Large-Area, Freestanding, Si Nanowire Arrays Encapsulated in Organic Matrices

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The Metal-Assisted Chemical Etch (MACE) is a novel etch technique capable of transferring sub-100nm patterned features¹ deep into Si, with minimal undercut or mask erosion. However, device fabrication by MACE processing still faces some fundamental issues: 1) feature aggregation during drying, due to capillary forces², 2) non-uniform feature removal from the mother wafer, and 3) damage to the underlying metal pattern. These impede the use of MACE in applications where discrete, uniform structures are needed, such as Li-ion battery electrodes.

Here we report methods permitting solutions to these three issues. We show: a) encapsulation of arrays of sub-100nm diameter nanowires in organic matrices, allowing preservation of the array pattern during drying; b) facile removal of the composite array from the mother wafer, and uniform cleavage of nanowires; c) minimal damage to the underlying metal pattern during removal (Figure 1d).

Initial nanopatterns, fabricated by thermal nanoimprint and metal deposition/liftoff, were square arrays of nanoholes 50nm in diameter with 200nm pitch in a 25nm-thick Au film on 4" p-type Si wafers. After patterning, the wafer was immersed in MACE solution, rinsed in solvent, and then immersed in a high wt% polymer solution for \sim 30 minutes. Next, the wafer was dried in air for >12hr at $\leq 60^{\circ}$ C. Finally, the composite layer was separated from the mother wafer by placing it on a hotplate at 100° C for \leq 5 minutes.

The composite structures were inspected by SEM, seen in Figure 1b, revealing well-preserved array structures, and organic sheaths surrounding individual wires. Additionally, decreased feature pitch, and notable "curling" of composite flakes, seen in Figure 1c, provided insight into mechanisms of film delamination from the wafer.

The resulting freestanding composite flakes could be easily transferred onto adhesive tape, representing a new avenue for the placement of high-aspect-ratio patterned arrays of Si nanowires onto various substrates.

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¹ Peng, K. Q., Yan, Y. J., Gao, S. P., & Zhu, J. (2002). Synthesis of large-area silicon nanowire arrays via self-assembling nanoelectrochemistry. *Advanced Materials*, *14*(16), 1164–1167.

² Logeeswaran, V.J. et al. (2010). Harvesting and Transferring Vertical Pillar Arrays of Single-Crystal Semiconductor Devices to Arbitrary Substrates. *IEEE Transactions on Electron Devices*, *57*(8), 1856–1864.

Figure. 1. a) 70° SEM micrograph of un-encapsulated, 50nm-diameter, $>20 \mu m$ tall Si nanowires produced by MACE; b) SEM micrograph of polymer sheath surrounding individual nanowires due to encapsulation in cellulosic polymer; c) SEM micrograph of composite "flakes", showing delamination from substrate, and dramatic "curling"; d) image of Au nanopattern remaining on Si "mother" wafer after composite removal, showing polymer residue, but no marring of the pattern.