Plastic deformation of indium nanostructures

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ABSTRACT

Mechanical properties and morphology of cylindrical indium nanopillars, fabricated by electron beam lithography and electroplating, are characterized in uniaxial compression. Time-dependent deformation and influence of size on nanoscale indium mechanical properties were investigated. The results show two fundamentally different deformation mechanisms which govern plasticity in these indium nanostructures. We observed that the majority of indium nanopillars deform at engineering stresses near the bulk values (Type I), with a small fraction sustaining flow stresses approaching the theoretical limit for indium (Type II). The results also show the strain rate sensitivity and flow stresses in Type I indium nanopillars are similar to bulk indium with no apparent size effects.

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1. Introduction

The functionality, lifetime, and future commercial success of novel nanoelectronic and nanoelectromechanical devices ultimately depend on a thorough fundamental understanding of the mechanical properties and reliability of nanostructures comprising them. To date, a considerable amount of effort has been dedicated to studying the room-temperature small-scale mechanical behavior of single-crystalline metallic nanostructures [1–22], with homologous temperatures less than 0.5. So far, there have been very few research investigations focusing on low melting temperature nanostructures. The most common technique for assessing small-scale mechanical properties without the presence of strong strain gradients is by uniaxial compressive loading of cylindrical pillars fabricated by focused ion beam (FIB) milling. This approach was first introduced by Uchic et al. [1,2] for pillars as small as ∼5 µm in diameter, and later extended by Greer et al. [6] and others for pillars with sub-micron diameters. Remarkably, all studies on the compressive strength of single-crystalline metal nanopillars with non-zero initial dislocation densities exhibit size effects which manifest themselves as a pronounced increase in yield strength when external dimensions are reduced to the micron and sub-micron scale [23]. Although these and other studies of size-dependent mechanical properties have spurred a non-trivial amount of computational efforts and theoretical discussions, experimental investigations addressing time-dependent deformation in nanometer scale structures remain limited to very few materials (i.e., molybdenum pillars [19,22]).

The main objective of this work is to expand the current understanding in small-scale mechanical properties to the deformation mechanisms governing low melting temperature nanostructures. Specifically, we focus our study on indium, which has a melting temperature of ∼156 °C, corresponding to a homologous temperature of approximately 0.7 in ambient conditions. Herein, the important mechanical parameters of this material are investigated, including the stress–strain characteristics, strain-rate effects on flow stress, and the influence of size on mechanical response. A thorough understanding of indium nanostructure deformation generated in the course of this work will further enrich the current small-scale plasticity state-of-the-art, especially in the area of low melting temperature materials. A further motivation for this work is the significant commercial applications of indium and indium-based alloys in the microelectronics industry. They are widely used as an alternative material in advanced lead-free elemental and alloy solders for microelectronic packaging [24–26]. In addition, novel electroluminescence devices based upon nanostructured indium powders are being developed [27].

Cylindrical indium pillars with sub-micron to nanoscale diameters were chosen for this study because they can be fabricated by the electron beam lithography and electroplating technique [28,29]. Since the homologous temperature of indium at room temperature is ∼0.7, factors that affect thermally activated processes, such as atomic/vacancy diffusion [30] and dislocation climb [31,32], are expected to contribute significantly to the time-dependent mechanical deformation mechanism. Indium has a face centered tetragonal (FCT) crystalline structure with a c/a = 1.075 [33], and...
in bulk is known to be one of the softest metals. Van der Biet and Van der Planken conducted a detailed study to characterize the plastic deformation mechanism of bulk indium single-crystals under tensile stress at 77 K, 195 K, and room temperature [34]. The major observation in their work is that twinning, with the composition plane of (1 0 1), is the prominent deformation mode for nearly all samples. However, the active slip systems depend on the testing conditions. At 77 K, easy glide was observed on (1 1 1) planes, which are the closest packed for this structure. In contrast (0 0 1) and (1 0 1) are the active glide planes in bulk indium deforming at room temperature.

It is important to distinguish the current study from the work of Lucas and Oliver [35], where creep and strain rate sensitivity of bulk indium single-crystals were measured by using nanoindentation with a sharp Berkovich diamond tip. Although nanoindentation is a versatile and powerful technique, it is limited by its ability to only directly measure the hardness of a material. The flow stress of the deformed material may then be estimated indirectly by assuming that measured hardness value for metals is linearly proportional to the yield strength [36,37]. In addition, the yield stress extracted by this method corresponds only to a characteristic strain value which is defined by the centerline-to-face angle of the indenter tip [36,38,39]. These uncertainties and other potential artifacts, such as sink-in, pile-up, and indentation size effects, pose drawbacks to using nanoindentation to obtain stress–strain relationships as a function of strain rates. Finally, the specific nanoindentation experiments conducted by Lucas and Oliver evaluated mechanical properties of indium at the micron scale only, dimensions significantly larger than that of the nanopillars in the current study. In order to accurately characterize the time-dependent mechanical properties of indium at the nanoscale, we conducted uniaxial compression tests on cylindrical indium nanopillars with diameters ranging from 920 nm to 350 nm.

Through these experiments on more than 100 indium nanopillars with different diameters, we observed two fundamentally different deformation mechanisms which govern the plasticity of indium nanostructures. The distinction between the two deformation mechanisms is apparent in the measured flow stress values. We found the majority of indium nanopillars to be relatively weak and plastically deform at engineering stresses near the bulk values of ∼7 MPa (Type I). This bulk value was estimated by using the nanoindentation hardness values reported by Lucas and Oliver [35] under the assumption that hardness is three times of the yield strength [36,37,40]. However, a small fraction of the samples were extremely strong and sustained compressive stresses approaching the theoretical limit of ∼435 MPa, or ∼10% of the indium bulk shear modulus, G = 4.35 GPa [41] (Type II). Results also indicate that the stress–strain response of indium nanopillars exhibiting flow stresses near the bulk value is strongly influenced by strain rate, but display no apparent size-dependence. Scanning electron microscopy (SEM) inspections of compressed nanopillars further reveal that the low strength samples display a deformation characteristic, which resembles material extrusion.

2. Experimental methods

Indium specimens examined in this work were fabricated by using electron beam lithography followed by electroplating to eliminate FIB-induced surface damage often associated with nanopillar fabrication [28,29]. This fabrication method involves lithographic patterning of polymethylmethacrylate (PMMA) resist with electron beam lithography, followed by selective metal electroplating into the prescribed resist template. The electron beam lithography and electroplating approach eliminates the additional complexity of ion effects and excessive pillar tapering, thereby rendering the uniaxial stress–strain curves correct. A schematic representation of the fabrication process flow is shown in Fig. 1. Indium nanopillar arrays were fabricated on silicon substrates with a 20 nm thick titanium adhesion layer and a 100 nm thick gold layer deposited by electron beam evaporation. The conductive gold/titanium seed layer was also used as the cathode in the subsequent electroplating steps. These metalized substrates were then spin coated with various dilutions of 950 kD PMMA dissolved in anisole (MicroChem Corp.). After spin coating, the PMMA layer was cured at 180 °C for approximately 15 min. Arrays of holes were then patterned in the PMMA resist by electron beam lithography using a Leica EBPG 5000+ operating at an acceleration voltage of 100 kV. The exposed pattern consisted of a 2.5 mm × 2.5 mm array of circles with 10 μm pitch. After exposure, the PMMA layer was developed in a 1:3 solution of methylisobutylketone and isopropyl alcohol (IPA) for 60 s, finishing with a 5 s rinse in IPA. Following PMMA development, the resist template underwent a 15 s oxygen plasma descum process to promote homogenous electroplating by increasing the hydrophilic nature of the pore interior [29]. This descum process was performed at room temperature, with a chamber pressure of 102 mTorr, plasma power of 20 W, and oxygen flow rate at 40 sccm. The PMMA etch rate corresponding to these process conditions was approximately 1.67 nm/s. The final nanopillar diameters produced in this work were 350 nm, 560 nm, and 920 nm.
Electroplating was performed under galvanostatic conditions using a two electrode configuration with the indium sulfamate based plating solution (Indium Corporation of America). The gold/titanium layer underneath the resist template acted as the cathode, and a high purity indium metal slab was used as a soluble anode. This sulfamate bath was maintained at ambient temperature and mechanically stirred during the deposition process. The plating process began with a 5 second current pulse at 10.0 mA/cm\(^2\) and then followed by electroplating at 1.0 mA/cm\(^2\). This initial high current step was used to increase the overall sample yield by producing an indium seed layer of less than 25% of the pillar height, promoting homogenous filling into the patterned features. With the processing technique described above, a production yield greater than 80% was achieved for patterns containing \(\sim 920\) nm diameter features. Fig. 2 shows the typical indium pillars fabricated by using the procedure described above with diameters of \(\sim 350\) nm, \(\sim 560\) nm, and \(\sim 920\) nm.

All indium nanopillar samples were annealed at room temperature for a minimum of 15 days prior to mechanical testing to allow for grain growth and defect annihilation. The initial microstructure was characterized and discussed in our previous work\[29\] by using a scanning micro-X-ray diffraction (\(\mu\)SXRD) technique. This technique has been widely used to characterize the microstructure and dislocation dynamics of nanostructures, such as advanced integrated circuit copper interconnect lines\[42,43\] and nanopillars\[29,44,45\]. The details of the \(\mu\)SXRD method have been described elsewhere\[29,42–46\]. The measurement resolution of the \(\mu\)SXRD technique is partly determined by the pixel size of the charge-coupled device camera used to record the Laue diffraction patterns, which corresponds to a resolution of \(\sim 0.02^\circ\) of the Laue diffraction peaks. Typical indium diffraction peaks characterized in this work have full-width-half-max in the range of \(\sim 0.298^\circ\) to \(\sim 0.778^\circ\). The \(\mu\)SXRD results\[29\] indicate that the electroplated indium nanopillar microstructure consists of several large grains at the most, which span the entire nanopillar diameter. \(\mu\)SXRD also revealed that these grains are either pristine, i.e., well annealed and contain no discernable dislocations, or have the initial density of fabrication-induced geometrically necessary dislocations (GNDs) on the order of \(\sim 10^9\) cm\(^{-2}\).

Uniaxial compression tests of indium nanopillars were performed at three nominal engineering strain rates: 0.01 s\(^{-1}\), 0.001 s\(^{-1}\), and 0.0001 s\(^{-1}\). The strain rate is defined as the ratio between the constant displacement rate and the initial height of the nanopillars being tested. Compression tests were performed ex situ with the dynamic contact module (DCM) of nanoindenter (Nanotestr™ G200, Agilent Technologies Inc.) equipped with a custom-made diamond flat-punch indenter tip with \(\sim 10\) \(\mu\)m diameter. Several compression tests were also preformed in situ using a custom-built instrument, referred to as the SEMentor, comprised of a scanning electron microscope (SEM, Quanta™ 200, FEI Company) and a nanoindenter-like nanomechanical module (DCM, Agilent Technologies Inc.)\[19,47,48\]. The SEMentor is equipped with a custom fabricated conductive diamond tip and allows for the visualization of deformation in tension or compression with simultaneous data collection. All mechanical tests were performed at room temperature.

3. Results and discussion

3.1. Deformation characteristics of indium nanopillars

The compressive flow stresses of indium nanopillars at \(\sim 2.0\%\) engineering strain plotted as a function of engineering strain rate are shown in Fig. 3, with the measured values summarized in Table 1. The plot reveals the presence of a wide scatter in the flow stress values, ranging from 9 MPa to 790 MPa, with the majority of data points being below 90 MPa. This division in the measured flow stresses is attributed to the indium nanopillars exhibiting two distinct loading responses, whereby attaining significantly different strengths. Typical stress–strain curves for \(\sim 560\) nm diameter indium nanopillars compressed at \(\sim 0.0001\) s\(^{-1}\) representative of

Fig. 2. SEM image of indium nanopillars with a nominal diameter of \(\sim 350\) nm, \(\sim 560\) nm, and \(\sim 920\) nm. The image was taken at \(\sim 70^\circ\) stage tilt.
Table 1
Summary of average flow stress values measured at ∼2.0% nominal strain for all indium nanopillars.

<table>
<thead>
<tr>
<th>Diameter (nm)</th>
<th>Strain rate (s⁻¹)</th>
<th>Deformation mechanism</th>
<th>Flow stress at ∼2.0% nominal strain (MPa)</th>
<th>Average ± std. dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>920</td>
<td>0.01</td>
<td>Type I</td>
<td>24.0, 31.0, 29.6, 39.1, 30.7, 30.0, 34.9, 31.5, 33.4, 29.8, 34.5, 27.8</td>
<td>31.4 ± 3.8</td>
</tr>
<tr>
<td>920</td>
<td>0.01</td>
<td>Type II</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>920</td>
<td>0.001</td>
<td>Type I</td>
<td>24.7, 21.7, 29.1, 23.4, 23.4, 17.5, 23.3, 24.8, 19.0, 25.2, 25.0, 17.4</td>
<td>22.9 ± 3.5</td>
</tr>
<tr>
<td>920</td>
<td>0.001</td>
<td>Type II</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>920</td>
<td>0.0001</td>
<td>Type I</td>
<td>20.6, 34.5, 16.2, 18.3, 25.2, 20.9, 14.0, 17.8, 25.8</td>
<td>21.5 ± 6.2</td>
</tr>
<tr>
<td>920</td>
<td>0.0001</td>
<td>Type II</td>
<td>97.4, 133.0</td>
<td>115.2 ± 25.2</td>
</tr>
<tr>
<td>560</td>
<td>0.01</td>
<td>Type I</td>
<td>34.3, 37.7, 47.7, 14.8, 37.0, 38.0, 20.3, 46.7, 27.7, 74.5, 69.9</td>
<td>40.8 ± 18.5</td>
</tr>
<tr>
<td>560</td>
<td>0.01</td>
<td>Type II</td>
<td>215.6, 369.9, 259.4, 361.0, 330.1, 326.5</td>
<td>310.4 ± 60.6</td>
</tr>
<tr>
<td>560</td>
<td>0.001</td>
<td>Type I</td>
<td>31.0, 21.4, 16.3, 21.4, 22.0, 33.3, 26.4, 29.0, 25.7, 19.4, 19.8, 16.6, 22.1</td>
<td>23.8 ± 6.2</td>
</tr>
<tr>
<td>560</td>
<td>0.0001</td>
<td>Type II</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>560</td>
<td>0.0001</td>
<td>Type I</td>
<td>10.3, 21.4, 10.6, 19.8, 14.1, 17.1, 28.4</td>
<td>17.4 ± 6.5</td>
</tr>
<tr>
<td>560</td>
<td>0.0001</td>
<td>Type II</td>
<td>328.4, 189.3, 324.0, 259.3</td>
<td>275.2 ± 65.4</td>
</tr>
<tr>
<td>350</td>
<td>0.001</td>
<td>Type I</td>
<td>323.3, 11.5, 20.6, 31.8, 12.2, 9.1, 18.9, 15.2, 10.1, 23.1, 14.6, 16.2,</td>
<td>21.8 ± 11.0</td>
</tr>
<tr>
<td>350</td>
<td>0.001</td>
<td>Type II</td>
<td>12.2, 24.1, 34.9, 37.6, 45.1</td>
<td>12.2, 24.1, 34.9, 37.6, 45.1</td>
</tr>
<tr>
<td>350</td>
<td>0.001</td>
<td>Type I</td>
<td>705.1, 590.5, 503.1, 667.9, 809.9, 381.1, 790.8, 624.2, 608.2</td>
<td>626.3 ± 151.5</td>
</tr>
</tbody>
</table>

Fig. 3. Log–log plot of engineering flow stress measured at 2.0% nominal strain as a function of strain rate for all indium nanopillars tested in this work.

these two deformation modes (Type I and Type II) are plotted in Fig. 4. Here, Type I deformation is characterized by low strengths which are close to the bulk value, with nanopillars yielding near 15 MPa and no significant subsequent strain hardening. Nanopillars undergoing Type I deformation also exhibit discrete displacement bursts under compressive stress. In contrast, Type II deformation results in nanopillars which are exceptionally strong, attaining stresses greater than 150 MPa, while undergoing significant strain hardening and exhibiting discrete displacement bursts. There is a clear bimodal distribution between the attained strengths for nanopillars exhibiting Type I versus Type II deformation, as the latter require applied loads approximately an order of magnitude greater than the former. Interestingly, nanopillars undergoing Type II deformation attain flow stresses of 422.6 ± 214.4 MPa measured at ∼2.0% nominal strain and averaged over 21 samples, corresponding to ~9.7% of the bulk shear modulus, G = 4.35 GPa [41]. This suggests that indium nanopillars of all sizes undergoing Type II deformation approach the theoretical strength, as the observed axial stresses are in the range expected for theoretical strength of metals (~G/30–G/10) [49,50].

Fig. 4. Representative engineering stress–strain curves for ~560 nm diameter indium nanopillars undergoing (a) Type I and (b) Type II deformation at a nominal strain rate of 0.0001 s⁻¹.

Post compression SEM images of indium nanopillars deforming via both types are shown in Fig. 5. Interestingly, there is no clear distinction between the post mortem appearance of 920 nm diameter indium nanopillars, which exhibited Type I deformation (Fig. 5a) and those which underwent Type II deformation (Fig. 5b). These images reveal wrinkling, bulging, and folding surface morphology, suggesting that the indium has extruded outward from the pillar surface—in response to compressive loading. This observation is quite different from the majority of post-compression SEM images of single-crystalline metallic nanopillars reported to date clearly show that the deformation occurred along well defined crystallographic slip planes and directions [1–13,15–21,23]. The lack of the discrete slip planes on the compressed indium pillars is expected due to increase number of active slip systems in indium at high homologous temperature. To confirm the lack of distinct

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![Fig. 3](image-url) Log–log plot of engineering flow stress measured at 2.0% nominal strain as a function of strain rate for all indium nanopillars tested in this work.

![Fig. 4](image-url) Representative engineering stress–strain curves for ~560 nm diameter indium nanopillars undergoing (a) Type I and (b) Type II deformation at a nominal strain rate of 0.0001 s⁻¹.
Fig. 5. Post compression SEM images of indium nanopillars with diameters of (a and b) 920 nm, (c and d) 560 nm, and (e and f) 350 nm. All images were taken at a ∼70° stage tilt.

Fig. 6. SEM images of bulk indium cylinder surface (a) before and (b) after uniaxial compression test with 30% nominal strain and deformation rate of 0.001 s⁻¹. Significant amount of nanoscale wrinkles and extrusions are found on the surface after the compression test.
Fig. 7. Progressive time series of SEM images taken during in situ compressive loading of ∼920 nm diameter indium nanopillar at nominal stain values of 0.0 (a), 0.04 (b), 0.06 (c), 0.13 (d), 0.23 (e), and 0.30 (f). The scale bar in each image corresponds to 1 μm.

slip plane appearance in Fig. 5a and b is not related to the size of pillars, cylindrical indium columns annealed at room temperature for more than 100 days with dimension of ∼3.3 mm diameter and ∼8.8 mm height were uniaxially compressed at a 0.001 s⁻¹ deformation rate to ∼30% engineering strain. The surface of this macroscopic sample was inspected before and after the compression tests at high magnifications by using high resolution SEM. The SEM image shown in Fig. 6a clearly reveals the initial bulk indium surface is flat and smooth. However, after the compression test (Fig. 6b), the bulk indium surface is wrinkled and material has extruded in a similar manner as the indium nanopillars undergoing deformation displayed in Fig. 5a and b. It is important to note that these are nanometer scale surface features and cannot be detected by optical inspection techniques. Results from Fig. 6 demonstrates that the extrusion like features shown in Fig. 5a and b are not unique to the nanopillars, rather they also form in the deformation of bulk indium structures. Fig. 5c and d shows the images of compressed 560 nm diameter nanopillars, and again both deformation types display characteristic extrusion patterns like those in the bulk indium. Post-compression SEM images of Type I deformation
of the 350 nm diameter indium nanopillars (Fig. 5e) show the extrusion pattern is nearly identical to those observed in nanopillars of other diameters. In contrast, the high strength Type II 350 nm diameter indium nanopillars do not deform via extrusion exclusively, as shown in Fig. 5f, well defined crystallographic shear offsets are also observed in these structures. To further understand the deformation characteristics of indium nanopillars, several in situ compression experiments were conducted in the SEMmentor. Fig. 7 shows a progressive time sequence of SEM images captured during uniaxial compression of a ~920 nm diameter indium nanopillar at a strain rate near 0.001 s⁻¹. The figure clearly shows that only continuous extrusion of indium occurred with no crystallographic slip. At strain values below 0.13 (Fig. 7a–c), localized bulging at the surface creates a wavy pattern. At higher strains, extrusion occurs simultaneously at the indenter/pillar interface and in the middle of the pillar near a ~45° oriented wrinkle. Subsequent extrusions continue along this wrinkle, folding and stacking on top of each other. Most of the extruded material is originated from the top pillar half. For strains greater than 0.30 (not shown) the bottom half also wrinkles and further extrusion occurs. No appreciable barrelling or Poisson’s effect is observed; instead it appears as though all plastic strain is carried by lateral surface extrusion.

In this study, both Type I and Type II deformation modes were observed for the identically processed nanopillars located on the same substrate and placed only 10 μm apart from each other. Therefore, the wide scatter of flow stresses in Fig. 3 cannot be attributed to the variations in the electroplating process. Another interesting observations of Type I or Type II deformation is the distribution of these two deformation modes amongst the three nanopillar sizes. A detailed breakdown of the Type I and Type II distributions is summarized in Table 2. The plot in Fig. 3 shows that out of 102 indium nanopillars successfully compressed, 81 nanopillars exhibit Type I deformation, while the remaining 21 nanopillars deformed via the Type II mechanism. Furthermore, the population of Type I and Type II deformation modes is dependent on the nanopillar size: for example, 35% of all compression tests on the ~350 nm diameter nanopillars undergo Type II deformation, while only 6% of ~920 nm diameter nanopillars do. Therefore, it appears that the reduction in sample size increases the fraction of high strength nanopillars. The data in Table 2 also helps to explain the lack of Type I signature in the previous study on indium nanopillars by Lee et al. [29]. All indium nanopillars tested in this prior work were approximately 400 nm in diameter and sample population examined was far more limited than the current study. According to Table 1, over one third of nanopillars this size should exhibit Type II deformation and approach theoretical strengths, Compared with the previous study, a significantly larger number of indium nanopillars were examined was far more limited than the current study. According to Table 1, over one third of nanopillars this size should exhibit Type II deformation and approach theoretical strengths, Compared with the previous study, a significantly larger number of indium nanopillars were tested in this work, including investigations of different sized nanopillars and different loading conditions. Therefore, it is not surprising that Lee et al. [29] observed only the Type II deformation behavior, highlighting the importance of having a large sample size when attempting to characterize nanoscale mechanical properties.

3.2. Strain rate sensitivity of indium pillars

Indium nanopillars displaying Type I versus Type II deformation modes have different responses to strain rate, as shown in Fig. 3. Nanopillars which undergo Type I deformation demonstrate a clear sensitivity to strain rate, while the flow stress of indium nanopillars exhibiting Type II deformation appears to be much less sensitive to deformation rate. In the following discussion, we focus on indium nanopillars undergoing Type I deformation only.

The average flow stresses at 2.0% engineering strain of Type I deformation for the three different strain rates are summarized in Fig. 8, where the error bars correspond to the standard deviation. The data shows a noticeable increase in indium nanopillar strength from ~17 MPa to ~37 MPa with the strain rate increasing from 0.0001 s⁻¹ to 0.01 s⁻¹. This strain rate sensitivity is consistent with the previous reports for bulk indium [31,54] and with the nanoindentation creep experiments [35,55]. A wide range of steady state high temperature (>0.5 × Tm) creep results from polycrystalline metals have demonstrated that a general relationship exists between the strain rate (\( \dot{\varepsilon} \)) and creep stress (\( \sigma \)):

\[
\dot{\varepsilon} = K \sigma^m
\]

where \( K \) is a material constant that depends on diffusivity and elastic modulus [54]. For bulk indium, the power-law exponent, \( n \), was measured by Weertman and has a value of ~6.1–7.7 at room temperature [31]. Using nanoindentation, Lucas and Oliver investigated the creep of indium single crystals [35]. Their work demonstrated that bulk indium hardness (\( H \)) also exhibited a power-law relationship with strain rate of the form:

\[
\dot{\varepsilon} \propto H^m
\]

where \( m \) is the hardness power-law exponent in the range of 6.3–7.3 [35]. With the assumption that the hardness is linearly proportional to the uniaxial flow stress, Eqs. (1) and (2) actually describe the same physical phenomenon.

To compare the indium nanopillar strain rate sensitivity with previous studies, the flow stress at 2.0% engineering strain results from Fig. 8 are fitted with Eq. (1). The power-law exponent (\( n \)) extracted from this fit is approximately 6.12 ± 1.42, where the spread corresponds to one standard deviation. This exponent is consistent with bulk indium room temperature creep results reported by Weertman [31] and further suggests that the Type I deformation mode is bulk-like, as it suggests that the deformation of low strength indium nanopillars is dominated by a bulk-scale mechanism. Additionally, the lack of strain rate dependence for the Type II pillars in Fig. 3 may be explained by the expected dislocation nucleation dominated mechanisms – rather than dislocation interactions – inherent to the deformation of pristine crystals.

3.3. Lack of size-effects in indium nanopillars

Another interesting observation is the absence of size-dependent strengthening in indium nanopillars undergoing Type I deformation. This is best illustrated in Fig. 9 where the Type
I data measured at a 0.001 s\(^{-1}\) strain rate is plotted as a function of nanopillar diameter. No size effect is present in the data, a finding diametrically opposite to nearly all other studies of single-crystalline FIB-fabricated [1–11,13,16–21] and electroplated [12] metallic nanopillars. It is important to recognize that this lack of size effects in indium is fundamentally different from that reported for copper nanowhiskers [49,56] and molybdenum micropillars [14] in the sense that stresses measured for indium nanopillars undergoing Type I deformation represent a small fraction of the theoretical strength of indium. In these previous studies the initial sample microstructure is pristine, i.e., containing no initial dislocations, and therefore attaining nearly theoretical strengths for all diameters [14,49,56].

Through the evidence presented, indium nanostructures categorized as Type I appear to deform plastically via similar mechanisms which operate in bulk scale indium crystals. Thus, the lack of size-effects in these specimens is not unexpected. The results show Type I nanopillars sustain flow stresses close to bulk indium values and also possess a similar strain rate sensitivity. In addition, Fig. 5 reveals that nanoscale surface features resembling material extrusion are universal in nano and bulk scale plastic deformation of indium. A similar report which demonstrated bulk-like behavior and lack of size effect in pillars is from the work by Bei et al. [15]. Their results show that the stress–strain behavior of 11% pre-stained molybdenum and molybdenum-alloy micropillars – which contain a significant dislocation population – is characteristically bulk-like with no apparent size effect. In contrast, for un-strained samples, Bei et al. showed that they achieved theoretical strength but without size effects, which is similar to the dislocation free Type II indium nanopillars demonstrated in Fig. 3.

### 4. Conclusions

We report that indium nanopillars display two fundamentally different deformation mechanisms upon uniaxial compression. In over one hundred nanopillars tested, approximately 80% exhibited low flow stresses, near that of bulk indium regardless of nanopillar diameter examined. These low strength nanopillars also displayed a clear dependence of compressive flow stress on deformation rate, similar to that of bulk indium. SEM inspections also show that low-strength indium nanopillars deform similar to the nanometer scale deformation extrusion features on bulk indium surfaces. The remaining 20% of compressed indium nanopillars deformed at nearly theoretical strengths, and as expected, demonstrated very little strain rate or size sensitivity.

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### References


### Table 2

Distributions of Type I and Type II indium nanopillars for all three sizes and strain rates.

<table>
<thead>
<tr>
<th>Strain rate (s(^{-1}))</th>
<th>350 nm diameter</th>
<th>560 nm diameter</th>
<th>920 nm diameter</th>
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<tr>
<td></td>
<td>Type I</td>
<td>Type II</td>
<td>Type I</td>
</tr>
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<td>N/A</td>
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<tr>
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<td>16</td>
<td>9</td>
<td>14</td>
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<td>N/A</td>
<td>N/A</td>
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<tr>
<td>Total</td>
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<td>9</td>
<td>32</td>
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<tr>
<td>% Total</td>
<td>65</td>
<td>35</td>
<td>76</td>
</tr>
</tbody>
</table>

Fig. 9. Log–log plot of engineering flow stress measured at 2.0% nominal strain as a function of diameter for all indium nanopillars undergoing Type I deformation.