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Quantitative Characterization of the Morphology of Multiwall Carbon Nanotube Films by Small-Angle X-ray Scattering

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Films of multiwall carbon nanotubes (MWCNTs) grown by thermal chemical vapor deposition were studied using small-angle x-ray scattering (SAXS). We assessed the extent of alignment of carbon nanotubes (CNTs) by examining relative SAXS intensities as a function of azimuthal angle. We also identified features in the SAXS patterns that correspond well to CNT diameters measured through high-resolution transmission electron microscopy. We determined that the alignment of CNTs as well as their average diameter can vary significantly throughout the film. This demonstrates the utility of SAXS for quantitative structural analysis of CNT films, indicating the potential to reveal new information about the CNT growth process, and relating variations in morphology to evolution of the catalyst and reaction conditions.

We have that shown small-angle x-ray scattering (SAXS) is a powerful tool for investigating the morphologies of multiwall carbon nanotube (MWCNT) films. CNT films have attracted significant interest from the engineering community because of their remarkable thermal, electrical, and mechanical properties, and because they show promise for use in nanoelectronics, energy-absorbing foams, superhydrophobic films, and power applications. MWCNT films can be grown by thermal chemical vapor deposition, resulting in films that range from vertically aligned to entangled and tortuous.

The extent of CNT alignment can only be assessed qualitatively using scanning electron microscopy, but we have shown that it can be characterized quantitatively in various regions of the CNT film by examining the relative SAXS intensities as a function of azimuthal angle. Additionally, by fitting the SAXS data to a model for cylindrical form factor, the average CNT diameter, and an estimate of the standard deviation, can be determined. The diameters determined through SAXS correspond well to high-resolution transmission electron microscopy (HRTEM) data. SAXS provides the advantage of sampling millions of CNTs in a single image, and also provides spatial resolution not accessible through HRTEM. The ability to characterize the CNTs as a function of position within the film allowed us to observe a systematic increase in the diameter as a function of height from the substrate as well as smaller diameter CNTs near the edge, relative to the center of the film. Systematic studies of the morphologies of the CNT forests as a function of variations in the growth conditions are underway and will provide insights into the fundamental mechanisms of the CNT growth process. Developing a complete understanding of this process is critical for enabling materials' structures and properties to be optimized and customized for specific applications.

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We have also investigated the effects of mechanical manipulations upon the morphologies of these MWCNT forests, including uniaxially compression and densification through solvent evaporation. A low-angle scattering feature in the data, corresponding to the interparticle structure factor, allows us to extract information about the packing density of the CNTs. We have recently performed USAXS measurements that confirm this result and additionally reveal the presence of CNT aggregates in these films. This analysis allows for determination of the average spacing between the CNTs, which varies significantly with mechanical manipulation.

SAXS provides rich morphological information that is not accessible through conventional microscopy techniques. This type of analysis can easily be applied to single-wall CNTs, zinc oxide nanowires, and a variety of other similar systems.

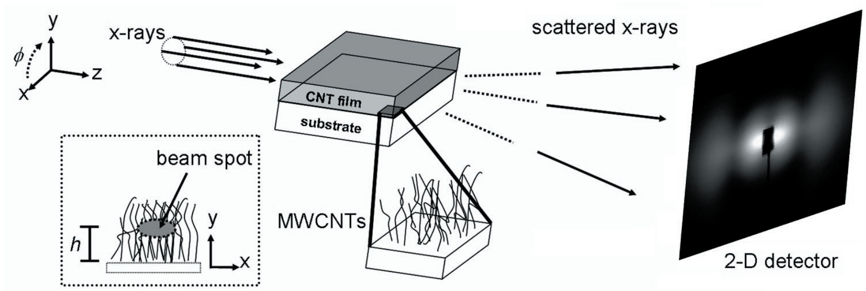

Figure 1. *Schematic of the experimental setup for small-angle x-ray scattering of multiwall carbon nanotube forests. A motorized stage provides spatial resolution allowing for the morphology to be investigated as a function of position within the film.*

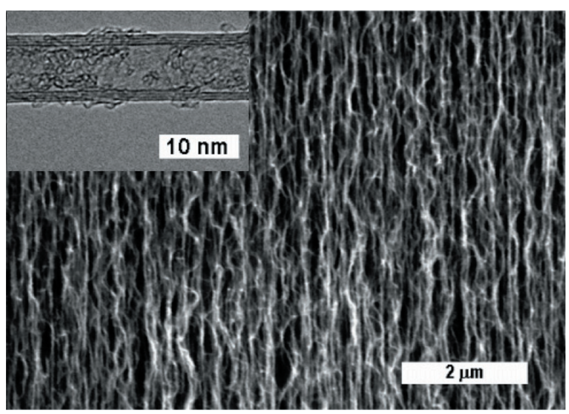

Figure 2*. Scanning electron microscopy (SEM) image of a vertically aligned multiwall carbon nanotube forest. Inset: transmission electron microscopy image of a single multiwall carbon nanotube. Small angle x-ray scattering (SAXS) provides quantitative characterization of the alignment, where only qualitative assessments of orientation can be made with SEM. SAXS measurements yield a "locally averaged" measurement of the CNT diameter, where TEM requires imaging of individual nanotubes.*