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# STUDY OF CARBON NANOTUBE FOR SCANNING PROBE MICROSCOPY

**Upendra Sharan Gupta[1], Tanay Sahu[2], Vikas Baghel[3]**
**Shailendra Singh Chouhan[4]**

*[1]Reader Dept. of Mech. Engineering, SVITS, Indore, (India)*
*[2,3,4]UG Scholar Dept. of Mech. Engineering, SVITS, Indore,( India)*

## ABSTRACT

*Atomic force microscopy (AFM) has become an indispensable tool due toits ability to image and manipulate matter at the nanometer scale in air,liquid, or vacuum. The AFM uses a micro- machined silicon or silicon nitride*

*probe mounted on a flexible cantilever that can sense or generate forcesbetween the probe tip and a sample surface. The AFM can thus be used aseither an imaging instrument or a manipulation device.Because it can acquire high-resolution topographical images in physiologically relevantaqueous environments, AFM has become especially important for structural biology and biophysics. AFM is distinct from other highly sensitive techniques for measuringintermolecular forces, such as the surface force apparatus and optical tweezers,due to the high spatial resolution possible and the capability to dynamicallymeasure and control time-dependent forces. In addition to obtainingtopographic images of biological structures, AFM can probe dynamic processesin solution, such as chemically and mechanically induced unfoldingmechanisms in proteins and DNA. The level of resolution possible in AFM for both single-molecule imagingand force transduction is ultimately limited by the structure of the tip.*

***Keywords- Atomic force microscope (AFM), carbon nanotubes, optical tweezers, micro- machined silicon, silicon-nitride probe,***

## I.INTRODUCTION

Carbon nanotubes are, in many respects, ideal high-resolution probe tipsfor AFM. Carbon nanotubes are hollow cylinders formed from rolled-upgraphene sheets that can be up to microns in length. A single-walled nanotube(SWNT) consists of a single graphene sheet, one atom thick, rolled upseamlessly into a cylinder with a diameter ranging from 0.7 to 6 nm. SWNTscan be used as high-aspect-ratio probes with radii comparable to molecular-scale dimensions. Individual SWNTs can bundle together, driven byattractive van der Waals forces, to form SWNT ropes containing up to hundredsof nanotubes each. Multiwalled nanotubes (MWNTs) consist of concentricgraphene cylinders and can have diameters ranging from 6 to 100 nm.Carbon nanotubes are chemically and mechanically robust. Both single-walled and multiwalled nanotubes are the stiffest material known, withYoung's moduli of about 1.25 to 1.3 TPa, which limits the noise due tothermal vibrations from degrading the ultimate obtainable resolution. Unlikeother materials, carbon nanotubes can buckle and bend

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elastically underlarge loads, limiting damage to both the tips and the sample. Becausenanotubes have well-defined molecular structures, the tip–sample interactionis better characterized and more reproducible than with conventionalprobes. This combination of mechanical properties and repeatable small sizeof SWNT probe tips makes them uniquely suited for robust AFM resolutionat the molecular scale.AFM probes fabricated with individual SWNTs vs. MWNTs or SWNTbundles have different imaging characteristics and properties. There aretrade-offs between various figures of merit for each type of nanotube probe,and the choice of which one to use will be different depending on theintended application. For example, AFM probes assembled using MWNTsor SWNT bundles having diameters greater than 5 to 10 nm are ideally suitedfor imaging rough terrain, such as narrow, deep recesses and otherhigh-aspect-ratio features that are inaccessible to conventional micro fabricatedprobes. Probes fabricated with individual SWNTs represent the ultimatein resolution, but are more susceptible to lateral bending and other deformationmodes that can impact image quality.The state of the art in fabrication methods and applications of carbonnanotube AFM probe tips up to 2001 has been published in review articles.In this chapter, we will summarize the earlier results and present developmentsthat have occurred in the intervening 3 years, from both experimentaland theoretical viewpoints. These recent advances have enriched the fieldand increased our understanding of the capabilities of carbon nanotube AFMtips, as well as some of their limitations. Although carbon nanotubes wereinitially intended as high-resolution topographical imaging probes, theirunique mechanical, electrical, and chemical properties have been exploitedfor more than just conventional AFM, impacting virtually every field in thescanning probe microscopy family, including scanning tunneling microscopy(STM), near-field scanning optical microscopy (NSOM), and scanning conductiveprobe microscopy, permitting a broad array of applications, frommicroelectronics to structural biology.

## II. FABRICATION OF NANOTUBE PROBE TIPS

Smalley's group reported the first example of the use of carbon nanotubesas scanning probe tips in 1996. They manually attached multiwalled carbonnanotubes and ropes of individual SWNTs to the apex of silicon pyramidal

tips using tape adhesive and a micromanipulator in an optical microscope.The attached nanotube tips were usually too long to be useful for high-resolutiontopographical imaging due to thermal vibrations. The length of the

nanotube could be shortened in situ in the AFM by electrical pulse etchingof the probe tip on a conductive surface. The drawbacks to this method werethat the mounting process was slow and painstaking, and larger nanotube

structures like MWNTs that could be imaged by the optical microscope weremore likely to be attached. Nevertheless, this study was the first to demonstrateseveral important advantages that nanotube probes have in generalover conventional AFM tips. The high aspect ratio of the nanotubes enabledmore accurate imaging of the sidewalls of deep silicon trenches. Strongadhesive forces between the sample surface and an AFM tip that complicateimaging with conventional probes were greatly reduced in the case of nanotubetips, due to their small size and cylindrical geometry. Finally, this studydemonstrated that the nanotube probes elastically buckled at

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higher contact forces.Since then, several reports have described manual assembly of nanotubeprobes, using optical microscopes as well as inside scanning electron microscopes(SEMs) for the fabrication of AFM tips, STM tips, and evennear-field optical probes. Nanotube AFM probes manually assembled fromMWNTs with this method are available commercially.In addition to the use of adhesives, other methods for bonding probes havebeen developed, including spot welding the attachment site of the nanotubeon the tip support and deposition of amorphous carbon from the electronbeam in the SEM. The ability to visualize the mounting of the probe and itsmorphology during fabrication allows one to optimize the characteristics ofthe probe almost in real time. Examples include controlling the length ofindividual MWNTs on support tips by Joule heating or application of mechanicalforce, sharpening a nanotube probe via extraction of an inner shellfrom an attached MWNT or by stripping away outer layers locally at thetip, and tuning the projection angle of attached SWNT bundles by repeatedAFM scanning of the probe across an array of tall pillars. Of course, thedegree of control possible will depend on the imaging resolution of the microscopeemployed. Ultimate resolution requires individual SWNT tips. Wongand coworkers manually attached bundles of SWNTs to AFM tips that wereapproximately 10 nm in diameter and contained up to hundreds of SWNTseach, but occasionally the electrical etching procedure would result in theexposure of one or a few SWNTs at the tip apex to give a high-resolutionprobe.Lieber’s group and Cooper et al were the first to show that individualsingle-wall carbon nanotubes could be directly grown by chemical vapordeposition (CVD) on the silicon tips themselves by first precoating the tipwith a metal catalyst. Direct-growth techniques were later applied to electrochemicallyetched tungsten tips for STM. Unlike manual assembly methods,CVD can be potentially applied to massively parallel fabrication ofnanotube probe tips on a wafer scale. In the CVD synthesis of carbonnanotubes, metal catalyst nanoparticles are heated in the presence of a hydrocarbongas or carbon monoxide; the gas molecules dissociate on the catalystsurface and carbon is absorbed into the particle. As the carbon precipitates,a carbon nanotube is grown with a diameter similar to that of the catalystparticle. Varying the concentration of the catalyst on the tip controls thedistribution of MWNTs to SWNTs, and the particle diameter controls thediameter of the tube.Early work with direct CVD growth involved creating nanopores, byetching the silicon tip in hydrofluoric acid; the nanopores could then havecatalyst particles deposited inside them. The CVD growth of the carbonnanotubes from pores located at the flattened apex of the silicon tip had thecorrect geometry for AFM imaging. Now, individual SWNT tips could beprepared, but the preparation of the porous layer in the silicon was stilltime-consuming, and often placement of the nanotube at the optimal locationnear the tip apex was not achieved. Later, direct surface growth of SWNTsby CVD on silicon tips was demonstrated, without the use of pores.Individual SWNT tips could be prepared this way by lowering the catalystdensity coating the silicon tip, although this also reduced the tip yield.

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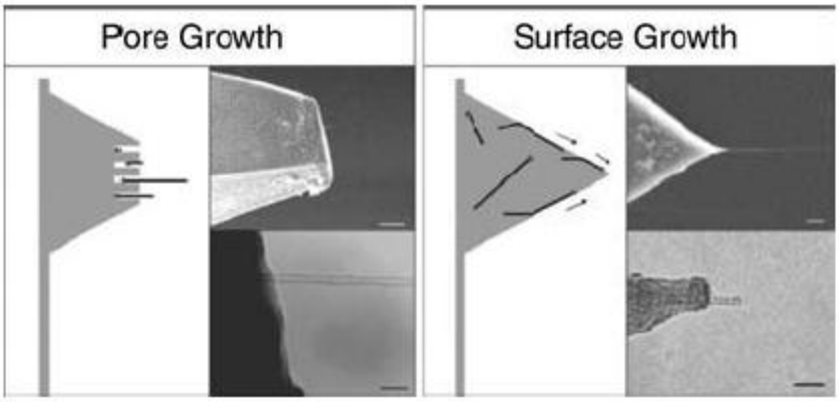


**Figure 1:- CVD nanotube tip growth methods.**

## III. AFM IMAGING WITH NANOTUBE PROBES

To determine the performance capabilities and obtainable resolution forcarbon nanotube probes, a more quantitative understanding of tip–sampleinteractions is needed. These interactions will, of course, be dependent onthe type of scanned probe microscopy performed (e.g., tapping-mode vs.noncontact mode AFM), the environment, the type of nanotube tipemployed, and the intended application. In a simple geometric model forthe nanotube tip–sample interaction, the ultimate resolution possible will bedetermined solely by the size of the tip. However, carbon nanotube probeshave unique properties compared to conventional tips that can stronglyaffect imaging fidelity in AFM, sometimes in unexpected ways.Colloidal gold nanoparticles are useful imaging standards to characterizenanotube tip resolution, because of their monodispersity in size andshape, and their incompressibility. The effective tip size can be calculatedfrom images of the particles based on the two-sphere model of Bustamanteand coworkers. Using this characterization method, Wong et al. reported that manually assembled MWNT probes had limiting tip radii of about 6nm, while manually assembled SWNT robes, which consisted of bundlesof 1.4-nm-diameter SWNTs, had effective imaging radii of about 3.5 nm.Direct-CVD-growth MWNTs from porous silicon AFM tips had limiting radii ranging from 3.5 to 6 nm. Both pore growth and surface growthSWNTbundles had effective radii in the 2- to 4-nm range. An effective radius ofjust 1 nm was reported for a pickup SWNT AFM tip. In some cases, comparable results have been reported for silicon or silicon nitride probes, butin those cases, high resolution was likely due to fragile tip asperities, whichwere not well defined. Notably, the range of limiting nanotube tip radii calculated from high-resolution AFM images of colloidal gold nanoparticleswere reported to agree with transmission electron microscopy (TEM) measurementsof the probes.Simple models of AFM resolution assume that the probe is a rigid,incompressible cylinder with a flat or hemispherical end. In practice, this isnot the case. While nanotubes have exceptional longitudinal stiffness,radially they are far more compliant, especially SWNTs, a characteristic thatrenders these tubes susceptible to bending or localized deformations of the nanotube walls. Snow et al. have shown that image artifacts and snap-to-contactbehavior can result from tubes that exceed either a critical length or acritical angle relative to the substrate surface normal. High-magnificationTEM images show that the

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nanotube probe ends are open due to ablationfrom the electrical pulse etching procedure used to shorten the tubes to usefullengths. The likelihood of deformation is further increased due to structuraldiscontinuities at the open end of the nanotube probe.Our group has carried out a rigorous examination of the influence ofnanotube probe morphology on AFM image resolution and quality bydirectly correlating scans taken with several pickup SWNT probes operatingin tapping-mode AFM in air with TEM images taken of these probes. Thesample in this study consisted of individual SWNTs lying flat on the samesilicon oxide growth substrate used to fabricate the nanotube AFM tips. Bycorrelating probe structure and orientation seen in the TEM images withtopographic AFM imaging performance, we have provided direct experimentalevidence consistent with the mechanical modeling studies carriedout by Snow et al. Other artifacts in addition to tip broadening can affectimaging. For example, we found that a SWNT projecting from the AFM tipat a 40° angle produced an image containing a positive height shadowingartifact approximately 10 nm in width parallel to each sample nanotube, dueto the non-ideal orientation of the probe. Additionally, the TEM image forthat probe showed that the nanotube was buckled near the silicon tip apex.Previous reports have described reversible elastic buckling of the nanotube,which did not have a serious impact on image quality. Our TEM correlationsindicated, however, that buckling can, under some circumstances,be inelastic, resulting in irreversible structural changes.We found that images taken with high-quality SWNT probes (i.e., thosethat were not too long and were oriented close to perpendicular with respectto the substrate) showed no sign of artifacts. By comparing the observedAFM resolution with the diameter of the nanotube probe measured from thecorresponding TEM image, it was found that the lateral resolution was, onaverage, 1.2 times the nanotube probe diameter, a value that approached theideal ratio of unity in the absence of thermal vibrations and bending effectsof the probe.Surprisingly, we found that for some cases, the apparent lateral resolutionwas actually better than expected on the basis of the probe diameter, asdetermined by TEM. The nanotube tip in Figure 1 is one such case. Thelateral resolution from this 5.5-nm-diameter probe was 1.2 nm, which is only22% of the probe diameter. Here, the lateral resolution of the probe is definedas the difference between the measured height of a sample, which can bedetermined to high precision with AFM, and the measured diameter (fullwidth at the noise floor).

## IV. APPLICATIONS OF CARBON NANOTUBE PROBES

### 4.1 Applications in structural biology

The use of carbon nanotube AFM tips for structural biology applications hasbeen well described in earlier review articles from Lieber’s group and willthus only be summarized here. One of the first types of samples to be imagedwith carbon nanotube AFM probes after their introduction in 1996 was DNA.Several groups reported imaging DNA and DNA–protein complexes withmanually assembled MWNT tips in air, in fluids, and in vacuum.The resolution reported for SWNT tip imaging of a RNA polymerase–DNAcomplex in air was about the same as that for MWNT tips (3.5 nm), althoughtruly tip-limited resolution in SWNT AFM images of 2.4-nm-diameter DNAwas reported by Chen et al., when imaged in aqueous solution with activeQ-control, as described above.Lieber’s group has imaged a number of isolated proteins using bothMWNT and SWNT AFM probes. CVD pore growth MWNT tips were usedto image immunoglobulin G (IgG) and IgM antibody proteins. IgG proteinsare approximately 15 nm in diameter and have a characteristic Y shape.This shape had been seen

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previously with AFM only at cryogenic temperatures.TheIgM antibody is a pentameric association of IgG proteins. Lieberand coworkers were able to image new structures not seen before with x-ray diffraction methods.CVD SWNT tips were used to image the smaller(8-nm) GroES protein, with sub-molecular resolution.

### 4.2 Nanolithography

The advanced imaging capabilities, well-defined morphology, and resistanceto wear of nanotube AFM probes, compared to conventional tips, weretechnological driving forces for their rapid development into new classes of high-resolution nanolithographic tools. Dai and coworkers demonstrated thecapability of MWNT probes as direct-write patterning tools by exploitingthe high electrical conductivity of the tubes to fabricate oxide nanostructureson silicon. The process relied on field-induced anodization of hydrogen-passivated Si surfaces in air with a negatively biased scanningprobe; however, tip wear was a serious issue that had limited the developmentof the technique for nanolithography. Dai et al. found that theMWNT tips did not suffer any noticeable degradation due to compressiveor lateral stresses, and were able to fabricate 10-nm-wide oxide lines at a100-nm pitch over a 100-$nm^2$ surface area in 100 sec while operating in thetapping mode. Later, direct-CVD-grown SWNT AFM probes were used tooxidize atomically flat titanium and achieve features as small as 8 nm indiameter at 20-nm spacing. If this technology were to be developed fordata storage, this would correspond to a bit density of 1.6 Tbits (1012 bits)per square inch. SWNT probes were also used to pattern 5- to 6-nm-widelines of titanium oxide as tunnel junctions in the construction of a single-electrontransistor that showed coulomb oscillations at room temperature.MWNT AFM tips have proven flexurally rigid enough to be used incontact-mode AFM, a mode in which the tip experiences significantlyhigher lateral forces than in the tapping mode. Okazaki and coworkers useda negatively biased MWNT tip scanning in contact mode to etch patternsinto a polysilane mask. MWNT tips are also suitable for indentation lithographyof polymer films and could be used to write bits into polycarbonatefilms used in DVD disks.

### 4.3 SWNT probe functionalization

Carbon nanotubes hold great promise in many areas of science and technologydue to their unique physical properties and molecular-scale dimensions.A significant technological advance for these materials has been their incorporationas specific molecular transducers in nanosensors, molecularelectronics, and as molecular manipulation tools. This potential is based onthe remarkable molecular recognition capabilities of carbon nanotubesthrough covalent chemical bonding, surface charge transfer, or electrostaticchanges when a specific molecule binds to a tube. Nanotubes can be chemically,physically, or biologically functionalized to recognize a particulartarget molecule and reject others in a complex environment.Perhaps the most exciting aspect of carbon nanotubes as AFM probe tipsfor probing the dynamics of biomolecules is that they can be chemicallyfunctionalized uniquely at their very ends. This can be initiated by an electricaletching process, which is also used to shorten the attached SWNTs inorder to achieve lengths suitable for high-resolution imaging. When SWNTtips are etched in an oxidizing environment (for example, in ambient air),the ends become functionalized with carboxyl groups. Wong et al. measuredthe chemical properties of oxidized nanotube AFM tips by measuring theiradhesion on hydroxyl-terminated self-assembled monolayers, and demonstratedthat

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carboxyl groups were present by observing a decrease in theadhesion force at pH 4.5, which corresponds to the deprotonation of carboxylicacid.The tips can be chemically modified further by coupling organicamines to the carboxylate group to form amide bonds. The use of reactiveamino chemistry is a common biochemical conjugation technique, and canbe exploited further to take advantage of a wide range of chemical andbiological means available for attaching fluorophores, antibodies, ligands,proteins, or nucleic acids to the ends of the nanotubes with well-definedorientations. With SWNT imaging, covalent and non-covalent forces can bemapped on single macromolecules or between individual biomolecules withgreater specificity than with conventional probes, due to the molecular-scaleresolution of the nanotube tip. Using functionalized SWNT probes, it is easier to ensure that there is only one molecule or complex attached to the probe.The manipulation of a ligand–protein interaction with specific single moleculescoupled to the nanotube tip has been measured with AFM by Wonget al.

### 4.4 Nanoelectrode scanning probes

Metallic nanotubes have extremely high conductivity; a single metallic SWNT can supportelectrical currents as high as tens of microamps. These characteristics haveexpanded the capabilities of these tools beyond simple topographical imagingapplications. Electrically conductive carbon nanotube AFM tips haveshown great promise for conductive probe methods, such as STM andelectrostatic force microscopy (EFM) techniques. SWNT bundles attachedto AFM tips have also been used as templates for metal nanowire-conductingprobes

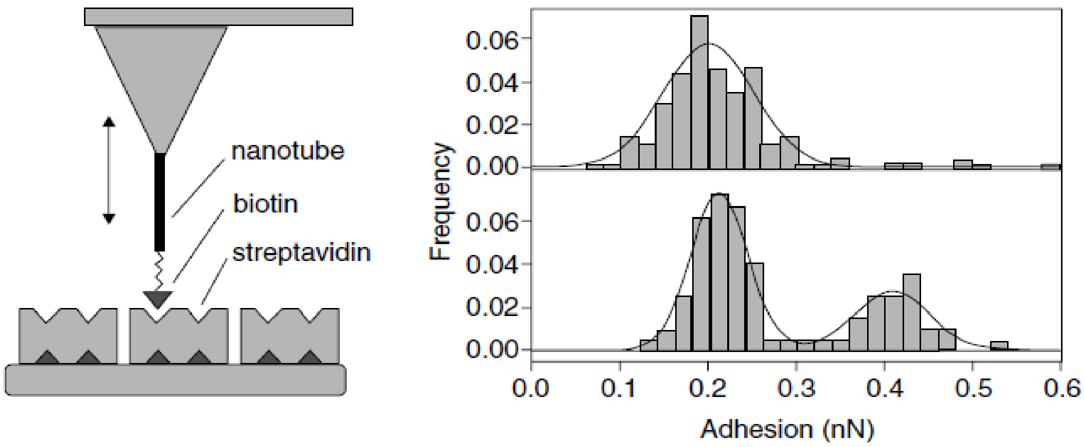


**Fig.2 Nanotube tips functionalized with biotin in force microscopy.**

that were robust enough to function in either the tapping or contactmode.Wilson and coworkers characterized the conductivity of SWNT probetips in detail by forming low-resistance electrical contacts to metal-coatedAFM tips and dipping the probes into a liquid mercury (Hg) droplet. Theywere able to discriminate between metallic and semiconducting SWNTs,separate the contact resistance of the nanotube on its AFM tip support fromits inherent resistivity, and detect the presence of multiple tubes on the tip.This work set the stage for constructing geometrically well-defined andreproducible nanoelectrodes. The development of such nanoelectrodes willbe particularly exciting for bio electrochemical applications, including thereal-time electrochemical probing of biochemical reactions in a single livecell with a minimally invasive probe.In electrochemical experiments, carbon nanotube-based electrodes andelectrode arrays have demonstrated exceptional electro catalytic activities.The nature of this enhanced electro catalytic performance relative to othermaterials is not clearly understood, but is thought to depend sensitively ondefect sites along the walls and open ends of the nanotubes. These mimicreactive edge planes of highly oriented pyrolytic graphite (HOPG), allowingfor more efficient electron transfer with electro active species in solution andfaster electrochemical kinetics. Cyclic voltammetry of the $Fe(CN)_6$3–/4–reduction–oxidation (redox) couple using MWNT bundles as the electrodeshowed purely Nernstian behavior, with no apparent activation barrier,which was not the case for a conventional platinum electrode. Conductivenanotube tips are therefore excellent candidates for electrochemical applicationsof scanning probe microscopes.

## V. CONCLUSION

Improved techniques for the manufacture of carbon nanotubes as robust andwell-characterized scanning probes have resulted in wider availability of thesetools to research groups performing AFM imaging. In addition to numerousdemonstrations of nanotube tips as high-resolution topographical and chemicalimaging tools, some exciting new applications have been developed within thelast few years that could significantly impact Nano biotechnology. Conductivenanotube tips attached to scanning probes can be assembled into functionalized nanoelectrodes capable of carrying out electrochemical reactions in physiologicallyrelevant environments. For example, these probes could be used asnanoscopic electro analytical tools to monitor cellular signaling pathways,including neurotransmitter release at synapses. Many signaling molecules,such as ligands, hormones, and neurotransmitters, are electrochemically active.Carbon nanotubes have been functionalized with biomolecules in numerousways in the construction of hybrid devices, such as field effect transistors,enzyme electrodes, and other biosensors. The integration of such a device onan AFM tip would represent the ultimate functionalized scanning probe.

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