Synthesis of Ultra-Long Carbon Nanotubes on Bulk Metal Catalysts via Directed Feed-Gas Delivery

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Background

Carbon nanotubes (CNTs) are often synthesized by chemical vapor deposition (CVD), whether simply thermal CVD or a plasma-enhanced version (PECVD). Many different strategies have been implemented to try to increase the density of CNT growth and/or the length of individual tubes. Typically, thin metal catalyst layers (nanoscale thicknesses) are deposited on carrier substrates.[1,2] Difficulties with many processes that succeed in increasing tube length or nucleation density include increased cost, processing time, system complexity, and difficulty of post-processing.

There is a demand to develop fabrication strategies for longer, more dense CNT growth while reducing the limitations often present in current methods. Improvements are needed to reliably produce tubes for applications that take advantage of the superb mechanical, electrical, and thermal material properties offered by longer tubes.

Methods

At Nanomatronix, LLC, we have explored a proprietary technique to provide high nucleation density of ultralong CNTs (those with macroscopic lengths). The technique developed thus far has relied on standard CNT growth in a high-temperature PECVD system with some modifications.

A bulk metal catalyst coupon serves as the substrate on which CNT forests are grown within a high-temperature furnace under vacuum and subjected to plasma. High temperatures are typically required for CVD and PECVD growth of CNTs.[1,2] Here, the plasma serves multiple purposes: a pretreatment of the catalyst surface, enhanced activation of nanotube synthesis, and vertical nanotube forest alignment.

High temperature (>700 ºC) plasma pretreatment of the catalyst was found to produce nanoscale protrusions, as shown in the SEM images of Figure 1. These nanoscale protrusions provided nucleation points for individual or bunches of nanotubes, depending on their size. Thus, the combined conditions of temperature, feed gas ratios and flow rates, and plasma power promoted CNT growth directly from bulk metal surfaces.

Plasma pretreatment was performed under 1:1 Ar:H_2 flow. Growth of nanotubes was primarily achieved with feed gas flow rates near 1:1 CH_4:H_2. Flow rates and plasma power were tested over a range of temperatures and growth times to extend nanotube forest length and density.

Results

Figure 1 shows SEM images of a Ni catalyst surface after plasma pretreatment, demonstrating the nanoscale protrusions required for subsequent CNT nucleation.

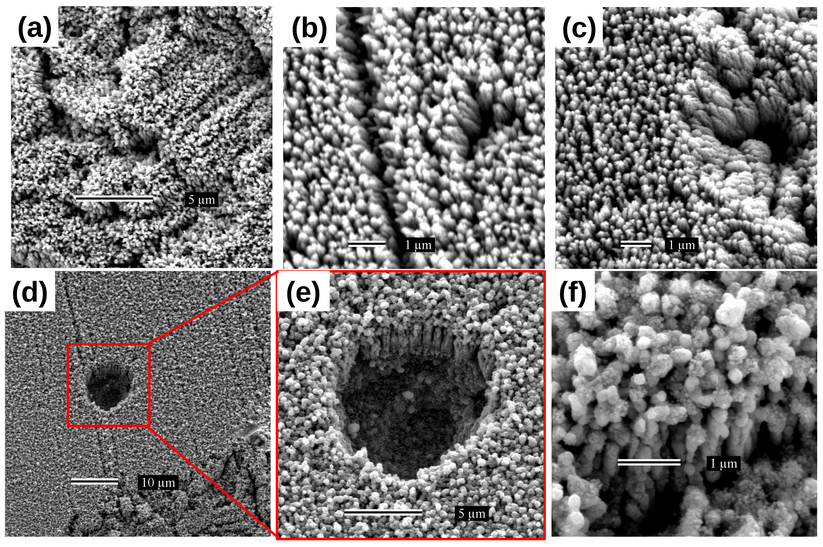

Figure 1

Figure 2 shows CNT forests (~ 5 µm in length) with high nucleation density across bulk Ni catalysts, grown after initial Ar/H_2 plasma pretreatment at 1100 ºC.

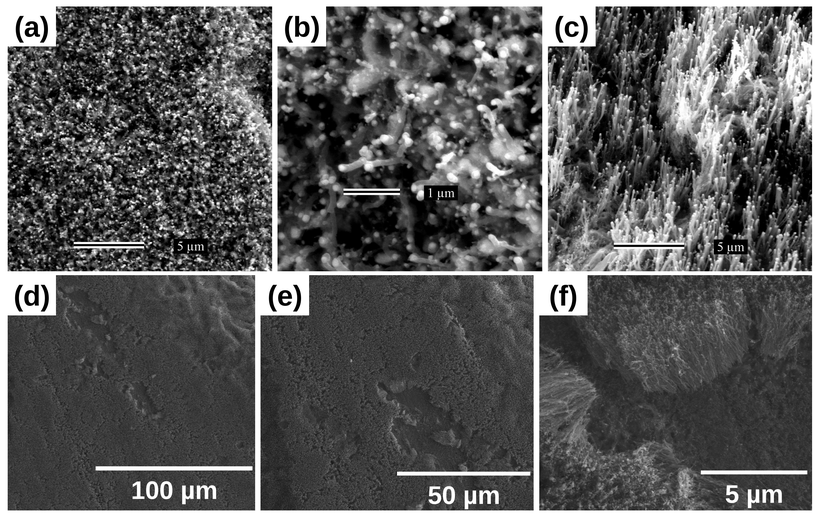

Figure 2

Figure 3 shows resulting CNT forests with ~20 µm length directly on bulk Ni catalyst surfaces.

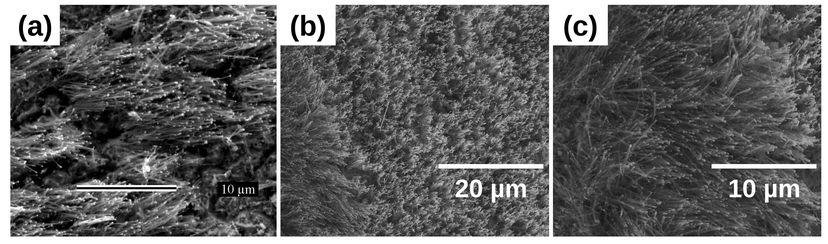

Figure 3

Conclusion and Future Work

Nanomatronix's modified PECVD furnace system was shown to enable dense CNT forest synthesis directly on bulk metal catalyst surfaces. Further developments will focus on increasing CNT lengths and nucleation density to take advantage of the processes' reduced processing complexity for various applications.

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References

[1] R. Zhang et al. *ACS Nano* 7(7) 6156–6161 (2013)

[2] M. Meyyappan et al. *Plasma Sources Sci. Tech.* 12 205-216 (2003)

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