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Strong Hall–Petch Type Behavior in the Elastic Strain Limit of Nanotwinned Gold Nanowires

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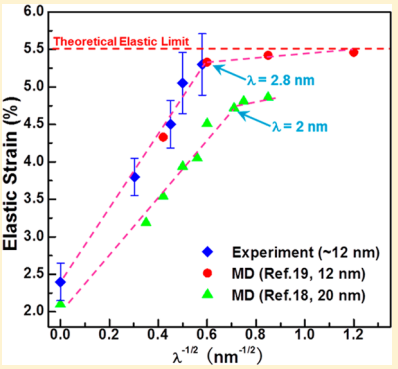
Supporting Information

ABSTRACT: Pushing the limits of elastic deformation in nanowires subjected to stress is important for the design and performance of nanoscale devices from elastic strain engineering. Particularly, introducing nanoscale twins has proved effective in rising the tensile strength of metals. However, attaining ideal elastic strains in nanotwinned materials remains challenging, because nonuniform twin sizes locally affect the yielding behavior. Here, using in situ high-resolution transmission electron microscopy tensile testing of nanotwinned [111]-oriented gold nanowires, we report direct lattice-strain measurements that demonstrate a strong Hall–Petch type relationship in the elastic strain limit up to 5.3%, or near the ideal theoretical limit, as the twin size is decreased below 3 nm. It is found that the largest twin in nanowires with irregular twin sizes controls the slip nucleation and yielding processes in pure tension, which is in agreement with earlier atomistic simulations. Continuous hardening behavior without loss of strength or softening is observed in nanotwinned single-crystalline gold nanowires, which differs from the behaviors of bulk nanocrystalline and nanotwinned-nanocrystalline metals. These findings are of practical value for the use of nanotwinned metallic and semiconductor nanowires in strain-engineered functional microdevices.



KEYWORDS: *Nanotwin, metallic nanowires, ideal elastic strain limit, Hall–Petch hardening, elastic strain engineering, in situ transmission electron microscopy*

The upper limit of strength in materials, or ideal theoretical strength, corresponds to the highest applied stress beyond which a perfect, defect-free crystal becomes irreversibly deformed at 0 K,[1,2] accompanied by the ideal elastic strain limit that can be applied to this crystal.[1,3,4] Previous studies have shown that elastic straining in surface-dominated materials such as thin films and nanowires can fundamentally change their electronic and phonon transport behaviors,[1,5−8] which is relevant for diverse applications from elastic energy storage and conversion to functional microelectromechanical devices. Attaining the elastic strains close to the ideal theoretical limits of materials, however, remains an outstanding challenge because pre-existing or new slip defects become active under stress.[9]

Previous experimental and theoretical studies have reported nanocrystalline or small-volume materials with ultrastrength resulting from a decrease in either grain size[1,10] or crystal dimension,[1,11,12] respectively. Recently, high elastic deformation has been measured in single-crystalline Cu nanowires[3] and submicron-sized metallic glasses[4] but these materials were found to yield at significantly lower limits than their ideal theoretical values.[1,13] Moreover, nanocrystalline metals exhibit significant Hall–Petch strengthening with decreasing grain size, until the onset of grain boundary (GB)-mediated plastic deformation processes leads to a softening, that is, the loss of strength, below a critical grain size.[14] The introduction of coherent interfaces such as nanoscale twin boundaries (TBs) has shown further strengthening in bulk nanocrystalline metals[15−17] and metallic or semiconductor nanowires,[13,18,19] yet bulk nanotwinned metals still exhibit softening mechanisms because of dislocation processes from incoherent GBs or preexisting TB defects.[16,20,21] In ultrathin nanotwinned nanowires containing no incoherent GBs, however, continuous Hall–Petch hardening without softening is expected up to the ideal elastic limit. Recently, a reversible shear strain as high as 34.6% was observed in Ni nanowires with irregular twins under bending deformation.[22,23] An earlier atomistic study by Deng and Sansoz[24] on nanowire models with regular twin sizes

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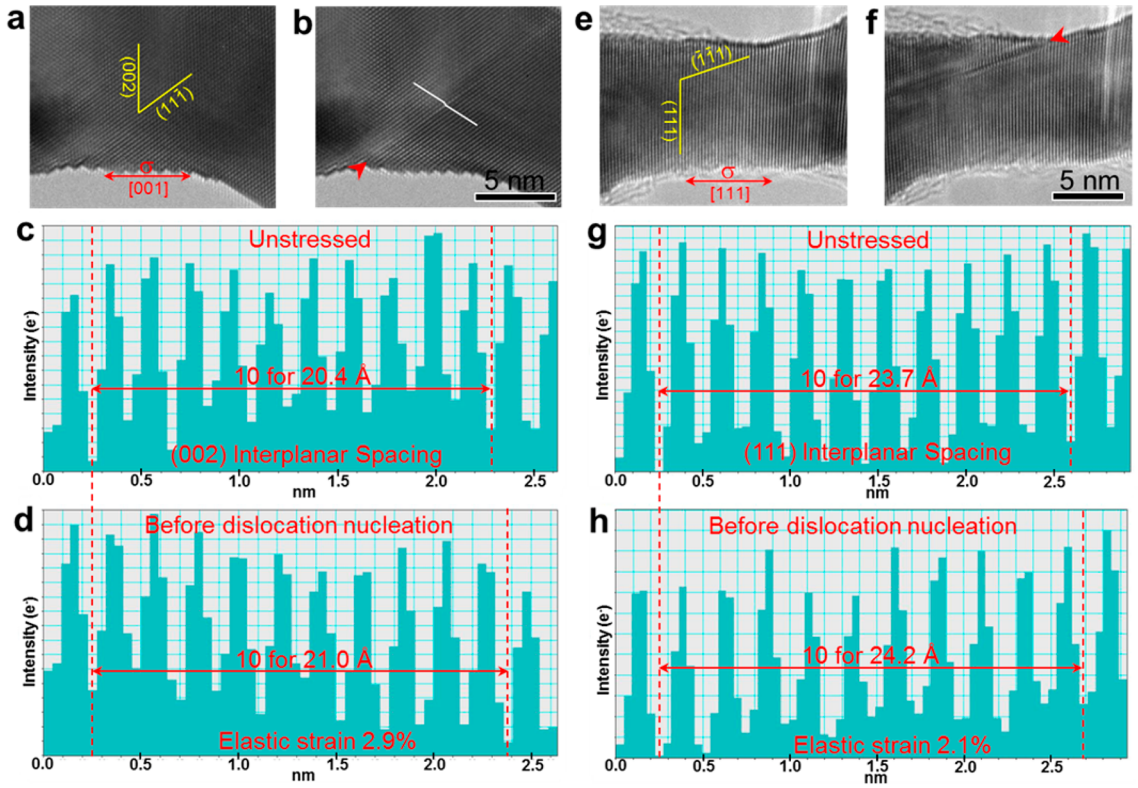

Figure 1. Tensile deformation and change of lattice spacing in single-crystalline Au nanowires with either [001] or [111] direction, respectively. (a,b) Deformation of single-crystalline Au nanowire (15 nm in diameter) under [001] tensile loading. (c,d) The evolution of [002] interplanar spacing under [001] tensile loading, (c) unstressed and (d) elastically deformed before a dislocation is nucleated in the nanowire. (e,f) Deformation of single-crystalline Au nanowire (10 nm in diameter) under [111] tensile loading. (g,h) The evolution of [002] interplanar spacing under [111] tensile loading, (g) unstressed and (h) elastically deformed before a dislocation is emitted in the crystal. The 20-planes measurement of the lattice spacing at the same location of nanowire shows no difference from the 10-planes measurements, as shown in Supporting Information Figure S2.

suggested that the maximum tensile elastic strain before surface nucleation of dislocation linearly scales with $1/\lambda$, where λ is the twin thickness. However, experimental verification of such theoretical predictions has been difficult because real TBs in as-grown nanotwinned nanowires are irregularly spaced,[8,13,19,25−29] which likely disrupts the local elastic limit along the nanowire axis. Therefore, in order to close the gap between experiments and simulations it is critically important to locally examine both yielding and hardening processes in nanotwins where crystal slip is first initiated.

Here, in situ high-resolution transmission electron microscopy (HRTEM) tensile testing and atomic-scale lattice strain measurements were used to study the twin-size dependence of elastic strain limit in nanotwinned Au nanowires with a particular focus on the direct relation between the initiation of crystal slip and local twin spacing. Our major result shows a strong TB-dependent Hall−Petch type behavior in the elastic strain limit of single-crystalline metallic nanowires by twin size reduction. Near-ideal theoretical elastic strain is demonstrated in deformed Au nanowires when the largest twin thickness is reduced below 3 nm. Also, fundamental differences in Hall−Petch type behavior between nanotwinned single-crystalline nanowires and nanocrystalline materials are discussed.

In-situ tensile deformation of ultrathin Au nanowires was performed under HRTEM, see experimental details in Supporting Information. Single-crystalline and nanotwinned Au nanowires with either [100] axis or [111] axis were investigated in this study. The ideal elastic strain limit corresponds to the maximum elastic strain that the lattice of a defect-free crystal can withstand. In perfect crystals without deformation, all atoms rest at their equilibrium lattice positions. Under mechanical loading, as the elastic strain rises, the atoms of the crystal deviate from their equilibrium positions, which is reflected by a change of interplanar spacing of the lattice planes perpendicular to the loading axis. At the yield point, the elastic strain energy accumulated in the crystal lattice can be partially released by the nucleation of lattice defects,[30] leading to yielding and permanent plastic deformation. As such, the elastic strain limit of Au nanowires is defined as the lattice strain at the onset of plastic deformation, which can be determined at atomic scale by analyzing the difference in interplanar spacings between the unstressed state and deformed state immediately before the nucleation of the very first lattice defect in in situ TEM.[3,9] For a direct comparison of lattice spacings between different nanowire structures, measurements over only 10 atomic planes were made due to the small twin thickness for nanowires with high density twins.

Figure 1 presents the tensile deformation of [001]-oriented and [111]-oriented Au nanowires without twins, along with measurements of the change in (002) interplanar spacing $d_{(002)}$, and (111) interplanar spacing $d_{(111)}$, respectively. Upon deformation, the tensile strain increases linearly with the deformation time (Supporting Information Figure S1). At a critical value of elastic strain, the initial yielding of these single-crystalline nanowires occurs via the surface nucleation of partial dislocations, as pointed out by the red arrows in Figure 1b,f. In an unstressed [001]-oriented nanowire, we find $d_{(002)}$ = 2.04 Å, which agrees well with the value in perfect FCC gold crystals,

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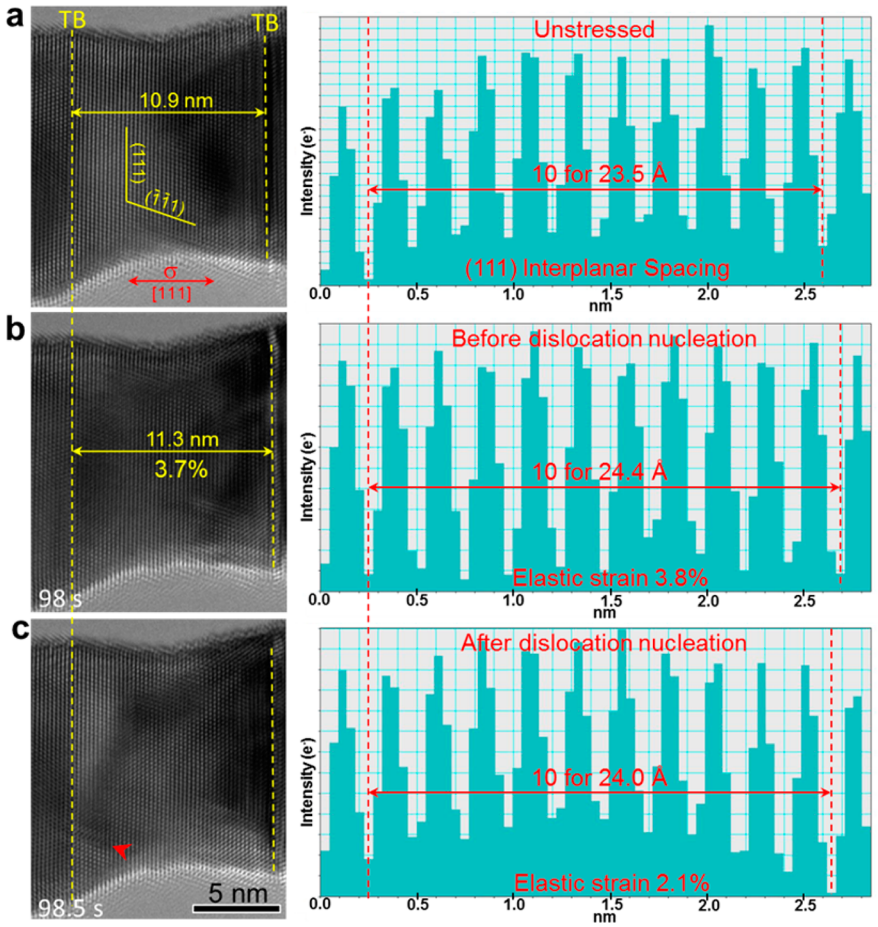

Figure 2. Change of twin thickness and lattice spacing in a nanotwinned [111]-Au nanowire with diameter of 13 nm and large twin thickness. Twin spacing and lattice spacing increase elastically under the [111] tensile loading. (a) The twin thickness and measured lattice spacing in unstressed Au nanowire. (b) The twin thickness and measured lattice spacing in the deformed Au nanowire before the nucleation of the first dislocation. (c) Nucleation of a dislocation from free surfaces, propagating into the crystal, and blocked by an adjacent TB, as pointed out by a red arrow. Measured lattice spacing suggests partial release of strain accumulated in the lattice.

and $d_{(002)}$ = 2.10 Å before the surface nucleation of the first partial dislocation (Figure 1c,d), respectively. This corresponds to a maximum elastic strain of 2.9% before yielding, close to the value of 3% observed in other experimental studies.[30] Likewise, it is found that $d_{(111)}$ increases from 2.37 to 2.42 Å in a twin-free [111]-oriented nanowire immediately before the yielding, which results in an elastic strain limit of 2.1%, as confirmed by both short and long distance measurements of lattice spacing (Figure 1g,h and Supporting Information Figure S2). This is close to the value of 2.4% for [111] single-crystalline Au nanowire predicted by MD simulations[18] and 2.1% for Au nanowire with a single TB.[31] Repeated measurements conducted at different locations of Au nanowires showed no difference. Therefore, the above results suggest that the maximum elastic strain in single-crystalline Au nanowires remain significantly lower than the theoretical limits of 7%[32] and 5.5%[18] in ⟨100⟩ and ⟨111⟩ Au nanowires, respectively.

Figure 2 shows the deformation of a [111]-oriented Au nanowire with low TB density. During deformation, the TB spacing can also serve as a gage length for the evolution of elastic strain in nanotwinned nanowires. For reference, the twin size highlighted in Figure 2a is 10.9 nm in the unstressed nanowire. As the tensile strain increases, Figure 2b shows that the same twin extends elastically up to 11.3 nm before yielding, corresponding to an elastic elongation of about 3.7%. Figure 2a,b shows the measurements of $d_{(111)}$ increasing from 2.35 to 2.44 Å before yielding, which corresponds to a maximum elastic strain of 3.8% ± 0.3% (the error represents the variations between repeated measurements at different locations on the nanowires), which is in good agreement with the value obtained from the change of twin thickness. Further loading gives rise to the surface nucleation of a partial dislocation and its subsequent blockage by the closest TB, as shown in Figure 2c. After dislocation nucleation, partial release of elastic strain down to about 2.1% is observed (Figure 2c and Supporting Information Figure S1).

Figure 3 displays the deformation of a [111]-oriented Au nanowire with thinner but irregular twins. Here, special attention is paid to two adjacent twins with different initial thicknesses, 16.4 Å (T_1) and 40.1 Å (T_2), as shown in Figure 3a. Under the current hypothesis, T_2 is expected to yield before T_1. Before yielding, Figure 3b and Supporting Information Figure S1 show that twins T_1 and T_2 undergo elastic deformation and increase in size to 17.2 and 42.0 Å, corresponding to a maximum uniform elastic elongation of ∼4.9% and ∼4.7%, respectively. The elastic strain obtained from the change of interplanar spacing $d_{(111)}$ measured directly in both twins T_1 and T_2 are 5.1% ± 0.4% before the yielding (Figure 3b-c and Supporting Information Figure S3; here the uncertainty was also calculated from the variations between

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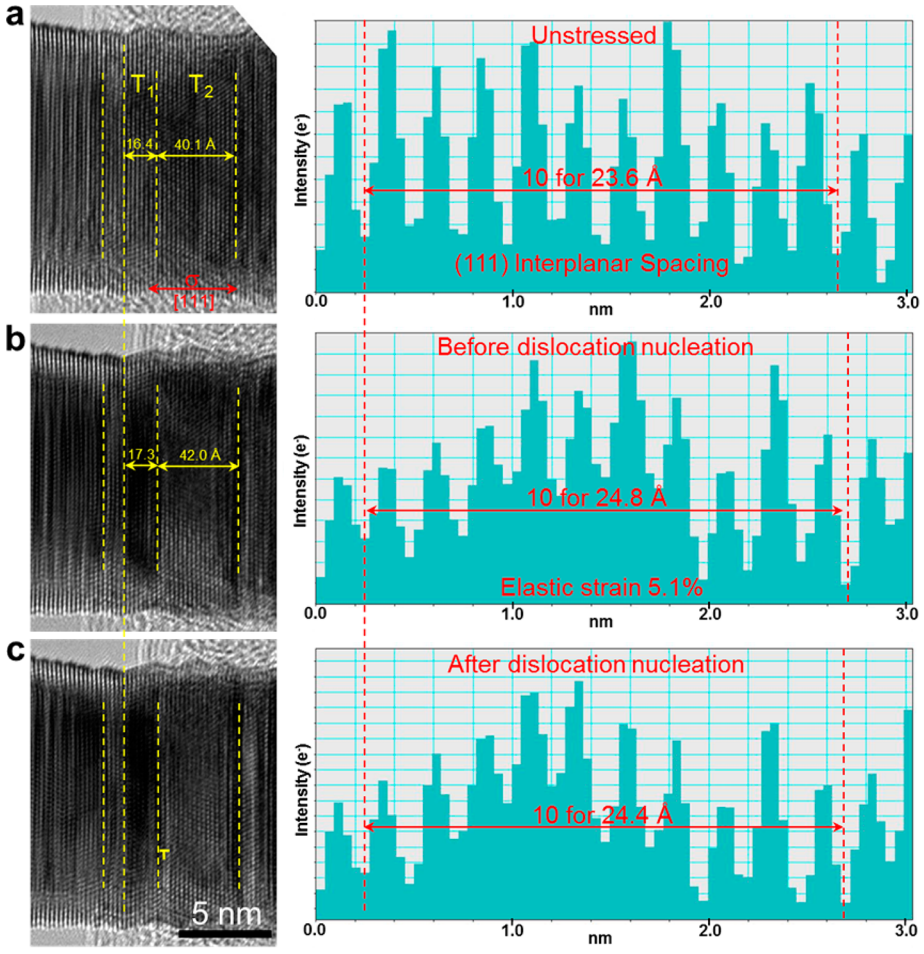

Figure 3. Change of twin thickness and lattice spacing in a nanotwinned [111]-Au nanowire with diameter of 12 nm and smaller twin thickness. Two twins are marked out and labeled as T_1 and T_2, respectively. The lattice spacing, $d_{(111)}$, were measured in T_2. (a) Unstressed Au nanowire with twins. (b) Deformed Au nanowire before the nucleation of the first dislocation. (c) Dislocation first nucleates in T_2, thus inducing the yielding.

repeated measurements conducted at different locations), which are in good quantitative agreement with the twin spacing measurement though some discrepancies exist between them due to the measurement error. Such elastic strain is much higher than that in [111]-oriented Au nanowires with no twin (Figure 1) and low twin density (Figure 2) and approaches the theoretical limit of 5.5% for gold deformed along the [111] direction.[18] The above results show a strong Hall–Petch type behavior in the elastic strain limit of nanotwinned nanowires, caused by the reduction in twin spacing. With further loading, a partial dislocation is nucleated in T_2, as shown in Figure 3c, accompanied by the release of lattice strain (Supporting Information Figure S1). This observation supports the atomistic prediction of Deng and Sansoz[33] showing that the elastic limit in nanowires with irregular twins is controlled by the nanowire segment with the largest twin spacing.

The dependence of elastic strain limit on twin size in [111]-oriented Au nanowires is represented in Figure 4, which includes both the results from present experiments and earlier atomistic simulations.[18,19] We note that all atomistic models used in Figure 4 consisted of {111} faceted Au nanowires of 12 and 20 nm in diameter, which is similar to the nanowires (in the 10–13 nm diameter range) used in the current experiments. Both the experiment and simulation results show that for twin thicknesses larger than λ = ~3 nm, the elastic strain limit can be linearly related to $\lambda^{-1/2}$, which suggests a strong

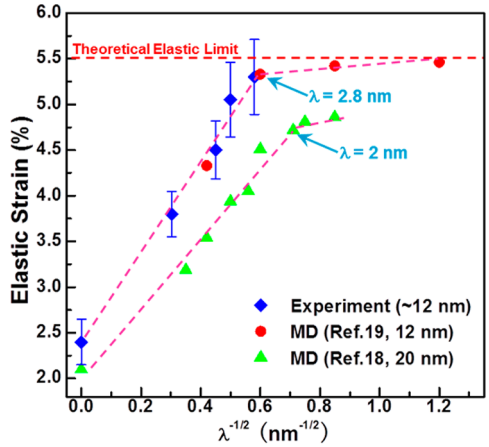

Figure 4. Hall–Petch type behavior in the elastic strain limit of nanotwinned [111]-Au nanowires. The red dots represent the MD simulation data from ref 19 with the nanowire diameter of 12 nm; while the green triangles represent the MD simulation data from ref 18 with the nanowire diameter of 20 nm. Both experiment and simulation results show that for twin thicknesses larger than 3 nm, the elastic strain limit can be linearly related to $\lambda^{-1/2}$, suggesting a strong Hall–Petch type hardening. Near ideal elastic strain limit is achieved as the twin size decreases below 2.8 nm.

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Hall−Petch type behavior due to the reduction of twin size. A salient feature of this figure, however, is the continuous hardening observed in nanotwinned single-crystalline [111] Au nanowires, up to the near ideal limit of 5.3% when λ is around 3 nm. This behavior differs markedly from that of bulk nanotwinned metals where TB-dependent Hall−Petch strengthening is followed by a significant softening as the twin size decreases, leading to the loss of Hall−Petch strengthening in bulk nanotwinned nanocrystalline materials.[16,20,21] The softening observed in bulk nanotwinned materials was attributed to the presence of GBs and preexisting TB defects that lead to dislocation nucleation, GB-mediated plastic deformation and detwinning processes at high stresses.[20,21] Although TBs can block the motion of dislocation contributing to the strain hardening, the present experiments have only focused on deformation mechanisms before and at yielding that did not involve any dislocation pile-ups. Therefore, the twin-size dependent Hall−Petch behavior observed in the elastic strain limit of nanotwinned nanowires is different from the classical Hall−Petch effect in which significant defect−defect and defect−interface interactions are assumed. The low dimensionality of ultrathin nanowires makes lattice defects less stable, and therefore more likely to be eliminated by free surfaces, leaving place to alternative plasticity mechanisms such as surface dislocation nucleation.[33] Here, the strong twin-size dependence of the elastic strain limit is more likely related to an image effect where TBs exert a repulsive force on gliding dislocations, scaling as $1/\lambda$.[33] As a result, a higher stress is required for the emission of dislocations from sources in those dislocation-free nanotwinned nanowires

Furthermore, Figure 4 also shows that the slope of the Hall−Petch type relationship found from our experiments agrees well with that predicted by atomistic simulations, which indicates the same deformation mechanism. In Figure 4, a critical separation size of ∼2.8 nm is observed in nanowires with the diameter of ∼12 nm. As the twin size decreases below 2.8 nm, the elastic strain limit is found to converge asymptotically to the ideal theoretical limit of 5.5% without much change with the twin size. This is because below the twin size of 2.8 nm, the mechanism of plastic deformation shifts from heterogeneous nucleation from free surface to homogeneous nucleation in the limited space between twin boundaries inside the nanowire.[19] The stress required for the homogeneous nucleation is close to the ideal shear stress of materials, which does not depend on twin size. The experimental results are found to support this theory. Moreover, the elastic strains of nanotwinned nanowires are affected by the nanowire diameter, as demonstrated by the MD simulation data in Figure 4. As the nanowire diameter increases to 20 nm, the critical twin size for this transition decreases to ∼2 nm. This can be attributed to the fact that the critical resolved shear stress for surface nucleation of nanotwinned nanowire decreases significantly with the increase of diameter.[33] This suggests that a smaller critical twin size will be observed as the nanowire diameter increases. However, this phenomenon relates to the size-dependent crystal plasticity effectsrather than an intrinsic twin size effect.

In summary, by using lattice spacing measurements under in situ tensile deformation at atomic scale, it was found that the elastic strain limit of ultrathin Au nanowires can be systemically improved through microstructure design by twin size reduction due to strong Hall−Petch type hardening effects. Near-ideal elastic strain limit up to ∼5.3% was measured in nanotwinned [111]-Au nanowires for the smallest twin size. These findings hold great promise for elastic strain engineering of nanotwinned metallic and semiconductor nanowires,[8,13,19,25−29,34−36] in which desirable physical and chemical properties can be obtained when deformed at their ideal theoretical strain limit.[1] This study therefore opens new opportunities for the use of nanotwinned nanowires in energy conversion and functional microdevice applications.

■ ASSOCIATED CONTENT

Supporting Information

The supporting figures showing the time-dependent elastic strain evolution, 20-plane measurement of lattice spacings, change of lattice spacing $d_{(111)}$ in T_1 of the nanowire in Figure 3, and experimental methods. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.nanolett.5b00694.

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Notes

The authors declare no competing financial interest.

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