3D mechanical measurements with an atomic force microscope on 1D structures.

Kallesøe, Christian; Larsen, Martin Benjamin Barbour Spanget; Bøggild, Peter; Mølhave, Kristian

Published in:
Review of Scientific Instruments

Link to article, DOI:
10.1063/1.3681784

Publication date:
2012

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.
3D mechanical measurements with an atomic force microscope on 1D structures

Christian Kallesøe, Martin B. Larsen, Peter Bøggild, and Kristian Mølhave

Citation: Rev. Sci. Instrum. 83, 023704 (2012); doi: 10.1063/1.3681784

View online: http://dx.doi.org/10.1063/1.3681784

View Table of Contents: http://rsi.aip.org/resource/1/RSINAK/v83/i2

Published by the American Institute of Physics.

Related Articles

Research on the fiber Bragg grating sensor for the shock stress measurement Rev. Sci. Instrum. 82, 103109 (2011)


Note: Direct force and ionic-current measurements on DNA in a nanocapillary Rev. Sci. Instrum. 82, 086102 (2011)

Optical cell for in situ vibrational spectroscopic measurements at high pressures and shear Rev. Sci. Instrum. 82, 073905 (2011)

Additional information on Rev. Sci. Instrum.
Journal Homepage: http://rsi.aip.org
Journal Information: http://rsi.aip.org/about/about_the_journal
Top downloads: http://rsi.aip.org/features/most_downloaded
Information for Authors: http://rsi.aip.org/authors

ADVERTISEMENT

HAVE YOU HEARD?

Employers hiring scientists and engineers trust physicstodayJOBS

http://careers.physicstoday.org/post.cfm
3D mechanical measurements with an atomic force microscope on 1D structures

Christian Kallesøe, a),b) Martin B. Larsen, Peter Bøggild, and Kristian Mølhaveb)
Department of Micro- and Nanotechnology, Technical University of Denmark, Kgs. Lyngby, DK-2800, Denmark
(Received 15 October 2010; accepted 13 January 2012; published online 9 February 2012)

We have developed a simple method to characterize the mechanical properties of three dimensional nanostructures, such as nanorods standing up from a substrate. With an atomic force microscope the cantilever probe is used to deflect a horizontally aligned nanorod at different positions along the nanorod, using the apex of the cantilever itself rather than the tip normally used for probing surfaces. This enables accurate determination of nanostructures’ spring constant. From these measurements, Young’s modulus is found on many individual nanorods with different geometrical and material structures in a short time. Based on this method Young’s modulus of carbon nanofibers and epitaxial grown III-V nanowires has been determined. © 2012 American Institute of Physics. [doi:10.1063/1.3681784]

I. INTRODUCTION

The mechanical strength and rigidity of nanostructures such as nanowires and carbon nanotubes (CNTs) are important properties for a range of applications, including CNT-AFM tips,1,2 composite materials,3–5 resonators,6–9 and relays.10–12 Important factors which have great influence on the mechanical properties of nanostructures are surfaces states and defects affecting Young’s modulus of the wires,13 heterostructure interfaces, and the epilaternal interface between wire and substrate. For multiwalled and singlewalled CNTs, several techniques on measuring Young’s Modulus have been established.13–17 For example, a scanning electron microscope (SEM) equipped with manipulation stages,14 scanning tunneling microscope (STM)15 and atomic force microscopes (AFM)13,16,17 setups have been employed to measure plastic and elastic deformation parameters of CNTs. San Paulo et al. reported on AFM mechanical measurements on both single and double clamped silicon nanowires grown by the vapor-liquid-solid (VLS) mechanism.18 Furthermore, vibration analysis has been successfully used to measure the mechanical properties of CNTs in situ TEM.19,20 However, not many experimental tests on mechanical properties have been performed for III-V nanowires, and a simple standard equipment method for 3D mechanical characterization has been missing, that can be used on e.g., nanowires standing up from a substrate.

In this work, we have developed a simple method to characterize the mechanical properties of nanostructures such as nanowires and tubes. With an atomic force microscope (AFM) the cantilever probe is used to deflect a horizontally aligned nanorod at different positions along the nanorod, which enables accurate determination of the spring constant, see Fig. 1. The mechanical properties are measured in 3D with nanoscale resolution along the nanowire, while the exact spatial and force resolution is determined by the AFM. Characterizing a single nanowire as in Ref. 13 can provide measurements of its mechanical properties, but the presented method allows measurement of the mechanical properties as the wire stands attached to its substrate as needed for any nanomechanical use of the nanostructure, where the clamping influence will be essential. Furthermore, the method can be used to map out individual nanostructures in many other geometries than Ref. 13, requiring a verification of the structures dimensions. From this, Young’s modulus of carbon nanofibers (CNFs) and epitaxial grown III-V nanowires has been determined. The method can find applications also in the new in situ SEM and TEM force sensing systems, such as the nanofactory AFM system.

II. ATOMIC FORCE MICROSCOPE SETUP

In this work, an AFM is used to directly measure the force-distance (FD) characteristics at varying distance from the substrate. During this process, the nanowires remain in their as-grown position, as illustrated in Fig. 1(a). The upper surface of the cantilever is used to deflect the nanowire at different positions along the nanowire, thus allowing for mapping of the mechanical properties in 3D with nanoscale resolution along the nanowire. The flat apex provides a well defined contact geometry, whereas the tip itself often has several facets at different angles. The substrate can be moved with nanoscale resolution and the deflection of the AFM cantilever was measured using photodiodes creating a A-B voltage signal (Fig. 1(a)). We estimate a 1 nm resolution of the stage.

The AFM used for the experiments was of type Veeco CPII and data was collected using Veeco Data Acquisition in connection with a free “AUTOHOTKEY” control software program21 controlling the x and y position of the stage and automating the data acquisition process. Each sample substrate was attached by carbon tape to a PMMA block, situated on a magnetic base plate, thus the substrate surface was perpendicular to the base plate. Both sample and cantilever were grounded in order to avoid electrical fields between them,
A linear tapering of the wire will cause the bending stiffness $El(y)$ to vary as function of $y$. The area moment of inertia is given by

$$I(y) = \frac{\pi D(y)^2}{64} = \frac{\pi}{64} \left(D_r - y \frac{D_r - D(L)}{L}\right)^4,$$

where $D_r$ is the diameter at the root of the wire and $D(L)$ is the diameter at the point load $y = L$. The angle, $\alpha$, can be found by integrating Eq. (1) and further integration results in the deflection which at the point load $y = L$ is given by

$$z_{nw}(L) = \int_0^L \int_0^L \frac{F(L - y)}{E \pi \left(D_r - y \frac{D_r - D(L)}{L}\right)^4} dy dy$$

resulting in the spring constant

$$k_{tap}(L) = \frac{3\pi E D^2 I D(L)}{64 L^3}.$$

The mechanical properties of the nanorods are calculated from the set of FD curves measured with the AFM. The spring constant of the nanowire will increase as the AFM contact point on the nanowire approaches the nanowire substrate, thus increasing the slope of the FD curves, $\alpha(y)$, see Figs. 2(b)

III. PURE BENDING OF A POINT LOADED NANOWIRE

Regarding the nanowire as a circular beam, fixed at one end and free at the other, see Fig. 2(a), the bending of the beam can be found for a point loaded beam,

$$\frac{d^2 z_{nw}(y)}{dy^2} = \frac{M(y)}{EI} = \frac{F}{EL}(L - y),$$

where $M$ is the applied moment at position $y$ given by $M = -F(L - y)$ and $F$ is the point force at position $L$. It is derived from the Euler-Bernoulli equation for the case of small deflection ($z_{stage}/L < 10\%$). Thus, a small deflection is assumed, i.e., a limit on the cantilever-nanorod angle of about $5^\circ$ which also include any initial angle between them. This, as will be shown later, is fulfilled as the nanowire slips off the cantilever before large deflections are achieved. The equation is fixed by the boundary conditions $dz_{nw}/dy |_{y=0} = 0$ and $z_{nw}(0) = 0$, as both angle and deflection at the root of the wire are zero.
and 2(c). Both the stage position change, $\Delta z_{\text{stage}}$, and the cantilever deflection, $z_{\text{cl}}$, are measured by the AFM, mechanically and optically, respectively, and from these the nanowire deflection can be found, see Fig. 2(a),

$$z_{\text{nw}} = \Delta z_{\text{stage}} - z_{\text{cl}}.$$  

The resulting forces are opposite and equal,

$$0 = F_{\text{cl}} + F_{\text{nw}}.$$  

Projecting them onto the $z$ axis and using Hooke’s law results in

$$0 = k_{\text{cl}} z_{\text{cl}} - k_{\text{nw}} z_{\text{nw}},$$  

and using Eqs. (5) and (7) the spring constant of the nanowire can be expressed as

$$k_{\text{nw}} = \frac{k_{\text{cl}} z_{\text{cl}}}{\Delta z_{\text{stage}} - z_{\text{cl}}}.$$  

Equation $z_{\text{cl}}$ is found from the A-B diode voltage, $\Delta V$, measured by the AFM. It is given by the linear relationship $z_{\text{cl}} = \alpha_{\text{ref}} \Delta V$, where $\alpha_{\text{ref}}$ is the reference response, i.e., a measurement of the cantilever deflection when the cantilever is pressed against a solid substrate (where $z_{\text{cl}} = \Delta z_{\text{stage}}$). Furthermore, the relationship between $\Delta V$ and $\Delta z_{\text{stage}}$ can be expressed by using a variable, $\alpha(y)$. Thus, the following two expressions are given

$$\alpha_{\text{ref}} = \frac{\Delta V}{z_{\text{cl}}}, \quad \alpha(y) = \frac{\Delta V}{\Delta z_{\text{stage}}},$$  

The spring constant can then be rewritten as

$$k_{\text{nw}} = \frac{k_{\text{cl}}}{\alpha_{\text{ref}} - \alpha(y)} - 1.$$  

Isolating for $\alpha(y)$ and inserting the spring constant for a tapered wire (Eq. (4)) with a point load at $y$ then gives the relation, which can be fitted to the measurements

$$\alpha(y) = \frac{\alpha_{\text{ref}}}{\frac{64k_{\text{nw}}}{3\pi D_{\text{nw}} D_{\text{cl}}}} \frac{2}{3} y^3 + 1.$$  

From the cantilever manufacturer the cantilever spring constant is known as $k_{\text{cl}} = 3$ N/m and the resonant frequency is $75 \pm 15$ kHz indicating an uncertainty of the spring constant of $\pm 1.2$ N/m. The reference slope $\alpha_{\text{ref}}$ was measured by pressing the cantilever against a solid substrate, thus $\Delta z_{\text{stage}} = z_{\text{cl}}$.

This was done for every cantilever used, with a value of $\alpha_{\text{ref}} = 2.5 \, \mu \text{V}/\mu \text{m}$. The FD curves are measured at different $y$ positions along the nanowire, using an automated autohotkey script developed for these measurements. The autohotkey program measures the FD curve, logs the $y$ position along with the FD data, and then adds a small predefined value to the $y$ position. The program then repeats these steps until the substrate and cantilever come in contact. To find Young’s modulus, $\alpha(y)$ is plotted as function of $y$, as illustrated in Fig. 2,
variation may be observed due to relative motion of the nanorod tip on the cantilever) and with a y dependent slope in region “C”. The FD curve contains several regions that correspond to specific contact regimes between the cantilever and nanorod. The minimum deflection and force that can be detected is determined by the amount of noise measured by the A-B diode signal in region “A”. From Fig. 4, \( \Delta V_{\text{noise}} = 0.01 \text{ V} \), corresponding to a deflection of \( z_{\text{min}} = \Delta V_{\text{noise}} / \alpha_{\text{ref}} = 4 \text{ nm} \). Using Hooke’s law the noise floor of the force detection is then: \( F_{\text{min}} = k_c z_{\text{min}} = 12 \text{ nN} \). A LabView program has been developed to calculate \( \alpha(y) \) as function of the y-position within a representing window, region “C”. In Fig. 4(b), \( \alpha(y) \) is plotted as a function of y and fitted to Eq. (12). Precise measurements of the wire dimensions are needed in order to achieve an accurate determination of the structural stiffness and Young’s modulus (using Eq. (13)); however, only the relative y-positions need to be known, as the exact root position (the y zero point) is not required but instead obtained from the fitting.

To understand the typical FD curve, the experiment was repeated inside a SEM using a Smaract nanomanipulator for detailed investigation of the cantilever-nanorod mechanical contact, without reading out the force, as shown in Fig. 5. The cantilever was tilted with respect to a CNF substrate as in the AFM measurements, thus the contact to the cantilever was established at the fiber tip when moving the CNF substrate downwards towards the cantilever, represented as “A” in Fig. 5. Moving the CNF sample further down, the cantilever exerted a force as a point load at the tip of the fiber, until the fiber and cantilever were parallel, region “B”. Therefore, the slope of the FD curve in this region will be independent of the y stage position. Moving the fiber further up, resulted in the cantilever acting as a point load at a y dependent position on the fiber, region “C” as the fiber pivots around the AFM edge, thus the slope of the FD curve in “C” will be y dependent. At some point the fiber started to slip in small steps along the cantilever before it finally slid sideways off the cantilever due to the triangular shape of the cantilever, region “D”. As a result, the slope of this part of the FD curve will decrease, where nonlinear effects due to detector saturation also can contribute. The fiber was observed in SEM to slide sideways off the cantilever, due to the triangular shape of the cantilever, and the A-B signal will thus drop to zero in region E as contact is lost. Comparing to Fig. 4, the different regions

![Fig. 5](image_url)
are clearly seen in the inserts labeled A, B, C, and D, and we hence have a qualitative model of the overall FD curve shape with the $y$-dependent part in region “C”.

V. MECHANICAL MEASUREMENT RESULTS

In Secs. VA and VB, measurements of GaAs nanowires grown on GaAs(111) and Carbon nanofibers (CNF) grown on SiO$_2$ are presented.

A. Mechanical properties of GaAs nanowires

Figure 6 shows SEM images of the measured GaAs nanowires. The wires are slightly tapered with little dimension variation. The root diameter is measured to be $D_r = 99 \pm 1$ nm and the tip diameter is $D_t = 68 \pm 1$ nm. The length of the wires is $L = 3.0 \pm 0.2$ $\mu$m. Using Eq. (13), Young’s modulus can be found from the fitted $A$ values in Fig. 7(a) from the following measurements:

$$E_{nw} = \frac{64k_{cl}y}{3\pi D_r^2 A} = \frac{20.8 \pm 8.5\text{GPa}}{A \cdot \mu\text{m}^2},$$

(14)

where the uncertainty includes both the nanowire dimension and AFM cantilever uncertainties. From Eq. (4), the spring constant as a function of wire length is given by

$$k_{tap}(y) = \frac{3\pi E D_r^3 D(y)}{64y^3}.$$ 

(15)

All results on GaAs nanowires are presented in Table I (including standard deviations ($\sigma$)) and Fig. 7(a).

The large standard deviations for $E$ seen in Table I stem mainly from the uncalibrated AFM cantilever. A calibrated cantilever would reduce the uncertainty to 10%. The mean value of Young’s modulus is estimated to $E = 94.1$ GPa, comparable to a bulk value of $E_{bulk} = 85.9$ GPa.$^{23}$ A mean spring constant at the end of the wire ($L = 3 \mu$m) is calculated to $k_{nw} = 0.034$ N/m.

B. Mechanical properties of carbon nanofibers

Figure 8 shows SEM images of the measured CNFs. A strong tapering is seen with $D_r = 350 \pm 30$ nm and $D_t = 90 \pm 40$ nm, with a very big spread in both diameter and length. The length of the fibers is $L = 5.9 \pm 1.0$ $\mu$m. Furthermore, the tapering is seen not to be linear, and some of the fibers are with two tips, thus the results are with a large uncertainty. Young’s modulus can be found from the fitted $A$ in Fig. 7(b),

$$E_{cnf} = \frac{64k_{cl}y}{3\pi D_r^2 A} = \frac{0.48 \pm 0.23\text{GPa}}{A \cdot \mu\text{m}^2}. $$

(16)

Tapered results on the CNFs are presented in Table II and Fig. 7(b). The mean value of the bending modulus of the tapered structures is estimated to $E_{tap} = 8.4$ GPa, with a very large uncertainty due to both uncalibrated AFM cantilever and the large spread in radius and shape of the fibers. A mean spring constant at the end of the tapered fibers ($L = 5 \mu$m) is calculated to be $k_{nw} = 0.04$ N/m.

VI. DISCUSSION

A qualitative model has been made for the overall FD signal describing the region of interest (region “C”) for

<table>
<thead>
<tr>
<th>$A$/$[\mu\text{m}^{-2}]$</th>
<th>$\sigma_A$/$[\mu\text{m}^{-2}]$</th>
<th>$E$/$[\text{GPa}]$</th>
<th>$\sigma_E$/$[\text{GPa}]$</th>
<th>$k_{tap}(y)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.24</td>
<td>0.02</td>
<td>86.7</td>
<td>42.7</td>
<td>(1.23 $\mu$m − 0.124$\nu$) $\mu$m$^2\cdot\text{g}^{-3}$ N/m</td>
</tr>
<tr>
<td>0.19</td>
<td>0.006</td>
<td>109.5</td>
<td>48.2</td>
<td>(1.15 $\mu$m − 0.156$\nu$) $\mu$m$^2\cdot\text{g}^{-3}$ N/m</td>
</tr>
<tr>
<td>0.20</td>
<td>0.06</td>
<td>104.0</td>
<td>77.6</td>
<td>(1.48 $\mu$m − 0.149$\nu$) $\mu$m$^2\cdot\text{g}^{-3}$ N/m</td>
</tr>
<tr>
<td>0.23</td>
<td>0.07</td>
<td>90.4</td>
<td>64.5</td>
<td>(1.28 $\mu$m − 0.129$\nu$) $\mu$m$^2\cdot\text{g}^{-3}$ N/m</td>
</tr>
<tr>
<td>0.26</td>
<td>0.005</td>
<td>80.0</td>
<td>34.2</td>
<td>(1.13 $\mu$m − 0.114$\nu$) $\mu$m$^2\cdot\text{g}^{-3}$ N/m</td>
</tr>
</tbody>
</table>
mechanical measurements. As mentioned previously, the minimum deflection and force that can be detected using the proposed method is determined by the AFM, i.e., the amount of noise measured by the A-B diode signal resulting in a minimum detectable force of $F_{\text{min}} = k_{c\ell} z_{\text{min}} = 12$ nN. Reducing the noise is therefore essential for the measurement of very thin nanowires. The triangular shape of the cantilever eases the contact procedure where only one wire should be contacted. However, the method is not capable of contacting individual nanowires if the wire-wire distance becomes smaller than the length of the wires. The fact that the mechanical properties are measured along the nanowire length, means that the method is suitable for a mechanical mapping of structures with varying material composition (heterostructures) or morphology (e.g., narrowing sections), measuring from the tip of the nanowire to determine the $y$-position. This requires a verification of the structures dimensions.

The $z$ range of the cantilever is not decreased as the cantilever approaches the substrate. Due to the triangular shape of the cantilever tip, the wire can, however, slide off sideways, thus region “E” in Fig. 5 is seldom reached. However, a measurement will still result in the wire breaking off as the point load approaches the substrate and the applied moment at the nanowire root will exceed the ultimate strength of the nanowire material. Thus, an $\alpha(y)$ value of 2.5 is never reached as also seen in Fig. 7. Gradual decrease in $z$ range along the wire would improve this.

The GaAs nanowires having diameter of less than 100 nm, are seen to have a Young’s modulus of 94 GPa which agree well with the bulk value of 85.9 GPa. The small discrepancy between the estimated and bulk values of Young’s modulus can be attributed to measurement uncertainties of the nanowire dimensions, but especially a systematic error from the spring constant of the cantilever. A calibrated cantilever should be used in the future. The agreement between bulk and measured wire modulus values stems with what have previously been reported by San Paulo et al. where a Young’s modulus of 186 GPa were measured for Si wires with diameters below 100 nm, comparable to a bulk value of 169 GPa for Si(111).  

CNTs are known for their incredible stiffness with Young’s modulus in the order of 100–1000 GPa. The low Young’s modulus found for the carbon nanofibers here using the tapered fitting, indicates that no strong tubular carbon structure is present. The scatter in the data for the CNFs can be explained by the fact that the measured bending stiffness is dominated by the physical dimensions of the fiber and is not a reflection of the model itself.

VII. CONCLUSION

We have established a method which enables mechanical measurements on 3D nanostructures such as individual epitaxially grown nanowires on their substrates, without any preparation steps, such as moving nanowires to other surfaces. The mechanical properties were measured with nanometer resolution along the nanowires determined by the AFM, and a force resolution of 12 nN. From these measurements Young’s moduli were found on many individual GaAs nanowires and carbon nanofibers, also taking their tapering structure into account. For the GaAs nanowires having diameter of less than 100 nm, a Young’s modulus in the order of 94 GPa were estimated, larger than the bulk value of 85.9 GPa. This increase was ascribed to a systematic error stemming from the cantilever spring constant. Increased precision would require determination of the exact cantilever spring constant.

Using arrays with much smaller densities would allow for a previous determination of the exact wire dimensions in SEM.
and optical location of individual nanowires with known dimensions. SEM or TEM combined with a piezoelectric stage moving a cantilever, would provide in situ mechanical measurements thus also revealing the transition between elastic behavior and brittle breakage of the wire.

The method opens up for mapping of the varying mechanical properties along, e.g., heterostructures, such as GaAs-GaP nanowires, or with varying diameters. Furthermore in situ experiments with force readout using, e.g., piezoresistive AFM probes would be useful for a perfect correlation of both nanorod dimension and the FD curves.

ACKNOWLEDGMENTS

We thank Ken Teo, Thomas Mårtensson, Monica Lexholm, and Lars Samuelson for providing samples and acknowledge financial support of the Danish Agency for Science, Technology and Innovation through grant No. 26-04-0258. Furthermore, we thank Matthias Goldschmidt and Lau Carlsen for experimental assistance.