New reference standards and artifacts for nanoscale physical property characterization

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ABSTRACT

This paper provides an overview of calibration artifacts being developed at the National Institute of Standards and Technology (NIST) that are intended to aid the accurate determination of nanoscale physical properties across a broad range of applications. We focus on three proposed reference standards: an SI traceable spring constant artifact for calibration of atomic force microscope cantilever stiffness in the nominal range between 0.02 N/m and 0.2 N/m, a piezoresistive force sensor for SI calibration of micronewton level contact forces, and a torsional oscillator for the absolute measurement of thin-film magnetic moments on the order of 1 μA m^2.

Keywords: atomic force microscopy, cantilever spring constant measurement, magnetic moment measurement, standard references and practices

1 INTRODUCTION

Products incorporating “new nanotechnology” are projected to generate one trillion dollars in revenues within the next decade [1]. Length [2] and force metrologies [3] are being developed at the National Institute of Standards and Technology (NIST) in anticipation of this trend. These metrologies are the basis for reference standards traceable to the International System of Units (SI) that can help calibrate scanning probe microscopes to measure physical properties in a traceable fashion. Artifacts traceable to well-defined length standards are already available from commercial sources to assist atomic force microscope (AFM) users with calibrating length measurements [4], and techniques to characterize probe tip geometry have been demonstrated to broad acceptance [5]. But accurate artifacts and techniques for calibrating AFM force sensitivity are still emerging [3]. We have shown that the force sensitivity of an AFM can be calibrated in a traceable fashion using a piezoresistive cantilever sensor that is first calibrated using an electrostatic force balance (EFB) [6]. In section 2, we give results using a new version of our EFB to calibrate a similar sensor, while in sections 3, 4, and 5 we report our efforts to create reference materials with calibrated physical properties.

2 FORCE SENSOR CALIBRATION

The force sensitivity and stiffness of a Kleindiek force measurement system* (FMS-MS, Kleindiek nanotechnik, Reutlingen, Germany) were calibrated using an EFB in a force versus displacement measurement mode like that proposed in Ref. 3. The goal was to calibrate a force sensor to verify the mean contact force applied during mechanical property measurements using atomic force acoustic microscopy (AFAM [7]).

2.1 Experiment

Figure 1 shows the force sensor in its holder, positioned near the latest NIST EFB. The sensing element is a 400 μm long, piezoresistive micromachined cantilever (too small to be visible in photo). The complete force measurement system includes an external bridge amplifier that measures changes in the electrical resistance of the piezoresistive element when forces are applied at the cantilever’s free end. A long standoff microscope was employed to observe the free end of the cantilever as it made contact with a 2 μm radius Hysitron cono-spherical indenter tip that was mounted on the balance’s weighing pan. A three-axis, fine motion stage was used for the gross manipulation, and then the balance null-control setpoint was varied to bring the indenter tip into contact. Contact was determined visually and also by monitoring the output of the force system’s measurement bridge, which was nulled before contact. Experiments were conducted in air, with the chamber sealed. The initial contact force was approximately 2.5 μN. A computer automated data acquisition and control routine incremented the balance displacement setpoint while maintaining feedback control. The resulting force,

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* Certain commercial equipment, instruments, or materials are identified in this article in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

displacement, and sensor output were recorded at four equally-spaced displacements. Thirty-six complete cycles (load and unload) were obtained.

2.2 Results

The force sensitivity was computed to be (24.2 ± 0.2) μN/V by fitting a straight line to each force versus voltage curve. Likewise, the nominal cantilever stiffness was computed to be (2.99 ± 0.02) N/m by fitting the force versus displacement data. Examples are shown in Figure 2. The bridge excitation voltage was left at the factory default (2.5 V) and was not calibrated against a voltage reference. The balance stiffness was nominally 0.03 N/m, and the force of its deflection was subtracted from the total. The stated uncertainty is simply one standard deviation of the 36 measurements. Uncertainty of the contact location may contribute another 0.5 % to the relative uncertainty of the reported force sensitivity (1.5 % to the stiffness, due to the cubic dependence on cantilever length). Load frame compliance was more than a thousand times less than that of the sensor and was neglected.

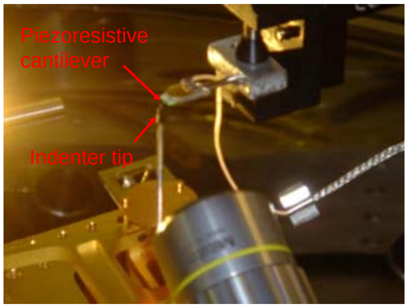

Figure 1. Force sensor approaching indenter tip mounted on NIST EFB

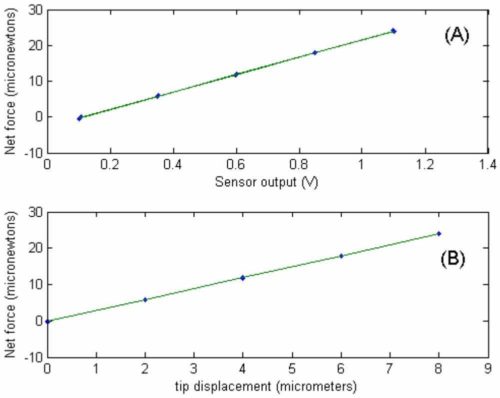

Figure 2. Force versus sensor output (A) and Force versus tip displacement (B) for a single loading cycle. Lines represent least squares fit of the data. The initial contact force was subtracted from the data prior to fitting.

3 STIFFNESS REFERENCE ARTIFACT

Forces measured with an AFM are usually computed from the measured tip deflection and spring constant of a cantilever sensing element. A convenient method for calibrating the spring constant is the cantilever on cantilever technique (e.g., [8]) which involves generating AFM force curves by pressing on the end of a cantilever of known stiffness. As demonstrated in section 2, it is possible to record both SI traceable force and displacement using an EFB to deflect a cantilever. So in principle, a reference cantilever can be tested using the balance, and an absolute value can be assigned to its stiffness. Such cantilevers could be made available to the public as NIST Standard Reference Materials (SRMs). However, microfabricated cantilevers are fragile, and we anticipate the need for a large quantity of low-cost, effectively disposable references, which is at odds with individual testing of each cantilever. Recognizing this, we are investigating whether wafer level measurements of resonance frequencies combined with selective absolute testing might adequately characterize the SRMs.

3.1 SRM designed for uniformity

Batch processing in semiconductor fabrication yields large quantities of uniform electronic devices on a single silicon wafer. In principle, uniform mechanical devices similarly could be achieved through photolithographic-based microfabrication of silicon cantilevers. The notion of making a wafer of uniform cantilevers for reference artifacts is not new [8,9]; however, previous attempts have lacked an absolute SI reference for performing statistical process control, or for error mapping and compensation of the variations within a lot. Here, we propose to monitor all cantilevers on the wafer using resonance frequency. Additionally, a subset of the cantilevers will be calibrated in an absolute sense, using the EFB to monitor for systematic deviations. Our experience testing the device of Ref. 9 illustrates the importance of having an SI traceable reference. Specifically, we observed deviations from reported stiffness values (as much as 40% in some instances) when a sample device provided by the National Physical Laboratory (NPL) was tested using both the NIST EFB and an SI calibrated instrumented indentation machine [10].

The NIST prototype cantilevers were designed and fabricated to minimize fabrication and measurement induced uncertainties. The design (Figure 3) consists of an array of seven rectangular cantilevers, microfabricated from single crystal silicon. The simple, prismatic beam geometry can be modeled analytically, and the cantilever stiffness k should be well described by the beam equation

$$k = \frac{Ebt^3}{4L^3} \quad (1)$$

where E is the elastic modulus and *b, t,* and *L* refer to width, thickness, and length respectively. Clearly, the most critical dimensional parameters are thickness and length.

Thickness was defined using high quality silicon-on-insulator (SOI) wafers (e.g., device layer was 1397 nm ± 17 nm according to manufacturer specification). Length control was established using calibrated e-beam lithography to both accurately pattern the cantilevers onto the SOI and achieve the proper alignment of the cantilever pattern with the edge of the handle wafer chip. Undercutting of the cantilever base during the backside etch is a problem that can lead to ambiguity in the effective length of the cantilevers [9]. Here, the e-beam was used first to precisely locate the edge of the silicon membrane by imaging through the device layer, and then to pattern the cantilever array in the proper position with respect to the backside etch.

The lengths vary from 300 μm to 600 μm in increments of 50 μm. The reference cantilevers are long enough so that relative uncertainties introduced by longitudinal misalignment during the calibration process should be less than 2%, since we believe we can ultimately probe the cantilever at a location that is repeatable to less than 2 μm.

3.2 Preliminary experimental results

Figure 4 shows a plot of stiffness values predicted by various methods for the cantilever array, along with experimental data obtained using the EFB to calibrate the shortest and longest cantilevers in the array. Agreement between the SI traceable determination, beam theory, and a determination based on beam theory combined with observed experimental resonance frequency is quite good: all values agree to within 3 % of the traceable numbers.

4 FORCE SENSOR AND STIFFNESS SRM

We are also attempting to create a piezoresistive cantilever force sensor that can serve as a force and/or stiffness SRM. The general layout of the sensor and the processing sequence for fabrication are shown in Figure 5.

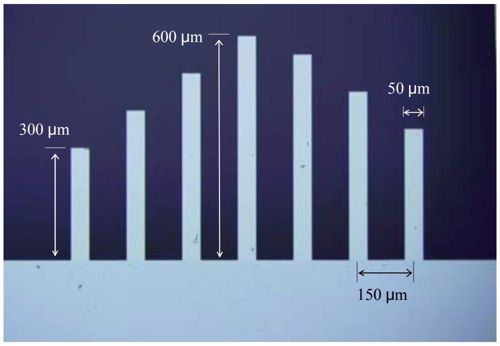

Figure 3. NIST stiffness SRM; cantilevers are 1.4 μm thick

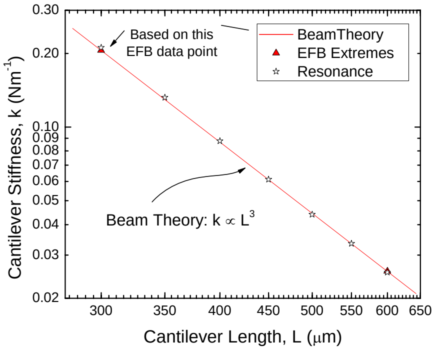

Figure 4. Plot of stiffness values for each cantilever as predicted by resonance measurement, beam theory, and as verified using EFB testing

Note that the beam thickness in the gage area has been reduced to produce higher sensitivity. The trade off for this sensitivity is a more complex artifact with additional critical dimensions to maintain. The numbered fiducials shown in Figure 5 indicate points along the cantilever's length where a calibrated stiffness will be determined, analogous to [9].

The functional dependence of stiffness on test location is more complicated in this design, but can be modeled assuming the gage area acts as a cantilever hinge and the remainder of the beam is a rigid lever arm. The exact functional relationship will be examined using the EFB and compared to the following model

$$k = \frac{EI}{\left(\frac{L+l}{2} x^2 - \frac{x^3}{6} \right)} \tag{2}$$

where *x* is the length from the cantilever base to the fiducial in question, *I* is the area moment of inertia of the two short beams that make up the hinge, *L* is the length of these beams, and *l* is the length of the rigid lever arm.

As with the previously described stiffness SRMs, the goal is to settle on a fabrication strategy which yields a high degree of uniformity within a wafer. Once again, we hope to use resonance frequency, this time conveniently measured from the piezoresistor, using a bridge as a process monitor to check for mechanical uniformity within a wafer run. This sensor is currently in prototype production, and testing results are not yet available.

5 THIN FILM MAGNETIC MOMENT SENSOR

The magnetic reference device of Figure 6 is a material property sensor itself, directly measuring the magnetic

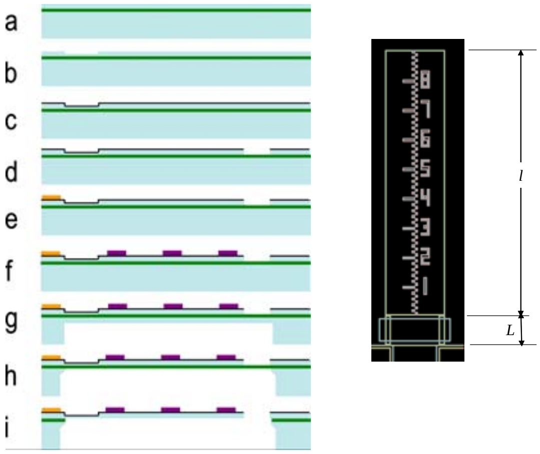

Figure 5. Processing sequence and plan view of piezoresistive cantilever (100 µm wide and 500 µm long) . Process steps include: a) start with SOI wafer, b) nitric/HF wet etch, c) boron diffusion, d) deep reactive ion etch (DRIE), e) Ti/Au contacts, f) Ti fiducials, g) DRIE, h) KOH wet etch, i) reactive ion etch release

moment of a thin film deposited on its surface [11]. The magnetic moment acts as a parallel spring, increasing the restoring torque that acts on the moving paddle when oscillating in the first torsional mode. The value of this restoring torque can be extracted from measurements of the resonance frequency as a function of the applied static bias field. The absolute calibration of this device in terms of SI units is dependent on knowing either the moment of inertia associated with the vibrating paddle, or on having an accurately determined value for its torsional stiffness [11].

We propose to measure the torsional stiffness by probing the paddle across its diameter using a calibrated instrumented indentation machine to record the stiffness normal to the plane of the sensor using techniques such as those described in [10]. The measured normal stiffness k_{meas} should follow the relation

$$k_{meas} = \frac{1}{\left(\frac{1}{k_n} + \frac{r^2}{\kappa}\right)} \tag{3}$$

where k_n is the normal stiffness at the center of the paddle, κ the torsional stiffness of the resonator, and r the offset of the indenter tip from the centerline. An initial measurement found k_n=211 N/m ± 8 N/m.

6 CONCLUDING REMARKS

We have presented results from ongoing efforts at NIST to develop artifacts and techniques that can aid in the determination of nanoscale physical properties measured using AFM, AFAM, and a torsional resonator. The common thread in these discussions is the pursuit of absolute accuracy through connection to SI traceable measurement standards.

The SI base quantities of length and mass are defined most accurately at levels nine orders of magnitude greater than those relevant to nanotechnology. Nevertheless, no comparison between nanoscale and bulk properties can take place absent this common measurement basis. Although it is increasingly difficult to preserve an unbroken SI measurement chain, especially for mechanical quantities, the effort remains worthwhile, and we remain optimistic that reference artifacts and standards for nanometrology, such as those we have described, ultimately will be achieved and become available to the technical community.

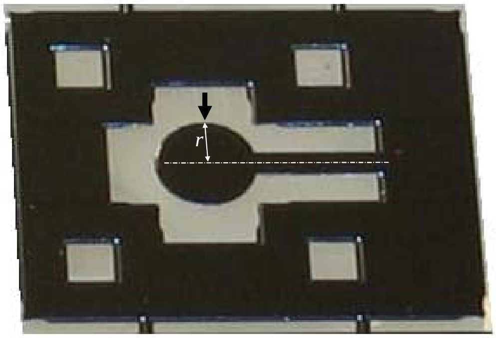

Figure 6. Thin film magnetic moment sensor ($r \approx 2$ mm)

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