

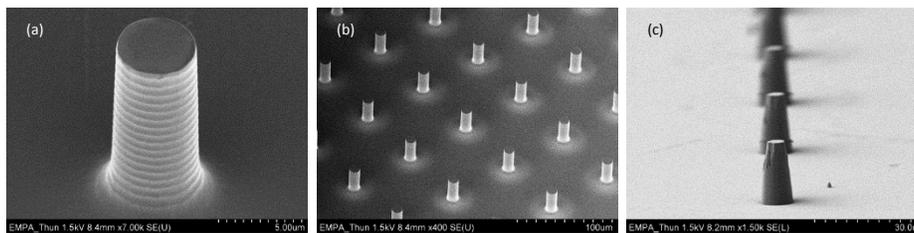
# NANOMECHANICAL TEST SPECIMEN PREPARATION TECHNIQUES BY MICROFABRICATION AND TWO-PHOTON POLYMERIZATION TO AVOID FIB INDUCED GALLIUM IMPLANTATION DAMAGE

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Traditional mechanical test specimen preparation methods require a subtractive approach to define the structure out of the bulk material. The most commonly used technology, focused ion beam patterning, leaves a modified specimen surface by gallium implantation resulting in for instance an altered grain structure. In this work, purely chemical approaches and an additive manufacturing technique are introduced to define test specimen.

Compression pillars have been fabricated out of single crystal silicon and glass. The developed silicon process is crystalline orientation independent; consists of optical lithography, reactive ion etching via an alternating fluorine plasma and polymer passivation, surface oxidation and HF wet etching for sidewall planarization. High aspect ratio structures are achievable with a sub-50 nm surface roughness and parallel sidewalls.

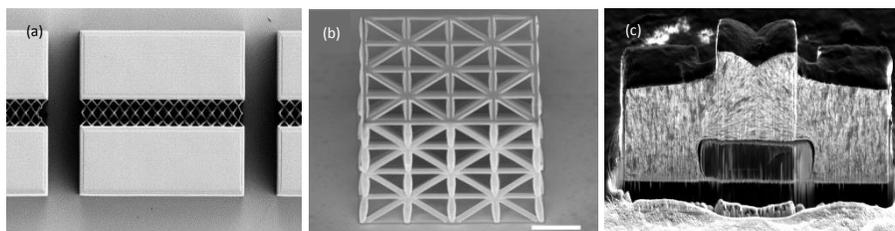
Glass pillar microfabrication requires a hard metallic mask due to the relatively low selectivity of any etchant plasma. Sputtered aluminum is patterned by photolithography and a chlorine based dry etch. The glass is reactively etched in a fluorine based plasma via this mask. The aluminum is then selectively removed by wet etching. The process has been demonstrated on a fused silica substrate with an average of  $84^\circ$  sidewall angle, although a wide variety of glasses may be used. Glass purity influences the sidewall angle.



*Figure 1 – Compression pillars created by microfabrication. (a) A 5- $\mu\text{m}$ -wide and 15- $\mu\text{m}$ -tall silicon pillar with sub-50 nm sidewall roughness. (b) An array of 15- $\mu\text{m}$ -wide and 45- $\mu\text{m}$ -tall silicon pillars. (c) A series of 5- $\mu\text{m}$ -wide and 15- $\mu\text{m}$ -tall fused silica pillars.*

A novel additive manufacturing technique, two-photon polymerization has been successfully combined with electroplating, electroless plating and atomic layer deposition to create hybrid nanocomposites of metal, ceramic and polymer structures with a precise unit cell definition on the submicron scale.

Crosslinking-type polymer structures are patterned with down to 100 nm lateral resolution using a Nanoscribe Photonic Professional GT laser lithography system. The polymer is then optionally surface coated with a ceramic (via ALD) or metal (PVD sputtering or electroless plating), and filled by electroplating. The polymer support can be removed by a fluorine based plasma.



*Figure 2 – Mechanical test specimen created by two-photon lithography. (a) Polymerized structure of a nanocomposite before filling the interstitial space with electroplated nickel. (b) Polymer structure to demonstrate engineered ductile to brittle transition; constructed of 1  $\mu\text{m}$  wide pillars and surface coated with metal via electroless plating. (c) A microshear test structure created by filling a polymer shell with electroplated nanocrystalline nickel.*