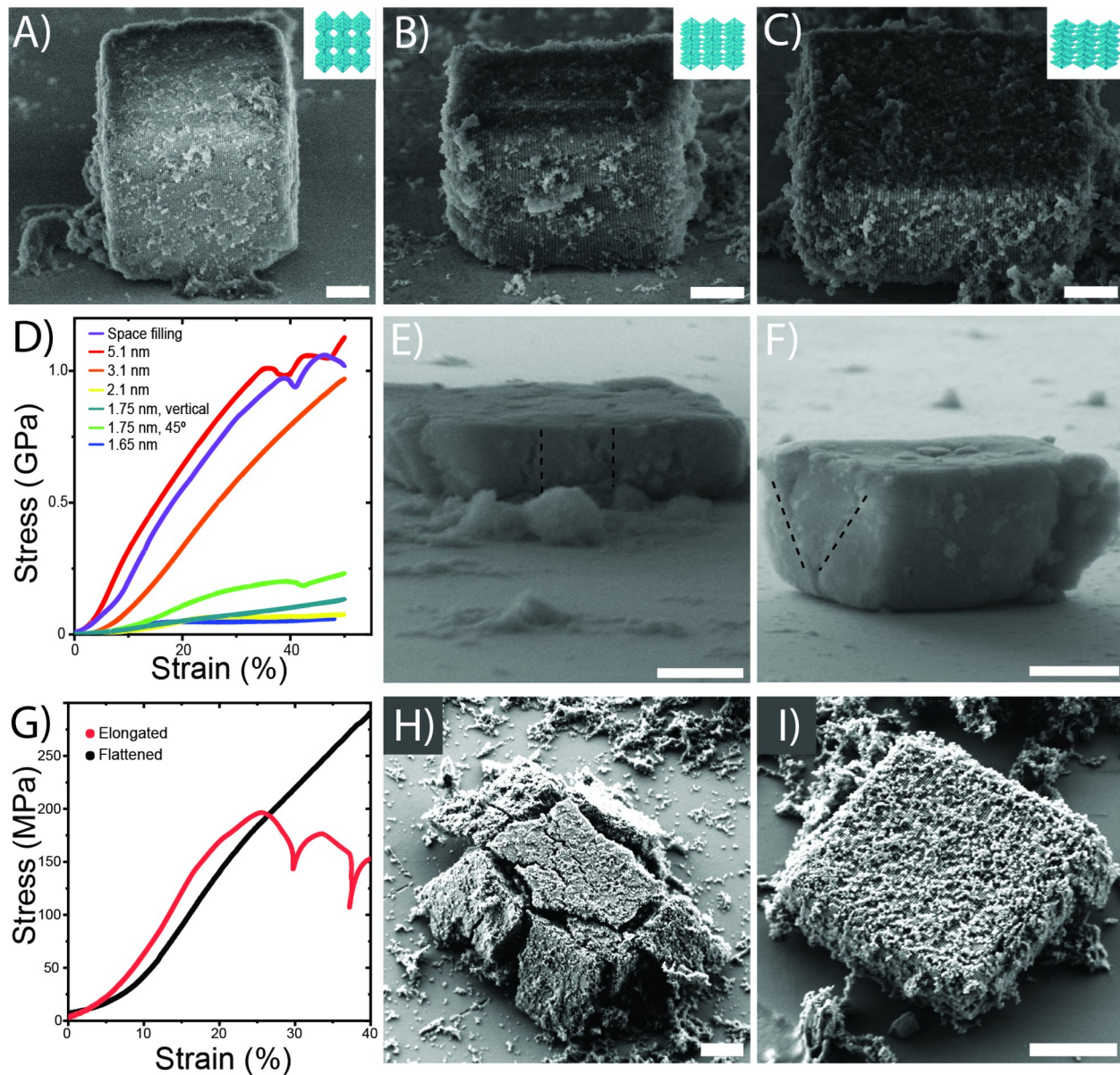


**Figure 1. Multiscale DNA-silica octahedral lattice.** From left to right: A.) DNA double helix strands are assembled into B.) six helix bundles. C.) A silica coating is grown onto the DNA bundle to form a core-shell structure. D.) The octahedron-based unit cell. E.) Cubic lattice structure. F.) SEM image of a DNA-silica lattice.

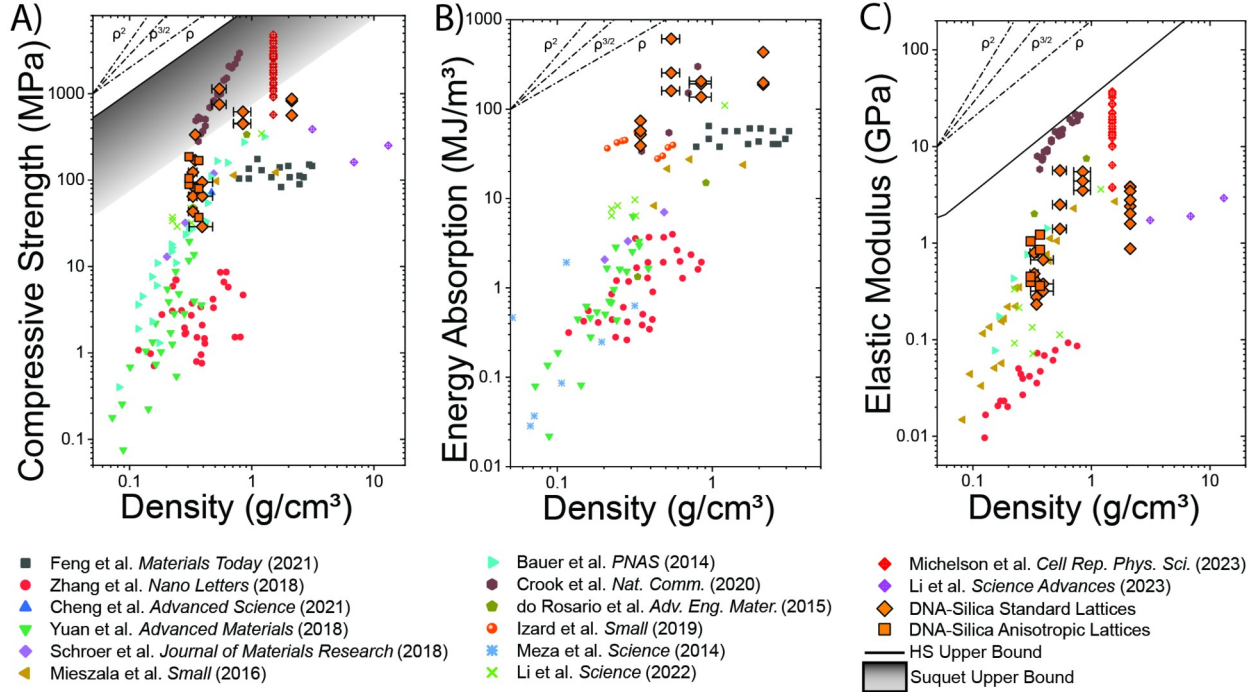


**Figure 2. Structural characterization of DNA- silica lattice structures.** A.) SEM image of a FIB cross sectioned lattice with 2.1 nm silica coating. B-D.) High magnification SEM images of B.) 2.1 nm, C.) 3.1 nm, and D.) space filling silica coating. E.) SEM image of lattice with space filling silica coating. F-G.) Atom probe tomography of space filling lattice. F.) Normalized radial concentration of select organic (N, Mg, CN, PO) and inorganic (Si, SiO, SiO<sub>2</sub>) ions with respect to distance from monoatomic C ions. G.) Heatmap from a 6 nm-thick slice of the reconstructed lattice showing the ratio of organic ionic species (containing C, N, P, and/or Mg) to inorganic (species containing Si). Dashed lines indicate regions where the organic to inorganic ratio is >1. These regions resemble a cross section of the octahedral nanolattice. Scale bars: A, E.) 1  $\mu$ m B.) 50 nm, C-D.) 200 nm.

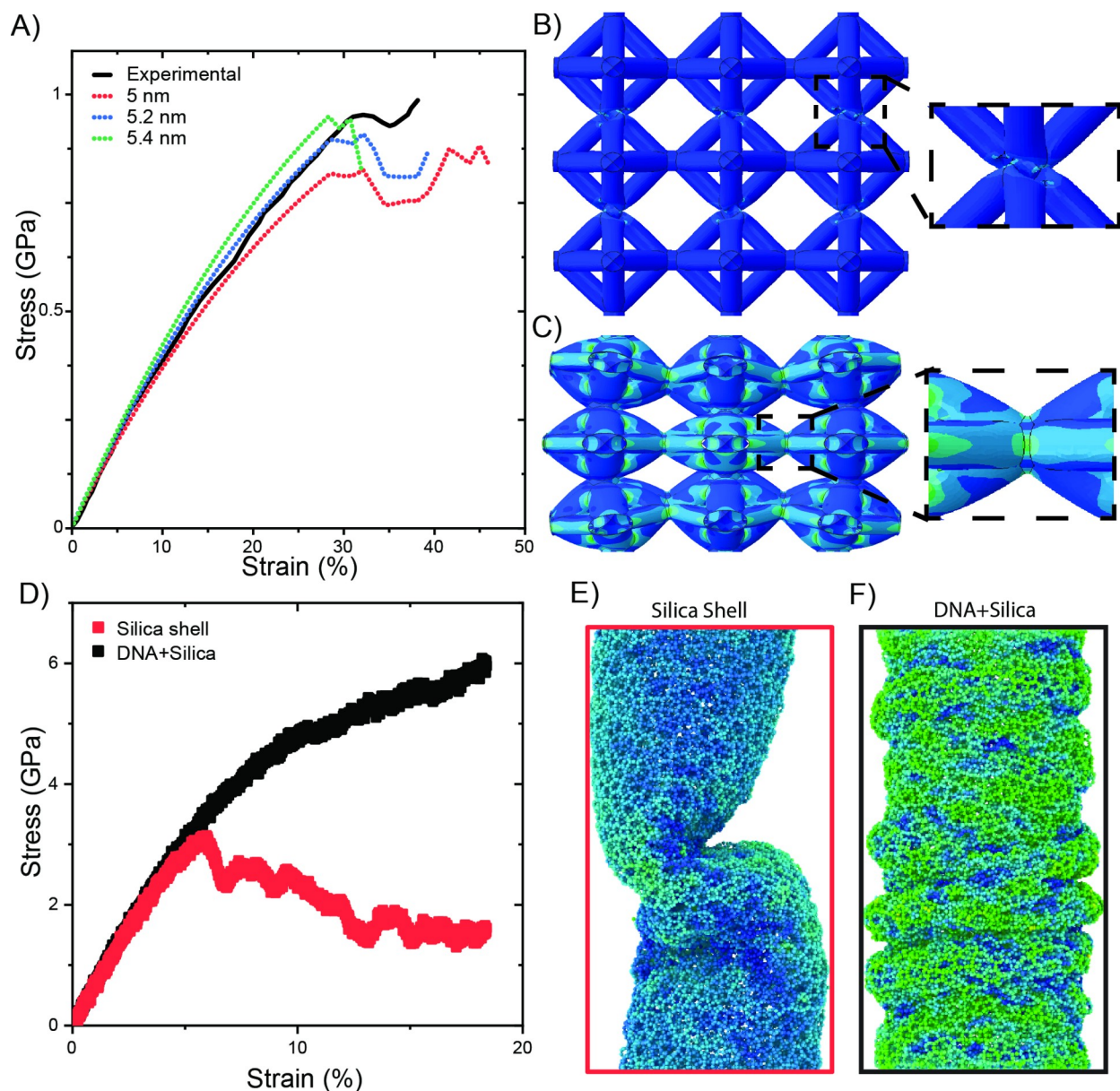




**Figure 3. Compression tests of standard, elongated, and flattened lattice structures.** A-C.) SEM images of A.) standard octahedron lattice, B.) elongated lattice, C.) flat lattice D.) Stress-strain curves for different silica coating thicknesses of the standard lattice. The blue curve corresponds to E.) post compression images. The vertical cracks that are formed during compression are marked with dashed lines. The green curve corresponds to F.) post compression images. The ~45° cracks are marked with dashed lines. G.) Stress-strain relations for the elongated and flat lattice structures. H.) post compression image for the elongated lattice. I) post compression image for the flat lattice. All scale bars are 1  $\mu\text{m}$ .



**Figure 4. DNA-silica lattices compared to other nano-lattices<sup>1–3,8,21,24,28,43,44,49–53</sup>.** A.) Compressive strength, B.) elastic modulus, C.) energy absorption vs. density. The gradient in A shows the Suquet upper bound (equation 5). The bottom of the gradient is the Suquet prediction using the strength of bulk silica. The top of the gradient is the Suquet prediction using the theoretical maximum strength of silica. The solid black line in C is the Hashin-Shtrikman upper bound (equation 4).



**Figure 5. Computational modeling and failure mode analysis.** A-C.) FEM simulation results of a 3x3x3 standard octahedral lattice made of hollow silica struts. A.) Stress-strain relations for silica shell thicknesses near 5.1 nm. B.) Lattice with 1.4 nm shell thickness fails through vertical collapse. C.) Lattice with 4.4 nm shell thickness fails through horizontal fracture. D-F.) MD simulation results of hollow silica strut and DNA core-silica shell strut. Both struts have a 6 nm diameter and a 1 nm shell thickness. D.) Stress-strain curves for single strut in compression. E.) Kinking buckling mode for the hollow silica strut. F.) Wrinkling buckling mode for the DNA core-silica shell strut.

**Video S1. Compression test for DNA-silica nanolattice with 1.75nm coating and 45° cracking.**



**Video S2. Compression test for DNA-silica nanolattice with 1.75nm coating and vertical cracking.**

**Video S3. Compression test for DNA-silica nanolattice with 3.1nm coating.**

**Video S4. Compression test for DNA-silica nanolattice with 5.1nm coating.**

**Video S5. Compression test for DNA-silica nanolattice with space filling coating.**

**Table S4. Sequences of staple DNA strands for regular octahedron.**

**Table S5. Sequence of sticky ends at vertexes of regular octahedron.**

**Table S6. Sequences of staple DNA strands for elongated octahedron.**

**Table S7. Sequence of sticky ends at vertexes of elongated octahedron.**

**Table S8. Sequences of staple DNA strands for flat octahedron.**

**Table S9. Sequence of sticky ends at vertexes of flat octahedron.**