ATOMIC FORCE MICROSCOPE PROBE CALIBRATION BY USE OF A COMMERCIAL PRECISION BALANCE

M. S. Kim, I. M. Choi, Y. K. Park, and D.I. Kang

Division of Physical Metrology, Korea Research Institute of Standards and Science, Korea

ABSTRACT

In this paper, we investigate the characteristics of commercial AFM cantilevers and force calibration cantilevers in the range of 10 nN ~ 1000 μN by use of a high precision balance with resolution of 1 nN and 1-D fine positioning stage.

his document is a guideline to the authors of papers to be presented at the TC3 2005 conference. These directions are written in the format required for papers. We advise you to download these directions as a MS Word document and use it as the template for your paper because it contains all necessary formats and styles. Papers should be submitted before October 30, 2004. The paper must be written in English, and length is limited to 6 pages, including graphs, figures, reference list.

1. INTRODUCTION

Atomic force microscopes (AFM) are widely used as a standard tool of imaging micro- or nano-scale structures. Recently, they are becoming the “small robot” that manipulates atoms or molecules in tiny world in many nanotechnology areas, such as nanolithography and nanomachining. Moreover, biotechnology researchers adopted the AFM as the instrument of choice for quantitative force measurement, achieving 10^{-12} N resolution in studies ranging from the adhesion of antigen-antibody to the measurement of DNA tensile strength. Such applications require accurate control or measurement of one of physical quantities, force at the nanonewton level. However, unfortunately silicon based AFM cantilevers do not provide reliable measurements of small forces, because the spring constant of cantilevers or force-voltage characteristics of piezoresistive cantilevers are not precisely determined due to limitations of micromachining process in dimensional (thickness, length, width) control of cantilevers. Thus, accurate force measurements require identifying characteristics of each cantilever through a calibration.

In respond to the academic and industrial requirements for accurate determination of cantilever stiffness, more than ten calibration methods have been proposed so far. Cleveland *et al* [1-2] proposed the resonant frequency method in which the geometrical size of a cantilever and mechanical properties of material (e.g. elastic modulus and density) are measured or estimated from manufacturer’s values thereof. Sader *et al* [3-4] incorporates the viscosity and density of the medium in which the cantilever is immersed along with experimentally determined values of the resonant frequency and quality factor, together with the cantilever dimensions, in order to calculate the stiffness. A calibration method via hydrodynamic drag is also proposed in which a cantilever under a point load is placed under laminar fluid flow [5].

Although the above methods provide accurate and reliable calibration results, it is not easy to calibrate the AFM cantilevers *in-situ*. Therefore, more convenient, non-destructive, and fast methods have been introduced by using artifacts (or references) for force calibration. Precalibrated cantilevers [6-8] and specially designed reference springs [9-10] give not only ease-of-use but also high calibration accuracy. However these methods need “reference” or “precalibrated artifacts.” That means these artifacts should be calibrated accurately to determine the spring constant of AFM cantilevers with acceptable accuracy.

Another calibration method involving precision balances is suggested [11-13]. Precision balances of compensating type measure the force that deflects a cantilever. A piezoelectric actuator or stage displaces the cantilever whose displacement is measured using its internal capacitive feedback sensor. Since the balance uses a compensation (or null-balancing) method, a balance stamp maintains its original position (i.e., the stiffness of the balance is very large) all the time. Thus, the deflection of the cantilever is nearly same as the displacement of it. This method provide a direct, reliable, and accurate spring constant calibration of the AFM cantilevers, but the force below 10 μN is not traceable to the International Standards of Units (SI) because the minimum calibrated mass artifact is 1 mg. Recently, there are a few approaches that realize traceable force standards from the electrical units of SI, such as electrostatic and electromagnetic force [14-15].

Our aim is to calibrate the spring constant of the AFM cantilevers in the range of 10 nN ~ 10 μN, which is the first attempt in that the force range is close to nanonewton regime as far as we know. In addition to spring constant measurements, we will investigate the linearity, hysteresis characteristics and some factors that induce the calibration errors such as temperature changes, frictions and contact angle. In this paper, we present the “nano force calibrator (NFC)”, which consists of a precise compensation balance and a single-axis precision moving stage and give the preliminary calibration results of a piezoresistive cantilever that is used as AFM probes.

2. NANO FORCE CALIBRATOR (NFC)

A photograph of our prototype NFC is shown in figure 1 and also the piezoresistive cantilever to be calibrated is shown in the inset of figure 1. It is similar to that used in Ref. [6] but the balance resolution is 0.1 μg corresponding to 1 nN.

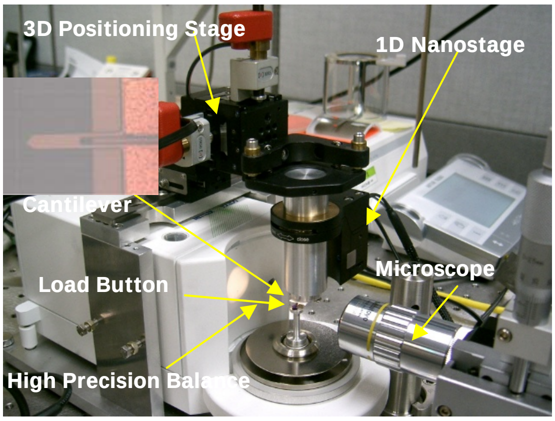

Figure 1 A photograph of the Nano Force Calibrator (NFC)

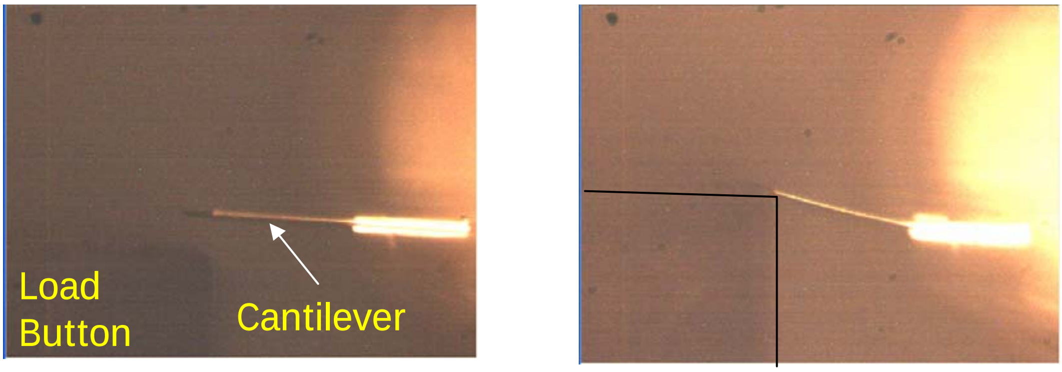

Figure 2 Photographs showing the cantilever (a)approaching the load button, and (b) deflected by the load button.

The cantilever to be calibrated is mounted on the one-dimensional (1-D) precision moving stage. The tip of the cantilever is pressed onto a load button that is made of sappier by moving the cantilever downwards. For the convenience of positioning cantilever onto the load button, the 1-D stage is mounted on a 3-D coarse positioning stage. An optical microscope is installed to give a vision when the cantilever approaches the load button. Figure 2(a) and (b) shows the cantilever, which is approaching the load button and deflecting due to the force acting between the tip and the load button.

3. SIMPLE MODEL OF THE MECHANICS OF THE CANTILEVER

In figure 3(a) and (b), the simple diagrams of the cantilever are illustrated, showing the side view of the cantilever unloaded and under loading, respectively. Although we did not find exact analytical solutions of the mechanics of the beam-bending arising in calibration of the AFM-cantilever, it seems useful to get a simple model in hopes that it may help us to figure out calibration results. Assuming the standard beam bending theory and beam curvature ρ is much higher than beam height h (i.e. σ>> h), the relationship of the contact force and the deflection at the tip is:

$$x = \frac{Fl^3}{3EI} \tag{1}$$

and the stiffness of the cantilever (*k*) is defined as follows:

$$k = \frac{F}{x} = \frac{3EI}{l^3} \tag{2}$$

where, l is the length of the effective lever arm, I is the axial moment of area, E is modulus of elasticity and F is the force acting at the tip, which is divided to two components, one (F_n) is acting normal to the load button surface, and the other (F_t) is tangential. Since we can only measure normal component of the force Fn, the calibrated stiffness of the cantilever (k_C) is:

$$k_C = \frac{F_n}{x} = \frac{3EI}{l^3}\cos\theta \tag{3}$$

where, θ is the slope at the tip, which is expressed as follows:

$$\theta = \frac{Fl^2}{2EI} \tag{4}$$

Equation (3) and (4) tell us that the calculated stiffness from the force measured by the precision balance and the displacement by 1D fine stage is lower than the original stiffness. Moreover, the much more the cantilever is deflected, the lower the calculated stiffness is. If the maximum slope at the tip is ten degree, the relative difference of the stiffness between at zero degree and ten degree (cosine error) is approximately -1.5 %. The tangential force due to the friction that occurs between the probe tip and balance load button may induce additional stress on the resistors located on the cantilever. Thus, the relationship between resistance and force might be more nonlinear than that between deflection and force. This interaction is not clear and more investigations or experiments should be performed in the future.

4. PRELIMINARY EXPERIMENTS

The piezoresistive cantilever used in this experiment had a normal resistance of 2 kΩ and spring constant of 100 N/m, all according to the manufacturer's specification. The cantilever chip is mounted on a ceramic base with gold contacts for wiring electrical leads. Changes in resistance were recorded using a 10-digits high precision multimeter. We used a tilting stage that holds the cantilever assembly and 1D precision nano stage, so as to adjust ceramic base was attached to the precision 1D stage through the fixture that holds the ceramic base of the cantilever was attached to a fine 1D stage, on which a tilting stage so as to adjust the orientation on of the cantilever with respect to the load button was adjusted almost zero degree from the horizontal defined by the sappier load acceptor to reduce the effect of the tangential force. plane diagram and a photograph of our prototype NFC is shown in figure 1 and figure 2, respectively. It is similar to that used in Ref. [6] but the balance resolution is 0.1 μg corresponding to 1 nN. The cantilever to be calibrated is mounted on the one-dimensional (1-D) precision moving stage. The tip of the cantilever is pressed onto a load button that is made of sappier by moving the cantilever downwards. For the convenience of positioning cantilever onto the load button, the 1-D stage is mounted on a 3-D coarse positioning stage. An optical microscope is installed to give a vision when the cantilever approaches the load button. Figure 3(a) and (b) shows the cantilever approaching the load button and deflecting due to the force acting between the tip and the load button.

Tables, graphs and equations should be included whenever possible on the page on which they are first discussed. Use one free line to separate them from the text before and after. They have independent numbering. Lettering in the figures should not be smaller than the size of the typed letters in the text. Do not use colored photographs and figures.

Table 1: Styles to be used

Use for	Style	Font
Title of the paper	Paper Title	14 pt, bold, all caps, centered
Author's names	Authors	12 pt, centered
Author's institutes or companies	Institutes	10 pt, centered
Abstract heading	Heading	11 pt, bold, align left
Abstract text	Abstract Text	10 pt, justified
Heading	Heading	11 pt, bold, align left
Paper text	Paper Text	11 pt, justified
Figures and tables titles	Figures/Tables	9 pt, align left/centered
References and addresses of authors	References	10 pt, justified

Digitize or paste down your graphs or figures. Captions start with boldface Table 1 or Figure 3 followed by a colon and a blank space. Equations are numbered (1), (2), ... at the right margin on the same line. Figure titles are 9 point (style "Figures/Tables") and should be placed below the figures; table titles are also 9 points (style "Figures/Titles") and should be above the tables.

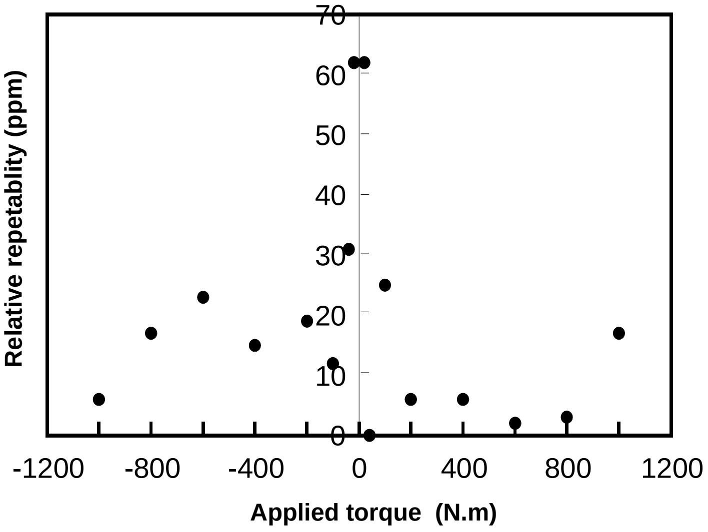

Figure1: Note how the text is centered under the figure

The table, figure or equation is centered between the left and right margins. It is not allowed to wrap text around a table, graph or equation.

$$\mathbf{U_{bmc} = 2.\sqrt{u_{ref}^2 + u_{drift}^2 + u_{temp}^2 + u_{reprod}^2}} \qquad (1)$$

The number of the equation is right aligned. Tables and equations must be made using the standard built-in features of the word processor, and features for automatic numbering may not be used.

5. SUBMITTING PAPERS

Papers must be submitted in electronic format as both MS Word document and as Portable document format (PDF) file. Send files as the attachment in a single e-mail message to e-mail: imeko@nlab.org , imeko@egyptww.com before October 30, 2004 Please include your paper number in message subject and mark two attached files as “Paper###.doc” and “Paper###.pdf”, where “###” stands for paper number.

ACKNOWLEDGEMENTS

Acknowledgements go into a separate section without section number before the REFERENCES. Note that the two blank lines before the heading of a section are not required for a section starting at the top margin of a new page.

REFERENCES

[1] Cleveland J P, Manne S, Bocek D and Hansma P K, “A nondestructive method for determining spring constant of cantilevers for scanning force microscopy,” *Rev. Sci. Instrum.*, **64**, 1993, pp. 403-5

[2] Walters D A, Cleveland J P, Thomson N H, Hansma P K, Wendman M A, Gurley G, and Elings V, “Short cantilevers for atomic force microscopy,” *Rev. Sci. Instrum.*, **67**, 1996, pp. 3583-3590

[3] Sader J E, Larson I, Mulvaney P, and White L R, “Method for the calibration of atomic force microscope cantilevers,” *Rev. Sci. Instrum.*, **66** 1995, pp. 3789-3798

[4] Sader J E, Chon J W M, and Paul M, “Calibration of rectangular atomic force microscope cantilevers,” *Rev. Sci. Instrum.*, **70** 1999, pp. 3967-3969

[5] Maeda N, and Senden T J, “A method for the calibration of force microscopy cantilevers via hydrodynamic drag,” *Langmuir*, **16** 2000, pp. 9282-9286

[6] Tortonese M, and Kirk M, “Characterization of application specific probes for SPMs,” *Proceedings of SPIE Vol. 3009*, San Jose/California, 1997, pp. 53-60

[7] Gibson C T, Watson G S, and Myhra S, “Determination of the spring constants of probes for force microscopy/spectroscopy,” *Nanotechnology*, **7,** 1996, pp. 259-262

[8] Cumpson P J, Clifford C A, and Hedley J, “Quantitative analytical atomic force microscopy: a cantilever reference device for easy and accurate AFM spring-constant calibration,” *Meas. Sci. Technol.*, **15** 2004, pp. 1337-1346

[9] Cumpson P J, Hedley J, and Zhdan P, “Accurate force measurement in the atomic force microscope: a microfabricated array of reference springs for easy cantilever calibration,” *Nanotechnology*, **14** 2003, pp. 918-924

[10] Cumpson P J, and Hedley J, “Accurate analytical measurements in the atomic force microscope: a microfabricated spring constant standard potentially traceable to the SI,” *Nanotechnology*, **14** 2003, pp. 1279-1288

Addresses of the Authors:

Min-Seok Kim, Mass and Force Group, Korea Research Institute of Standards and Science, Science Town, Daejeon, P.O. Box: 102, Korea. minsk@kriss.re.kr