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Silicon nanosprings fabricated by glancing angle deposition for ultra-compliant films and interfaces

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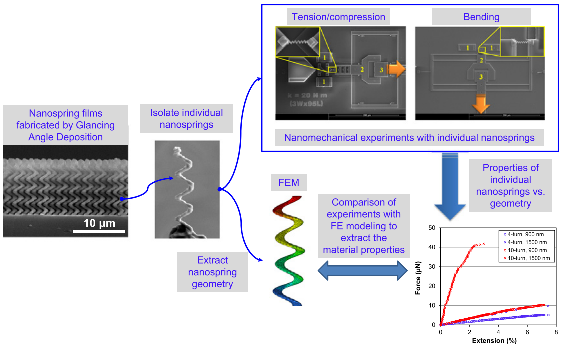
HIGHLIGHTS

- Measured the mechanical behavior of Si nanosprings fabricated by Glancing Angle Deposition in tension, compression & bending.
- Obtained the material modulus of aSi nanostructures fabricated by GLAD, ranging from 5 to 50 GPa vs. 94 GPa for bulk aSi.
- The normal and shear stiffness of seeded aSi nanospring films was tuned in the range of 90-1,000 MPa and 15-150 MPa.

GRAPHICAL ABSTRACT



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ABSTRACT

Micro and nanostructures with well-defined shape and dimensions are the hallmark in the design of scalable nanomaterials, yet the properties and precise geometry of such nanoscale building blocks are largely unknown. This work sheds light into the microstructure, material properties and mechanical behavior of individual nanosprings fabricated by seeded Glancing Angle Deposition (GLAD), with the purpose of designing highly compliant interfaces with drastically reduced coupling between normal and shear deformation. The mechanical response in tension/compression and bending of individual amorphous Si (aSi) nanosprings with 4 or 10 coil turns and different seed spacings was obtained with the aid of MEMS devices: The normal and bending spring stiffness values were in the range of 7–215 N/m and 1–31 N/m, respectively, resulting in estimates for the normal and shear film stiffness in the range of 90–1000 MPa and 15–150 MPa, respectively. The true geometry of GLAD Si springs was determined via SEM tomography and was incorporated in modified analytical and finite element models which, in turn, were used to compute the material modulus of aSi nanostructures fabricated by GLAD. TEM studies revealed that GLAD Si nanosprings are comprised of tightly bundled fine fibrils which impart flaw tolerance and reduce the effective elastic modulus.

1. Introduction

Interfaces are often the weakest link between materials with elastic property mismatch and/or significant differences in the coefficient of thermal expansion. In such material systems, insertion of a compliant interface could mitigate the interfacial stress while increasing the system toughness and resistance to failure. Soft materials, such as polymers and low homologous temperature metals, provide high interfacial compliance, however, at the expense of temperature sensitivity and low strength. Therefore, it becomes apparent that an effective interface material should possess the thermal stability and properties of common

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ceramics or metals, and a microstructure that is designed to provide high compliance and resistance to fracture. In the recent years, several applications have been shown to benefit from films comprised of discrete nanoelements, such as thermal interfaces for high power microelectronics [1] and nanostructured thin films for high capacity electrochemical anodes that are subject to large volumetric expansion [2–4]. Films and interfaces comprised of metallic or ceramic nanosprings, for example, reduce the coupling between normal and shear deformation and transform macroscopic shear forces into locally normal and bending forces on individual nanosprings. Such films are often fabricated by Glancing Angle Deposition (GLAD) which does not require photolithography to fabricate intricate, 3D micro and nanostructures. Among the advantages of this method are its compatibility with microelectronics processing, the capability for large area fabrication on virtually any flat surface, and the option to terminate the nanospring arrays with a fully integrated cap that facilitates uniform load transfer to all nanosprings in a film. In general, GLAD can produce films of slanted, straight, or helical micro- or nano-elements of monolithic or hybrid materials [5–10] by controlling the rotation speed of the substrate, the deposition rate, and the deposition angle. By this method, micro or nanostructures grow competitively due to “self-shadowing” because of the very shallow “glancing” angle (>80°) of the incident vapor with respect to the substrate. The nuclei that finally grow form a dense array of slanted columns. If slow substrate rotation is applied, the slanted columns evolve into helices: a full rotation of the substrate results in the growth of one full turn of a helix (spring). Unseeded nanospring films have been the most commonly fabricated GLAD materials, however, according to a recent study [11] they have inconsistent wire thickness and coil diameter, which limit our ability for versatile design of films with desirable mechanical stiffness. On the contrary, substrate seeding yields orderly arrays of nanostructures [12] with uniform geometry and tailorable properties. The latter still remain unexplored, because very little is currently known about the coupled relationships between the GLAD parameters, the material properties of GLAD-deposited structures, and the geometry and mechanical behavior of individual GLAD structures.

This study focuses on the material and mechanical behavior of individual nanosprings in seeded GLAD films comprised of tall Si nanosprings, in order to establish relationships between the deposition parameters and the resulting nanospring geometry and mechanical properties. These fabrication-structure-properties relationships will facilitate the informed design of thin films and interfaces with desirable mechanical behavior that departs from that of bulk metals and ceramics. The few early studies on the mechanics of SiO, Ti, and Cr GLAD films used nanoindentation with a spherical tip and treated the nanospring films as monolithic materials [13,14]. It was assumed that the individual nanosprings are formed by perfectly circular nanowires, which permitted the use of the analytical models by Ancker and Goodier to estimate the spring stiffness [15,16]. However, the shadowing process taking place during GLAD does not imply circular wire cross-sections. Furthermore, the shear response of GLAD spring films, which is of interest in most applications, has only been studied by side-loading the capping layer of Ta_2O_5 nanospring films with an Atomic Force Microscope (AFM) tip [17,18]. Among the shortcomings of these efforts are the application of unaccounted moments by the AFM tip during shear loading, the inability to apply a purely normal force by the tilted AFM cantilever, and the application of a point force on the cap of the film rather than uniform pressure. A direct measurement of the stiffness of individual springs was attempted by Liu et al. [19], who also used an AFM tip to load individual Si springs inside a film. The authors reported spring stiffness values that were lower than expected, attributing the issue to the application of off-axis loading by the AFM tip. However, major uncertainties could also arise from the assumption of a circular wire cross-section to facilitate the use of analytical spring models [15,16] as well as the unknown material properties of GLAD nanostructures.

The present study aims at quantifying and simulating the mechanical behavior of GLAD nanosprings to enable the design of advanced interfaces with desirable normal and shear stiffness. A systematic study of the geometric parameters affecting the mechanical behavior of individual amorphous Si (aSi) nanosprings was carried out. aSi was selected because it is elastic and brittle at room temperature, thus eliminating the convolution of structural spring deformation with material plasticity. Experiments were conducted via Microelectromechanical Systems (MEMS) type devices to measure the mechanical behavior of GLAD nanocolumns and nanosprings in tension, compression and bending. Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) studies provided the microstructure and the precise geometry of individual nanosprings. For the first time, an accurate description of the coil and wire geometry was employed in modified analytical models and a Finite Element (FE) model for comparison with the experimental results, which allowed us to estimate the effective material properties of different nanostructures fabricated by GLAD.

2. Experimental methods and materials

2.1. Fabrication of Si nanospring films

Si nanospring films were fabricated by GLAD on unseeded (Fig. 1(a, b)) and seeded (Fig. 1(c–f)) Si substrates at Micralyne in Edmonton, Canada. The seeded films were deposited on a Si wafer in a hexagonal pattern of 500 nm tall Si seed posts with 900 nm or 1500 nm spacing, which were created via Deep Reactive-Ion Etching (DRIE). This seed arrangement generates relatively uniform shadowing during substrate rotation, and thus films with quasi-isotropic in-plane properties. The substrate was titled at 85° and rotated at constant speed to control the wire diameter, coil diameter, and helix angle. The deposition rate was 10 Å/s for all spring types, while the substrate rotation rate was 4.2°/min for 4-turn springs and 10.6°/min for 10-turn springs. The geometry of the resulting structures was spring-like, except for the 10-turn nanosprings with 1500 nm seed spacing that were screw-like (Fig. 1 (f)). All nanospring types were 10 ± 0.5 μm high. Similarly, 10 μm long aSi columns were fabricated on seeded substrates to measure the material properties of the GLAD-deposited columns. The unseeded films resulted in highly intertwined springs, with a broad distribution of wire diameters (Fig. 1(a,b)). Therefore, unseeded films provide limited control on the mechanical properties and structural uniformity. The spring intertwining can be reduced by increasing the number of coil turns per unit length, as deduced from a comparison of Fig. 1(a) and Fig. 1(b) where the separation between individual springs was increased by reducing the spring pitch height from 2500 nm (4-turn coils) to 1000 nm (10-turn coils), or by substrate seeding: increased seed spacing reduces the overlap between adjacent springs (Fig. 1(c–f)). Notably, the seed spacing and pitch height not only affect the arrangement of nanosprings but also influence their geometry and properties.

2.2. Mechanical characterization of individual Si nanosprings and nanocolumns

Single nanosprings were isolated under a high magnification optical microscope with a use of a micromanipulation stage, and were placed onto a MEMS device for testing in tension/compression and bending via a MEMS-based optical metrology method [20]. The Si nanosprings were fixed onto the MEMS devices via platinum (Pt) tabs deposited with a Focused Ion Beam (FIB) (Fig. 2(a–d)), which fully constrained the spring ends and approximated fixed-fixed boundary conditions. Ultra-low FIB current averted any deposition of Pt onto the springs. Only springs from seeded substrates (Fig. 1(c–f)) were tested because the density and the irregular geometry of unseeded springs (Fig. 1(a, b)) made it impossible to isolate them from their films. Finally, uniaxial tension experiments with straight Si columns (Fig. 2(e)), also fabricated