

Mounting single-walled nanotube fibers on atomic force microscope probe tips

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A method of mounting fibers composed of single-walled nanotubes (SWNT) on the tip of a silicon atomic force microscope (AFM) probe has been developed. Visual characterization by scanning electron microscopy of the tips is presented. The visual evidence suggests the method is capable of producing tips capped by finely tapered fibers. The method involves generation of a fiber of SWNT material from a suspension of SWNT in N,N-dimethylformamide, manual alignment of the fiber on an AFM probe tip, and separation of the fiber by a focused laser. The tips are coated with a thin polymer layer to increase adhesion and provide stiffness and then re-cut with the laser to expose the nanotube material.

1. INTRODUCTION

Carbon nanotubes were discovered by Sumio Iijima in 1991 [1] in soot produced by an electric arc discharge between carbon electrodes. Nanotubes are characterized by diameters typically on the order of tens of angstroms to tens of nanometers, and their lengths, which are limited by the growth process, reach several microns. These rope-like molecules have a structural form that can be conceptualized as a long, thin graphene sheet rolled into a cylinder such that opposing sides are bonded. Carbon nanotubes occur as both single-walled nanotubes (SWNT) and nested structures of single-walled tubes called multi-walled nanotubes (MWNT). Nanotubes have physical properties that make them technologically attractive, including high thermal and electrical conductivity, high Young's modulus, and the ability to bend elastically and return to their original configuration.

Atomic force microscopy (AFM) is a technology that has benefited from the exceptional properties of carbon nanotubes. AFM relies on a sample-tip interaction to collect a variety of information about the sample, including topographic, chemical

potential, and magnetic features. AFM can provide nanometer scale resolution since the interaction distance is typically on the nanometer scale and the tips are sharpened to provide a microscopically fine point. Fixing bundles of MWNT and SWNT to the end of the probe can enhance the performance of the AFM tip due to the nanotubes' strength and wear properties. These nanotube tips remain sharp due to the durability of the carbon-carbon bonds. In addition, the nanotube diameter does not change as the tip wears down, unlike conventional pyramidal tips that flatten out with continued use.

Previous work has been done enhancing AFM probes with nanotube tips. Hafner *et al.* [2] swept an AFM tip across a silicon chip covered with vertical SWNT, causing some of them to detach and bond to the AFM tip. Hafner *et al.* also have grown nanotubes directly onto the AFM probe tip by chemical vapor deposition. [3]. Dai *et al.* demonstrated a method for manually attachment of nanotube material to the end of AFM probes. [4] The current project has developed a novel method of manually attaching single walled nanotubes to the end of an AFM probe tip using fibers of SWNT pulled from a dispersion of N,N-dimethylformamide (DMF) [5]. Orientation of SWNT fibers was visually characterized with a scanning electron microscope (SEM).

2. PROCEDURES

The AFM probe consists of a fine tip affixed to a flexible cantilever mounted on a flat silicon substrate. Because of the pyramidal shape of the probe tip, it is difficult to mount individual SWNT directly on the tip at the angle needed for AFM imaging. The author first attempted to circumvent this limitation by mounting nanotubes in successive stages: first a bundle of MWNT was mounted to the end of the probe tip normal to the

cantilever and then a fiber of SWNT was mounted on the bundle of MWNT. Finally, the system was stabilized with a polymer coating. However, under SEM imaging, it became clear that the limited control over such a complicated system made achieving a well-aligned nanotube tip prohibitively difficult.

The author then resorted to an alternative plan: mounting the SWNT fiber directly on the probe tip. A suspension of SWNT was prepared by sonicating purified SWNT buckypaper [6] in DMF for ten minutes. Unsuspended material was allowed to settle and a right-angle capillary tube was filled with the supernatant nanotube-DMF suspension. Electrical contact was made with the suspension by inserting a 5 cm long platinum wire into the capillary tube. Electrical contact was also made by a copper alligator clamp to a graphitized carbon fiber mounted with colloidal silver paint on a 1 cm long copper wire. The end of the fiber was inserted into the end of the capillary tube and connected to the positive terminal of a power supply (Kepko Power Supply, model 125-1M) held at 1.5 V, while the platinum wire was connected to the power supply's negative terminal. Current was passed for 10 minutes and then the fiber was slowly withdrawn. Nanotubes, which pick up negative charge in the DMF solution, were attracted to the carbon fiber which forms the positive terminal in this system. Once in intimate contact, the nanotubes adhered to each other and the highly graphitized carbon fiber by Van der Waals forces. A fine fiber of SWNT material formed on the end of the carbon fiber upon withdrawal from the capillary tube. The AFM probe tip was brought into contact with the SWNT fiber at the end of the carbon fiber and attached by Van der Waals attraction using the micromanipulator. The SWNT fiber was cut to a length of 2 μm by a laser (Uniphase μGreen SLM Laser, CW, 50mW, 532nm) focused by the microscope objective. In order

to prevent disruption of the weak Van der Waals force holding the SWNT fiber to the tip, the nanotube modified AFM tip was then coated with a thin layer of polymer by a method to be described fully in future literature. After coating, the SWNT fiber was shortened by laser cutting to a length of 0.8 μm under computer control (Lab View program courtesy of Zhihong Chen). This process exposed the SWNT material at the tip of the fiber as the polymer burned off at a faster rate than the nanotubes. Manipulation of the components was performed under an optical microscope (Axiovert 100 A optical stereomicroscope, 16X ocular, 5X and 50X objective) using two micromanipulation stages each with x, y, and z control (Newport OptoSigma 461 series).

4. DISCUSSION

Figures 1 and 2 represent the best orientation of a SWNT fiber on the AFM probe tip that the author attained. The long, thin taper of the SWNT fiber suggests that this method of nanotube mounting could produce tips capable of AFM imaging. Figures 3 and 4 illustrate one of the difficulties of this method: the tendency of the SWNT fiber to deform under laser cutting. This produces a curved tip end that is unsuitable for use in AFM imaging. Further work might focus on increasing tip uniformity and stability, developing a method to cut the SWNT fiber without deforming it, characterizing the tip with more sensitive detection methods to determine if it terminates in a single tube, and testing the tip as an AFM probe.

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