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Electron Microscopy Characterization of Carbon Nanotube Based Electrical and Mechanical Test Structures

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The potential for integration of carbon nanotubes (CNTs) into working devices has long been recognized. However, to realize this potential, test structures must be fabricated to evaluate critical parameters such as pattern geometry, growth orientation, catalyst density, etc.[1] We present here a method for the controlled growth of CNTs in configurations suitable for mechanical, thermal and field emission testing. These structures display high pattern definition and uniform growth morphology.

CNT growth was achieved through chemical vapor deposition (CVD). A p-type silicon wafer (100) was etched for 30 seconds in Buffered Oxide Etch (BOE) to remove the native oxide layer. An iron nitrate (0.72M, 15mL) sol gel catalyst was spun onto the wafer at 3000 rpm for 30 seconds and dried. A generic photoresist was then spun on at 3000 rpm for 30 seconds. Patterning was obtained using various chromium and transparency photo-masks with an exposure time of 12 seconds and an intensity of 10 mW/cm^2. After development of the photoresist mask and etching the catalyst film in BOE for 40 seconds, the sol gel patterned die were placed in a horizontal tube furnace for CNT growth. Growth was achieved at 700°C for 30 minutes with an admixture of H_2 and C_2H_2 (385 sccm and 25 sccm respectively) as in Dong *et al* [2].

Characterization was performed using a field emission scanning electron microscope (FEI Sirion FESEM) and a high-resolution transmission electron microscope (Tecnai F-20 HRTEM). Figures 1(a) and 1(b) depict two post-growth patterns evaluated for integration into a thermal switch. The insets of both figures depict angled, higher magnification images of the pattern detail. Figure 2(a) demonstrates the ability to create complex arrays of CNT bundles with varying diameters for field emission experimentation. A more uniform array [Fig. 2(b)] was created for mechanical and thermal testing. Note that CNT growth is restricted to patterned catalyst locations in each case. Fig. 3(a) is a low magnification SEM image of an individual CNT bundle. The inset is of one section of the bundle's sidewall showing the vertically aligned CNTs at high magnification. This alignment is achieved through the density of growth provided by the catalyst. Fig. 3(b) is an HRTEM image revealing that the CNT consists of 5-6 graphitic layers. The surface of the tube is partially covered by amorphous carbon. This internal morphology is consistent across all growth patterns. The low number of layers present in these MWCNTs also suggests the potential of this method for producing double-walled or single-walled nanotubes. The method for controlled growth of MWCNTs presented here is thus highly adaptable and consistent in its internal morphology. Work is currently underway to optimize these structures for their mechanical, thermal and field emission characteristics.

References:
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[2] L. Dong, J. Jiao, C. Pan, D. Tuggle, Appl. Phys. A, 78, 9-14 (2004).
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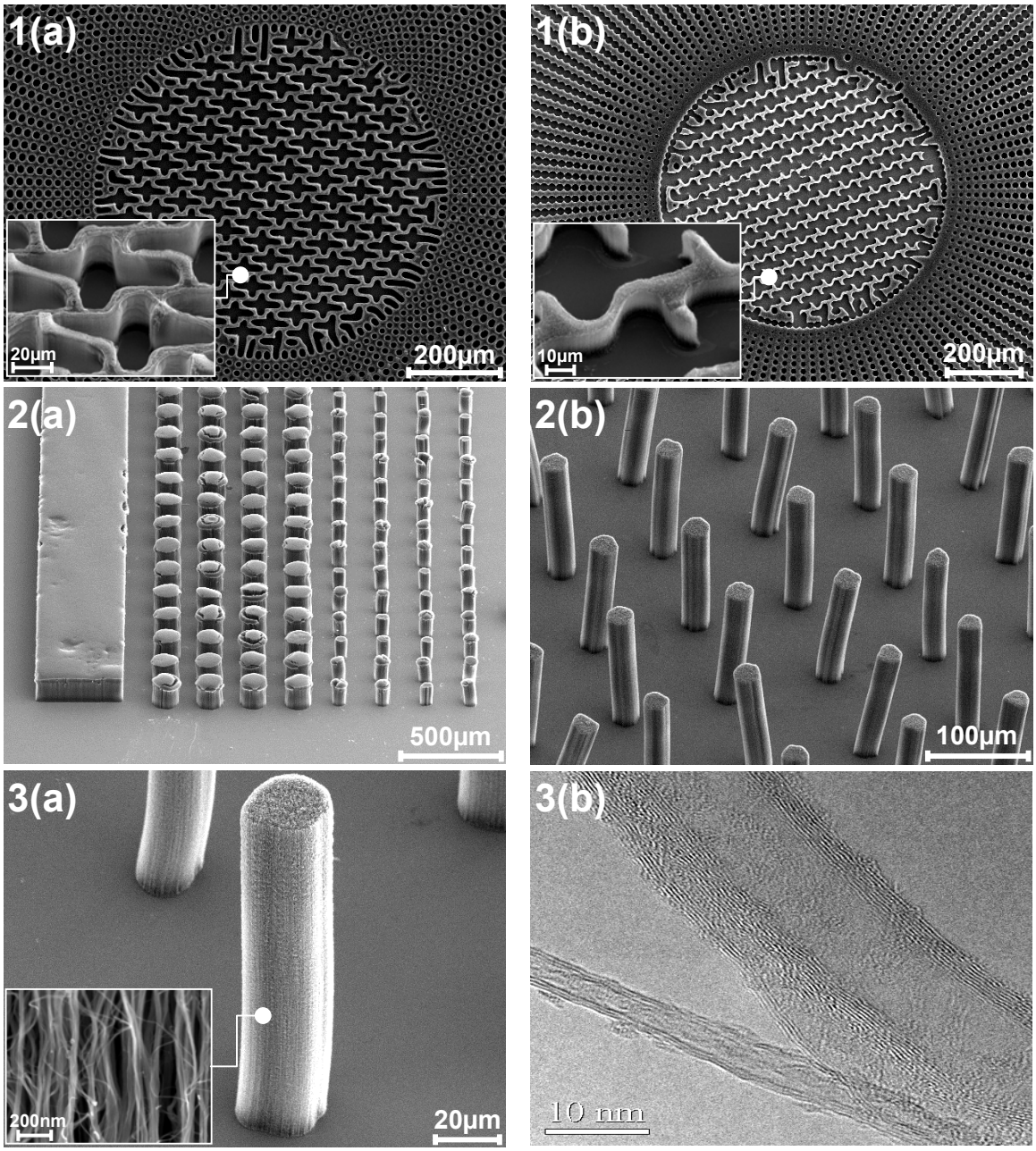

Fig. 1(a) and **Fig. 1(b)** demonstrate the complexity of patterns attainable with the reported CVD process. The insets in each case are magnified images of the MWCNT growth.
Fig. 2(a) and **Fig. 2(b)** show the definition and alignment possible in CNT bundle arrays.
Fig. 3(a) shows a vertically aligned CNT column rising from the location of a catalyst dot while **Fig. 3(b)** is a HRTEM showing a small number of carbon layers forming the outer wall of a single MWCNT.

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