Imaging and Characterization of Carbon Nanotube Growth and Transfer

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Abstract:

Carbon nanotubes (CNTs) have extremely high strength, flexibility, and excellent electrical properties as either a metallic or semiconducting material. Semiconducting CNTs are thus potential candidates for logic devices, such as carbon nanotube field effect transistors. Dense parallel arrays of CNTs are grown by chemical vapor deposition (CVD) on a quartz substrate and then transferred onto a silicon wafer, while, ideally, maintaining the same high density and alignment as the growth. However, issues with the current process include incomplete nucleation of CNTs at the catalyst strip along with a lower percentage of multiple transferred CNTs. We use atomic force microscopy (AFM) and scanning electron microscopy (SEM) to measure the approximate diameters and densities of CNTs and nanoparticles present during various stages of the growth and transfer.

In this work we characterized the diameters of carbon nanotubes and other nanoparticles present on the surface of various growth samples. We also characterized the surfaces of various lithography treatments to determine the plausibility of patterned ferritin as a CNT catalyst. This helped us to better understand these processes by determining how complete the chemical nucleation of CNTs was during the growth and how to improve future growths. A more complete understanding and further optimization of the growth and transfer processes parameters will allow for fabrication of devices with high CNT density, which is desirable for logic applications.

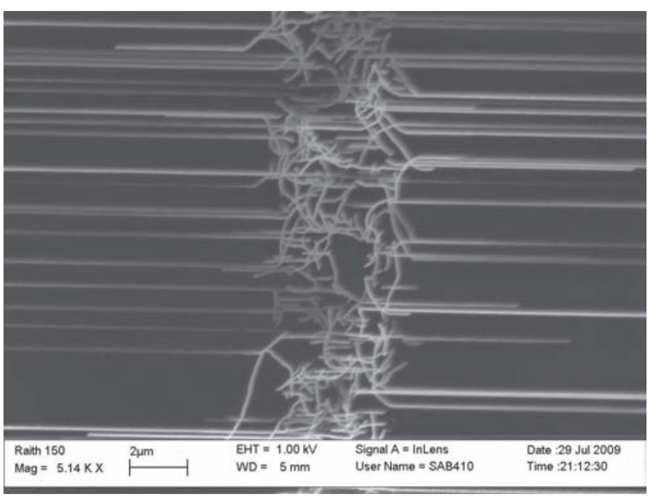

Figure 1: Catalyst strip with dense parallel growth.

Background:

Carbon nanotubes are essentially tubes of rolled up graphene with a typical diameter of 1-5 nm. In this work, we used tapping mode atomic force microscopy to obtain a topographical image of the CNT growth and transfer surfaces. The SEM was also utilized as a larger scale imaging tool to determine CNT density, Figure 1.

Methodology:

CNTs were grown onto a single crystalline quartz wafer and later transferred onto a silicon wafer to allow for complementary metal oxide semiconductor (CMOS) processing.

Iron nanoparticles were patterned into 5 μm wide strips on a quartz wafer. The wafer was then calcined at 900°C to remove any contamination. CNTs were then grown by CVD in a methane atmosphere which was raised to about 850°C. After the growth was complete, a gold thin film (~1500Å) was evaporated on the quartz, covering the CNTs. Thermally releasing tape was then applied to the gold/CNT combination, peeled off, and transferred to the silicon wafer. The wafer was then placed in an oven at 130°C, at which the tape released. The wafer then underwent three minutes of oxygen plasma cleaning and four minutes of argon plasma cleaning to remove any contamination from the tape.

After three minutes of liquid gold etch (sodium iodide, iodine, and water), parallel arrays of CNTs should be present on the silicon wafer.

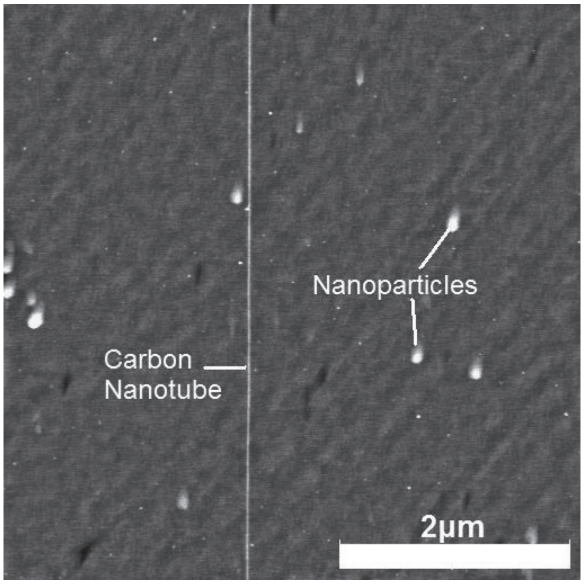

Figure 2: AFM image of CNT and nanoparticles.

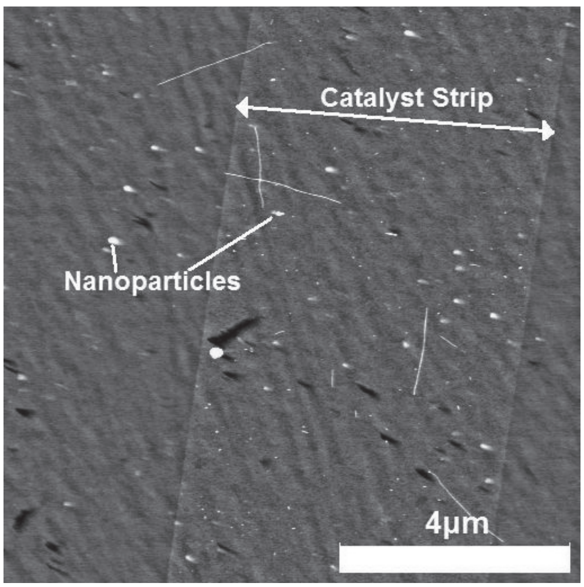

Figure 3: AFM of nanoparticles in and around the catalyst strip.

Results and Discussion:

Unsuccessful growths were investigated using the AFM to measure the diameters of the CNTs in areas of sparse growth. These diameters were then compared to the diameters of particles within the growth area, Figure 2. The average height of the CNTs was ~ 1.7 nm while the average particle height was ~ 2.4 nm.

Particles on and around catalyst strips with no nucleation, Figure 3, were then imaged with the AFM. These also had an average diameter of about 2.5 nm. It was concluded that these contaminant particles were amorphous carbon. This was supported by the relatively large diameter of particles, location of particles throughout the growth area, and the fact that amorphous carbon is a common impurity in CVD growth which indicates incomplete nucleation at the catalyst sites [1].

Issues with the current growth process led us to investigate ferritin, a globular protein complex that encompasses an iron nanoparticle, for patterned CNT growth. Ferritin has already been proven as catalyst for robust CNT growth [2], however traditional iron nanoparticle patterning techniques cannot be used and an alternative method of patterning has not been developed. Our goal was to pattern ferritin using the photoresist LOL2000 in order to yield a more patterned and aligned growth.

To test the solubility of ferritin in photoresist, we used AFM to image the following samples: ferritin with distilled water, patterned ferritin with two layers of photoresist, and patterned ferritin with the top layer of resist removed to determine the surface texture and roughness. We also imaged a lift off area of resist, as a surface texture control, and the areas close to the patterned region, to see if any particles were present on this un-patterned area.

By comparing the topographical images from the AFM, we were able to determine if the samples contained ferritin nanoparticles. The results of this experiment can be seen in Table 1. It was found that ferritin nanoparticles were clearly evident in the ferritin water solution. However, there was no evidence of ferritin nanoparticles in the patterned resist area or on the area adjacent to the patterned resist.

Conclusion:

In conclusion, it was determined that particles on catalyst strips and around CNTs of sparse growths were most likely amorphous carbon. It was also determined that ferritin is only soluble in water, not in the photoresist LOL2000, therefore a new method is required to pattern ferritin for growth.

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References:

[1] Franklin, N., Li, Y., Chen, R., Javey, A., and Dai, H. "Patterned growth of single-walled carbon nanotubes on full 4-inch wafers" Applied Physics Letters, Vol. 79, No. 27, pp. 4571-4573, 31 December 2001.

[2] Patil N., Lin A., Myers E., Wong H.-S.P., Mitra S. "Integrated Wafer-Scale Growth and Transfer of Directional Carbon Nanotubes and Misaligned-Carbon-Nanotube-Immune Logic Structures"; VLSI Technology, 2008 Symposium on, pp. 205-206, (2008).

Sample	Average Height (nm)	Peak Height (nm)	Ferritin Particles? (Y/N)
Ferritine DI Water	1.7	5.5	Yes
2 Layers of Resist	1	2.6	No
1 Layer of Resist	0.8	2	No
Blank Area (next to Dual Layer)	0.6	0.7	No
Blank Area (next to Single Layer)	0.6	0.9	No
Lift Off Only	0.6	0.7	No

Table 1: Average heights of AFM scans for ferritin test.

Process & Characterization