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Polycrystalline micropillars by a novel 3-D printing method and their behavior under compressive loads

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ABSTRACT

We present an entirely new method of bottoms-up fabrication of polycrystalline micropillars using direct printing and sintering of nanoparticles in 3D and study their behavior under compression for different microstructures. The pillars showed brittle behavior with higher effective modulus for small grain sizes with high porosity, while highly ductile behavior with a lower effective modulus and larger grain sizes but low porosity. These unusual trends are explained by a porosity model. The results point to a novel method of fabricating micropillars with different microstructures to study fundamental materials science of polycrystalline materials at micro to meso-length scales.

Micropillar compression test is a popular method to study the mechanical behavior of various engineering materials for more than a decade [1–4]. Micropillars of different materials with a variety of sizes, textures, and microstructures [5] have been realized by using fabrication methods such as focused ion beam (FIB) milling [1,6,7], nano-imprinting [8], vapor deposition [9], and electrodeposition [10]. Each of the above methods has led to experiments that have contributed greatly to our knowledge of the behavior of materials at microscales. These methods, however, have their advantages as well as limitations in allowing the manufacture of samples of desired materials, geometries, and microstructures. For example, FIB milling can produce nano and microscale samples with precise dimensions from a wide variety of materials, but also results in ion implantation into the samples that may induce artificial size effects, or result in voids that affect their mechanical behavior [11]. In addition, for polycrystalline materials, obtaining different grain sizes (and hence the microstructures) for the same material is not straightforward. In case of electrochemical deposition and/or etching, the limitations are based on the chemical compatibility of the material being tested and difficulty in changing the microstructure [10]. For microscale samples, it was seen that the deformation mechanisms of elemental metals differ over three grain size ranges, namely, below 100 nm (nanocrystalline), between 100 nm and 1 μm (ultrafine grain), and above 1 μm (medium to large grains) [12,13]. For materials having ultrafine grains, the deformation was shown to be accommodated by grain boundary sliding and dislocation activity (unit or partial) sourced from the boundary [12,13]. The above work further suggests that creating new microstructures and geometries at microscales can help in the investigation of new scientific principles and the engineering of materials for specific applications.

Several developments in the manufacturing sciences have opened up the possibility of making devices and samples not possible in the past [14]. Bottoms-up manufacturing technique such as microscale 3D printing is among one such method, where nano or microparticles can be printed on 2D surfaces followed by sintering, that creates microscale geometries with a microstructure defined by the starting nano or microparticles [15–18]. We have recently developed a new manufacturing process where nanoparticles were arranged in 3D space at microscales in different shapes using pointwise printing in three dimensions followed by binder removal and nanoparticle sintering [16]. Complex 3D micro-lattice structures with truss members having diameters of 10s of microns with the microstructure defined by the nanoparticle size were fabricated using metal nanoparticles. During sintering of the nanoparticles, the porosity in the solid truss members can be controlled by the sintering temperature and time [16]. An example illustrated in Fig. S1 shows a silver micro lattice with near-fully dense truss members fabricated by this method. The truss members (Figs. S1b–c) can be considered as solid two-force elements, especially when their aspect ratio

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(length to the diameter) is high, as is Fig. S1b. The behavior of micropillars is thus a highly important engineering problem for microscale cellular/lattice materials.

The impetus for the present work is thus twofold. First, we wanted to fabricate polycrystalline micropillars using the recently discovered method of assembling metal nanoparticles in 3D space followed by sintering [16] and demonstrate its flexibility in creating samples with different microstructures and internal porosities. Second, we wanted to carry out a micropillar compression test on such samples with grain sizes ranging from hundreds of nanometers (fine grain microstructure) to a few microns (coarse grain microstructure) with different porosities and obtain their behavior under compression. The work thus advances fundamental materials science by using the latest developments in novel microscale manufacturing methods.

The micropillars fabrication by nanoparticle printing is shown in a schematic in Fig. 1(a), with details in section S1 of supporting information. Fig. 1(b, c) show square array of 25 pillars, and the pillar dimensions (90 μm outer diameter, 70 μm inner diameter, and about 600 μm in height) and the grain size distribution within a given pillar, respectively. We note that the pillar surface is "rough" and made up of silver grains formed from the sintered nanoparticles. Within the process optimization used for this study, the pillar axis could be maintained within about ±3°. We note that the hollow geometry was selected in this study to enable fabrication of taller pillars suitable for optical observation and strain recording under compression as described below. It is clear from Fig. 1 that the pillars can be directly printed to the final net shape in a single printing step followed by sintering. The time taken to print a single pillar is within a minute. Note that the grain size can be changed by either varying the starting nanoparticle size and/or the sintering conditions. Lastly, any material in nanoparticle form with particle size <500 nm can be printed, with the nanoparticles transforming into the grain, which can provide flexibility in choosing materials and microstructures.

The compressive mechanical behavior of micro-pillar arrays was investigated in a customized apparatus (Fig. S2) described in the supporting information, Section-S2. The SEM images of the grains and grain size distribution, and focused ion beam (FIB) sections of the pillars for different sintering conditions is shown in Fig. 2 and the statistics are given in Table 1. The grain sizes were measured by detecting boundaries in an SEM images by image processing software (Image J, NIH, Bethesda, MD) over at least three areas for each specimen. Gaussian normal curve was fitted on the area percentage distribution to calculate the mean grain size and bandwidth. At a lower sintering temperature of 250 °C, the mean grain size is of the order of about 250 nm, while at higher sintering temperatures of 350 °C and 450 °C, the mean grain size increased to about 2.9 μm, and 3.8 μm, respectively. For a sintering temperature of 550 °C, however, a near bimodal distribution with mean grain sizes at 3 μm and 7 μm (bimodality factor of 2.1) was observed as shown in Fig. 2(a). Since the pillars are made by sintering and growth of the nanoparticles, it is expected that the microstructure will have internal porosity. We carried out focused ion beam (FIB) section for a few cases to get an estimation of the porosity. The FIB section shown in Fig. 4 (b–d) show porosities at about 17–20% (pore size 250–300 nm), 15% (pore size ~1 μm), and <1% (pore size ~170 nm) for sintering

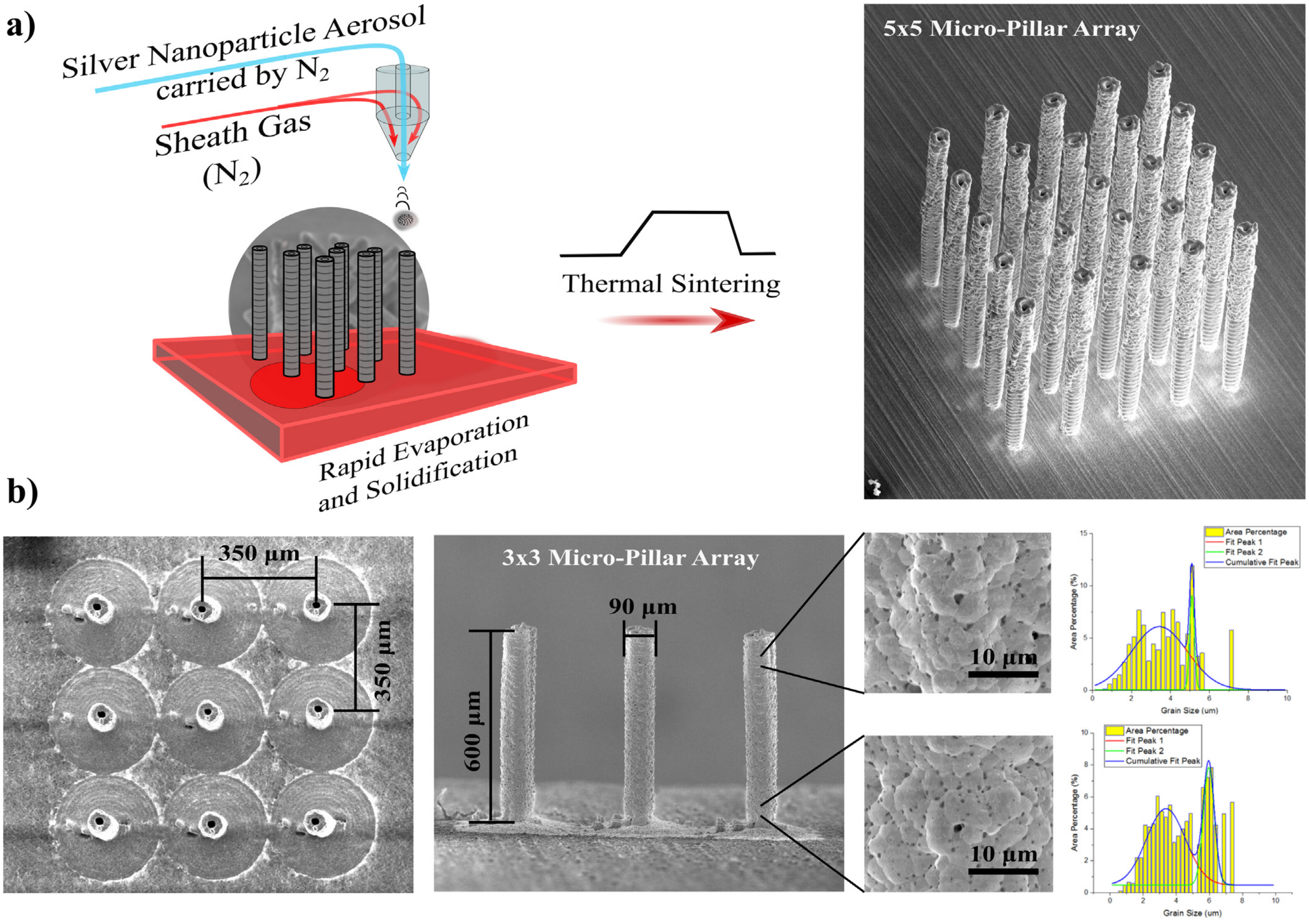

Fig. 1. (a) Schematic of the printing process showing the fabrication of the micro pillars. (b) A printed micropillar array in a 5 × 5 matrix. (c) A 3 × 3 micropillar array with pillar dimensions of 90 μm outer diameter, about 70 μm inner diameter, 350 μm spacing, and about 600 μm in height.