ERDC/EL SR-15-1

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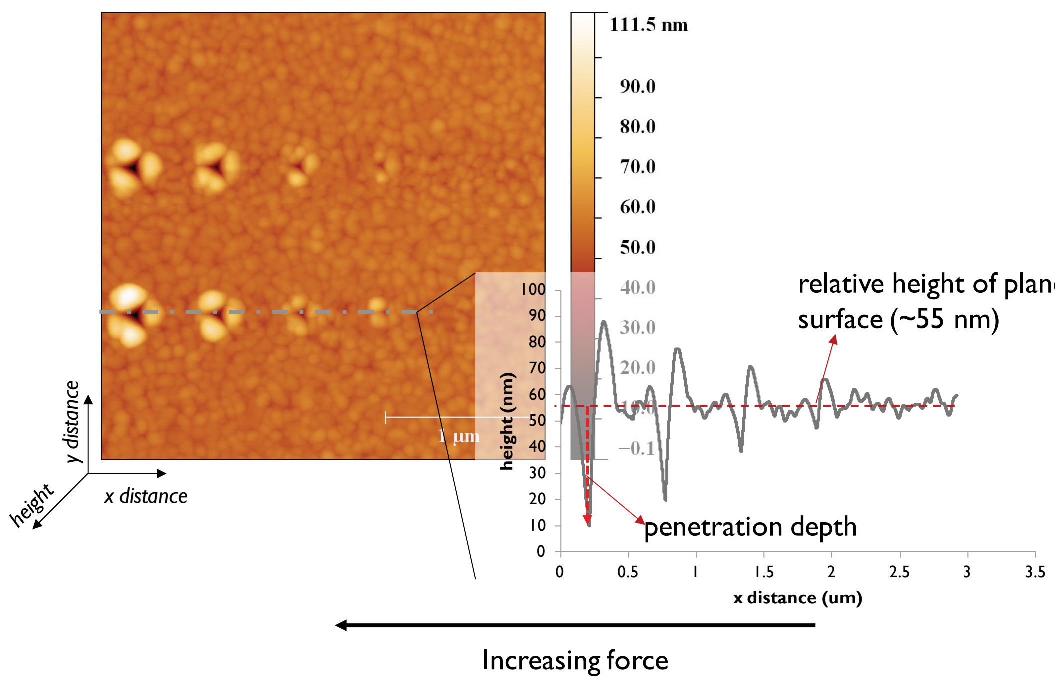
Environmental Consequences of Nanotechnologies

Determination of Nanomechanical Properties by Atomic Force Microscopy

Scientific Operating Procedure SOP-C-#

Michael F. Cuddy, Aimee R. Poda, and Matthew S. Hull

February 2015



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Abstract

The following methods provide a guide to measure mechanical properties of materials by means of an atomic force microscope (AFM). Traditional nanoindentation measurements do not afford immediate complementary surface imaging to visualize the residual indent. This obstacle is overcome using AFM. By indenting a surface with a diamond-tipped, stiff cantilever, local nanoscopic materials properties may be deduced. Briefly, an appropriate AFM cantilever is calibrated to determine its deflection sensitivity and spring constant; it is then used as both an imager and indenter at the surface of material of interest. The load applied by the cantilever is accurately controlled by knowledge of the deflection sensitivity. The maximum applied load is mediated by the cantilever spring constant. Following data collection, image and force curve analyses are completed to determine projected indent areas and load/unload profiles. This yields materials properties that include the material hardness and the Young's modulus along with corresponding surface topography.

Contents

Figures and Tables

Figures

Tables

Preface

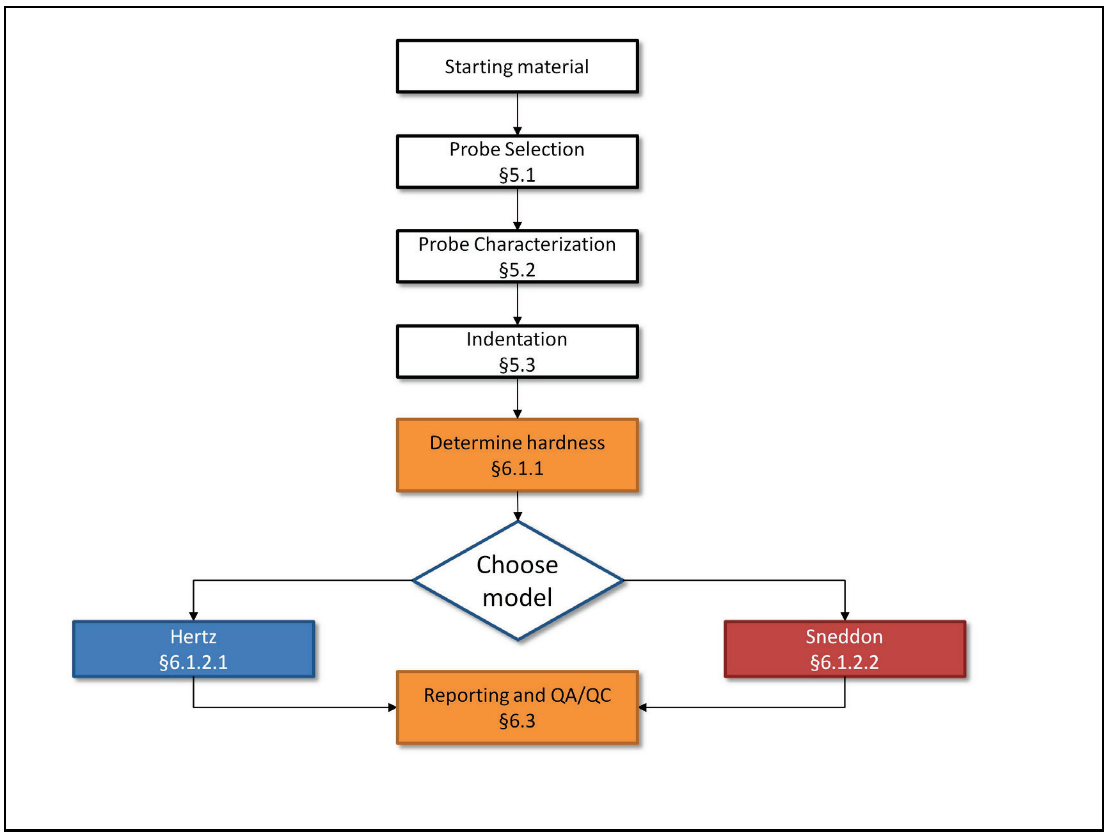
The Scientific Operating Procedure (SOP) described herein for assessing the properties of nanotechnologies was developed under Task 2: Optimized Scientific Methods of the ERDC/EL Environmental Consequences of Nanotechnologies research program. The primary goal of this Task was to develop robust SOPs for investigating the environmental health and safety (EHS) related properties of nanotechnologies including nanomaterials and products incorporating nanomaterials.

This SOP describes how to determine the nanomechanical properties, hardness and Young's modulus, at solid surfaces using atomic force microscopy. The present SOP combines best laboratory practices available from the literature and professional experience of ERDC research scientists.

1 Scope

This SOP is used to investigate the mechanical properties of nanostructured materials or the properties of materials at the nanoscale. In particular, materials parameters including hardness and Young's modulus are determined. This SOP is applicable to soft and semi-hard solid materials. Under the scope of this SOP, a diamond-tipped nanoindentation cantilever is used to indent gold as a reference for validation of the techniques.

Figure 1-1. Scientific operating procedure flow chart.



2 Background

Nanomechanical characterization by means of atomic force microscopy (AFM) offers an advantageous route over other materials qualification methods as it provides an all-in-one testing/imaging/analysis approach. Nanoindentation, in particular, represents a versatile capability that may be realized using most modern AFM instrumentation. Nanoindentation is used to measure both the hardness and Young’s modulus at the nanoscale level. Accurate values for these parameters are dependent upon sufficient characterization of the indenter tip and an appropriate model describing the tip-surface interaction. The protocol presented here will outline the steps necessary to qualify the indenter tip and extract nanomechanical information from AFM-based force-curve analysis using a nanoindentation procedure.

Tranchida and coworkers [1] described the preliminary determinations, calibrations, and analyses necessary to extract useful localized nanomechnical information from AFM force curves. The main points for consideration include:

1. Sample properties
2. Cantilever properties

Contributions from an underlying substrate can obfuscate the nanomechanical properties of the sample of interest at large penetration depths of the AFM tip. Therefore, it is important that the analysis be confined to the sample itself. Generally, a penetration depth of no more than 10% of the total sample thickness is recommended. Similarly, localized roughness of the sample can introduce distortions at the tip itself, such as a torsional force applied to the tip during approach. Thus, tip effects must be considered for even moderately rough samples (i.e., 1-3 nm RMS in a 1 um^2 area). Finally, it is critical that the cantilever be well-characterized to achieve reliable results. The applied load (F) is calculated from

$$F = kd = kd_{sens}t_{thresh} \quad (1)$$

where d represents the cantilever deflection and k the cantilever spring constant. Thus, the value k should be known with excellent accuracy; this value is generally supplied by the manufacturer for a specific tip and may be validated experimentally. In addition, the feedback loop for the cantilever/detection system must be calibrated. This involves determination of the cantilever deflection sensitivity (d_{sens}; i.e., the sensitivity of the optics to changing deflection). The deflection is calculated as the product of the deflection sensitivity and trigger threshold voltage (t_{thresh}) as shown in Equation 1. Tip shape is a third important factor; this will influence the models chosen to investigate tip-surface interactions [2]. Tip shapes are generally known and provided by tip manufacturers and may be verified by electron microscopy imaging.

ISO standard 14577 [3] provides an international standard for indentation testing using nanoindenters and hardness testers. The analyses of the load-depth curves obtained from the methodologies defined in ISO 14577 will be adapted for nanomechanical testing using AFM. In particular, the hardness value (Hv) will be calculated through Equation 2, where F_{max} is the maximum applied load and A is the projected area of contact based upon the tip shape.

$$Hv = \frac{F\,\mathrm{max}}{A} \tag{2}$$

The sample indentation modulus (E) will be obtained through a model of tip-surface interaction appropriate for the tip shape. E is effectively analogous to the Young's modulus at the nano scale. Although a wide range of models exist that describe various interactions and tip shapes, the Hertz or Sneddon models are generally employed. For isotropic samples, the Hertz model is a useful, simplified interaction descriptor. It is often used to describe indentation by a spherical indenter, and is applicable for shallow indentations, such as on a rigid film. The Hertz model is given in Equation 3, where δ is the indenter penetration depth and ν is the sample Poisson's ratio which can be estimated from tabulated values. Generally, for soft samples, or for unknown Poisson's ratios, the value may be set to 0.5. R is the radius of the spherical indenter.

$$F = \frac{4E\sqrt{R}}{3\left(1-v^2\right)}\delta^{3/2} \tag{3}$$

The Sneddon model [4] is applicable to a punch of conical profile and Equation 4 is used to determine E from an AFM force-curve, where α is the tip half angle.

$$F = \frac{2E\tan(\alpha)}{\pi(1-\upsilon^2)}\delta^2 \tag{4}$$

3 Terminology

3.1 Related Documents

- ASTM STP 889: Microindentation techniques in materials science and engineering
- ISO 14577-1: Metallic materials – instrumented indentation test for hardness and materials

3.2 Definitions

- Aspect ratio, n—as applied to AFM cantilevers, the ratio of cantilever length to width.
- Contact mode, n—the common method of AFM imaging, wherein a tip scans a surface of interest while remaining in close contact with the surface.
- Deflection sensitivity, n—a conversion factor to translate voltage into cantilever deflection, obtained from the slope of a force curve at a hard surface (i.e., sapphire) where indentation does not occur (d_{sens}).
- Force curve, n—a plot of AFM cantilever deflection as a function of applied voltage. With accurate tip qualification, may be transformed to a load-displacement curve.
- Half angle, n—slope of the AFM tip from center axis to face (α).
- Hardness, n–resistance of material to plastic deformation, i.e., by indentation (Hv).
- Isotropic, adj.—a mechanical property of a material that does not have a directional dependence.
- Poisson's ratio, n—the ratio of transverse to axial strain; a measure of material response to compression (ν).
- Simple harmonic oscillator (SHO), n—a system that describes deviation from equilibrium (i.e., of a spring) using a restoring force linearly proportional to displacement (i.e., $F = k\mathrm{x}$).
- Spring constant, n—a measure of relative stiffness, defined as the ratio of applied force to the displacement effected by the force.
- Tapping mode, n—in AFM, a method of surface imaging wherein the tip is vibrated very close to the specimen plane as it is rastered laterally to scan topography. This mode is used to avoid gouging or otherwise modifying the surface of interest.

- Young's modulus, n—material elasticity; a measure of the ratio of stress to strain, expressed in Pa.

3.3 Acronyms

- AFM – Atomic Force Microscopy
- <R> – reference material (50 nm gold film on Si)

4 Materials and Apparatus

4.1 Materials

Nanoindentation probe

Cantilever with spring constant and tip appropriate to withstand pressures exerted in the nanoindentation experiment. In general, the stiffness of the cantilever should be close to the expected sample stiffness. Refer to §5.1 for more detailed guidance on probe selection.

4.2 Apparatus

An integrated atomic force microscope with nanoindentation capability shall be used for this SOP. Many modern instruments are equipped with the capability to perform indentation experiments and force curve measurements. The main requirements of the testing apparatus are that it be capable of applying and removing a known load; and that it be capable of imaging the indented surface at a microscopic level without instrument reconfiguration.

5 Procedure

- First, an appropriate tip is selected for the material to be investigated.
- Next, the tip/cantilever is characterized to accurately determine its mechanical properties (deflection sensitivity, spring constant).
- Finally, the sample is scanned, nanoindented, and rescanned over the same area.

5.1 Probe Selection

A suitable tapping mode tip should be selected for nanoindentation experiments to allow for indentation as well as high-resolution imaging that is not practical using standard indenters (i.e., Berkovich indenters). An appropriate spherical or conical tip shall be chosen. The ultimate choice of probe depends upon: (1) the maximum desired applied load and (2) the fragility of the sample to be indented. Many tips manufactured specifically for AFM nanoindentation are diamond with a sharp radius of curvature (~50 nm or less) and well-defined angles. Cantilever stiffness is important to consider for the expected hardness of the sample to be indented. Fragile and soft materials (i.e., cells), for example, are prone to rupture if encumbered with too great a load or too sharp a tip, and should therefore not be analyzed using a very high spring constant probe. Generally, these guidelines are applicable to measurements made in a gaseous environment, at a solid surface. If measurements are to be conducted in liquid, specialized probes and cantilever holders are required, though the general principles still apply. The scope of this method is applicable to both spherical and conical tips; although other shapes may be used for nanoindentation experiments, they will not be covered in the scope of this SOP.

For indentation of cells and delicate biomaterials, cantilevers with spring constants in the range of mN/m should be suitable. For hard materials (i.e., bone), cantilevers with spring constants of >100 N/m may be required. For soft and delicate materials, it is recommended to use a probe with a spherical tip to avoid rupture of the material. For harder samples, or for materials with extremely small dimensions (i.e., <tip radius, R), a conical indenter may be appropriate. A general guidance is provided in Table 5-1.

Table 5-1. Suggested cantilever and tip characteristics for soft through semi-hard materials.

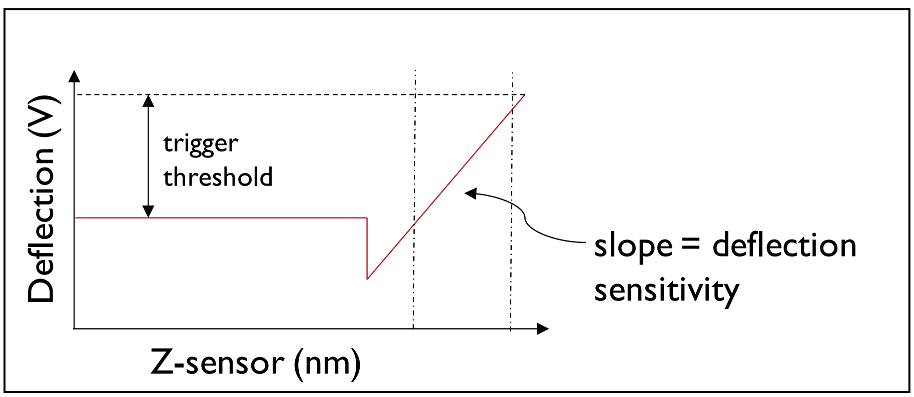
Sample	Spring constant range (N/m)	Appropriate indenter shape
soft biological material (i.e., cells)	0.005-0.050	spherical
semi-soft material (i.e., agarose gel)	0.01-0.10	spherical
stiff material (i.e., muscle tissue)	1.0-10	spherical
semi-hard material (i.e., bone)	10-300	spherical or conical

5.2 Cantilever Characterization

5.2.1 Determine Deflection Sensitivity

To determine the deflection sensitivity, use a hard, uniform, and clean surface such as sapphire (9 on Mohs hardness scale) or at least as hard as silicon (6-7 on Mohs hardness scale) if sapphire is unavailable. Engage the indentation tip and collect a force curve. The plot should display deflection as a function of z-height/displacement. The slope of the curve in the contact region is the deflection sensitivity in V/nm (Figure 5-1).

Figure 5-1. Example force curve for nanoindenter tip interaction with a hard surface (i.e., sapphire) for deflection sensitivity calibration.



5.2.2 Determine spring constant

Several methods exist to calibrate the spring constant of AFM cantilevers, if these are not provided from a tip manufacturer. The recommended approaches are based on the availability of the techniques with particular instrumentation and the ratio of cantilever length, L, to width, b. For instruments capable of thermal tune calibrations, proceed to step 5.2.2.1. Otherwise, skip to step 5.2.2.2.

5.2.2.1 Thermal tune method for cantilever calibration

The thermal tune method is widely available with modern AFM instrumentation. If this method is unavailable, it is suggested that the steps outlined in section 5.2.2.2 be used to estimate k regardless of cantilever aspect ratio. This method for spring constant determination treats the cantilever as a simple harmonic oscillator (SHO). A power spectral density plot of the cantilever vertical deflection signal is generated by the instrument and is fit with an SHO model in fluid (air) to determine the mean square displacement of the cantilever [4]. This, in turn, is indirectly proportional to the cantilever spring constant. The ambient temperature should be recorded during the thermal tune. Ultimately, the approach results in an overall uncertainty of <10% in k [5].

5.2.2.2 Sader method for cantilever calibration

The Sader method [6-8] is a hydrodynamic predictive model for spring constant (k) calibration of a cantilever in fluid (air). The governing equations for a rectangular cantilever are provided in Equations 5-7. In Equation 5, K_1 and K_0 represent modified Bessel functions of the second kind, and Re denotes the Reynolds number. Additional constants, definitions, and values, where appropriate, are defined in Table 5-2.

$$k = 0.1906 \rho b^2 L Q \Gamma_i(\omega)\omega \tag{5}$$

$$\Gamma(\omega) = \Omega(\omega) * \left[1 + \frac{4iK_1\left(-i\sqrt{i\mathrm{Re}}\right)}{\left(\sqrt{i\mathrm{Re}}\right)K_0\left(-i\sqrt{i\mathrm{Re}}\right)}\right] \tag{6}$$

$$\mathrm{Re} = \frac{\rho b^2 \omega}{4\eta} \tag{7}$$

Table 5-2. Summary of Sader method variables for rectangular-shaped cantilevers.

Variable	Definition	Notes
ρ	density	1.18 kg m^{-3} (air at STP)
η	viscosity	1.86E-5 kg m^{-1} s^{-1} (air at STP)
b	cantilever width	
L	cantilever length	
Q	quality factor	
ω	radial resonant frequency	= 2π*f* (*f* being the fundamental frequency)
$\Gamma_i(\omega)$	hydrodynamic function	imaginary part only

The spring constant may be estimated using the above equations, given the length, width, quality factor, and fundamental frequency of the cantilever in use. Note that the given values for air constants given in Table 5-2 are applicable at 25 °C and atmospheric pressure. Typical errors arising from this method are generally <5% [7,8]. Alternatively, several web-based calculators and smartphone applications are dedicated to Sader-based calculations of spring constants for rectangular and arbitrarily-shaped cantilevers alike:

- iPhone application: Sader Method [http://itunes.apple.com/us/app/sader-method/id402544930]
- Online: [http://www.ampc.ms.unimelb.edu.au/afm/webapp.html]

5.3 Sample Analysis

Nanoindentation experiments should be carried out at ambient temperature (i.e., 25 °C) and significant temperature fluctuations (>±5 °C) during the course of the experiment should be avoided. The sample should be securely mounted such that it is stable for the duration of the experiment. If possible, the supported material should be vibrationally isolated.

- Tune cantilever
    - Set drive frequency at center of resonant frequency peak
    - Adjust drive amplitude to achieve an RMS amplitude of ~240 mV
- Locate a clean, uniform, level area at the sample surface
    - Area should be clear of debris and scratch marks/mars
    - Area should be representative of the sample
- Engage the instrument in tapping mode
- Scan a region approximately 3 x 3 μm^2 in area at a rate of <1 Hz
- Save scanned image
- Following scan, collect force curves in the same area
    - Select a trigger threshold or range of trigger thresholds that will yield appropriate load(s) [refer to Equation 1]
    - Perform a series of indents at different, representative locations within the imaged area

    * Indents should be evenly spaced, with enough separation between them to avoid spillover from indentation pileup
    * Indents can be made in a matrix array (i.e., 3x3 or 5x5) for reproducibility analysis

  - Save force curves (i.e., deflection vs. z-sensor plots)
  - Avoid penetration depths >~80% of the total film/material thickness so as to eliminate contributions from substrate

    * If penetration depths exceed the thickness of the material of interest, be sure to perform model fits to the data only in the region where the material of interest resides.

- Return to image analysis and image the same area to reveal indentations

  - If indentations are not visible, it may be necessary to repeat with increasing trigger threshold values
  - If indentations overlap, repeat at a different location and use fewer indentations or a larger area

- Save scanned image

6 Reporting

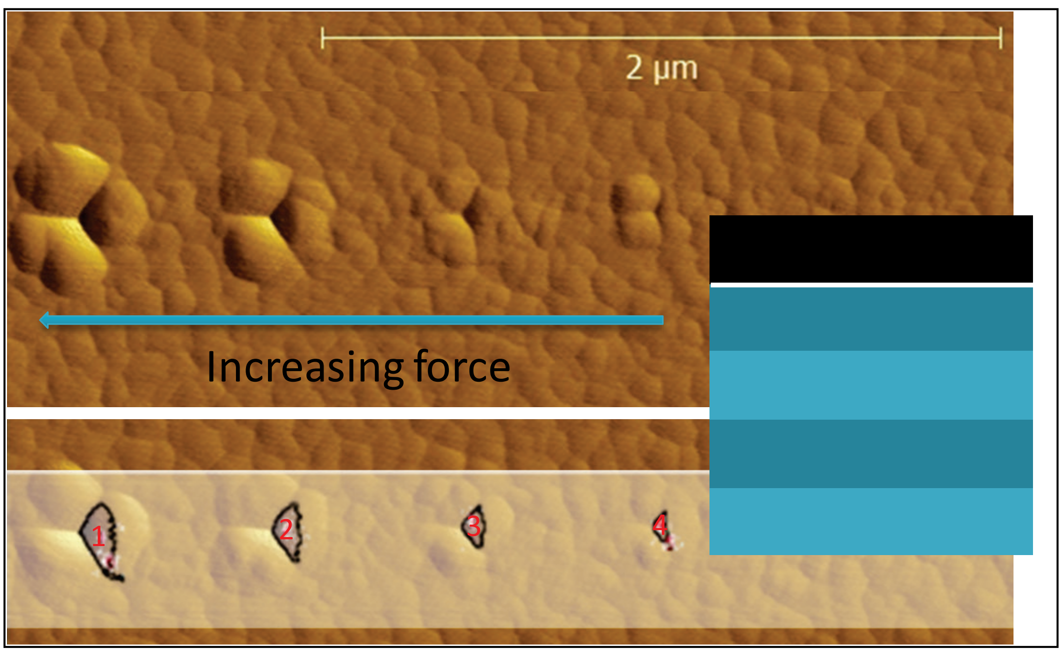
6.1 Analysis of Results

The deflection sensitivity (§5.2.1), spring constant (§5.2.2), and trigger threshold (§5.3) are used to determine F for each indent using Equation 1.

6.1.1 Determining nano hardness

Hardness measurements at the nanoscale are made according to Equation 2. Here, F_{max} is determined in step 6.1 above. The projected area for each indent may be determined using image analysis software such as ImageJ. An example of area analysis for indents as a function of increasing force is provided in Figure 6-1 for <R>.

Figure 6-1. Indentations of increasing force (from right to left in the image) created using a diamond-tipped cantilever indenting a gold surface. The projected areas of the indentations are given in the inset. The maximum load applied at each indent was 41.1, 31.6, 22.1, and 12.6 µN for indents 1 through 4, respectively.



Note that hardness values determined by this means are susceptible to error. This error arises due to microscopic contributions that cannot be neglected on the nanometer scale. Further reading on these phenomena may be perused in ASTM notes [9].

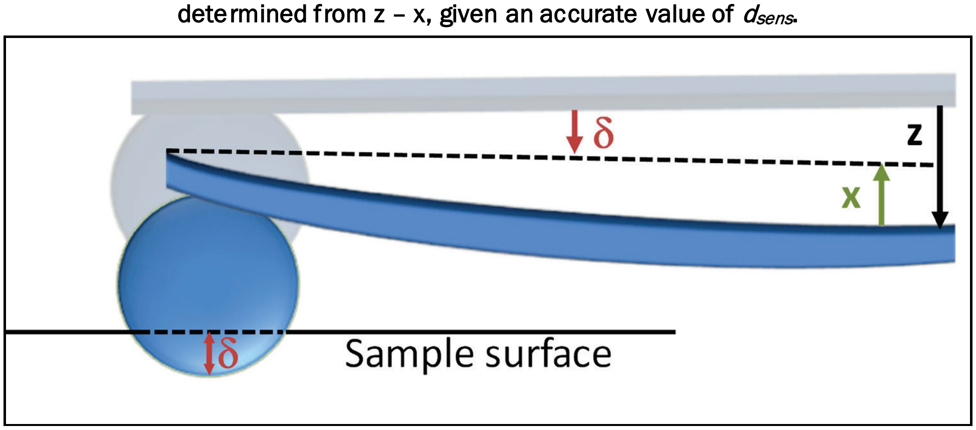
6.1.2 Determining indentation modulus

An analogous value to the Young's modulus is derived from the shape of the extending force curves collected during indentation (§5.3). Note that this value, E_{eff}, contains information regarding the plastic deformation of both the surface (E) and the indenter tip (E_i), as in Equation 8, where v_i is the Poisson's ratio of the tip itself. Note that for comparative purposes, the trends in Young's modulus for different materials indented by the same tip should be valid. In order to compare the Young's modulus of materials analyzed by different AFM tips, it is necessary to determine the Poisson's ratio and modulus of the tip material (i.e., diamond at 0.2 and 1220 GPa, respectively).

$$\frac{1}{E_{eff}} = \frac{1-\upsilon^2}{E} + \frac{1-\upsilon_i^2}{E_i} \tag{8}$$

The value for the reduced modulus is obtained through a model that represents the interaction of the AFM tip with the flat surface of the material analyzed. In order to neglect tip-surface interaction effects, the approach or *extend* force curve must be analyzed, as the *retract* curve may contain artifacts of adhesion. To accurately model force curve data, it is necessary to ensure that the data are appropriately plotted. In particular, it is critical that the force curve represents the force vs. tip-sample separation rather than the cantilever height measured by the instrument; this is the <u>*corrected force curve*</u>. The problem is depicted in Figure 6-2 for the approach. Once the force curve is plotted on the appropriate axes, the baseline should be flattened, if needed, to remove tilt in the non-contact region of the curve.

Figure 6-2. Deflection induced by tip-sample contact bends the cantilever by x. The instrument measures the piezo displacement, z. However, the penetration depth, δ, is required for analysis of the indentation modulus, and can be determined from z – x, given an accurate value of d_{sens}.



The scope of this SOP covers the interaction of either a spherical tip probe or a conical tip probe with the surface. In either case, it is necessary to identify the contact point, or where the value of tip-sample separation is zero. This may be done using built-in analysis software on the nanoindentation AFM. For analysis of indents made using a spherical tip, proceed to §6.1.2.1; to analyze results from a conical probe, proceed to §6.1.2.2.

6.1.2.1 *Spherical tip – Hertz model*

The Hertz model is used to describe the interaction of a non-deformable, spherical-tipped probe indenting an infinitely-extending half space. It is applicable for indentations whose depths do not exceed that of the indenter radius. Equation 3 should be applied to corrected force curves obtained in §5.3. A fitting routine is commonly supplied in AFM force-curve analysis software. Several indentations of various depths should be analyzed for precision, but take care to analyze only those data for penetration depths <10% of the total film/material thickness to avoid confounding contributions from the underlying substrate. For more information on fitting force curves for indentations in the Hertzian regime, refer to [10].

6.1.2.2 *Conical tip – Sneddon model*

A modification of the Hertz model, the Sneddon model is applicable to conical indenters and should be employed when the penetration depth of the tip is greater than the tip radius. It is especially applicable to soft biological materials, such as cells, where the deformation depth imposed by indentation is often too deep to apply Hertz models. The Sneddon model is applied to the corrected force curve using Equation 4. A fitting routine is commonly supplied in AFM force-curve analysis software. Several indentations of various depths should be analyzed for precision, but take care to analyze only those data for penetration depths <10% of the total film/material thickness to avoid confounding contributions from the underlying substrate. Fitting of the Sneddon model proceeds similarly to that of the Hertz model [10].

6.2 Key Results Provided

The results obtained from nanomechanical testing using AFM techniques are comparable to those gleaned from conventional nanoindentation measurements. There is an added benefit, however, in the ability to directly image the indented area immediately after performing the indentation steps. This can yield information about material nano-hardness and elastic modulus. Both materials characteristics are relevant for assessing the durability of the material given its in-use exposure.

6.3 QA/QC Considerations

After indentation in certain films/materials/surfaces/etc., the indenter tip may become contaminated with undesired residue. This can result in skewed force curves and/or aberrations in AFM images. Should the tip become contaminated (as evidenced by inconsistent force curves or streaking in AFM images), it may be necessary to remove the contaminant. In this case, indent a gold sample using a high force. It may be necessary to repeat the indentation on gold several times until the contaminant is removed.

Note that the models presented herein ignore attractive and repulsive forces that may arise between the tip and surface even in the *extend* force curves. For a comprehensive approach to addressing intermolecular interactions during nanoindentation, one must consider more complicated interaction theories [11]. In most cases, ignoring these forces is acceptable.

In light of the various sources of error that arise throughout the experimentation, from tip characterization to assumptions made regarding fitting models, to non-uniformities of examined surfaces, the expected error associated with the final reported Young's modulus may reasonably exceed ±10%. Strict adherence to tip qualifications and choice of the appropriate model will mitigate this error significantly.

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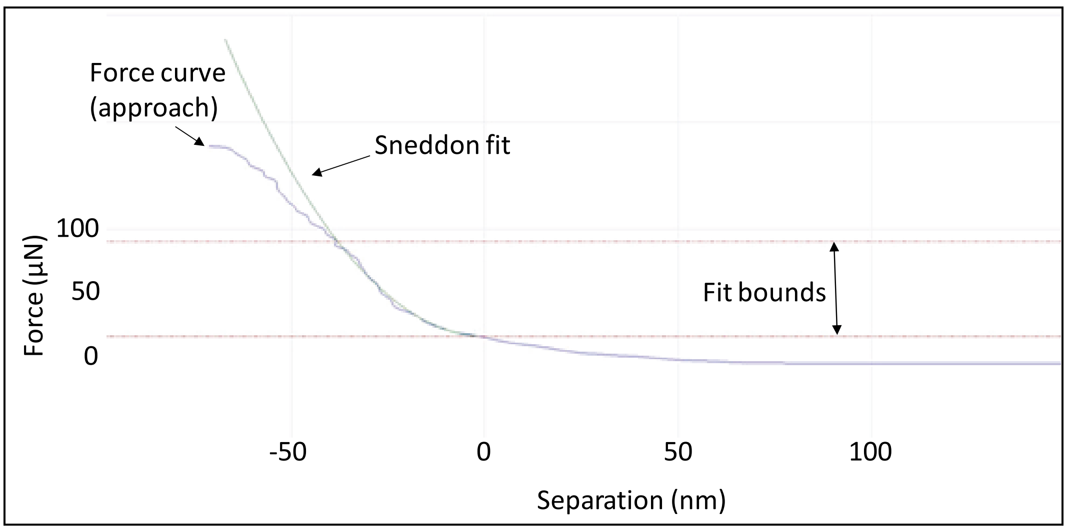
Appendix A: Notes and Supplementary Data

The contained SOP was applied to the reference material, <R> (50 nm thick gold on the [100] face of silicon), and the results are shown in the figure below. The particulars of the experiment are as follows:

Probe: Bruker PDNISP diamond-tipped stiff cantilever

$$d_{sens} = 185.34 \text{ nm/V}$$
$$k = 218.2 \text{ N/m}$$
$$\alpha = 51^\circ$$
$$\nu = 0.4$$

Figure A-1. Approaching force curve and best-fit for nanoindentation of gold reference material.



The approaching force curve is depicted in blue and the Sneddon fit to the data in green. The contact point is adjusted to 0 nm for tip-surface separation. The curve is fit over the range of 0-40 nm separation as depicted by the fit bounds in red. This range allows us to ignore the contribution of the underlying silicon substrate (encountered at separations > 50 nm). For this curve, the effective modulus is 79.8 GPa with R^2 = 0.99. From a matrix of [3 x 15] = 45 indents, the average modulus of gold was found to be 80 ± 7 GPa (compare to the reported value of the reference material of 78 GPa).

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13. SUPPLEMENTARY NOTES

14. ABSTRACT

The following methods provide a guide to measure mechanical properties of materials by means of an atomic force microscope (AFM). Traditional nanoindentation measurements do not afford immediate complementary surface imaging to visualize the residual indent. This obstacle is overcome using AFM. By indenting a surface with a diamond-tipped, stiff cantilever, local nanoscopic materials properties may be deduced. Briefly, an appropriate AFM cantilever is calibrated to determine its deflection sensitivity and spring constant; it is then used as both an imager and indenter at the surface of material of interest. The load applied by the cantilever is accurately controlled by knowledge of the deflection sensitivity. The maximum applied load is mediated by the cantilever spring constant. Following data collection, image and force curve analyses are completed to determine projected indent areas and load/unload profiles. This yields materials properties that include the material hardness and the Young's modulus along with corresponding surface topography.

15. SUBJECT TERMS

AFM	Thin films	Young's modulus
Nanoindentation	Force curve	
	Hardness	

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			**19b. TELEPHONE NUMBER** ***(include area code)***
Unclassified	Unclassified	Unclassified		24	