COMPOSITE MATERIALS WITH CONTROLLABLE MACROMECHANICAL PROPERTIES BASED ON MEMS-ASSISTED STRUCTURAL MANIPULATION OF LOW-DIMENSIONAL SUBCOMPONENTS

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ABSTRACT

We report an approach to achieve composite materials with wide-ranging macroscale mechanical properties through the three-dimensional structural manipulation of low-dimensional subcomponents. Such composites could be useful in MEMS actuators based on highly compliant mechanisms, or in mechanical metamaterials with highly anisotropic mechanical properties (e.g., negative Poisson ratio). The presented composites possess a multilayer structure comprising alternating high modulus and low modulus subcomponent materials (i.e., permalloy ($Ni_{80}Fe_{20}$) and polydimethylsiloxane (PDMS) elastomer), within which lithographically-patterned pores are present. By controlling the pore geometries/orientations and the individual metal/elastomer layer thicknesses in the microscale, in-plane and out-of-plane mechanical properties (i.e., tensile and bending moduli) are substantially tailored.

INTRODUCTION

Composite materials, i.e., materials comprising two or more heterogeneous subcomponent materials, may exhibit useful electrical/mechanical/optical properties that cannot be achieved from a homogeneous, single material system. Various composites for MEMS applications include inorganic fillers (e.g., silver nanoparticles [1], carbon [2,3]) and "soft" polymeric materials (e.g., PDMS [1], Ecoflex™ [3]); such composites exhibit desired electrical/electromechanical functionality (that results from the fillers) while being highly mechanically compliant (due to the polymeric materials). As an example, carbon nanofiber/polymer composites exhibit both high piezoresistivity and high flexibility, making them suitable for strain sensing [3].

To some extent, additional control over the mechanical properties of a composite can be achieved by using subcomponent filler materials with anisotropic geometry (e.g., platelets [4] and fibers [5]); however, it is difficult to achieve composite materials with controlled, wide-ranging mechanical properties since the positions and geometries of the subcomponents cannot be precisely defined based on conventional mixing processes.

Materials with rationally designed, extremely high anisotropy (e.g., negative Poisson ratio) have been developed based on projection lithography [6]; however, the manipulation of out-of-plane mechanical properties is still challenging, since photolithography is typically limited to planar structures. Three-dimensional composite structures with designed mechanical properties have been presented [7]. The microfabrication of such structures typically relies on direct writing process, e.g., two-photon polymerization, which exhibits limited production throughput.

Here, we present a MEMS-based, scalable route to composite materials with well-defined, three-dimensional subcomponent microstructures. Based on the presented approach, the vertical and lateral extents/positions of the micrometer-scale subcomponents can be independently defined by multilayer electrodeposition and photolithography, respectively. As a result, composites with wide-ranging, controllable macromechanical in-plane/out-of-plane properties can be achieved. Two subcomponent materials, i.e., permalloy ($Ni_{80}Fe_{20}$) and PDMS, are utilized to demonstrate the proposed composite materials.

COMPOSITE STRUCTURE

The composite materials possess three-dimensionally designed subcomponent structures that comprise lithographically-defined, pore-patterned laminated metal permalloy layers of micron-scale individual thickness, sandwiched by PDMS of similar thicknesses (Figure 1). Although Figure 1 depicts a composite with rectangular pores, the pore geometries are not limited unless the size scale of desired patterns are not smaller than the (lower) limit of conventional photolithography. Permalloy and PDMS are chosen as the subcomponent materials to highlight wide-ranging mechanical properties of the composites with different structural designs. Composites with similar subcomponent compositions may exhibit very different mechanical properties by design.

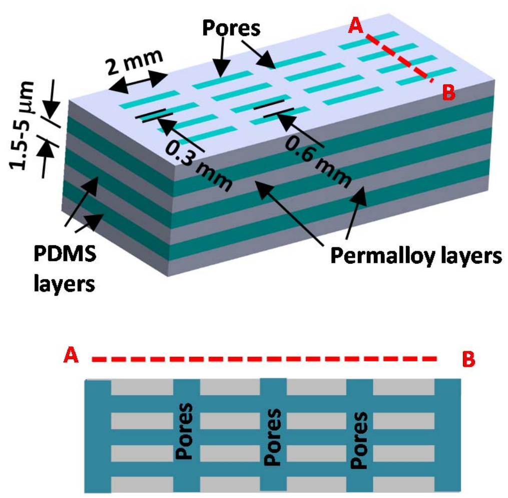

Figure 1: (a) Schematic of a composite (b) and its cross-sectional view.

FABRICATION

The fabrication process is based on the combination of standard projection lithography and multilayer electrodeposition. The process is comprised of four steps: (1) multilayer electrodeposition of alternating permalloy structural layers and copper sacrificial layers; (2) anchor fabrication; (3) selective removal of copper sacrificial layers; and (4) PDMS infiltration.

Fabrication begins with through-mold electrodeposition of permalloy/copper multilayers (Figure 2(a)) [8]. The electrodeposition molds, i.e., lithographically-patterned negative photoresist, possess both larger-scale features that define the lateral extent of the electrodeposition, and smaller-scale features that define the pore geometries of each subcomponent layer. The individual layer thicknesses are defined by controlling deposition time; they can range from 50 nm to a few micrometers [9]. For this work, copper and permalloy thicknesses were identical. After mold removal, a selective, timed copper etch creates microgroove structures (Figure 2(b)) on the sidewalls of the lithographically-defined pores.

Spin-cast micromolding [10] follows to form PDMS anchor structures within the deposited multilayers (Figure 2(c), (d)). Photolithography is performed to fill half of the patterned pores with a negative photoresist. PDMS is spun-cast to fill the non-resist-containing holes with elastomer (Figure 2(c)). Then, the patterned photoresist is removed by acetone (Figure 2(d)); the PDMS residues, i.e., PDMS remaining "on" the patterned photoresist immediately after the spin casting, are effectively removed. This process can be utilized to form the anchor structures with desired polymeric materials that cannot be defined by photolithography.

Copper is selectively removed from the entire material volume leaving the permalloy layers mechanically supported by the PDMS anchors (Figure 2(e)). An ammonium hydroxide based copper etchant is used for the selective etching. The etching begins from the patterned pores that are not occupied by the PDMS anchors. The etching time is defined by the distance between the peripheries of the exposed pores and the anchors; typically, etching is performed more than 24 hours to ensure the complete removal of copper. After the copper removal, the etched multilayers are rinsed in isopropyl alcohol and dried in an oven to minimize the stiction between the suspended permalloy layers.

PDMS vacuum infiltration follows (Figure 2(f)). PDMS can infiltrate through micrometers-width gaps to fill the pores with high aspect ratios; this is possible since (1) PDMS is air-permeable and (2) all the pores are interconnected with each other (in other words, this case is different from infiltrating PDMS into insolated nanoscale holes or grooves, where achievable infiltration depths are significantly limited). Since the PDMS now occupies the space formerly filled by copper, the PDMS thickness is nominally the same as this original copper thickness. The residual thin PDMS film comprising the uppermost layer of the samples is removed (not shown in the figure). These samples could be cut; the cutting orientation (with respect to the patterned pores) can be a key design variable in defining the ultimate composite mechanical properties.

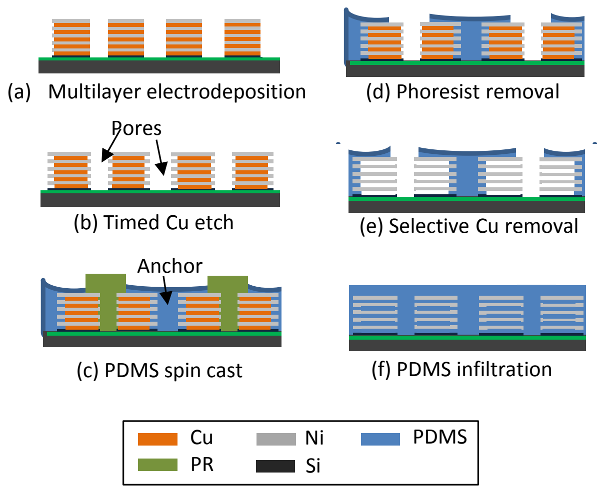

Figure 2: Fabrication procedure.

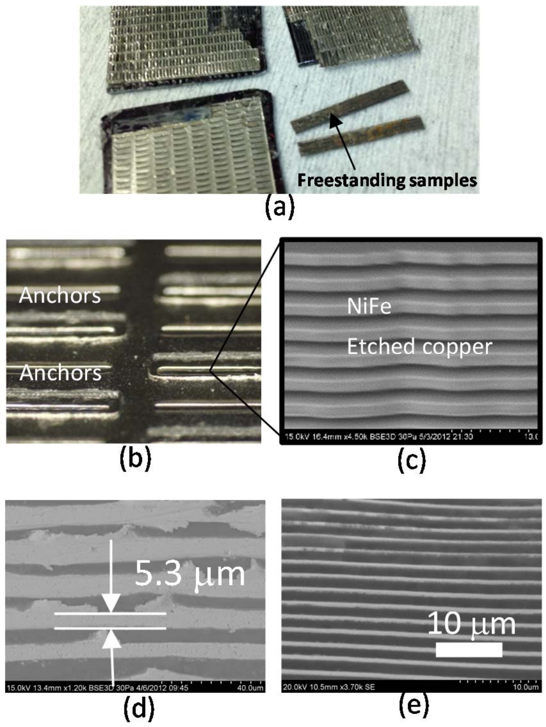

Figure 3: (a),(b) Optical and (c)-(e) scanning electron microscope (SEM) images of fabricated samples. (b) Image of a sample prior to the PDMS infiltration. The pores that are not filled by anchors are exposed. (c) is a magnified image of an exposed sidewall of the composite. (d), (e) Cross-sectional view of the samples.

FABRICATION RESULTS & PROCESS VALIDATION

Figure 3(a) shows the fabricated samples on a silicon substrate; these structures are mechanically detached from the substrate prior to mechanical characterization. Figures 3(b), (c) show the magnified views of the copper-etched multilayers with suspended permalloy layers, prior to the PDMS infiltration. Figures 3(d), (e) show the cross-section images; infiltrated PDMS (dark grey) can be observed between the permalloy layers (light grey). Although the cross-sectional imaging validates the composite structures in part, it is still difficult to assess whether (1) the copper is completely removed throughout the volume of the deposited multilayers, and (2) the individual permalloy layers are electrically insulated by the infiltrated PDMS.

This is validated by two approaches. First, the weights of the multilayers are measured as a function of etching time. The deposited multilayers exhibit nearly 50% weight loss after ~36 hours; this corresponds to the complete removal of copper. Second, the electrical isolation between the permalloy layers after copper removal, sample drying and PDMS is validated through an *in-situ* impedance measurement during the process detailed elsewhere [8, 11]. Initially, the as-deposited multilayers are wrapped with Litz wires to form inductors. The measured inductances of the samples decrease as a function of operating frequency due to the induced eddy current losses within the thick conducting multilayers. As the copper is gradually removed from the multilayers, the inductances at high frequencies are improved since the permalloy interlayer conductances are decreased. After no further change is observed on the inductances (which means the complete removal of copper), sample drying and PDMS infiltration followed. Figure 4 compares the inductances of a permalloy (individual layer thickness: 1.5 μm)/copper multilayer sample prior to copper removal, and the corresponding permalloy/PDMS composite. The measured inductances of the composite corresponds to the theoretical calculation based on the perfect electrical insulation between permalloy layers [8]; this suggests that there is negligible process-induced, electrical shorting between the permalloy layers. Although the designed subcomponent permalloy/PDMS thicknesses are identical in this study, both PDMS and permalloy may range from 500 nm to over a few micrometers. The total thickness (in other words, the total volume) of a composite is limited by the maximum achievable thickness of the negative photoresist (~100 μm for NR26-20000P, Futurrex), which is utilized as electrodeposition mold; different photoresists (such as AZ NX15, MicroChem) could be employed to achieve thicker composite materials.

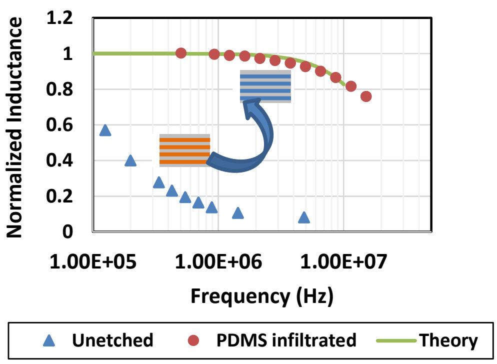

Figure 4: Measured inductances of the samples before copper removal (unetched) and after copper removal followed by PDMS infiltration (The inductances are normalized by the measured inductance of the fabricated composite at the lowest frequency of interest).

MECHANICAL CHARACTERIZATION

Permalloy/PDMS composites with (1) different individual layer thicknesses with (2) different pore geometries (or, in other words, different orientations with respect to the lengths of the samples; either orthogonal, or parallel) are fabricated. The number of permalloy layers (N) is designed so that the total thickness of permalloy is identical (35 μm) for all samples.

Three-point bending and tensile testing are used to determine the bending and tensile moduli (Figure 5) of the composites, respectively. Bending and tensile stiffness of the composites are estimated from the linear regime of stress-strain curves; "effective" bending/tensile moduli are then calculated by the equations; note the total sample thicknesses are considered to calculate the moduli for respective samples.

Effective bending moduli of the samples comprising different number of permalloy layers are characterized. The measured bending modulus decreases with increasing N; the effective bending moduli of 24-layer samples (with 5 μm-thick subcomponent layers) are ~25 times smaller compared to identically patterned, single layer permalloy (Figure 6). Note that the effective modulus for a 24-layer sample (with 1.5 μm-thick subcomponent layers) is nearly 1/3 that of a 7-layer sample, even though the weight portions of permalloy in the composites are identical. These results imply relatively weak mechanical coupling between the individual permalloy layers due to the low shear modulus of PDMS. Although not covered in this paper, it would be interesting to study whether this decreasing trend of effective modulus will continue as the thicknesses of both permalloy and PDMS subcomponents decrease.

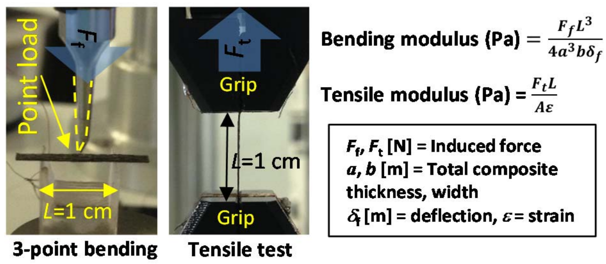

Figure 5: Test setup (3220-AT Series, Bose) for bending/tensile tests.

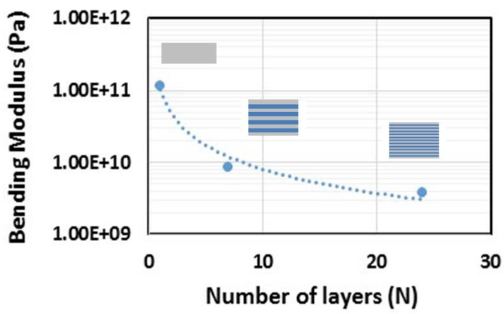

Figure 6: Effective bending modulus measured from the multilayers with different number of layers. The total permalloy thicknesses were nominally identical. (All samples' pores patterned parallel to the lengths).

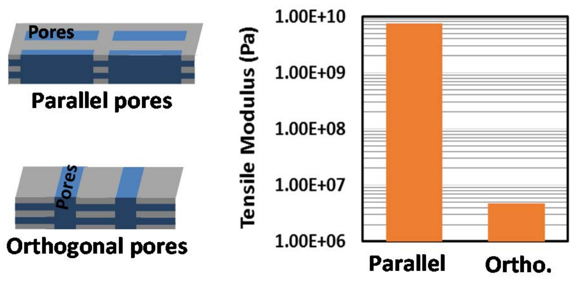

Figure 7: Tensile modulus measurement results for different pore geometry (or, in other words, different orientation with respect to the lengths). Samples have 21 pairs of permalloy and PDMS (N=21).

The composite in-plane tensile modulus are drastically variable by proper design of pore patterns. Very low moduli (4.75 MPa, similar to PDMS [12]) are measured from the samples with orthogonally-patterned pores (Figure 7), which is three orders of magnitude smaller than the measurements from the samples with parallel pores. The induced tensile loads are transferred to the pure PDMS filled in the patterned pores, thereby resulting in a high strain. The results imply that extremely wide-ranging composite mechanical properties can be achieved through a proper control of subcomponent geometries and orientations without a significant change of subcomponent composition.

CONCLUSIONS

A permalloy/PDMS composite material was demonstrated of which in-plane and out-of-plane macromechanical properties can be controlled by three-dimensional structural manipulation. The composite materials comprised micrometers-thick pore-patterned permalloy layers sandwiched by PDMS layers with similar individual layer thicknesses; a MEMS-based multilayer electrodeposition was employed to define the lateral/vertical extents of micrometer-scale subcomponents. Wide-ranging macromechanical properties (i.e., effective bending/tensile moduli) were measured for the samples comprising different number of layers (or, different individual layer thicknesses) and pore orientations. In addition to their utility in mechanically compliant MEMS, the presented approach could be a viable route to composite materials with anisotropic thermal/electrical properties.

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