Effects of Sample Geometry on the Uniaxial Tensile Stress State at the Nanoscale

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ABSTRACT

Uniaxial compression of micro- and nanopillars is frequently used to elicit plastic size effects in single crystals. Uniaxial tensile experiments on nanoscale materials have the potential to enhance the understanding of the experimentally widely observed strength increase. Furthermore, these experiments allow for investigations into the in-strength and to help to study tension-compression asymmetry. The sample geometry might influence mechanical properties, and to investigate this dependence, we demonstrate two methods of uniaxial nanotensile sample fabrication. We compare the experimentally obtained tensile stress-strain response for cylindrical and square nanopillars and provide finite element method simulation results and discuss the initiation of plastic yielding in these nanosamples.

KEYWORDS

nanoscale, plasticity, mechanical, tension

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1. INTRODUCTION

Continued miniaturization of devices used in bio- and nanotechnology requires a thorough understanding of crystal deformation at small scales. In recent years, uniaxial compression experiments on micro- and nanosized samples have been used to study the size effect of materials [1–8]. These studies show that single crystals are capable of sustaining significantly higher stresses than bulk crystals when their diameters are reduced below 10 microns. The discrete dislocation framework is a multiscale model that has recently been used to investigate the deformation in nanosamples [9, 10]. Molecular dynamics simulations of single crystalline Au nanowire deformation [11] predict a strength increase as well as tension-compression asymmetry and claim that the surface tension of nanometer-sized structures leads to a domain of compressive forces in the core. The applied stress would compensate the surface-induced compressive stress in tension, but the surface-induced stress in the core helps to apply stress during compression. It is proposed that the core compression leads to a higher magnitude of yield stress than in compression for samples of identical size. Classical tensile experiments on copper, iron, and silver pristine whiskers in the micron regime [12] have shown that the flow stress can reach values close to the theoretical strength of the material. Recently, Kiener et al. [13] performed tensile experiments on square cross-sectional samples milled out of Cu single crystal via focused ion beam (FIB). They observed a power law size dependence with the exponent of −0.4 and stresses significantly below the theoretical strength of Cu. The differences in sample geometry might have contributed to such disagreement. Pillars with a circular cross section have lower surface-to-volume ratios compared to those with a square cross section, which might lead to additional surface-induced stresses in the pillar core for square-shaped samples. The additional compressive stresses might, in turn, contribute to the tension-compression asymmetry and cause higher tensile yield stresses for the square shaped samples. Because the surface/volume ratio decreases with increasing sample dimensions, the effects of surface stress on the overall flow stress become negligible for macrosized samples. Atoms at the corners in the square cross-sectional geometry are less confined by their neighbors, leading to a significantly lower energy for homogeneous defect nucleation from the corner than for nucleation on a flat surface, as shown by the computations of Zhu et al. [11] and Li [14]. This difference in free energy leads to the lower yield stresses for corner dislocation nucleation (observed in rectangular cross-sectional samples) than at a homogeneous surface (circular cross-sectional pillars). In this work, we report experimental and finite element method (FEM) results investigating the influence of square versus round cross-sectionally shaped samples on uniaxial tensile deformation behavior of single crystal ⟨001⟩-oriented gold nanopillars. We also describe new fabrication methods for creating uniaxial tension samples via FIB.

2. NUMERICAL STUDIES

We utilize FEM simulations to quantify the elastic stress distribution within the sample to optimize the sample geometry such that the inhomogeneities in stresses and strains within the sample are minimized. The stress concentrations might lead to localized incipient plasticity, and the experiment would not be representative of the general material response at small scales. Flow stresses are often assumed to be homogeneous for nanopillars compressed along high-symmetry crystallographic orientations. However, geometrical constraints at the top and base of the pillar may lead to the elevated local stresses. At the base, the deformation is constrained by the substrate, and friction between the pillar and the flat punch indenter tip restricts the deformation of the pillar top. We use the conventional eight-node linear finite elements (ABAQUS C3D8) and simulate the deformation of square and round cross-sectional samples on a substrate. These elements allow us to quantify the geometrical effects, while allowing for time-efficient simulations. Due to the double symmetry in the sample geometry, it is sufficient to model a quarter of the pillar/substrate combination with 10 three-dimensional elements in the radial direction and 30 in the axial. We assume linear isotropic elasticity to evaluate the overall stress state at 10% strain. Isotropic properties of Au are represented by Young’s modulus 79 GPa and Poisson’s ratio 0.42. Since the constraint at the pillar top cannot be determined unambiguously, we do not include it in the model. However, the findings due to the constraint pillar base can used to infer a
Figure 1 shows the von Mises yield stress distribution inside the pillars of the two described geometries, which is the most appropriate criterion for the incipient yield position in the continuum framework. While the stresses in the two geometries are nearly identical in the plane of symmetry, we find that the maximum axial stress in the circular column is 1.14 GPa, while that in the square one is 1.80 GPa and is localized in the corner of the square sample decaying away from it. We find that the substrate constraint leads to the formation of higher local stresses in the square-shaped sample, even though the average applied stress is the same. Assuming that the yield is governed by the nucleation of dislocations from the region where the stress state exceeds that of the von Mises yield criterion, this inhomogeneous elastic stress distribution leads to an inhomogeneous dislocation nucleation from the areas of elevated shear stresses, assuming that the dislocation sources are homogenously distributed and require equal activation energy. The post yield stress-strain behavior of the nanopillars consists of multiple discrete slip events indicative of dislocation avalanche propagation, rendering the continuum theory inapplicable to describe this behavior. To model plasticity in these nanoscale volumes with free surfaces, powerful 3-D mesoscopic simulations, such as discrete dislocation simulations [9, 10, 15–20] as well as molecular dynamic simulations [11], are better suited to investigate the post yield behavior of nanopillars.

3. SAMPLE DESIGN AND FABRICATION

Kiener et al. [21] have also reported a method for tensile sample fabrication from single crystal wire tips, which were also orthogonally etched in the FIB. Their experimental procedure requires subsequent remounting of the entire sample onto a different sample holder for each tensile experiment, result-
ing in frequent misalignment issues and activation of preferential single slip even for high-symmetry-oriented samples. The experimental approach described here is an improvement over the previously reported work because it allows for (1) the fabrication of several samples in the same bulk single crystal, (2) the compression and tension testing of the same sample, and (3) a greatly reduced misalignment between the sample and the tensile grips. Figure 2 shows the sequence of tensile sample fabrication steps performed in FEI Nova Nanolab 200 FIB. The resulting structure (Fig. 2(d)) is a circular pillar with rectangular handles on either side. The handle thickness, $b$, was designed to be equal to the pillar radius, $r$, and their height, $h$, was optimized to induce lower shear stresses in the handles than in the sample. Since the maximum shear stresses in the sample are induced at the angle $\alpha = 45^\circ$ from the axial direction, and the area of the handles is required to be greater than the cross-sectional sample area, $2bh > \pi r^2 \cos \alpha$, the handle height has to satisfy the following criteria:

$$h > \frac{\pi}{\sqrt{2}r} \approx 2.22r$$  \hfill (1)

The samples were prepared by the use of a FIB from a $\langle 100 \rangle$-oriented Au single crystal, which was cleaned with acetone and isopropyl alcohol (IPA) and then mounted on a scanning electron microscope (SEM)-sample holder with carbon paint. In the first fabrication step, we use a perpendicular incident beam and a 7 nA current at 30 kV acceleration voltage to mill out a large ring with the outer diameter sufficiently large to allow the tensile grips to be lowered into the cut out volume. The diameter of the remaining material is 2 µm for $\sim 300$ nm-diameter samples and 5 µm for 1 µm-diameter ones. In the second step (Fig. 2(b)), we lower the current

**FIGURE 2.** Schematic of sample fabrication in focused ion beam: (a) milling out ring, leaving core in the middle behind; (b) define milling pattern on surface of core using polygons; (c) mill out center piece and handles; and (d) make undercuts using an i-beam at $45^\circ$.
to 0.1 nA and define the handles by using the polygon drawing tool in the FIB software. To achieve a smooth surface on the central part of the sample, 15 polygons on either side were milled in parallel. Special attention is paid to the raster direction such that the polygons are milled from the outside to the inside, which reduces the redeposition of atoms. In the third step (Fig. 2(c)), the sample is tilted to 45°, and equally sized rectangles are used to make the undercuts with 10 pA ion current. Special attention is paid to align the rectangular undercuts at the sample side and at the underside of the handles. The rectangular shaped pillars are prepared in a similar fashion. Initially, a large pedestal in the center is created. Using rectangular milling patterns, a rectangular pillar is produced. Afterward, the single crystal is transferred onto a 86°-wedged sample holder, and the undercuts are milled. Postfabrication, the samples are inspected and measured by SEM.

4. RESULTS AND DISCUSSION

We fabricated nanopillars with diameters ranging from ∼ 300 nm to ∼ 900 nm out of Au bulk single crystal in (100) orientation. SEM images of the smallest samples of each geometry are shown in Fig. 3. The handles are 2 × 1.4 µm, and the pillar tensile gage length is 4.74 µm for the circular cross-sectional pillar. The rectangular one generally has a gage length of 3 µm, and the handles extend ∼ 1 µm on either side and have a height of 1–1.5 µm. Tension experiments were carried out inside the SEMentor, a one-of-a-kind in situ mechanical deformation instrument with a specifically fabricated tensile/compressive tip, as shown in Fig. 4. The SEMentor provides simultaneous visualization and video capturing capability with mechanical data collection. The applied force was determined by the feedback loop such that the displacement rate is kept constant at 2 nm/s. The applied forces and the measured displacements are recorded instantaneously throughout the test, while the deformation is observed and recorded in the SEM. Still images are extracted during the plastic deformation of the pillar. These images are used to directly measure the gage length, \( L \), which is assumed to be equal to the plastic length, \( L_p \), since elastic deformation is significantly smaller than the plastic contribution. Assuming plastic volume conservation, the true stress is evaluated by \( \sigma = P L_p / (A_0 L_0) \), where \( P \) is the applied force, and \( L_0 \) and \( A_0 \) are the initial gage length and cross-sectional area, respectively. The obtained stress-strain data are not averaged since the discrete slip events, which characterize the plastic deformation, are different from sample to sample due to the differences in the initial dislocation distribution and the stochastic nature of the deformation. During compression, the statistical distribution of slip burst size follows a power law, as de-

![Image of tensile sample at 45° tilt angle. The sample has a diameter of 334 nm and a length of 4.74 µm in the gage length](image_url)
Figure 4. Tensile/compressive grips milled into a diamond nanoindenter tip

Derived from the statistical model of Csikor et al. [22] and the experimental investigations of Dimiduk et al. [23] and Brinckmann et al. [6]. This power law appears to be independent of the material and the sample size. Detailed statistical studies of the tensile behavior will be executed in the future. The experiments indicate that the slip lines form primarily in the middle section of the sample, with clear, evenly distributed offsets. No indication of deformation near the substrate was observed, most likely because plasticity at the nanoscale does not necessarily initiate at the regions with highest von Mises stress but is rather controlled by the local environment in the sample. Figure 5 compares the representative stress-strain curves for a circular and for two rectangular-shaped pillars of similar dimension. We evaluate an effective diameter \( d_{\text{eff}} = \sqrt{4/\pi a_1 a_2} \) for the rectangular pillars to compare pillars of equivalent cross sections. Both pillar shapes exhibit similar elastic stress-strain responses, and in the following plastic regime, it appears that below \( \sim 7\% \) strain, the flow stress of the rectangular pillar is more continuous compared with the discrete burst-ridden signature of the circular one. The difference between the two shown rectangular-shaped samples is evidence of the experimental scatter observed in nanopillar experiments. At higher strains, however, the stress-strain curves are similar, consisting of numerous discrete slip events intermitted by elastic loading segments. This signature is similar to the previously reported compression results (e.g., [2]). Our gold nanopillars show pronounced ductility in tension, with the deformation intentionally aborted at \( \sim 30\% \) strain, where necking occurred prior to fracture.

5. CONCLUSIONS

Our studies indicate that the pillar geometry appears to play a role in the deformation response of nanosized samples, as has been suggested by molecular dynamics simulations [11, 14]. These simulations show that defect nucleation from the corners requires less activation energy than nucleation from a flat surface. The FEM results presented here reveal regions of confinement-induced higher localized stresses in the rectangular-shaped pillars compared with the circular ones at the same overall applied stress. Our uniaxial tensile experiments on nanosized crystals indicate that unlike cylindrical ones, rectangular cross-sectional pillars exhibit a continuous increase in flow stress during early stages plasticity. However, at 10% strain, a value commonly used for comparison of the flow stress...
FIGURE 5. Stress-strain curves for circular and square cross-sectional samples under tensile deformation. The cross-sectional sample has a diameter of 692 nm. The effective diameter $d_{eff} = \sqrt{4/\pi}a_1a_2$ of the rectangular cross sections with the side lengths $a_1$ and $a_2$ is defined for the rectangular sample for pillars of different diameters, the geometry influence on flow stress becomes insignificant. Further tensile experiments on pillars of different dimensions are necessary to study the size dependence. Furthermore, to shed more light on the discrete events that govern plastic deformation, atomistic simulations of nanosized pillars are necessary.

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