

Nanoporosity-Driven Deformation of Additively Manufactured Nano-Architected Metals

Corresponding Author: Dr Wenxin Zhang

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This file contains all reviewer reports in order by version, followed by all author rebuttals in order by version.

Version 0:

Reviewer comments:

Reviewer #2

(Remarks to the Author)

In this article, the authors utilise a novel approach to fabricate nanoscale lattice structures. They use two-photon lithography to fabricate 3D structures which are then immersed in nickel nitrate solution. This allows for Ni ions to infuse into the polymer, forming Ni-infused hydrogel structures, which are then heat treated to create the final Ni nanolattices. The fabrication method has been previously assessed for creating nanopillars and nanolattice coupons in Zhang et al, Nano Lett. 2023, 23, 8162–8170.

This work demonstrates how this fabrication method can be used to create beam- or sheet-based nanolattices of different topologies and feature thicknesses (i.e., beams or sheets). The authors first evaluate key morphological characteristics and the quasi-static compressive response of fabricated coupons. Based on the experimental data, they evaluate the effect of different feature thicknesses on compressive strength, and then develop an FEA model that correlates their results with the entrapped porosity within the structures. The FEA model is being implemented into two phases: (1) in the unit cell scale which informs for the localised accumulation of porosity, and (2) in the macroscopic scale (reduced model) to evaluate the response of the whole lattice. The FEA model was applied only to the beam-based lattice.

Given the rational challenges of fabricating and testing samples at this length-scale, the manuscript provides a clear methodology and solid results. Its novelty comprises (1) the fabrication of different nanolattice topologies and (2) the mechanical assessment of these structures by considering fabrication defects for increasing feature thickness. The authors should add few details on methods and results and expand their literature review to discuss any limitations and future works. Below there are few comments I suggest being addressed by the authors prior to publication.

Comment 1

Regarding the measurement of feature dimensions. In methods and the supplementary materials, it is described that beam/sheet thickness was estimated by measuring 10 features per lattice. All samples demonstrate a wide spread of thickness, which is reasonable due to the challenging fabrication at the nanoscale. It would be interesting to refer to the location of measured features within the structures and discuss any potential differences, for example near the edges or at the middle of the samples.
In addition, the octahedral lattice consists of beams at 0° and 45° inclination in respect to the building bed, have you noticed any difference in the thickness in respect to the inclination angle? That could be relevant for the spinodal lattice which has sheets at different orientations.

Line 105: It would be good for readers to clarify that feature dimensions refer to beam or wall thickness.

Comment 2

The grain size has been previously quantified in vertical nanopillars, but it can be affected by the building orientation of the beams. It might be useful to add a comment about it and refer to relevant literature for conventional additive manufacturing (e.g., powder bed fusion). Investigating the grain size distribution within nanopillars built at different angles would be a potential future work.

Comment 3

Could you please explain how roughness was calculated? Which form of roughness (Ra, Rz, Rq) is being reported?

Comment 4

In Figure 2A, I would expect relative density ρ to increase with increasing d . Is the notation of the images (i, ii, iii) correct?

Comment 5

In Figure 2A, it would be helpful to add a circle indicating the primary collapse zone at phase 2. It seems that failure occurs consistently at the lower part of the specimens. If this is true, to what would you attribute it to? Is the lower part of the specimens – which is also the supported section – weaker due to the manufacturing process?

Comment 6

I believe that the walls of the Schwarz-P lattices undergo bending rather than buckling for the relative densities reported. This could be checked with the developed FEA model too.

Comment 7

Lines 188-190: Indeed, there is a clear trend distinguishing the periodic and non-periodic lattices. Do you believe that the difference can also originate from the type of lattice you chose? The non-periodic (spinodal-like) appears to comprise of walls with smooth transition compared to the octahedron or the Schwarz-P. Both periodic lattices form nodes with high curvature which can act as stress concentration sites, making it more sensitive as mentioned in the text.

This can be also associated with the increased porosity at the nodes of both periodic lattices as discussed in the section “Size-dependent defect distribution as the origin of nanomechanical behavior of nano architecture”. It will be interesting to highlight in your discussion a possible sensitivity of your fabrication method in lattice topologies with high nodal connectivity or very acute angles between features. Can this be attributed to the flow of Ni solution during infusion?

Comment 8

The reduced FEA model for the full lattice appears to match the trend observed in the experimental data for increasing d . Could you provide a percentage difference for pairs of same d between the simulation and experiment?

Comment 9

The experimentally tested specimens appear to have a high aspect ratio (height-to-width) compared to the ones used in the FEA models. Aspect ratio can affect measured properties and failure behaviour of lattices and might have caused differences in the simulation vs experimental data. It is worth discussing it using relevant literature.

Comment 10

In Figure 5A, values in the Y-axis are given as specific strength for each lattice. A better comparison might utilise the compressive strength of the lattice normalised to the yield strength of the base material.

Comment 11

In the methods, please provide the as-designed thickness of beams/walls, the size of unit cells, and the arrangement of unit cells ($H \times W \times L$) that were fabricated. How close to the as-designed dimensions were the as-built ones?

Comment 12

Lines 471-472: a better description on how the scaling factors are decided might be useful for readers.

Below are few minor comments on editing and formatting that can improve readability:

Line 63: Consider adding a parenthesis explaining what “building block” refers to. Later in the introduction, it seems that you refer to the beams or walls of the architected materials, but on a first read, it can also be misinterpreted as the unit cells that form the lattices.

Figure 1: please do the scale bar equal length for all macro-images (B_i , C_i , D_i). In addition, consider replacing D_{iii} with a magnified view of the pore that you have imaged using SEM.

Figure 2: use scale bars of equal length for all SEM images here as well.

Lines 41-42, 60-63, 89-90, 175-178: Replace the numbering (1, 2) with either letters (a, b) or Roman numerals (i, ii) to avoid confusion with the reference numbering.

Be consistent with the symbol used for feature size, in some parts of the text you use d while in others you use t .

Reviewer #3

(Remarks to the Author)

This study presents a well-executed investigation of hydrogel infusion-based 3D printing for fabricating various Ni nano-architectures, achieving a specific strength that may surpass previously reported results. While this is an interesting and valuable outcome, the overall contribution to merit publication in Nature Communications is questionable, particularly given that the authors have already demonstrated the well-known “smaller is stronger” size effect in a power-law manner in their

previous works.

1. The Main Text section refers only briefly to the authors' earlier publications, making it difficult for readers to understand how the current results build upon previous efforts. I believe the authors should take the initiative to clearly highlight the advancements made in this study relative to their earlier work. It would provide valuable context and help readers better appreciate the progress achieved.

- The statement "Despite significant advances in the additive manufacturing (AM) of metals with complex microstructures and geometries(10, 11), the direct fabrication of designed-for-function metal parts remains a challenge", acknowledges existing challenges. However, citation (11), the authors' own work published as "M. A. Saccone, R. A. Gallivan, K. Narita, D. W. Yee, J. R. Greer, Additive manufacturing of micro-architected metals via hydrogel infusion. *Nature* 612, 685–690 (2022)", demonstrated the successful fabrication of various metallic nano-architectures using the same technique. Explicitly linking this prior challenge/achievement to the ongoing development in the current work could enhance the perceived significance and continuity of the research.

- The statement "as well as from the throughput and structural complexity and integrity constraints of emerging nano-printing methods, most of which are field-assisted or nanocluster-based(14–18)", cites reference (18), which is the authors' own previous work: "A. Vyatskikh, S. Delalande, A. Kudo, X. Zhang, C. M. Portela, J. R. Greer, Additive manufacturing of 3D nano-architected metals. *Nat. Commun.* 9, 593 (2018)". In that study, Ni nano-architectures with octet geometries were successfully fabricated using the same technique, demonstrating high specific strength. However, the current statement does not clearly acknowledge this prior achievement and confusingly refers to the technique inconsistently, citing it as a different method. This inconsistency obscures the connection between previous work and the claimed fabrication limitations, making the relevance of past efforts unclear.

- The statement "Leveraging on the fabrication of monolithic metal nanopillars, which can serve as the nano-building blocks with superior strengths(20), we fully develop nano-HIAM process for complex 3D nano-architectures with 100-nm-fine feature dimensions and 10s-nm-scale surface roughness", cites reference (20), which is the authors' own previous work: "W. Zhang, Z. Li, R. Dang, T. T. Tran, R. A. Gallivan, H. Gao, J. R. Greer, Suppressed Size Effect in Nanopillars with Hierarchical Microstructures Enabled by Nanoscale Additive Manufacturing. *Nano Lett.* 23, 8162–8170 (2023)". While this prior work indeed demonstrates progress in the fabrication of hierarchical nanopillars, the claim that "we fully develop the nano-HIAM process" for high-resolution complex 3D nano-architectures does not clearly present a novel finding beyond what has already been reported. The features cited 100-nm-scale resolution and 10s-nm surface roughness appear to have been achieved in the authors' earlier studies.

- Overall, this work further expands upon the authors' previous studies by fabricating and comparing a series of periodic beam-based and shell-based Ni nanolattices, as well as non-periodic spinodal-like Ni nano-architectures, supported by finite element analysis.

2. Recent studies by other groups have also demonstrated significant progress in fabricating high-specific-strength Ni-based nanoarchitectures using two-photon lithography (TPL) combined with thermal treatment. For example, "3D Printing of Metal and Alloy Nanoarchitectures through a Complexation-Driven Strategy, *Nano Letters* (2025)" reports advances from a feature-size perspective. Additionally, TPL-fabricated shell-based carbon nanoarchitectures, supported by finite element simulations, have achieved near-theoretical strength limits, as cited reference (21), "Achieving the theoretical limit of strength in shell-based carbon nanolattices, *PNAS* (2022)".

3. On Page 3, Line 97, the authors mention that the TPL printing speed was "increased by 10x". It would be helpful to provide the actual printing speeds used for comparison to better quantify this improvement.

4. On Line 99, the manuscript claims that this method ensures "thereby ensuring its reliability, precision, and throughput for producing large, complex 3D geometries". This statement may be somewhat overstated. It would be useful to clarify what is meant by "large" in this context, especially in relation to industrial scalability.

5. First, how many samples were tested for each feature size in Figure 2? Was there any notable variability in the results? Second, in Figures 2B and 2C, clear structural failure modes are shown. However, on Page 5, Line 142, the authors mention: "We were able to easily identify these samples by their signature of having visual defects (e.g., cracked or wrinkled walls) and low compressive stiffness during initial loading and not include them in the analysis". This raises concerns regarding repeatability and precision. A discussion on how these exclusion criteria affect the statistical robustness and reproducibility of the compression may be needed.

6. The finite-element simulation results are meaningful; however, compared to previously reported micro-architectures built via 3D printing in the literature, the findings for the nano-architectures shown in Figure 4 and Figure S6 do not appear to offer new insights.

7. On Page 12, Line 313, it states "In the meso-to-macroscale regime of metallic lattices, with feature dimensions of 30 μm to 2 mm, there exists a "smaller is weaker" trend". However, this should not be considered a universal rule. Numerous studies have demonstrated exceptions across various metallic alloys. For example, in titanium alloys, powder bed fusion-built micro-lattice Ti6Al4V has shown a specific strength of approximately 326.6 MPa/(g/cm³), which is about 1.7 times higher than that of bulk Ti6Al4V (180 MPa/(g/cm³)), as reported in the literature "Ultra-high specific strength Ti6Al4V alloy lattice material manufactured via selective laser melting, 2022".

8. On Page 13, Line 363, the statement "These structures surpass the fabrication precision and structural complexity

limitations of all existing metal additive manufacturing methods today” appears to be somewhat overstated, considering both the authors’ previous work and findings reported by other research groups.

Version 1:

Reviewer comments:

Reviewer #2

(Remarks to the Author)

The authors have addressed all of my comments in detail. In the revised manuscript, they have included additional methodological information, provided further clarification of their results, and discussed the study’s limitations. Based on these revisions, I consider the manuscript suitable for publication.

Reviewer #3

(Remarks to the Author)

The authors have replied all concerns. Therefore, the revision is acceptable for publication.

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We sincerely thank the editor and reviewers for their insightful comments and constructive suggestions on our manuscript. Below is our detailed point-by-point response to each comment. The reviewers' comments are shown in blue italics, while our responses are provided in black. We have thoroughly revised the manuscript, with all modifications highlighted in red, which are also included in the response letter.

Reviewer #2 (Remarks to the Author):

In this article, the authors utilise a novel approach to fabricate nanoscale lattice structures. They use two-photon lithography to fabricate 3D structures which are then immersed in nickel nitrate solution. This allows for Ni ions to infuse into the polymer, forming Ni-infused hydrogel structures, which are then heat treated to create the final Ni nanolattices. The fabrication method has been previously assessed for creating nanopillars and nanolattice coupons in Zhang et al, Nano Lett. 2023, 23, 8162–8170.

This work demonstrates how this fabrication method can be used to create beam- or sheet-based nanolattices of different topologies and feature thicknesses (i.e., beams or sheets). The authors first evaluate key morphological characteristics and the quasi-static compressive response of fabricated coupons. Based on the experimental data, they evaluate the effect of different feature thicknesses on compressive strength, and then develop an FEA model that correlates their results with the entrapped porosity within the structures. The FEA model is being implemented into two phases: (1) in the unit cell scale which informs for the localised accumulation of porosity, and (2) in the macroscopic scale (reduced model) to evaluate the response of the whole lattice. The FEA model was applied only to the beam-based lattice.

Given the rational challenges of fabricating and testing samples at this length-scale, the manuscript provides a clear methodology and solid results. Its novelty comprises (1) the fabrication of different nanolattice topologies and (2) the mechanical assessment of these structures by considering fabrication defects for increasing feature thickness.

The authors should add few details on methods and results and expand their literature review to discuss any limitations and future works. Below there are few comments I suggest being addressed by the authors prior to publication.

Reply 2:

We thank the reviewer for their careful reading and positive assessment of our manuscript. We have revised the manuscript to include additional details in the Methods and Results sections and to expand the discussion of limitations and future work. Below is our point-by-point response to the comments.

Comment 1

Regarding the measurement of feature dimensions. In methods and the supplementary materials, it is described that beam/sheet thickness was estimated by measuring 10 features per lattice. All

samples demonstrate a wide spread of thickness, which is reasonable due to the challenging fabrication at the nanoscale. It would be interesting to refer to the location of measured features within the structures and discuss any potential differences, for example near the edges or at the middle of the samples.

In addition, the octahedral lattice consists of beams at 0° and 45° inclination in respect to the building bed, have you noticed any difference in the thickness in respect to the inclination angle? That could be relevant for the spinodal lattice which has sheets at different orientations.

Line 105: It would be good for readers to clarify that feature dimensions refer to beam or wall thickness

Reply 2-1:

We thank the reviewer for this insightful question. We agree with your careful observation of a wide spread of thickness in each nanolattice. In particular, feature thickness near edges shows greater variation due to fabrication reasons. Thus, the dimension measurements were limited to the middle of the samples.

Beam cross-sections are commonly longer in the vertical direction — partly due to inclination, partly due to inherently elongated shape of the two-photon lithography laser voxel along Z. Thus, for beam elements, we measured both horizontal and vertical dimensions and took the geometric mean of the two as the characteristic dimension of a cross-sectional feature. For shell elements, we measured the thickness perpendicular to the shell, which was found to be not significantly dependent on the inclination.

We have added the following note in the method section where the feature dimension measurement is mentioned: “As observed, there exists a spread of feature dimension from location to location and in horizontal vs. vertical directions. Thus, all feature dimension measurements were taken at the inner 4x4 lattices of a cross-section, avoiding fabrication-induced distortion near edges, and orthogonal to the feature edge/surface.”

Comment 2

The grain size has been previously quantified in vertical nanopillars, but it can be affected by the building orientation of the beams. It might be useful to add a comment about it and refer to relevant literature for conventional additive manufacturing (e.g., powder bed fusion). Investigating the grain size distribution within nanopillars built at different angles would be a potential future work.

Reply 2-2:

We thank the reviewer for this valuable suggestion. We agree that the building orientation may strongly influence local thermal gradient, solidification front, and consequently the grain size and morphology in conventional additive manufacturing methods (Akram, J., et al., 2018, *Additive Manufacturing*, 21, pp.255-268.; Raghavan, N., et al., 2017, *Acta Materialia*, 140, pp.375-387; Leicht, A., et al., 2018. *Materials Characterization*, 143, pp.137-143.). In the current hydrogel-infusion based nano-additive manufacturing, the final grain structure is formed after the calcination and reduction of whole Ni-ion-infused hydrogel 3D structures. This process results in a more homogenous grain morphology as shown in our work in contrast to the highly anisotropic

(columnar-shaped) grain structure in conventional metal additive manufacturing. While the building orientation might influence the local heat flux, the difference in the local thermal gradient is believed to be much smaller than that in the layer-by-layer printed structures. The grain size will be mainly controlled by the conditions of thermal treatment, i.e., calcination /reduction temperature and time. We have revised the manuscript to add a brief discussion of the grain size on page 3:

“...it should be noted that the conditions of calcination and reduction treatment will affect the grain growth kinetics and final grain structure which requires further systematic study...”

Comment 3

Could you please explain how roughness was calculated? Which form of roughness (Ra, Rz, Rq) is being reported?

Reply 2-3:

Thank you for asking the clarification question. The roughness is mainly estimated based on TEM images based on our observation that the angular grains form the feature material's surface and defines the averaged fluctuation of the surface height being equal or less than the length of one grain. The roughness thus we report to be on the same order of magnitude as the observed nanocrystalline grain size being 10s of nanometers. Due to the 3D nature of the present samples, we were unable to use AFM or similar techniques to quantify the surface roughness in the conventional way. We appreciate your understanding and consideration of the technical limitations.

Comment 4

In Figure 2A, I would expect relative density ρ to increase with increasing d . Is the notation of the images (i, ii, iii) correct?

Reply 2-4:

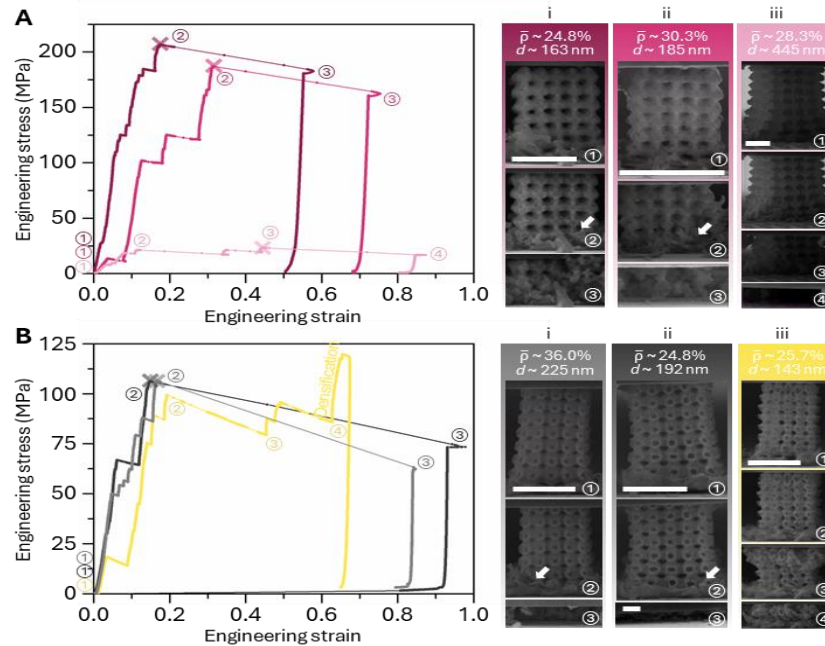
We appreciate your careful observation. We double-checked our measurements analysis. Indeed, the reported value is accurate. It might appear not intuitive relative to the other two counterpart nanolattices, because this specific lattice's beam cross-section has a lower aspect ratio between the horizontal and the vertical dimensions, so in the SEM micrograph, the beams could appear tall, void space may appear smaller, while the beams are thinner in the into-the-page direction.

Comment 5

In Figure 2A, it would be helpful to add a circle indicating the primary collapse zone at phase 2. It seems that failure occurs consistently at the lower part of the specimens. If this is true, to what would you attribute it to? Is the lower part of the specimens – which is also the supported section – weaker due to the manufacturing process?

Reply 2-5:

We thank the reviewer for this valuable suggestion. We have added white arrows in Fig. 2 A and B to indicate the location of the premature failure at the base layers of the periodic lattices in phase 2.



This premature failure in phase 2 often happens at the base layers where processing shrinkage induces distortion and internal pre-stress/strain, resulting in small bursts in the stress-strain curves. Nevertheless, the final failure modes of the specimens characterized by the catastrophic strain burst are either concentrated porosity-controlled materials failure in samples with small feature dimension or large imperfections induced structure failure in samples with large feature dimension.

Comment 6

I believe that the walls of the Schwarz-P lattices undergo bending rather than buckling for the relative densities reported. This could be checked with the developed FEA model too.

Reply 2-6:

We thank the reviewer for this insightful question. We agree that bending is the default deformation mode for the Schwarz-P nanolattices in current study with ratio of shell thickness to unit-cell size ~ 0.09 - 0.14 and relative density $\sim 25\%$ - 40% as also discussed in the literature (Xing, H., et al., 2025. *Small Structures*, p.e202500440.). FEM simulation of the typical Schwarz-P unit cell (Fig. 4Aii) also supports the bending-dominated deformation mode as evidenced by the typical stress distribution across the shell of the unit cell (Fig. S6Bi). However, we do find some rare examples like the one in Fig. 2Biii, where the fabrication-induced imperfections, i.e., cracks, induce localized deformation and premature layer-by-layer failure, lowering the strength for more than 30%. This initial imperfection-induced localized failure has also been reported in other architected structures, i.e. polymeric microlattices (Wang, Y., et al., 2022. *PNAS*, 119(34), p.e2119536119.). Since these rare cases have significant lower strength and clearly visible defects, we classify these structures as poorly manufactured samples and don't include them in the scaling factor analysis where we focus on the intrinsic mechanical behavior of the AM nanoarchitected structures. To avoid confusion, we have revised the related narrative on page 4 "...we also observed

a secondary failure type, a “structural failure” mode (Fig. 2Biii and Ciii), which manifests as gradual, layer-by-layer **localized failure** and densification”.

Comment 7

Lines 188-190: Indeed, there is a clear trend distinguishing the periodic and non-periodic lattices. Do you believe that the difference can also originate from the type of lattice you chose? The non-periodic (spinodal-like) appears to comprise of walls with smooth transition compared to the octahedron or the Schwarz-P. Both periodic lattices form nodes with high curvature which can act as stress concentration sites, making it more sensitive as mentioned in the text.

This can be also associated with the increased porosity at the nodes of both periodic lattices as discussed in the section “Size-dependent defect distribution as the origin of nanomechanical behavior of nano architecture”. It will be interesting to highlight in your discussion a possible sensitivity of your fabrication method in lattice topologies with high nodal connectivity or very acute angles between features. Can this be attributed to the flow of Ni solution during infusion?

Reply 2-7:

We thank the reviewer for this valuable suggestion. We agree that the difference in the types of the lattices and the associated distinguished geometrical and microstructural features lead to the observed clearly different scaling factors in periodic and non-periodic lattices. On the one hand, the periodic octahedral and Schwarz-P nanolattices have well-defined regions of high stress concentration at junctions (Fig. S6) with high curvature as opposed to the more randomly distributed stress field in spinodal-like nanolattices, which makes the junctions the more susceptible regions for material failure. On the other hand, the junctions in the octahedral and Schwarz-P nanolattices are also shown to have concentrated porosity which induces size-dependent degradation in strength, which is not observed in non-periodic spinodal-like lattices due to their stochastic microstructural variation. These features in periodic nanolattices collectively suggest a junction region-dominated deformation behavior with much stronger feature-dimension dependence of ultimate strength that is intrinsically controlled by the distribution of nanoporosity. The lack of such “junctions” and the stochastic nature of the distribution of stress field and internal defects in spinodal-like lattices lead to the reduced size-dependence.

We agree that lattice topologies might have a profound impact on the microstructure and properties of the nano-architected structures from our hydrogel infused based nano-additive manufacturing. Our work shows that local feature dimension directly affects the formation of nanopores which controls the local material strength and fracture behavior of the architected structures. The formation of nanoporosity arises from the significant volumetric shrinkage, primarily during the air-calcination step and secondarily during the forming gas reduction step. The local topological features might alter several aspects in the fabrication process including the driving force for grain formation of the NiO intermediate phase in air-calcination and the kinetics of the removal of the oxides through the reduction under hydrogen gas. However, with regard to your comment about “flow of Ni solution during infusion,” we believe it has significantly less impact than the latter thermal steps on the final product from a topology-sensitive perspective. The infusion step time

was determined to allow the Ni infusion to stabilize at an equilibrium Ni distribution and amount — meaning minimal influence on the final product with further infusion time.

We believe that lattices with high nodal connectivity could have many junction regions with increased feature dimensions and higher nanoporosity similar to our octahedral nanolattices, resulting in a concentrated nanoporosity-governed deformation and fracture behavior. We have added a brief discussion on page 13 to highlight the possible link between lattice topologies and mechanical properties as follows.

“....It is also worth noting that the topological features of the nano-architectures such as local feature dimension variation, connectivity and curvatures might affect the kinetics of nanopore formation during the calcination and reduction processes and the mechanical behavior, which requires further systematic studies ...”

Comment 8

The reduced FEA model for the full lattice appears to match the trend observed in the experimental data for increasing d. Could you provide a percentage difference for pairs of same d between the simulation and experiment?

Reply 2-8:

Thank you for the opportunity to further discuss the comparison between our experimental results and FEM simulations. In the reduced FEA model, we adopt two different material models to account for the size-dependent strength of the materials, i.e., direct scaling method and porosity-based degradation method. Both methods perform quite well in matching the scaling factor of the size-dependent feature strength (Suquet upper bound) with respect to feature dimension (Fig.4iv) as observed in experimental results, which suggests that the nanoporosity-governed deformation is the key factor controlling the mechanical response of these nano-architected structures. Regarding the percentage difference in feature strength, below is a table directly comparing the experimental and simulated results at three specific feature dimensions of the nanolattice.

Feature dimension (nm)	Experimental results	direct scaling method	porosity-based degradation method
185	766	639 (-16%)	731(-5%)
220	511	479 (-6%)	524(+3%)
300	257	290 (+13%)	311(+20%)

Table R1: comparison of the size-dependent feature strength between experiment and simulation at different feature dimensions.

The simulation results match reasonably well with the experimental results given the difficulties in accurately predicting the ultimate strength. There could be two major sources leading to the discrepancy between the experiments and simulations. One is the statistical variation in the strength of the building blocks (i.e., nanopillars) (Fig. 2D) and the nanoporosity distribution (Fig.

3B and Fig. S4A) due to the demanding manufacturing process, collectively leading to a certain level of variation in the ultimate strength of the nanolattices. This material and processing statistical variation has not been considered in our FEM simulations. The other source is the adoption of the Suquet upper bound for the evaluation of feature strength (Eq.4), which allows us to discuss the size effect of the feature dimension separately from effects of the relative density. In current nano-additive manufacturing, the relative density of the final product is collectively determined by the feature dimension and the length of the unit cell after reduction, which is quite challenging to control precisely in experiments. On the other hand, FEM explores the geometric space evenly and widely by varying the feature dimension and length of the unit cell in a structural way. Therefore, there are some differences in the relative density between the samples in experiments and simulation in Table R1, which are considered through the adoption of the Suquet upper bound to obtain the feature strength from the lattice strength. However, this translation could induce a certain level of discrepancy. Nevertheless, we would like to highlight the key message of this coupled experiment–simulation study—the nanoporosity-governed deformation mechanism, as supported by the quantitative agreement in the scaling factors.

Comment 9

The experimentally tested specimens appear to have a high aspect ratio (height-to-width) compared to the ones used in the FEA models. Aspect ratio can affect measured properties and failure behaviour of lattices and might have caused differences in the simulation vs experimental data. It is worth discussing it using relevant literature.

Reply 2-9:

We thank the reviewer for this insightful question. We would like to clarify that the aspect ratio of the octahedral lattice is designed to be the same in the experiment and simulation and the inclination angle of the beams are designed to be 45 degrees. The FEM model of the unit cell (Fig.4i) is directly taken from the CAD file of the printed nanolattice. However, we acknowledge that the actual printed nanolattices have slightly different ratios due to varying levels of shrinkage during the reduction process. The aspect ratio (h:w) in the experiment is observed to be generally below 1.2 in the upper portion of the lattice, representing a ~20% increase. We believe this discrepancy will not change the basic fracture behavior which is governed by the concentrated nanoporosity and characterized by the catastrophic global collapse of the nanolattices, as buckling is not activated in those nanostructures. Nevertheless, aspect ratio (lattice angle) has been shown in the literature to be an important geometric factor affecting the mechanical properties. For example, a higher aspect ratio has been shown to increase strength and stiffness of solid polymer microlattices (Jacobsen, A.J., et al., 2007. *Advanced Materials*, 19(22), pp.3892-3896.; Deshpande, V.S. and Fleck, N.A., 2001. *International Journal of Solids and Structures*, 38(36-37), pp.6275-6305.). We expect some quantitative variation in the measured ultimate strength arising from differences in the aspect ratio of the experimental samples. This variation could contribute to the discrepancy between the ultimate strengths obtained from experiments and simulations, as

discussed in more detail in Reply 2-8. The manuscript has been revised to include additional discussion of the aspect-ratio differences between the simulations and experiments on page 13.

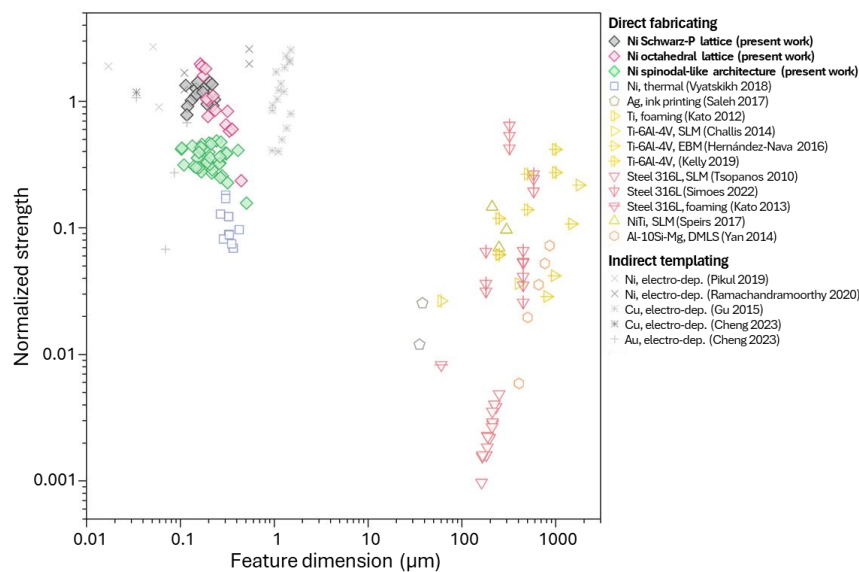
“...Owing to the complex thermal and chemical reactions during the calcination and reduction processes, the height-to-width aspect ratio of the as-fabricated nano-architected metals (Fig. 1Biii–Diii) is slightly higher than that of the as-designed samples (Fig. 1Bii–Dii) and the FEM models (Fig. 4). This difference in aspect ratio may influence the mechanical behavior of the nanolattices(43, 44) and contribute to the quantitative discrepancy in ultimate strength between the experimental results and FEM simulations...”

Comment 10

In Figure 5A, values in the Y-axis are given as specific strength for each lattice. A better comparison might utilise the compressive strength of the lattice normalised to the yield strength of the base material.

Reply 2-10:

We thank the reviewer for this valuable suggestion. We use Fig.5A to demonstrate that the nano-architected Ni metals manufactured through our nano-additive manufacturing methods can reach the same high level of specific strength as meso-to-macro lattices with more mature manufacturing methods, while being three orders of magnitude smaller. Since specific strength is widely quoted in the study of mechanical properties of lattice structures, we prefer to keep Fig.5A in its current form. We have included the following new figure in the SI (Fig. S12), where the compressive strength of the lattice is normalized by the typical yield strength of the corresponding bulk materials. This plot allows further comparison of the mechanical properties of our nano-architected Ni metals across different material systems in the literature.



Comment 11

In the methods, please provide the as-designed thickness of beams/walls, the size of unit cells, and the arrangement of unit cells ($H \times W \times L$) that were fabricated. How close to the as-designed dimensions were the as-built ones?

Reply 2-11:

Thank you for the comment. **Fig. 1A** shows the true 3D representation of the two-photon lithography laser line trace in the DeScribe software using the as-designed, i.e., as-printed feature dimensions. Besides, our current **Fig. 1B-D** already shows a zoom-in version comparison of the CAD model references (**Fig. 1Bii–Dii**) and the respective as-fabricated features (**Fig. 1Biii–Diii**). The main deviations and root causes of the as-fabricated vs. the as-designed are:

1. Two-photon lithography voxel: the two photon lithography laser has a focusing size that is smaller in X-Y plan and taller in Z-direction, causing the smallest as-printed features to be elongated in Z-direction.
2. Absolute dimension: overall linear shrinkage to 10-20% of as-printed dimension, depending on Ni infusion solution concentration.
3. Feature (beam and wall) aspect ratio: depending on calcination/reduction pressure, temperature, and duration, the bulk dimension and local feature dimension shrinkage could have different contributions as a result of varying atom diffusion kinetics under varying conditions.

Overall, it is commonly observed that the as-fabricated features may have increased aspect ratio and relative density compared with the design. We have incorporated additional comments in Supplementary Text 1.

Comment 12

Lines 471-472: a better description on how the scaling factors are decided might be useful for readers.

Reply 2-12:

We thank the reviewer for this valuable suggestion. In the FEM simulations, we assign scaling factors for the size-dependent fracture strength based on the results from our previous study on nanopillars (*Nano Letters*, 2023, 23(17), pp.8162-8170), where we show that when the pillar diameter is smaller than 350 nm, the scaling factor of fracture strength is around ~ -0.2 while for larger pillar, the increased porosity will lead to a scaling factor of ~ -2 . Therefore, we assign a scaling factor of -2 to the nodal elements with increased dimensions and a scaling factor of -0.2 to non-nodal elements, which mostly fall within the size insensitive region in our previous study. We have revised the Methods section on page 16 to further clarify this point.

“... The choice of the scaling factor of -2 for nodal elements and -0.2 for non-nodal elements is based on the observation from our previous study²⁰ of Ni nanopillars where we show that the scaling factor of fracture strength is ~ -0.2 for nanopillars with diameter smaller than 350 nm and changes to ~ -2 for larger nanopillars with increased porosity...”

Below are few minor comments on editing and formatting that can improve readability:

Line 63: Consider adding a parenthesis explaining what “building block” refers to. Later in the introduction, it seems that you refer to the beams or walls of the architected materials, but on a first read, it can also be misinterpreted as the unit cells that form the lattices.

Figure 1: please do the scale bar equal length for all macro-images (Bi, Ci, Di). In addition, consider replacing Diii with a magnified view of the pore that you have imaged using SEM.

Figure 2: use scale bars of equal length for all SEM images here as well.

Lines 41-42, 60-63, 89-90, 175-178: Replace the numbering (1, 2) with either letters (a, b) or Roman numerals (i, ii) to avoid confusion with the reference numbering.

Be consistent with the symbol used for feature size, in some parts of the text you use d while in others you use t .

Reply 2-13: We thank the reviewer for the valuable suggestions.

- In line 63, we have added the definition of “Building blocks” as “...the properties of the “building blocks”—the constituent elements of the structures such as beams and shells — directly influence the performance of the overall “architecture...”.
- We have used scale bars with equal lengths of 5 μm in Fig. 1 and 4 μm in Fig. 2. In Fig. 1Dii and Fig. 1Diii, we provide a direct comparison of the as-printed and as-designed spinodal-like nano-architectures. Therefore, we would like to keep Fig. 1Dii as it is.
- We have used Roman numerals to replace the numbering in the revised manuscript.
- We have used d for feature dimensions throughout the revised manuscript.

Reviewer #3 (Remarks to the Author):

This study presents a well-executed investigation of hydrogel infusion-based 3D printing for fabricating various Ni nano-architectures, achieving a specific strength that may surpass previously reported results. While this is an interesting and valuable outcome, the overall contribution to merit publication in Nature Communications is questionable, particularly given that the authors have already demonstrated the well-known “smaller is stronger” size effect in a power-law manner in their previous works.

Reply 3: We thank the reviewer for their careful reading and detailed assessment of our manuscript. We have revised the manuscript to better highlight the unique contributions of this work, including the fabrication of high-integrity, high-strength nano-architected metals and the mechanistic understanding of nanoporosity-governed deformation, comparison with our prior study and other recent publications. Below is our point-by-point response to the comments.

1. The Main Text section refers only briefly to the authors’ earlier publications, making it difficult for readers to understand how the current results build upon previous efforts. I believe the authors should take the initiative to clearly highlight the advancements made in this study relative to their earlier work. It would provide valuable context and help readers better appreciate the progress achieved.

- The statement “Despite significant advances in the additive manufacturing (AM) of metals with complex microstructures and geometries(10, 11), the direct fabrication of designed-for-function metal parts remains a challenge”, acknowledges existing challenges. However, citation (11), the authors’ own work published as “M. A. Saccone, R. A. Gallivan, K. Narita, D. W. Yee, J. R. Greer, Additive manufacturing of micro-architected metals via hydrogel infusion. Nature 612, 685–690 (2022)”, demonstrated the successful fabrication of various metallic nano-architectures using the same technique. Explicitly linking this prior challenge/achievement to the ongoing development in the current work could enhance the perceived significance and continuity of the research.*

- The statement “as well as from the throughput and structural complexity and integrity constraints of emerging nano-printing methods, most of which are field-assisted or nanocluster-based(14–18)”, cites reference (18), which is the authors’ own previous work: “A. Vyatskikh, S. Delalande, A. Kudo, X. Zhang, C. M. Portela, J. R. Greer, Additive manufacturing of 3D nano-architected metals. Nat. Commun. 9, 593 (2018)”. In that study, Ni nano-architectures with octet geometries were successfully fabricated using the same technique, demonstrating high specific strength. However, the current statement does not clearly acknowledge this prior achievement and confusingly refers to the technique inconsistently, citing it as a different method. This inconsistency obscures the connection between previous work and the claimed fabrication limitations, making the relevance of past efforts unclear.*

- The statement “Leveraging on the fabrication of monolithic metal nanopillars, which can serve as the nano-building blocks with superior strengths(20), we fully develop nano-HIAM process for*

complex 3D nano-architectures with 100-nm-fine feature dimensions and 10s-nm-scale surface roughness”, cites reference (20), which is the authors’ own previous work: “W. Zhang, Z. Li, R. Dang, T. T. Tran, R. A. Gallivan, H. Gao, J. R. Greer, Suppressed Size Effect in Nanopillars with Hierarchical Microstructures Enabled by Nanoscale Additive Manufacturing. Nano Lett. 23, 8162–8170 (2023)”. While this prior work indeed demonstrates progress in the fabrication of hierarchical nanopillars, the claim that “we fully develop the nano-HIAM process” for high-resolution complex 3D nano-architectures does not clearly present a novel finding beyond what has already been reported. The features cited 100-nm-scale resolution and 10s-nm surface roughness appear to have been achieved in the authors’ earlier studies.

• Overall, this work further expands upon the authors’ previous studies by fabricating and comparing a series of periodic beam-based and shell-based Ni nanolattices, as well as non-periodic spinodal-like Ni nano-architectures, supported by finite element analysis.

Reply 3-1:

Thank you for the opportunity to further clarify the novelty of this work and its relation to our previous studies. In the current work, we demonstrate the hydrogel-infusion based nanoscale additive manufacturing (nano-HIAM) capable of fabricating high-quality nano-architected Ni metals with complex microstructures and superior specific strength, which are not achievable by the current state-of-the-art metal additive manufacturing methods to the best of our knowledge. Integrated experiments and multi-hierarchy finite element analysis further collectively reveal the nanoporosity-governed deformation mechanism and the feature-dimension dependence of the ultimate strength, completing the mechanical validation of the additively manufactured (AM) nano-architectures.

- As pointed out by the reviewer, our previous work (*Nature* 612, 685–690, 2022) is the first demonstration of the hydrogel-infusion-based mesoscale additive manufacturing method, that is versatile for fabricating various high-quality architected metals with feature dimensions of approximately 40 μm . Our current work has developed the capability for additive manufacturing of nano-architected structures, **reducing the feature dimension by more than 200-fold** compared with previous work.
- In our previous nano-HIAM work (*Nano Lett.* 23, 8162–8170, 2023), we demonstrated the fabrication and testing of nanocrystalline Ni nanopillars with diameters as small as 100 nm and revealed the underlying size-dependent atomistic deformation mechanisms. Our current work has significantly advanced nano-HIAM by developing the capability to fabricate **high quality, high specific strength nano-architected structures with complex microstructures**, which are well-positioned for applications in emerging nanotechnology fields, such as Nano-MEMS and nanorobotics.
- In addition, we would like to point out that our hydrogel-infusion-based additive manufacturing method has a very different manufacturing process from the one introduced in previous work (*Nat. Commun.* 9, 593, 2018), although two-photon lithography was adopted in both cases. As a result, the previous study (*Nat. Commun.* 9, 593, 2018)

fabricated nano-architected structures with ~ 100 nm resolution, and the concentration of metal in the structure is over 90% as shown in the following EDS spectrum. The current hydrogel-infusion-based method succeeds in achieving **pure metallic structures** (as demonstrated by the XRD analysis in *Nature* 612, 685–690, 2022) after calcination and reduction and further demonstrates versatility in fabricating multi-component alloys.

[Figure Redacted]

In summary, our current work has made significant advances in nanoscale metal additive manufacturing and developed the capability to fabricate pure metal nano-architectures with

unprecedented high structural integrity and mechanical performance. This work is built on our previous studies on mesoscale HIAM and nano-HIAM of simple nanopillars, and achieves control of complex microstructure at the nanoscale, laying the foundation for emerging applications in nanotechnology. The integrated experimental–simulation study also reveals the fundamental nanoporosity-governed deformation mechanism of the nano-architectures, which is essential for the mechanical validation of AM products. We have revised the Introduction part on page 2 to clearly state the current advances relative to our earlier work.

“...The current nano-HIAM is built upon our previous development of meso-HIAM for the fabrication of micro-architected metals (18) and nano-HIAM for monolithic metal nanopillars with superior strength (20), while substantially advancing nanoscale fabrication of complex 3D structures. In this work, we fully develop the nano-HIAM process for complex 3D nano-architectures with 100-nm-fine feature dimensions and 10s-nm-scale surface roughness...”

2. Recent studies by other groups have also demonstrated significant progress in fabricating high-specific-strength Ni-based nanoarchitectures using two-photon lithography (TPL) combined with thermal treatment. For example, “3D Printing of Metal and Alloy Nanoarchitectures through a Complexation-Driven Strategy, Nano Letters (2025)” reports advances from a feature-size perspective. Additionally, TPL-fabricated shell-based carbon nanoarchitectures, supported by finite element simulations, have achieved near-theoretical strength limits, as cited reference (21), “Achieving the theoretical limit of strength in shell-based carbon nanolattices, PNAS (2022)”.

Reply 3-2:

We thank the reviewer for bringing the relevant literature to our attention. The recently published work (*Nano Lett.* 2025, 25, 8294–8302) has demonstrated the fabrication of 3D metal/alloy nanoarchitectures through a complexing-capable polymer scaffold with feature size of ~200 nm and specific strength comparable to the ones in *Nat. Commun.* (9, 593, 2018) as shown below.

[Figure Redacted]

As shown in Fig. 5A of the manuscript, the specific strength of our nano-architected Ni metals can reach 100 MPa.g-1cm³, almost **10 times higher** than the value of ~10⁴ Pa.kg-1m³ (10 MPa.g-

1cm³) reported in *Nat. Commun.* (9, 593, 2018) and *Nano Letters* (2025, 25, 8294–8302), suggesting a significantly improved structural integrity with our nano-HIAM methods.

In contrast to the study of TPL-fabricated shell-based carbon nanoarchitectures (*PNAS* 2022), our current work tackles the challenges in fabricating **high-integrity nano-architected metallic structures** with superior mechanical properties and reveals the origins of feature-size dependence with integrated experimental characterization and multi-hierarchy FEM simulations. The feature-size dependence, originating from the microscopic features in metallic structures, is absent in carbon nanostructures. Therefore, our current work not only demonstrates the capability of nano-HIAM in fabricating high-strength nanostructures but also advances the fundamental understanding of feature-dimension effects in AM nano-architected metals. While size effects have been extensively studied in nanocrystalline metals through conventional manufacturing methods, our work represents **the first comprehensive study of the feature-dimension-dependent mechanical behavior of AM metals at nanoscale** to the best of our knowledge.

We have included the relevant literature in the revised manuscript on pages 2 and 12:

“...Emerging nano-AM technologies, including two-photon-lithography-based 3D printing (18, 19) ...”

“Our work ... achieving specific strengths that are...10 times higher than all other reported additively manufactured Ni nanolattices (18, 19) ...”

3. On Page 3, Line 97, the authors mention that the TPL printing speed was “increased by 10x”. It would be helpful to provide the actual printing speeds used for comparison to better quantify this improvement.

Reply 3-3: Thank you for asking for clarification. We have specified the printing speed in the Methods section. The laser rastering speed of 500-1500 $\mu\text{m/s}$ was increased to 7500-15000 $\mu\text{m/s}$. Increasing the laser rastering speed while keeping the other parameters constant is reducing the effective laser dosage and reducing the degree of polymerization. We have observed the benefit of higher laser speed in mitigating local heating and preventing bubbling, but overly increasing the laser speed causes (1) the collapse of as-printed polymeric structures due to low mechanical strength at low polymerization, (2) the collapse of as-calcined NiO structures due to insufficient amounts of infused Ni, (3) distortion of X-Y plane feature aspect ratio due to limited X-Y stage motor moving speed.

4. On Line 99, the manuscript claims that this method ensures “thereby ensuring its reliability, precision, and throughput for producing large, complex 3D geometries”. This statement may be somewhat overstated. It would be useful to clarify what is meant by “large” in this context, especially in relation to industrial scalability.

Reply 3-4: We thank the reviewer for this valuable suggestion. In our original statement, “thereby ensuring its reliability, precision, and throughput for producing large, complex 3D geometries”,

the term “large” refers to 3D nano-architected structures that are large **relative to** the basic “building blocks” such as monolithic Ni nanopillars. The intended implication is the capability to handle geometric complexity at the nanoscale, rather than scaling up nanostructures to macroscale dimensions where conventional metal additive manufacturing methods are typically employed to fabricate metallic metamaterials. We have revised the statement to avoid confusion as follows.

“...thereby ensuring its reliability, precision, and throughput for producing **upscaled, complex 3D geometries that are composed of simple “building blocks”**...”

5. First, how many samples were tested for each feature size in Figure 2? Was there any notable variability in the results? Second, in Figures 2B and 2C, clear structural failure modes are shown. However, on Page 5, Line 142, the authors mention: “We were able to easily identify these samples by their signature of having visual defects (e.g., cracked or wrinkled walls) and low compressive stiffness during initial loading and not include them in the analysis”. This raises concerns regarding repeatability and precision. A discussion on how these exclusion criteria affect the statistical robustness and reproducibility of the compression may be needed.

Reply 3-5: We thank the reviewer for this insightful question. In Figs. 2D and E, we have shown the results of all the tested samples with varying feature dimensions for each type of nano-architecture to investigate the feature-dimension-dependence of strength. Due to factors including the larger focusing size along Z direction in two-photon lithography laser, linear shrinkage of the nanostructures after reduction, and so on, the as-printed structures show some deviations from the as-designed structures (more details in Reply 2-11 and revised SI). From the distribution of feature strength, $\sigma_{feature}$, we observe some variation for samples with similar feature size. This variation could be due to the statistical distribution of concentrated porosity in periodic nanolattices and the local microstructure variation in spinodal-like architectures. We appreciate your understanding and consideration of the challenges in nanoscale fabrication and testing.

Regarding the statement “We were able to easily identify these samples by their signature of having visual defects (e.g., cracked or wrinkled walls) and low compressive stiffness during initial loading and not include them in the analysis”, only 3-4 data per architecture-type were excluded from the analysis where obvious visual defects were found in the nanolattice and the strength was significantly lowered. This treatment shouldn’t affect our subsequent analysis of the feature-dimension dependent behavior, which originates from the evolution of microscopic features rather than large structural fabrication defects in those excluded samples. We have revised the manuscript on page 5 to rationalize this treatment as follows:

“...This treatment excludes the few outliers with apparently large fabrication defects and allows us to focus on the **intrinsic size-dependent materials deformation behavior of nano-architected structures from nano-HIAM...**”

6. The finite-element simulation results are meaningful; however, compared to previously reported micro-architectures built via 3D printing in the literature, the findings for the nano-architectures shown in Figure 4 and Figure S6 do not appear to offer new insights.

Reply 3-6: Thank you for the opportunity to further clarify the novelty and implications of our multi-hierarchy FEM analysis. We acknowledge that FEM simulations have been widely employed to investigate mechanical properties, including elastic modulus and strength, of architected structures. In this work, our multi-hierarchy FEM investigates the deformation of nano-architected structures from the unit cell level (Fig. 4A and Fig. S6) up to the structural level (Figs. 4B and 4C). There are two important insights from this FEM analysis: (1) the different features of stress distribution from simulations of unit cells of octahedral, Schwarz-P and spinodal-like nanolattices contribute to the reduced feature-dimension dependence of ultimate strength in non-periodic architectures (spinodal-like nanolattices) compared to periodic nanolattices (octahedral and Schwarz-P nanolattices) and (2) the reduced model incorporating material parameters extracted from nanopillars and structural-level information on the distribution of nanoporosity, can quantitatively match the scaling factor of ultimate strength from experiments, confirming a nanoporosity-governed deformation mechanism in those additively manufactured nano-architected structures. In particular, the physics-based material models adopted in current FEM simulations of octahedral nanolattices (Fig. 4C) consider the important size-dependent strength and nanoporosity distribution, which is absent in previous analyses of micro-architectures and is the unique feature of the nano-architectures presented in current work. Through the physics-based multi-hierarchy FEM analysis, we reveal the feature-dimension dependence and the fundamental deformation mechanism in nano-architected structures, advancing the digital mechanical validation of nanoscale additively manufactured products for applications in emerging nanotechnology.

7. On Page 12, Line 313, it states “In the meso-to-macroscale regime of metallic lattices, with feature dimensions of 30 μm to 2 mm, there exists a “smaller is weaker” trend”. However, this should not be considered a universal rule. Numerous studies have demonstrated exceptions across various metallic alloys. For example, in titanium alloys, powder bed fusion-built micro-lattice Ti6Al4V has shown a specific strength of approximately 326.6 MPa/(g/cm³), which is about 1.7 times higher than that of bulk Ti6Al4V (180 MPa/(g/cm³)), as reported in the literature “Ultra-high specific strength Ti6Al4V alloy lattice material manufactured via selective laser melting, 2022”.

Reply 3-7: We thank the reviewer for this valuable question. Our original statement is based on the observation that in multiple material systems including steels and Ti-6Al-4V (Fig. 5A), the structures produced by conventional metal additive manufacturing methods experience a reduced specific strength when the feature dimension is reduced to μm scale, which is likely due to the compromised structural integrity and increased manufacturing defects in these conventional additive manufacturing processes. In *Materials Science and Engineering: A* 840 (2022), 142956, the enhanced specific strength is due to the design of the lattice structure, and the feature dimension

is ~1 mm as shown in the table below. Therefore, these structures are considered relatively large and are not affected by the increased manufacturing defects, typically presented at smaller feature dimensions.

[Figure Redacted]

8. On Page 13, Line 363, the statement “These structures surpass the fabrication precision and structural complexity limitations of all existing metal additive manufacturing methods today” appears to be somewhat overstated, considering both the authors’ previous work and findings reported by other research groups.

Reply 3-8: Thank you for the comment. As discussed in more details in Reply 3-1 and Reply 3-2, our current work of nano-HIAM significantly advances nanoscale metal additive manufacturing with enhanced capability in handling structural complexity and fabricating nano-architectures with superior mechanical strength, compared to our previous work and works from other research groups. We have revised the manuscript on page 13 to avoid confusion as follows:

“...These structures surpass the fabrication precision, structural complexity and mechanical robustness limitations of prevailing metal additive manufacturing methods(18, 26–33, 36–38)...”