Fabrication and Mechanical Properties of Porous 3D Nanostructures

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Many naturally occurring hard materials such as bones, sea sponge skeleton, and wood have superior mechanical properties, and are simultaneously lightweight compared to man-made materials of same composition. The high stiffness and toughness of these cellular materials can be attributed to the combination of material composition and geometric architecture at micro/nano scale. The elastic modulus for cellular solids are related to density through $E/E_s \propto \rho_R^n$, where E_s and E are elastic modulus for solid and cellular material, respectively, ρ_R is relative density, and n is scaling factor dependent on geometrical arrangement of components. Synthetic cellular materials such as silica aerogels, metal foams, and polymer foams have random constituent elements and the material modulus scales poorly with density ($n \sim 3$). Recent research in ultra-light materials is focused on fabricating ordered structures of polymer microframe using multiple beam interference lithography [1], hollow metallic and ceramic microstructure [2-4] to achieve favorable scaling of elastic modulus with density.

Here we present a fabrication technique combining colloidal lithography with atomic layer deposition (ALD) to make ordered ZnO porous nanostructure. The fabrication process begins with illumination of monodispersed periodic nanospheres of diameter 500 nm with UV light source ($\lambda = 325$ nm). The resulting complex intensity pattern is recorded in the underlying positive-tone photoresist layer, to fabricate complex 3D periodic nanostructure [5] as shown in figure 1(b). The polymeric nanostructure is then conformally coated with ZnO using ALD process. Figure 1(c) shows a photoresist template coated with 30 nm ZnO. The sample is then heated at 550°C for 30 minutes in a furnace to burn the photoresist template and get a free standing hollow 3D porous nanostructure as shown in figure 1 (d).

The fabricated 3D porous nanostructures are tested for their mechanical characteristics using nanoindentation. A probe with radius of 10 μ m is used for nanoindentation and a cyclic incremental loading mechanism is used to measure mechanical response of samples. The elastic modulus of bulk ZnO measured using nanoindentation is about 146 GPa. Figure 2(a) shows the SEM image of 30 nm ZnO structure after nanoindentation at 500 μ N force. The averaged elastic modulus of 30 nm ZnO hollow nanostructure is plotted in figure 2(b) and is about 7.68 GPa. We will study the effect of porosity of ZnO 3D structures on the scaling factor (*n*) for elastic modulus. The porosity will be varied by changing the thickness of ZnO layer during ALD process. We will also study the effect of scaling factor on hardness and yield strength of the structure.

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Figure 1. Fabrication process for 3D hallow ZnO nano-porous structures. (a) Schematic diagram showing stack design for making 3D photoresist template. Thickness of ARC and photoresist layer is 98 nm and 1 µm respectively. The arrows indicate direction of UV light for photoresist exposure. (b) SEM image of Photoresist template after lithography showing complex 3D structure. (c) Photoresist template coated with conformal 30 nm ZnO layer using ALD process. (d) SEM image of free standing hollow 3D ZnO structure after burning the photoresist template.



Figure 2. (a) SEM of 30 nm ZnO nanostructure after nanoindentation. Dotted circle marks the circular indent made in the structure (b) Elastic modulus plotted as a function of contact depth for a series of experiments. The black dotted line indicates averaged elastic modulus value.

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