

Nanomanipulation And Characterization Of Individual Carbon Nanotubes

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ABSTRACT

A new tool that operates in a scanning electron microscope for measuring the mechanics of nanostructures is briefly presented, and its use to measure the response to tensile loading of carbon nanotubes is outlined.

1 INTRODUCTION

The rapid advancement in miniaturization in the last decade has seen the discovery a variety of nanostructures. Carbon nanotubes (CNT) have received particular attention. Multi-walled carbon nanotubes (MWCNT) were discovered in 1991[1] and single wall carbon nanotubes (SWCNT) in 1993 [2,3], and over 4100 scientific publications have appeared on CNTs to date. The mechanical and electrical properties of CNTs have been the subject of numerous theoretical and experimental studies. CNTs can be considered to be a seamless cylinder (SWCNT), or a set of nested cylinders (MWCNT), of graphene sheet(s), and can have open or capped ends. The properties of a graphene sheet, of quantum confinement in small dimensions, and the detailed geometry including the chirality of the shell structure of CNTs have been studied, and potential applications are likely in materials reinforcement [4,5], field emission panel display [6-8], electronic nanowires [9,10] and devices [11-13], chemical sensors [14], H₂ gas storage [15], as tips for scanning microscopy [16,17], for use in batteries [18], and so on.

CNTs should possess mechanical properties far superior to commercially available carbon fibers, due to their expected structural perfection. Young's modulus (~ 1 TPa) similar to the in-plane modulus value for high quality graphite and high tensile strength (~ 50 to 100 GPa, thus much greater than any other available material) are predicted, and for the modulus experimentally verified, for CNT [19-22]. For comparison, the highest strength carbon fiber in industry has a strength of ~ 7 GPa [23].

The small dimensions of CNTs offers significant challenges for experimental study of their mechanical properties: (i) the challenge of CNT placement in an appropriate testing configuration; (ii) in certain cases the fabrication of appropriate clamps and thus achieving control of the boundary conditions for loading; (iii) successful application of the desired loading; (iv) characterizing and measuring the mechanical deformation at the nanometer and perhaps even the atomic length scale.

High-resolution microscopes allow the characterization of nanostructures, and developments in the new area of "nanomanipulation," based on inserting or adapting new tools to such microscopes, have enhanced our ability to mechanically test nanostructures such as

Here, we briefly summarize our effort in the development of new tools for nanoscale characterization and the study of various mechanical properties of CNTs.

2 A BRIEF REVIEW OF RELATED INSTRUMENTS FOR NANOSCALE MATERIAL CHARACTERIZATION

Scanning probe microscopy and electron microscopy have been the most widely used methods for resolving and characterizing nanoscale objects. We and others have primarily used SPM and EM instruments to study nanotube mechanics. For this reason, we give a brief review of the methods of operation of these types of microscopes.

Electron microscopes (EM) use high-energy electron beams (several keV up to several hundred keV) as a source for scattering and diffraction from a sample, which results in high resolving power down to sub-nanometer resolution because of the extremely short wavelength (a fraction of a nanometer) of high kinetic energy electrons.

In scanning electron microscopy, SEM, a focused electron beam (nanometers in spot size) is rastered across the sample surface and an amplified image of the sample surface is formed by recording the secondary electron signal or the back scattering signal generated from the sample. SEM is limited by the scattering volume of the electrons interacting with sample material, and high end instruments are capable of achieving a resolution of a few nanometers.

Scanning probe microscopes (SPM) use extremely sharp probes (that can have 10 nm or smaller radius of curvature at the tip) controlled by sensitive sensing and actuation feedback electronics for obtaining nanoscale and even atomic scale information. In a typical imaging experiment, the tip is rastered over the sample and the sample geometry is thereby mapped out. SPM can also be used to nanoindent samples, to nanomachine surfaces, and to bend and manipulate nanostructures. Depending on the type of interaction force involved for sensing, an SPM includes a family of microscopes such as the atomic force microscope (AFM), magnetic force microscope, electric force microscope, friction force microscope, and so on. Depending on the mechanism used for measuring the force interaction, the SPM also includes many modes of operation, such as contact mode, tapping mode, force modulation mode and so on. We refer to, for example, references [24-26].

3 NEW TOOLS FOR NANOSCALE MECHANICAL MEASUREMENT

The high resolution and large sample chamber space make SEM a good candidate for inclusion of three

probe, and the applied force was measured at the other end by the cantilever deflection of the other AFM probe. The measured force vs elongation were converted, by SEM measurement of the MWCNT geometry, to a stress versus strain curve and the breaking strength of each MWCNT was obtained by measuring the maximum tensile loading force at break. The experiment also clearly resolved that a MWCNT normally breaks in a sword-in-sheath breaking mechanism, where the MWCNT so attached under tensile load breaks at its outmost layer followed by the sliding out of the inner shells during the continuous pulling.

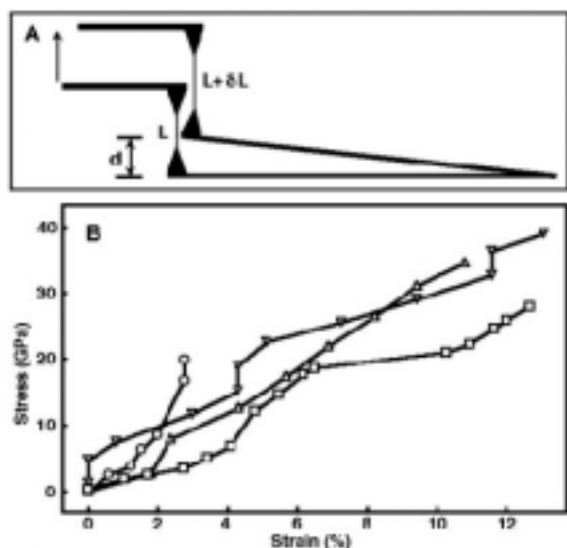


Figure 3: (A) Schematic showing the principle of the tensile-loading experiment. When the top cantilever is driven upward, the lower cantilever is bent upward by a distance d , while the nanotube is stretched from its initial length of L to $L + dL$ because of the force exerted on it by the AFM tips. The force is calculated as kd , where k is the force constant of the lower cantilever. The strain of the nanotube is dL/L . (B) Plot of stress versus strain curves for individual MWCNTs.

The shear strength between the shells of a MWCNT is also an interesting subject for experimental study. Yu et al. were able to directly measure the friction force between the neighboring layers while pulling the inner shells out of the outer shells of a MWCNT using the same apparatus for measuring the tensile strength of individual MWCNTs [29]. The realization of such measurement was based on the discovery that a tensile-loaded MWCNT normally broke with a sword-in-sheath breaking mechanism. The separated outer shell can still be in contact with the underlying inner shell in certain cases (in other cases, the “snap back” of the loading and force-sensing cantilevers leads to two separated fragments). A model was then developed to include forces such as (i) F_a , the applied force from the deflection of the soft AFM cantilever; (ii) F_s , the static shear interaction force between shells present during the “stick” event; (iii) F_d , the dynamic shear interaction force between shells in the “slip” event; (iv) F_c , the solid-solid surface tension interface force that is due to the creation of a new shell surface area in the pullout event

dangling bonds on the edge of the fractured MWCNT cylinder with the internal shell surface. Shear strength was related to the shear interaction force. The continuous measurement of force and “contact length” (the overlap length between the outer shell and its neighbor) in the pullout process provided then the necessary data for obtaining the dynamic (0.08 MPa) and static shear strength (0.30 MPa in one case and 0.08 MPa in another case) between the shells. Such measurement also allowed the direct estimation of the surface energy of graphite.

5 CONCLUSIONS

The new developments in the area of nanoscale manipulation and measurement as reflected in the studies presented in the last section have certainly helped our understanding of CNT mechanics. Since CNTs possess unique structures that maintain their conformation while being manipulated they represent a “nano-tinker toy” for manipulation on the nanoscale. Therefore, such types of approaches also provide a window on current capabilities for exploring and exploiting the “nano-world,” and provide an avenue for future advancement in methods and tools useful in nanotechnology.

But what has the community not yet achieved? We have not yet measured the tensile loading response of an individual SWCNT, nor have we applied a known torque or controlled, and reversible, twist along a CNT. The influence of environment on NT mechanics has not yet been explored in any detail—such as effects of temperature, chemical environment, loading rate, defect density, nor do we have a clear and detailed picture of the nucleation, propagation, and ultimate failure resulting from, defects. From the experimental perspective, such advances will come with new approaches and tools generated by innovative thinking. It is clear that focused effort in developing new measurement tools that can be integrated into high spatial resolution imaging instruments is necessary for further advances in nanostructure mechanics.

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