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Methods for TEM analysis of NIST's SWCNT SRM*

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The National Institute of Standards and Technology (NIST) is developing a series of single-walled carbon nanotube (SWCNT), reference materials (RMs), to provide researchers with well characterized materials for their applications. The SWCNT reference materials will be introduced as a series of three types of material: (1) raw soot characterized for composition, (2) purified (> 90 % SWCNT) bucky paper, and (3) dispersed, length-sorted populations characterized by length. The first material, bulk raw soot, is expected to be certified for atomic composition by NIST's highest standards, making it a Standard Reference Material (SRM). For the second material, the raw soot will be further processed through dispersing, filtration and washing to yield a bucky paper sample, which will be certified for composition. In the third material, raw soot will be taken through a purification and length-sorting procedure to yield a series of surfactant-suspended, length-sorted tubes classified as "long," "medium," and "short." General measurements made on these materials include transmission electron microscopy (TEM), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and Raman, ultraviolet-visible-near infrared and fluorescence spectroscopies. TEM is used in the analysis of SWCNT as a qualitative technique which provides a measure of purity for a given sample. TEM allows for the characterization of nanotube type (i.e., multi-walled, single-walled) and the degree of bundling in the structures. Electron-diffraction information from isolated SWCNT samples obtained with TEM can also be used to characterize the chirality.

To prepare a sample for TEM, SWCNT raw soot is suspended in a solvent, in which the concentration of a solution is determined by its color (a small tweezer pinch of nanotubes in solvent yields a light gray solution). The solution is deposited onto a carbon-coated copper TEM grid by pipette and allowed to dry in air. In figures 1 – 6, show examples of TEM images from a variety of sample preparations of the SWCNT SRM 2483 raw soot. A selection of dispersing solvents including organic solvents, aqueous surfactants, and DNA dispersions were examined.

All carbon nanotubes used for this study were samples taken from NIST's single-walled carbon nanotube raw soot SRM 2483, which was grown through the cobalt molybdenum-catalyst (CoMoCat) process and then further refined. From the raw soot material, a length-sorted material was produced by dispersing the nanotubes in a sodium deoxycholate surfactant solution. Once suspended, the carbon nanotubes were subjected to a series of ultracentrifugation steps to yield a length separated product. This product was characterized for length by atomic force microscopy (AFM). The samples were prepared for analysis in the TEM by depositing small drops of the various SWCNT dispersions onto holey carbon films on Cu TEM grids by use of a micropipette. All images presented here were taken at 200 keV under low-dose operating conditions in the TEM and were captured with a one megapixel CCD camera located below the viewing chamber at a TEM optical magnification of 200 kX to 400 kX.

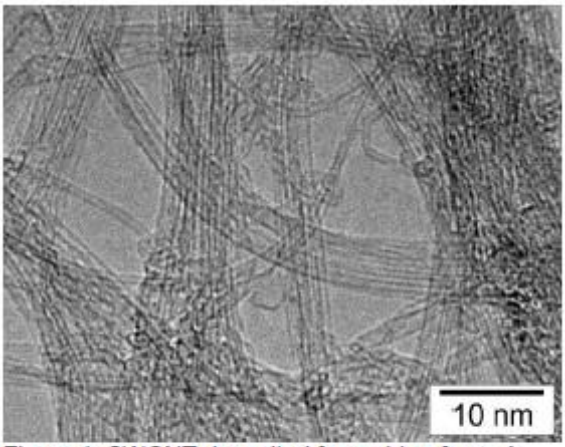

Figure 1: SWCNT deposited from chloroform. A representative area shows ropes and single nanotubes dispersed with one another.

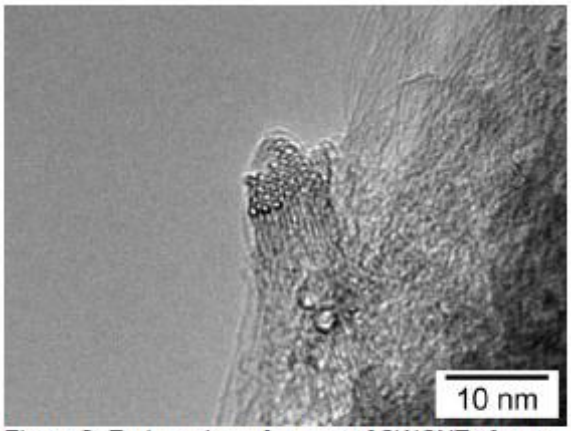

Figure 2: End on view of a rope of SWCNTs from a SWCNT sample deposited from chloroform. The little circles seen in the center of the image are ends of an array of individual CNT of tubes.

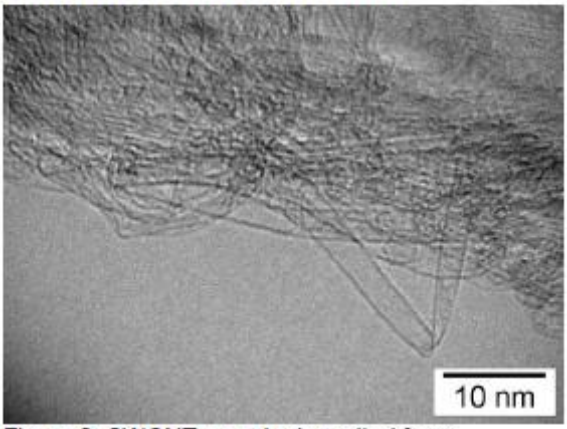

Figure 3: SWCNT sample deposited from chloroform. The wide region is a graphene sheet, approximately 2.5 nm in length, protruding from a CNT bundle.

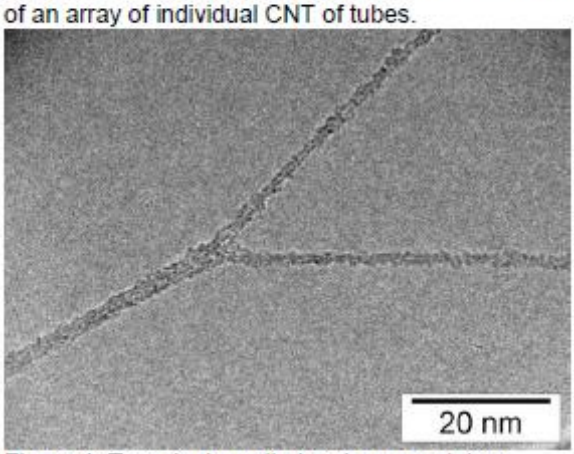

Figure 4: Two single-walled carbon nanotubes branch off from one another in a sodium deoxycholate sample.

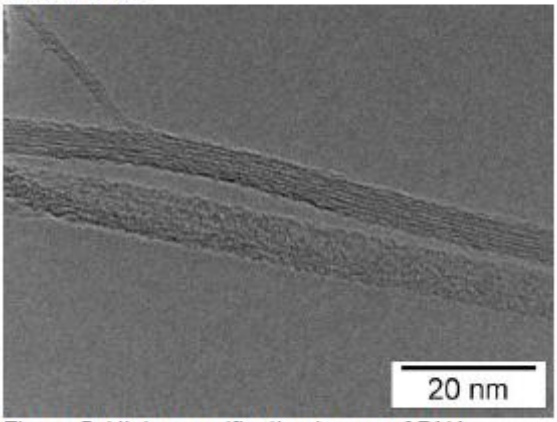

Figure 5: High magnification image of DNA-SWCNT. DNA is draping the SWCNT rope.

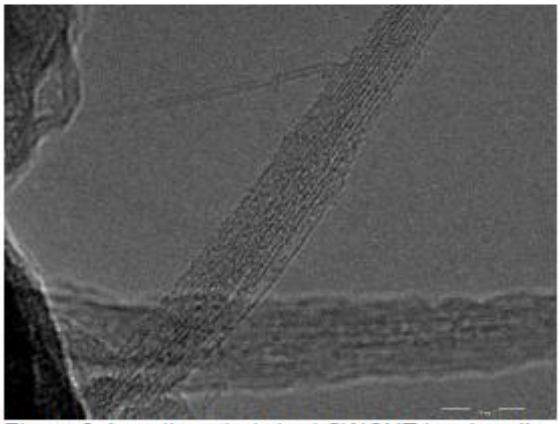

Figure 6. Length sorted short SWCNT in a bundle with an individual SWCNT separated at top.

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