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Fabrication, structure and mechanical properties of indium nanopillars

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Abstract

Solid and hollow cylindrical indium pillars with nanoscale diameters were prepared using electron beam lithography followed by the electroplating fabrication method. The microstructure of the solid-core indium pillars was characterized by scanning micro-X-ray diffraction, which shows that the indium pillars were annealed at room temperature with very few dislocations remaining in the samples. The mechanical properties of the solid pillars were characterized using a uniaxial microcompression technique, which demonstrated that the engineering yield stress is ~9 times greater than bulk and is ~1/28 of the indium shear modulus, suggesting that the attained stresses are close to theoretical strength. Microcompression of hollow indium nanopillars showed evidence of brittle fracture. This may suggest that the failure mode for one of the most ductile metals can become brittle when the feature size is sufficiently small.

Keywords: Plastic deformation; X-ray diffraction; Compression test; Electroplating; Yield phenomena

1. Introduction

With the recent progress in small-scale fabrication techniques, it is now possible to readily manufacture structures with a minimum feature size at the nanometer scale. In the integrated circuit industry, high-performance transistors with critical dimensions as small as 20 nm have been successfully produced. However, the mechanical behavior of these nanoscale structures has not been thoroughly understood. The majority of research conducted in this area has focused on metals with cubic structures, i.e. nickel [1–3], gold [4–6], copper [7], molybdenum [8–10] and niobium [11]. The results of all these studies show that the uniaxial compressive yield strength of crystalline cubic metals increases with reduced sample sizes [12]. Uchic and Dimiduk [2] developed focused ion beam (FIB) techniques to fabricate pure nickel and nickel alloy pillars with a diameter as small as 5 μm. Their compression data revealed that the yield strength of small pillars can be ~100 times greater than the bulk values. Greer et al. [4] and Volkert et al. [13] show similar size effects with the compression yield strength of FIB-fabricated solid and porous gold pillars, respectively. Recently, the yield strength of body-centered cubic (bcc) Mo-alloy single crystal pillars fabricated by a solid solution directional precipitation methods was examined by Bei et al. [14]. This pillar fabrication technique removes the potential damage created by the FIB milling process and results in a pristine microstructure, with no initial defects [15]. They demonstrated that the Mo-alloy pillar flow stress approached the theoretical strength value, ~1/25 of shear modulus. In contrast to the earlier works, no size-dependent hardening was observed in these Mo-alloy pillars prepared by a FIB-less technique most likely due to the pristine initial microstructure in these pillars. These findings suggest that both the initial microstructure, i.e. dislocation density, as well as the size, have to be taken into account when considering mechanical testing at small scales.

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In this work we investigated small-scale plastic deformation of indium nanopillars, a previously unstudied material and crystal structure. This material has a tetragonal crystalline structure and a low melting temperature of ~156 °C. Indium and its compounds have many important applications in the field of nanotechnology. For example, it has been demonstrated that indium phosphide (InP) and indium arsenide-phosphide (InAsP) nanowires can be used as highly polarized photoluminescence and infrared photodetection devices [16,17]. Vertical field-effect transistors have also been constructed with indium oxide (In_2O_3) nanowires [18]. Therefore, understanding the mechanical properties of nanometer-scale indium structures may have a significant impact on the commercial success of these novel devices.

The low melting temperature of indium allows it to undergo room temperature annealing, which helps to reduce or eliminate fabrication-induced stresses, defects, and dislocations. In addition, the small as-deposited indium grains grow into large crystals at ambient conditions. Two different sample geometries were studied – solid core and hollow pillars. The deformation mode of hollow pillars is particularly interesting because of the large surface area to volume ratio. In these samples the dislocations should glide to the surface much more readily than in the solid core pillars with the same diameter due to the significant effects of the image stress. To avoid potential ion damage caused by the FIB milling technique, our indium pillars were fabricated via an electroplating method.

2. Procedure

Fig. 1 illustrates the process integration steps for the fabrication of nanometer-scale indium pillars. These pillars were fabricated on silicon substrates coated with electron beam evaporated chromium/gold bi-layer. A gold seed layer was selected as the cathode in subsequent electroplating steps while the chromium film was used to promote adhesion between gold and silicon substrate. The thickness of these layers was deemed non-critical, but generally maintained at 50–100 nm for the gold layer and 20–50 nm for the adhesion layer. Following deposition of the seed layer, polymethylmethacrylate (PMMA) diluted in anisole (supplied by Microchem Corp.) was spun on the substrates and then cured at 180 °C for ~3 min to form the final resist layer. These PMMA thin films were then patterned by using the electron beam lithography techniques to produce holes in the resist with the diameter of ~400 nm. The resists were exposed with a 30 kV electron beam at dosages of ~1250 $\mu C\,cm^{-2}$ and developed using a 1:3 mixture of methylisobutylketone and isopropyl alcohol (IPA). After the PMMA templates were produced, samples were separated into two different groups. One group underwent a 15 s oxygen plasma descum process while the other did not. This descum process was applied to reduce pillar tapering caused by electron beam lithography and to promote homogenous electroplating. This descum process was performed with a Trion Phantom Reactive Ion Etcher (RIE) system operated at room temperature, with a chamber pressure of 102 m Torr, plasma power of 20 W, and oxygen flow rate of 40 sccm. Results from a separate descum experiment on a 1.5 μm thick blanket PMMA thin film showed the etch rate under these conditions is ~1.67 nm s^{-1}.

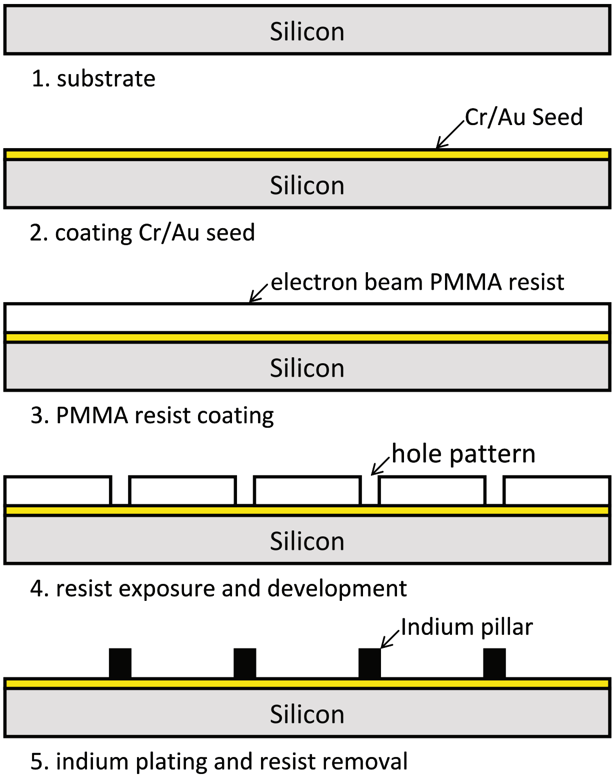

Fig. 1. Schematic illustration of the indium pillar fabrication process.

After patterning the PMMA films, indium was electroplated into the template structures by using a two electrode configuration system and a commercially available indium sulfamate solution (purchased from Indium Corporation of America). This sulfamate bath was maintained at ambient temperature and mechanically stirred during the deposition process. High-purity indium metal slab was used as a soluble anode while the conductive gold seed layer underneath the patterned PMMA resist acted as the cathode. The plating process began with a 3 s current pulse of 11.4 mA cm^{-2} and then followed by galvanostatic electroplating at 1.14 mA cm^{-2}. The initial high current step was used to produce a thin indium seed layer which promoted homogenous filling of the patterned features and increased the final yield of indium pillars. Scanning electron microscopy (SEM) inspections show that greater than 50% of the patterned features were filled with indium metal when processed with the procedure described above. To achieve the pillar height of ~800 nm, the deposition time for the virgin and plasma-treated samples were set at 510 and 620 s respectively. After plating, specimens were rinsed in de-ionized water and the PMMA was removed using a commercial photoresist stripper (Microchemicals GmbH AZ® Kwik Strip®) at room temperature for at least 18 h.