Top-Down Fabrication of Patterned, Vertically Aligned Silicon Nanowires

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Abstract and Introduction:

Silicon nanowires (SiNWs) have recently attracted significant attention due to their one-dimensional structure [1] and semiconducting properties [2]. With many potential applications in micro- and nano-electro-mechanical systems (M/NEMS) [3], the development of various SiNW conformations is a popular area of current research. Challenges, however, such as controlled fabrication, presently keep this promising technology from reaching mainstream industry.

The aim of this project, therefore, was to develop a method of synthesizing patterned, vertically-aligned SiNWs with highly controlled diameters and lengths using a top-down fabrication method. The ultimate use of this methodology will be to develop various three-dimensional (3D) shapes and curvatures of SiNWs, with the hopes that the manipulation of these structures can offer many applications, especially in M/NEMS devices.

In order to realize the ultimate goal of 3D SiNW microstructures, controlled synthesis must first be accomplished. Recently, there has been success in NW fabrication with relatively controlled dimensions and densities using both bottom-up and top-down methods [4]. In the present work, the latter approach was used, in order to properly pattern the nanowires into desired microstructures.

The process began by photolithographically patterning Si wafers, and then etching them so that Si micropillars remained on the wafers. Then, in order to form SiNWs from the pillars, a metal-assisted chemical etching (MACE) mechanism [5] was employed, during which the wafer was submerged in a solution of hydrofluoric acid (HF) and silver nitrate ($AgNO_3$), resulting in SiNWs that collectively were shaped in the original patterned structure.

For the current purpose, however, there were restrictions present. During the etching process, the desired mechanism was for etching to occur only at the top of a micropillar, and not at its sides nor at the substrate level. In order to achieve this, an etch mask had to be laid down to coat the sidewalls and the substrate to protect them from the MACE process. Various masks were tested, and the results showed that a Teflon®-like polymer deposited by deep reactive ion etching (DRIE) from C_4F_8 plasma produced an effective mask.

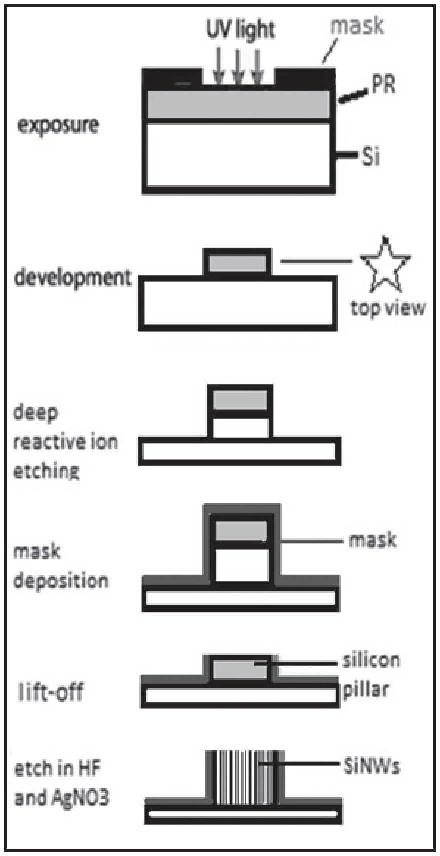

Figure 1: A map of the step by step SiNW fabrication method.

Experimental Methods:

A basic photolithography process combined with DRIE was employed to pattern micropillars on bare Si wafers (Figure 1). After the spin-coating of photoresist (PR, SPR 220-3.0, 3.5 μm), the wafers were exposed to ultraviolet light for six seconds in a mask aligner, exposing the areas intended for patterns. After development of the PR for one minute, Si micropillars (20-40 μm tall) were formed by DRIE. Then, the etch mask was deposited using only the passivation layer (no etchant) in the DRIE process, flowing C_4F_8 plasma (200 sccm, 0.5-1.5 minutes).

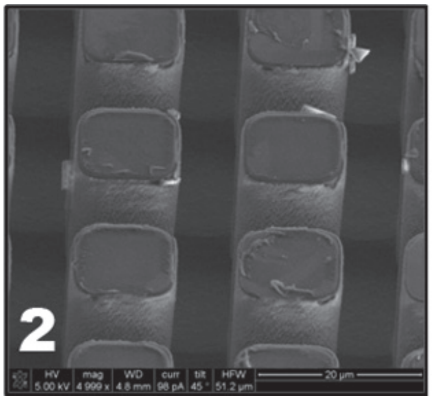

Figure 2: Patterned micropillars of Si coated by Taffy that are now ready for the MACE process.

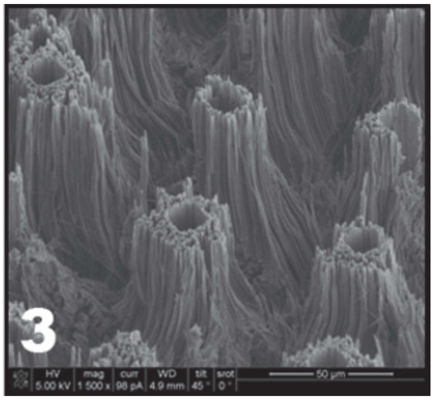

Figure 3: SiNWs that have self-densified into structures during a one hour etch.

Next, after lifting off the mask from the top of the pillars by sonicating in acetone (16 minutes), the samples were ready to etch (Figure 2). The etch bath consisted of 5M HF and 0.04M $AgNO_3$ at 50°C, and the samples were placed in it for 10 minutes. They were then removed and rinsed with nitric acid. Finally, the samples were imaged using scanning electron microscopy (SEM).

Results and Discussion:

Our first tests set out to investigate the effect of patterning Si substrates on the SiNW fabrication process. After photolithography and DRIE were done to form shaped micropillars, samples were placed in the etch bath for one hour. As we can see in Figure 3, the process was successful. SiNWs were synthesized with aspect ratios of up to 3000, and they, in fact, seemed to self-densify based on the pre-patterning of the substrate. Although it was promising, this behavior did not occur everywhere, however. Certain patterns (like the one shown) formed SiNWs as anticipated, but for others, there was more etching at their sides and at the substrate level than there was at their tops. Therefore, it was evident that the original etch mask—aluminum oxide, Al_2O_3—was insufficient and needed to be replaced.

After testing many materials, one was found to effectively mask the micropillar sides and substrate—a Teflon-like polymer deposited by the DRIE process. Depositions between 0.5-1.5 minutes resulted in thicknesses of the polymer, or "Taffy," that ranged from 100-500 nanometers. These thicknesses worked well to protect the sidewalls of the pillars, as well as the substrate, while still allowing for etching to occur at the top. In Figure 4, we can see that bundles of SiNWs, about 50-300 nm in diameter, are evident only at the top of a micropillar. This process was therefore very promising in fabricating vertically aligned SiNWs that assume a pre-patterned shape, and ultimately, 3D structures.

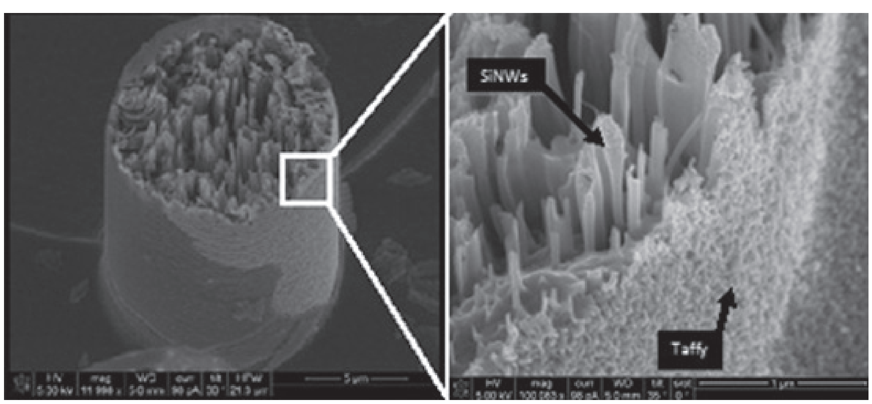

Figure 4: A micropillar that has SiNWs forming at the top, while the sides remain coated by Taffy.

Future Work:

Despite the promising results, there is still optimization to be done. In some areas between patterns, the Taffy did not seem to adhere to the substrate. For this reason, it came off during the etching and exposed the underlying area to the HF/$AgNO_3$ combination. Further studies should try to understand why this occurs and change the parameters so that uniform masking can occur. Once the minor hurdles are settled, controlled synthesis of patterned, 3D SiNW structures should be realized.

Acknowledgements:

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References:

[1] Chang, S., et al. "Densely Packed Arrays of Ultra-High-Aspect Ratio Si Nanowires." Advanced Functional Materials (2009): 2495-2500.

[2] Cui, Y. et al. "Nanowire Nanosensors for Highly Sensitive and Selective Detection of Biological and Chemical Species." Science (2001): 1289-1292.

[3] Feng, X.L., et al. "Substrate Bending for Reversible Control of Frequency and Quality Factor of Nanostring Resonators." Nano Letters (2007): 1953-1959.

[4] Hahm, J. and C.M. Lieber. "Direct Ultrasensitive Electrical Detection of DNA and DNA Sequence Variations Using Nanowire Nanosensors." Nano Letters (2004): 51-54.

[5] Hochbaum, A.I., et al. "Enhanced thermoelectric performance of rough silicon nanowires." Nature (2008): 163-167.