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Fabrication of single vertically aligned carbon nanotubes for cellular electrochemistry

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In the field of nanomaterials, since the discovery of carbon nanotubes (CNTs) [1], these have found numerous applications due to their unique properties, e.g., electrical conductivity, chemical, thermal and mechanical stability and suitability for functionalization. The potentials of the CNTs have been demonstrated, e.g., electrochemical biosensing, biomedical, supercapacitor and field emission applications [2-5]. Vertically aligned CNTs (VACNTs) have emerged as an interesting application area based on either chemical assembly of functionalized CNTs on surfaces [6] or catalyzed growth of CNTs using plasma enhanced chemical vapor deposition (PECVD) [7]. To control the dimension, density and positioning of the grown VACNT arrays, nanometer sized patterns of catalyst have been prepared using, e.g., electron beam lithography (EBL) [5] or nano-imprint lithography (NIL) [7].

In this work we describe optimization of a lithographic process to fabricate devices with individually addressable VACNTs with diameters in the range of 50-150 nm. The fabricated devices are developed for biological cell-based electrochemical applications to allow intracellular measurements. To facilitate individual addressability, the VACNTs were grown at the ends of lithographically patterned lead structures (made of titanium tungsten (TiW)) that form an array of 54 growth sites (see Fig. 1). Furthermore, to ensure that each of the VACNTs is the only electrically conducting entity, the leads were passivated using 100-nm PECVD silicon nitride (Si_xN_y) layer (see Fig. 2 for the schematic view of the device). EBL patterning of Zep resist facilitated both reactive ion etching of the Si_xN_y to reveal the discrete positions of the underlying TiW and subsequent lift-off of the Ni catalyst (15-nm layer). This approach eliminate the need of lithographic processing after CNT growth. VACNT growth was done through PECVD process in BlackMagic (Aixtron) at 750 °C and 6 mTorr using nitrogen atmosphere and C_2H_2 as a carbon source. To eliminate the charging effect of the applied plasma, which due to focusing on the thin conducting TiW leads could break the electrically conducting paths, a 50-nm polysilicon layer was sputtered on the wafer prior to EBL process to facilitate grounding during the PECVD process.

To optimize the dimensions of the openings etched in Si_xN_y , different diameters (ranging from 60 nm to 300 nm) were patterned in the EBL process. Figure 3 shows a VACNT grown from the deposited Ni islet in a nominally 240-nm opening in the Si_xN_y layer. The optimized process outlines the necessary steps for device fabrication. However, to increase the throughput of the process for wafer scale, further optimization will be conducted to facilitate lithographic processing using Cannon UV-stepper that allows patterning of sub-micrometer sized structures on an entire wafer. This approach will simplify the overall lithographic process, still enabling patterning of openings in the size regime optimized in this work using EBL.

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Figure 1. Optical micrograph of a micro-electrode array (MEA) fabricated on thermally oxidized silicon wafer (2.5 μm of oxide), with leads made of sputtered TiW (120 nm) and silicon nitride top coating (100 nm).



Figure 3. Electron micrograph (FEI Nova 600) of a single carbon nanotube grown from an opening of 240 nm. The bright spot at the nanotube's tip is the catalyst (Ni) nanoparticle.



Figure 2. Schematic view of a cross section of a single electrode pad: the carbon nanotube is grown directly from an opening etched in silicon nitride insulating layer.