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Near-ideal compressive strength of nanoporous silver composed of nanowires



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ABSTRACT

Nanoporous materials exhibit promising applications in energy storage, catalysis, and sensing. They are typically synthesized by dealloying, a costly and environmentally detrimental technology, valid only within a narrow compositional range of alloys. Surmounting these disadvantages, we assembled nanoporous silver materials via bottom-up nanoscale joining of nanowires, a technique also suitable for other metals. Furthermore, the resulting nanoporous materials exhibit an unprecedented, near-ideal compressive yield strength (~2.6 GPa). Such an ultra-high strength, however, does not belong to the nanoporous materials composed of nanowires with the minimum length in our samples, challenging the smaller-is-stronger tenet. According to molecular dynamics simulations, such a strength degradation as nanowires shorten is attributed to the internal compressive stress arising from the five-fold twins within nanowires. Such internal stress maximizes at the center and diminishes near free surfaces, making the center part harder to compress than that adjacent to the free surfaces. The volume fraction of the latter increases as the nanowire shortens, diminishing the overall Young's modulus. For nanowires having aspect ratios smaller than six, a reduced Young's modulus lowers the yield strength since the yield strain is independent of aspect ratios. However, if the aspect ratio exceeds six, compression induces bending, reducing both yield strength and yield strain. Overall, this work not only provides new physical insights on the structure-mechanical property relationship for nanoporous silver, but also paves a new way for bottom-up synthesizing nanoporous metals with ultra-high compressive strength efficiently, economically, and environmental-friendly.

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1. Introduction

Characteristic of an open sponge-like morphology, nanoporous metals have abundant voids and enlarged specific surface area [1]; both features enhance their catalytic and chemical activities, making them well-suited in energy storage [2,3], catalysis [4], and

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sensing [5,6]. Besides superior functional characteristics, nanoporous metals also integrate various advantageous structural properties. For example, the high porosity makes nanoporous metals ultralight, allowing ease of transportation and assembly; due to their high compressibility and fracture toughness, nanoporous metals can deform to various customer-made shapes without being damaged [7]. Hence, nanoporous metals also have exciting structural application prospects in 3D graphene synthesis [8], catalyst architectures [9,10], and hierarchical electrode [11].

Conventionally, nanoporous metals are synthesized by dealloying. Based on the difference in the corrosion resistance of the alloyed components, dealloying has successfully generated

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nanoporous Pt [12], Pd [13], Au [14], Ag [15], and Cu [16]. However, the preparation of alloys is time-consuming and costly. Also, the heavy metal ions left in solutions after dealloying are harmful to the environment. Furthermore, dealloying can produce nanoporous metals from alloys only within a narrow compositional range [17–19]. Below the parting limit of the less noble element in the alloy, such as 60 at% silver in Ag-Au [20], only surface dealloying occurs, enriching the surface with the more noble element and eventually passivating the surface. On the other hand, beyond the upper limit, such as 80 at% silver in Ag-Au [20], dealloying results in disconnected remains and significant volumetric shrinkage [18]. Accordingly, what is highly desirable is an environmental-friendly, efficient, and economical fabrication of nanoporous metals, a technique which can produce nanoporous structures with a much wider range of porosity and less volumetric contraction than that generated via dealloying.

Besides, the yield strength of nanoporous metals increases evidently as the diameter of ligaments reduces to the nanoscale [21–24]. However, smaller is not always stronger. For example, when the average grain size in nanocrystalline metals is in the deep sub-micrometer regime (~10 nm), further reducing the grain size switches the dominant deformation mechanism from dislocation glide to diffusional creep [25] and grain boundary sliding [26], degenerating the yield strength. So far, such an optimal dimension of nanoporous metals corresponding to the maximum yield strength has yet to be experimentally confirmed. Since decreasing the diameter of the ligament does not affect the average grain size, the underlying mechanisms governing such an optimal dimension in nanoporous metals should be different from that in nanocrystals.

Furthermore, previous studies on nanoporous metals have observed that not only the yield strength but also the fracture mode depends on the length of the ligament [21,23]: short ligaments exhibit ductile failure and serrated stress-strain curves, while long ligaments deform via extreme shear localization and finally fail abruptly [27]. However, the ligaments in dealloying nanoporous metals have a constant aspect ratio (length/diameter) independent of the porosity [27,28], limiting a systematic investigation on the influence of the aspect ratio of the ligaments on mechanical properties of nanoporous metals.

This work proposed an innovative, environmental-friendly, bottom-up synthesis of nanoporous silver, a method that can be generalized to other metallic systems. Next, the compressive yield strength of the resulting nanoporous silver was measured by nanoindentation. Then, a new scaling relationship was proposed to quantify the aspect-ratio effect on the compressive yield strength of the nanoporous silver. Finally, molecular dynamics simulations provided new physical insights underlying the structuremechanical property relationship between the aspect ratio and the compressive yield strength of nanowires.

2. Methodology

The silver nanowires used in this study were synthesized in polyol solution based on a chemical method from the literature [29,30]. All the chemicals and reagents used in this study were of analytical grade and in the as-received form without any purification. First, silver nanowires were prepared in a polyol using 330 mg polyvinylpyrrolidone (PVP, $(C_6H_9NO)_n$, K25, M.W. = 24000, Alfa Aesar) and 12.5 mg silver chloride (AgCl, Alfa Aesar) mixed with 40 ml ethylene glycol (EG, Fisher Chemical). The mixture was heated in a round-bottom flask between 160 and 170 °C [31,32]. Then, we dissolved 110 mg silver nitrate (AgNO₃, Alfa Aesar) in 10 ml ethylene glycol liquid while intensely stirring the solution for 0.4–4 h. By varying this reaction time, seven groups of nanowires with different lengths are obtained (named as NWs in Table 1). For

comparison, silver nanoparticles (denoted as NPs in Table 1) were also prepared with the same method described in previous works [33,34]. Next, the as-synthesized silver nanomaterials were condensed by centrifugation at 4000 rpm using a 50 ml centrifuge pipe. The clean supernatants were removed from the centrifuge pipe by a pipette, leaving the highly concentrated silver nanomaterial pastes at the bottom. The excess organic compounds in the silver nanomaterial pastes were washed and removed by deionized (DI) water and repeated centrifugation. The washed pastes, each with a 0.6 M concentration and 0.2 ml volume, were then drop-cast at 60 °C using a 1-ml syringe onto Ag-coated cylindrical copper substrates, 4 mm in diameter and 1 mm in height. These samples were covered by glass slides and hot pressed at 150 °C for 30 min with 50 N force using a micro-tensile tester working in compression mode (Instron 5548, UK). This 30-min nanojoining process creates metallic bonds at the contact points between nanowires by diffusion, without affecting the microstructure and shape of nanowires. Last, rather than mechanical polishing, hot pressing was used to create a flat surface to avoid damage of the silver nanostructures on the free surface.

The mechanical properties of nanoporous silver materials were tested with a depth-sensing nanoindenter (Hysitron-Tribo indenter) equipped with a Berkovich tip calibrated on fused silica. Indentations were performed on a flat surface in a load-controlled mode at a constant loading rate of $60 \,\mu N/s$ and different loads of 300, 600, and 900 µN. For each loading magnitude, five indents were collected on each sample. The relative density of nanoporous materials was calculated via image processing using Image] software (version 1.43u); it was also estimated by the quantitative relationship between the relative measured modulus and relative density. The relative densities obtained from these two different methods are compared and discussed in the results section. The indentation hardness, *H*, according to the Tabor relation [35,36], was assumed to linearly correlate with the strength of materials, σ , as $H \approx 3\sigma$, which is a general rule for ductile, work-hardening materials.

To elucidate experimental results, we performed large-scale MD simulations of uniaxial compression tests on five-fold twinned silver nanowires using LAMMPS code [37] with a time step of 2 fs. The interatomic interaction was described by the embed-atommethod (EAM) potential developed for silver [38], which could accurately reproduce the mechanical properties of silver and has been used in previous simulations on five-fold twinned silver nanowires [39,40]. It has been suggested in previous studies that Ag nanowires synthesized in polyol solution using a chemical method have truncated decahedron structure with [110] growth direction and bounded by five [100] planes [34,41–43]. This structure has been used in other studies on silver nanowires as well [39,44–50].

Our nanowire models have the same thickness of 20 nm but different lengths, resulting in different aspect ratios ranging from 1 to 69. Prior to loading, all samples were relaxed by conjugategradient energy minimization, followed by an isobaric relaxation increasing the temperature from 0 K to 300 K in 100 ps and further relaxing the model at 300 K for another 100 ps. Then, straincontrolled compression was emulated at a strain rate of $5 \times 10^8/s$ at 300 K. Strains were applied by dilating the model along the axial direction of nanowires and then applying an equal affine transformation to atomic positions. Next, the deformed models were subjected to isothermal-isobaric (NPT) relaxation in the transverse directions, but the cell dimensions in the axial axis were held constant during the relaxation to preserve the overall strain. The relaxation time thus determines the strain rate. Atomic configurations are visualized using OVITO package [53], which utilizes the Common Neighbor Analysis (CNA) [51] to classify defects in crystals [52] and the Dislocation Extraction Algorithm (DXA) to identify

Table 1	
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The relative density and label of each nanoporous materials. The thickness, length, and the aspect ratio (AR) of the component nanowires within different nanoporous materials are also listed.

Category	NP	NW1	NW2	NW3	NW4	NW5	NW6	NW7	
1D nanostructure									
Thickness (nm)	76.1 ± 10.9	40.8 ± 1.7	50.2 ± 4.5	46.3 ± 4.3	51.5 ± 4.7	62.0 ± 7.3	91.4 ± 12.1	64.4 ± 7.2	
Length (µm)	-	1.0 ± 0.3	3.4 ± 0.6	7.0 ± 1.4	9.4 ± 3.2	13.3 ± 2.8	21.6 ± 7.9	29.3 ± 5.4	
Aspect Ratio/AR	1	24	68	151	182	215	237	455	
Nanoporous materials									
Relative Density	95%	39%	58%	45%	73%	48%	49%	52%	
Label/npm-AR	npm-1	npm-24	npm-68	npm-151	npm-182	npm-215	npmi-237	npm-455	

dislocations [54]. The input file used in this paper can be found in the supporting information.

nanowires, we can design the geometrical parameters of the ligament within the nanoporous structures, an advantage not available with the traditional dealloying methodology.

3. Results

3.1. Morphology of the silver nanostructures composed of nanowires

Morphologies of the silver nanoporous materials composed of nanowires are illustrated in Fig. 1. The longer the nanowires, the harder they can assemble seamlessly, leaving a substantial volume of internal pores, thereby forming nanoporous structures. Without the preparation of alloys, this method is more economical and efficient than dealloying, and no extra environmentally detrimental metal ions are left in solution. Moreover, via assembling nanoporous silver structures through the bottom-up joining of



Fig. 1. The microstructure of various nanoporous materials containing different nanowires. (a-h) The surface of nanoporous materials. Insets figures exhibit the corresponding morphologies of the component nanowires. For the nanoparticle samples (npm-1) shown in (a), the scale bar represents 200 nm; for other nanowires samples, their scale bars represent 2 μ m.

The geometry parameters of the synthesized nanowires and the resulting nanoporous materials are listed in Table 1. Each nanoporous material is labelled as npm-AR, where AR stands for the aspect ratio of the component nanowires. Every thickness and length of nanowires are averaged over more than 30 samples. Different nanoporous structures are composed of nanowires with varying thicknesses spanning from 41 to 91 nm and lengths ranging from 1 to 29 μ m, resulting in different relative densities. For comparison, nanoparticles with a diameter of 76 nm are also fabricated into a nanoporous structure, with a relative density close to 1.

3.2. The mechanical properties of nanoporous silver

To understand the effect of the dimension of nanowires on the mechanical properties of the nanoporous silver, we measured the hardness via load-controlled nanoindentation at a constant loading rate of $60 \,\mu$ N/s and separate loads of 300, 600 and 900 μ N. Shown in Fig. 2 are the load-depth curves of different samples. The intensity of load, loading cycles, and loading rates have negligible effects on mechanical properties (more details in supporting information). However, while the loading curves for npm-1 are relatively smooth, fluctuations occurred on that of other samples, indicating a crescendo plastic deformation singularity as the aspect ratio of the component nanowires increases. Because of its highest relative density, npm-1 has the highest modulus among all structures, while for other structures their modulus is reduced due to their lower relative density.

The reduced Young's modulus can be quantitatively correlated with the relative density, ρ / $\rho_{\rm bulk}$, where $\rho_{\rm bulk}$ is the density of silver. According to Ashby's theory for foam and lattice [55], the relative Young's modulus, E/E_{bulk} , where E_{bulk} is the Young's modulus of polycrystalline silver, has a linear relationship on logarithmic scales with $\rho / \rho_{\text{bulk}}$, whose slope is one for stretch-dominated deformation $(E/E_{\text{bulk}} \propto \rho/\rho_{\text{bulk}})$ and two for bending-dominated deformation $(E/E_{\text{bulk}} \propto (\rho/\rho_{\text{bulk}})^2)$. The latter was used in a previous study to calculate the relative density of porous silver produced by inkjet printing [56]. As shown in Fig. 2e, the measured relative density from the image analysis of our nanoporous structures are close to the relative densities predicted by the equation describing the bending-dominated deformation, $E/E_{\text{bulk}} \propto (\rho/\rho_{\text{bulk}})^2$, while the relative densities predicted by the relationship suitable for idealstretch deformation $(E/E_{\text{bulk}} \propto \rho/\rho_{\text{bulk}})$ are only one-quarter of that from image analysis, obviously deviating from the experimental measurement of relative densities. Therefore, the deformation of our nanoporous structures is bending-dominant, and the relative density obtained from the image analysis is reasonable and consistent with that predicted by relative Young's modulus.

Aside from the relative Young's modulus, the relative strength, $\sigma / \sigma_{\text{bulk}}$, is also plotted against the relative density, $\rho / \rho_{\text{bulk}}$ (Fig. 2f).



Fig. 2. Relative-density effects on the mechanical properties of silver nanoporous materials. Loading-depth curves of different nanoporous materials are shown in (a) npm-1, (b) npm-24, (c) npm-68 and (d) npm-215. Relative modulus and relative strengths are plotted against relative density on logarithmic scales in (e) and (f), respectively. The relative strengths predicted by the conventional Gibson-Ashby relation are also plotted for comparison.

σ_{bulk}, the measured strength of bulk silver determined by nanoindentation, are 520 ± 30 MPa, 604 ± 51 MPa, and 557 ± 24 MPa for 300 μN, 600 μN, and 900 μN loads, respectively. For all different loads, the relationship between σ/σ_{bulk} and ρ/ρ_{bulk} is linear on logarithmic scales, with a slope of 1.5, which is an inherent feature of the bending-dominated deformation of metallic foams under compression [55]. Despite having the same slope, the relative strengths measured from our samples are much higher than that predicted by the traditional Gibson-Ashby relation for metallic foams [57,58] (Fig. 2f). Such deviation could be attributed to the size-effect on yield strength, which was ignored in the conventional Gibson-Ashby relation.

To determine the geometry effect of the component nanowire on the yield strength, we factored out the influence of the relative density according to the Gibson-Ashby relation, $\sigma_{npm} = \sigma_{bulk} / (\rho / \rho_{bulk})^{3/2}$. As may be seen in Fig. 3a, σ_{npm} increases not only with the decreasing thickness, like that of other dealloying nanoporous metals [21–23], but also as the component nanowire shortens. Specifically, npm-68 and npm-24 exhibit very high yield strengths of 2.6 GPa and 2.4 GPa, respectively, more than twice of that of nanoporous silver produced by inkjet printing (~1.1 GPa) [56], even close to the theoretical ideal shear strength, $G/2\pi$, of polycrystalline silver (4.8 GPa when G is 30 GPa [59]).

Generally, the yield strength increases by decreasing the dimension of nanowires, following the famous "smaller is stronger" tenet. However, the function of the yield strength with the length of nanowires exhibits two regimes (Fig. 3a): for nanowires with lengths larger than 5 μ m, the shorter the nanowires, the larger the σ_{npm} ; when the length is less than 5 μ m, further reducing the length diminishes the yield strength, challenging the well-known smaller-is-stronger tenet. To take into consideration the effect of both thickness and length on yield strengths, we plotted the strength as a function of aspect ratios in Fig. 3b. Clearly, two different behaviors of yield strength are illustrated: the yield strength first increases with decreasing aspect ratios, peaking at npm-68, but then declines. Specifically, both the thickness and length of the nanowires within npm-68 are larger than that of npm-24, but the maximum yield strength belongs to npm-68, not



Fig. 3. Nanoporous silver materials assembled by nanowires have near-ideal compressive yield strengths. The measured yield strength is shown as a function of the (a) thickness, length, and (b) aspect ratio of component nanowires. Indents on (c) npm-1, (d) npm-24, (e) npm-68 and (f) npm-215 are exhibited with scale bars representing 500 nm.

npm-24. Also, npm-151 is composed of nanowires having a smaller diameter than that within npm-68, but its yield strength is only approximately 1.5 GPa, 50% of that of the npm-68. These results indicate that not only diameter (or thickness) but also the length or the aspect ratio are essential in determining the mechanical properties of nanoporous silver materials.

3.3. The plastic deformation mechanism of nanoporous silver

Like the yield strength, the plastic deformation mechanism of nanoporous silver materials also depends on the aspect ratio of the component nanowires. On the indent of npm-1 (Fig. 3c), as well as the line profile in Fig. 4a crossing the indentation area, we find plowing near the indent, a result of severe plastic deformation. Such protrusions, however, disappear at the indentation of other samples ((Fig. 3d–f) composed of nanowires with larger aspect ratios. Instead, at the center of the indentation buckling of nanowires emerges (Fig. 4b–d). Buckling during compression also occurs in aluminum foams, whose compressive stress-strain curves are characterized by an initial linear elasticity followed by a roughly constant stress plateau that continues up to very large strains (~60%) beyond which the stress increases sharply [60,61]. Therefore, the occurrence of buckling cannot be used as a criterion for the ductility of a material.

Different plastic deformation behaviors can also be reflected in the number and depth of jumps on the load-controlled forcedisplacement curves, labelled as pop-in events. As the aspect ratio increases, the pop-in events increase in depth but decrease in number. For example, Fig. 4e—h shows the loading-depth curves of npm-1, npm-24, npm-68, and npm-215, respectively, as well as the slope of the loading curve (named as the derived depth change). On the loading curves of npm-1 are numerous faint pop-ins with 4 nm in depth. As the aspect ratio increases from 4 to 68, the depth soars to 25 nm while only 2 jumps remain, implying a crescendo deformation singularity, evidenced by the local buckling observed in npm-24 (Fig. 4b), npm-68 (Fig. 4c), and npm-215 (Fig. 4d). Accordingly, unlike yield strength that has two opposite relationships with aspect ratios, increasing the aspect ratio of the component nanowire always promotes bending and buckling during compression.

4. Discussion

4.1. The relationship between the strength and dimension of nanowires

Although the conventional Gibson-Ashby model can capture the dependence of relative yield strength on relative density, it underestimates the relative yield strength due to the size effects on yield strength. Furthermore, the size effect described by the traditional Hall-Petch relation only involves the diameter of nanowires, neglecting the aspect-ratio effect. Thus, we propose a new scaling relation between yield strength and geometrical parameters of nanowires, including not only the thickness but also the length of nanowires as follows,

$$\sigma_{\rm nw} = c_{\rm s} \left(\sigma_0 + k_{\rm nw} (L \times T)^{-\frac{1}{4}} \right) \tag{1}$$

 $c_{\rm s}$ is 0.3 for metallic foam [57,58]. σ_0 is the yield strength of bulk silver (~54 MPa [48]). $(L \times T)^{-1/4}$ is a function of both length *L* and diameter (or thickness) *T*, replacing the term $T^{-1/2}$ used in the traditional Hall-Petch relation. When *L* is equal to *T* for spherical or cubic nanoparticles, the expression $(L \times T)^{-1/4}$ degrades to the traditional Hall-Petch relation $T^{-1/2}$. Interestingly, the compressive



Fig. 4. The plastic deformation mechanism of nanoporous materials composed of nanowires having different aspect ratios. (a) The gray value of the SEM image in subfigure represents the position of the surface near the indentation of npm-1. Zoom-in images of the indent areas on npm-24, 68, and 215 are shown in (b-d); scale bars represent 200 nm. The loading-depth curve and derived depth changes of the curve are shown in (e), (f), (g), and (h) for npm-1, npm-24, npm-68, and npm-215, respectively.

yield strength of nanowires within nanoporous materials increases approximately linearly with $(L \times T)^{-1/4}$ (Fig. 5a) when the aspect ratio is larger than 68. For nanoparticles, the Hall-Petch coefficient $k_{\rm np}$ (~0.35 MPa m^{1/2}) is consistent with that of silver microwires (0.36 MPa m^{1/2}) [62], polycrystalline silver [62], and sintered silver nanoparticles [63]. However, for our nanoporous silver composed of nanowires, $k_{\rm nw}$ is 0.84 MPa m^{1/2} as a result of the bendingdominate compression to be discussed in section 4.3.

4.2. Theoretical yield strength determination via molecular dynamics simulations

Although both the length and thickness of nanowires are considered in the empirical Eq. (1), the mechanism by which the aspect ratio influences the compressive yield strength remains elusive. In the nanowire-based nanoporous structure, dislocation glide is restricted in each nanowire. In addition, during the nano-indentation test, nanowires parallel to the indented surface are only locally compressed, while the nanowires perpendicular to the

indented surface are compressed or bended entirely. Thus, the aspect-ratio effect on the compressive yield strength is mainly associated with nanowires perpendicular to the indented surface.

The physical insight of the aspect-ratio effect on the yield strength was studied by large-scale Molecular Dynamics simulations of uniaxial compression tests of nanowires. The nanowires simulated has a five-fold twin, (001) side surface, and [110] axial direction (as shown in the inset image of Fig. 5b). All nanowires in simulations have an identical thickness of 20 nm but various aspect ratios ranging from 1 to 69. A strain-controlled loading is applied along the axial direction at 300 K and a constant strain rate of 5×10^8 /s.

As the stress-strain curve in Fig. 5b shows, the stress abruptly decreases after reaching the yield strength, which has two different relationships with aspect ratios. If the aspect ratio is smaller than 6, the yield strength increases by rising the aspect ratio (Fig. 5c), and the yield strain remains approximately constant (right panel of Fig. 5b). In contrast, if the aspect ratio is larger than 6, both the yield strength and the yield strain decrease as the aspect ratio increases.



Fig. 5. The breakthrough of the "smaller is stronger" tenet in nanoporous silver materials. (a) The scaling relationship between the strength of the nanoporous materials composed of nanoparticles or nanowires and the two-dimensional size $(L \times T)^{-1/4}$; (b) The simulated stress-strain curves and the yield strength and strain of silver nanowires with different aspect ratios. Inset figure is the structure of a five-fold twin nanowire. (c) Yield strength vs. aspect ratio of the 20 nm-thick nanowires in simulations. For each geometrical parameter, we simulated five samples with identical geometry but different atomic velocity initialization to quantify the error bar due to thermal fluctuations. (d) The relationship between simulated yield strength of individual nanowire or nanoparticle and the two-dimensional size $(L \times T)^{-1/4}$.



Fig. 6. The physical insights of the aspect-ratio effect on the compressive yield strength and deformation mechanism of nanowires. (a) The longitudinal section of the nanowire with an aspect ratio of 1; each atom is colored by its local internal stress in the axis direction. (b) Stress-strain curves of different parts of nanowires with an aspect ratio of 1. (c) The stress at the yield point for different parts of nanowires as a function of the aspect ratio of nanowires.

Such a transition in the aspect ratio-yield strength relationship implies a transition in deformation mechanisms as the aspect ratio varies. According to the theory of elastic stability [64], the critical compressive load to initiate one-point bending is $P = \pi^2 E I / l^2$, where *E*, *I*, and *l* are the Young's modulus, the moment of inertia, and the length of nanowires, respectively. Given a constant diameter of nanowires, the aspect ratio determines the deformation mechanism. When the aspect ratio is less than 6, *P* is larger than the compressive yield strength of nanowires; thus, no bending mode can occur, and the maximum stress obtained on the stress-strain curve is the compressive yield strength. When it is larger than 6, however, the critical load for one-point bending becomes smaller than the compressive yield strength, so compression induces bending. This time, the critical stress for buckling decreases with the increasing aspect ratio.

The transition in the deformation mechanism from compression to bending is also reflected in the relationship between the yield strength and $(L \times T)^{-1/4}$ (Fig. 5d), similar with that shown in Fig. 5a. For comparison, we also performed simulations of the uniaxial compression of five-fold twinned nanoparticles with varying thicknesses but the same aspect ratio of one. This time, the yield strength of different nanoparticles decreases monotonously with increasing thicknesses (Fig. 5d), indicating the aspect ratio is the main reason for the invalidation of the smaller-is-stronger tenet.



Fig. 7. Dislocation generation in silver nanowires. (a) Nucleation and propagation of a Shockley partial dislocation on a {111} plane. (b) Dislocation generation on the transverse cross-section of nanowires. TEM observation at the indent area of (c) npm-24, (d) npm-1, and (e) npm-215, with scale bars of 2 nm, found surface steps, Shockley partial dislocations, and screw dislocations.

Underlying the aspect-ratio effect on the yield strength is a unique structure of our nanowires: five-fold twin boundaries. Since the angle between two twin boundaries in a five-fold twin structure (72°) is larger than that in an unstretched twinned structure (70.53°), intrinsic stress is generated to accommodate the angular difference [39,40,50]. The resulting internal stress along the loading axis (σ_{vv}) is compressive but gradually decreases to zero near free surfaces (Fig. 6a). Based on such internal stress distribution, we divide the nanowire into three regimes: top, bottom, and middle. The top and bottom portions contain atoms within 50 Å to the top and bottom surfaces, respectively; the rest atoms belong to the middle region. Due to the surface effect, the average internal compressive stress of both the top and bottom regimes are smaller than that of the middle part. Since a larger internal compressive stress in the middle region reduces the interatomic distance, so the middle part becomes harder to compress than the top and bottom regions. For example, in Fig. 6b, we plotted the local stress-strain curves of the top, bottom, and middle regions of a nanowire with an aspect ratio of 1. At the same applied strain, both the stress and the slope of the stress-strain curve for the middle parts are higher than those for the top and bottom parts. Quantitively, the stressstrain curve of the middle and top parts can be fitted as $\sigma = 1279.8\epsilon^2 + 80.389\epsilon$ and $\sigma = 1135.3\epsilon^2 + 71.465\epsilon$, respectively. If we fit the stress-strain curves of the top and bottom regions starting from an initial compressive stress 1.3 GPa, the resulting fitting equations ($\sigma = 1296.7\epsilon^2 + 60.373\epsilon$) is very close to that of the middle part, which estimates the difference in the residual stress between the middle region and that of the other two regions.

Due to the effect of internal stress, the local yield strength of the middle part σ_{middle} (8.1 GPa) is always larger than σ_{top} and σ_{bottom} (7.1 GPa) (Fig. 6c), and the local stress of different parts at the yield point is independent of the aspect ratio unless the compression-induced bending occurs. Nonetheless, as an averaged value over all atoms, the yield strength of the whole nanowires still increases from 7 to 8 GPa (Fig. 5c and d) as the aspect ratio rises from 1 to 6. This is because the volumetric fraction of the middle regime becomes increasingly significant within nanowires having a larger aspect ratio. The aspect ratio corresponding to the maximum yield strength (~6), however, is much smaller than the experimental value (~68). This difference is attributed to the fact that nanowires in the nanoporous silver are assembled randomly, so the effective aspect ratio of the ligament in the nanoporous structure should be much smaller than that of the component nanowires.

The internal stress discussed above will become the main factor determining yield strengths only when the yield strain is independent of aspect ratios. Due to dislocation starvation in nanowires, plastic deformation is triggered by the dislocation generation from the junction between side surfaces and twin boundaries, as indicated in Fig. 7a exhibiting a Shockley partial dislocation on its gliding plane. Such a generation process is also delineated in Fig. 7b on the cross-section of the same nanowire. At a strain of 5.2%, a chain of atoms at the intersection of free surfaces and twin boundaries



Fig. 8. Compression induces bending of nanowires. (a) to (d) exhibit the local stress distribution along the longitudinal direction of nanowires. Stress and strain concentration due to bending occurs in nanowires with aspect ratios of 15 (c), and 30 (d). (e) The structure of a nanowire with an aspect ratio of 50 after yielding. Many bending points are induced by compression.

migrate in a direction perpendicular to the loading axis (Fig. 7b). Then another chain of atoms below the free surface starts moving, forming a Shockley partial dislocation on a {111} plane shown in Fig. 7a (more details on the dislocation generation mechanism are in supporting information). Such dislocation generation from the surface is supported by the TEM images showing the steps at the free surface of npm-24 after the compressive test (Fig. 7c). Besides, partial dislocation pairs inside nanowires are highlighted in Fig. 7d, with an interval of 3.8 nm, close to the theoretical value, 3.5 nm [65]. Screw dislocations are also generated, causing the Moiré pattern shown in Fig. 7e. Overall, unlike yield strength, which is an averaged effect of the whole structure, plastic deformation is a result of local atomic movements, involving only a few atoms on free surfaces. Therefore, during pure compression, the yield strain is independent of both the internal stress and the aspect ratio of nanowires.

4.3. The compression-induced bending of silver nanowires with different aspect ratios

Besides the one-point bending discussed in section 4.2, according to the theory of elastic stability [64], the critical compressive load initiating the bending of nanowires with *n* bending points is $P = n\pi^2 EI/l^2$. When the aspect ratio is less than 6, even the critical load for one-point bending is larger than the compressive yield strength of nanowires. Consequently, bending cannot occur

(Fig. 8a and b); dislocations are homogeneously distributed throughout the whole nanowire; the local stress at the yielding strain of nanowires is approximately equal everywhere (Fig. 8b). When the aspect ratio is larger than 6, however, the critical load for one-point bending $(P = \pi^2 E I/l^2)$ becomes smaller than the compressive yield strength, so compression induces bending, concentrating dislocations at the bending point (Fig. 5c and d). For example, when the compressive strain of a nanowire with an aspect ratio of 15 reaches 5.4%, the concentration of dislocations at the bending point reduces the local stress to 3 GPa, while the stress in other parts remains at 8 GPa (Fig. 8c). The averaged stress of the entire nanowire, as a result, decreases significantly. Furthermore, as the critical load initiating one-point bending reduces with the increasing aspect ratio, so does the difference between different bending modes $(\pi^2 EI/l^2)$. Consequently, in nanowires with aspect ratios >30, as soon as the nanowire starts deforming, many-point bending occurs (Fig. 8d and e), intensifying the local curvature of nanowires, concentrating dislocations at more bending points, and thereby further diminishing the critical stress for buckling (Fig. 5c).

5. Conclusions

In conclusion, we fabricated nanoporous silver structures via nanojoining of nanowires, a technology which is more economical and environmentally friendly than the traditional dealloying method and can be applied in other metallic systems as well. Furthermore, the compressive yield strength of the nanowires within the nanoporous silver material can reach 2.6 GPa, close to the theoretical shear strength of polycrystalline silver. However, the nanowires corresponding to the maximum yield strength do not have the minimum length in our samples, challenging the smalleris-stronger tenet. Such violation is attributed to the lengthdependent internal stress distribution that makes longer nanowires harder to compress. Overall, this study provides new physical insights into the structural-mechanical properties relationships for nanoporous structure, as well as novel paradigms for the design and synthesis of hierarchical nanoporous materials for various applications and strength enhancement.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.actamat.2019.05.011.

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