FIB Milling and Replica Molding of Complex Surfaces with Atomic-Scale Precision

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The deterministic fabrication and efficient replication of nanometer-scale structures with atomic-scale precision has long been a foundational goal of nanotechnology, but is still not routinely realized in many important application areas. Here, we advance and integrate focused ion beam (FIB) milling and replica molding, in an innovative approach to prototyping and manufacturing complex three-dimensional surfaces with atomic-scale control over height and depth in a variety of essential materials. Our approach will facilitate the development and application of nanotechnologies in fields involving optical, fluidic, and electronic devices directly benefitting from such dimensional control.[1-2]

Our patterning process depends on characterizing the atomic- and nanometer-scale topography of a silicon surface in response to gallium FIB bombardment, as shown in Figure 1. At doses $\gtrsim 10^{-2}$ pC·µm^{-2}, the surface swells up to heights of $\gtrsim 0.2$ nm, as a result of silicon amorphization. At a dose of ≈ 0.15 pC·µm^{-2}, the surface swells up to the maximum height of ≈ 1.1 nm, with accompanying sputtering and redeposition at pattern edges. Higher ion doses mill the surface back down, returning to its initial height at a dose of ≈ 6.5 pC·µm^{-2}.

We optimize the FIB parameters for milling silicon at higher doses, as shown in Figure 2. FIB milling enables the three-dimensional (3D), direct-write patterning of silicon surfaces with heights and depths varied across the nanometer scale. Root-mean-square (RMS) surface roughness increases from the atomic scale and saturates for deeper structures at ≈ 1.1 nm due to secondary milling of asperities.

We can pattern complex 3D surface topographies of arbitrary design, as shown by the bitmap images patterned in Figure 3 and Figure 4, which is highly advantageous for rapid prototyping. Atomic-scale control over surface heights and depths is evident in atomic force microscopy (AFM) images.

Finally, we transfer such structure patterns into silicone by replica molding with nearly perfect fidelity, as shown in Figure 4. The shallower silicon and silicone staircases have single step depths of (0.43 ± 0.13) nm and (0.40 ± 0.14) nm, and RMS surface roughness values of (0.26 ± 0.01) nm and (0.59 ± 0.06) nm (average ± standard deviation), respectively. The deeper silicon and silicone staircases have single step depths of (0.86 ± 0.08) nm and (0.92 ± 0.05) nm, and RMS surface roughness values of (0.17 ± 0.01) nm and (0.35 ± 0.05) nm, respectively.

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[1] A. M. Bowen et al., *Advanced Functional Materials*, **22**, 2927 (2012).
[2] E. J. Smythe et al., *ACS Nano*, **3**, 59 (2009).

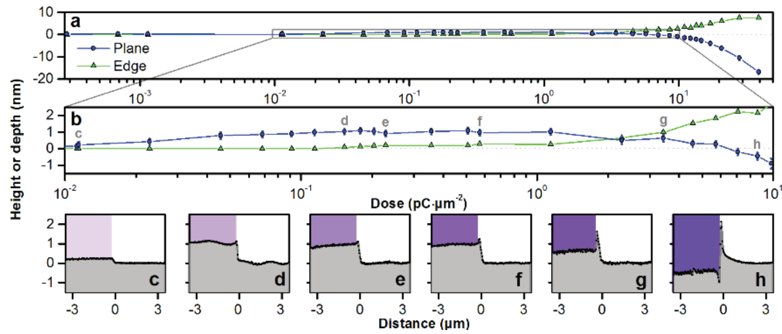

Figure 1: *Characterizing the topography of a silicon surface in response to FIB bombardment.* (**a**–**b**) > 5 orders of magnitude of dose variation causes swelling up, milling down, sputtering away, and redepositing at the edge of a step pattern in silicon. (**c**–**h**) AFM sections showing the profiles of the surface as the dose increases. The bombarded area is indicated by violet shading. Swelling and milling are evident on the pattern plane, while redepositing and secondary milling are evident at the pattern edge.

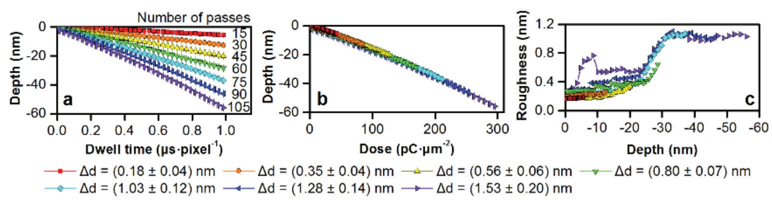

Figure 2: *Milling staircase structures in silicon*. (**a**) Step depths increase approximately linearly with both dwell time and pass number. (**b**) Step depths increase approximately linearly with total dose delivered. The effect is reproducible for different increments of dose per step. (**c**) The root-mean-square surface roughness increase from ≈ 0.3 nm to a maximum value of ≈ 1.1 nm at a depth of ≈ 60 nm.

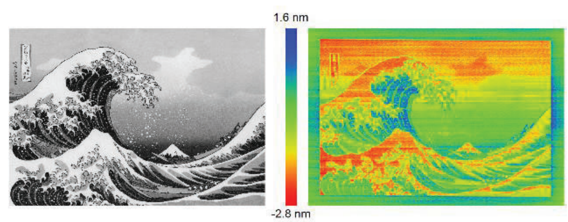

Figure 3: *Rapid prototyping of complex patterns in silicon*. "The Great Wave off Kanagawa" was published by Katsushika Hokusai between 1829 and 1833. A bitmap image of this work of art is converted from color to grayscale and milled into a silicon substrate, as characterized by AFM. Atomic-scale control over heights and depths is evident.

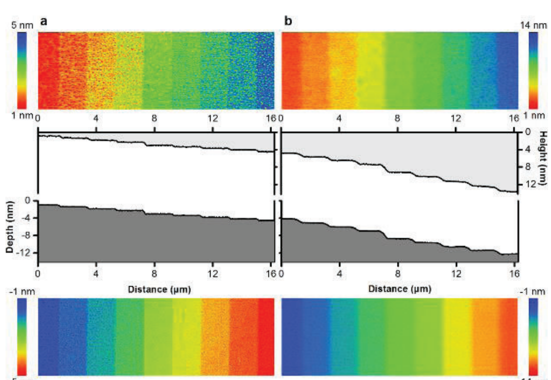

Figure 4: *Transferring patterns from silicon to silicone with atomic-scale precision*. AFM images and sections of silicon masters (dark gray) and silicone replicas (light gray). (**a**) Shallower and (**b**) deeper staircase structures are shown, with additional patchy features imprinted into silicone from the mold release.