Microforce and Instrumented Indentation Research at the National Institute of Standards and Technology, Gaithersburg, MD

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ABSTRACT

This paper provides an overview of recent efforts at the National Institute of Standards and Technology (NIST) to develop metrology and standards to support general users of instrumented indentation and scan-probe devices. Research directed towards a primary realization of force in the regime below 10 µN, development and characterization of novel compact microforce sensors, scan-probe force calibration, and standardization of instrumented indentation practices and calibration procedures is included. Descriptions of various laboratories and facilities are also highlighted.

BACKGROUND

Many commercial and custom force-measuring instruments, including nanoindenters and atomic force microscopes (AFM), have recently been developed with resolutions extending into the nanonewton regime. These have been used for studying micromechanical material properties such as hardness and modulus [1], for testing fatigue and fracture of thin films [2], for studying adhesion of ultrathin films [3], and even for the measurement of covalent bond forces[4] in the piconewton range. In Figure 1 we attempt to convey a sense of the tremendous scope of the forces, phenomena, and instruments encountered.

As miniaturization trends continue in advanced technology industries, for example in the manufacture of microelectronics, photonics, data storage devices, and micro-electromechanical systems (MEMS), it will be increasingly necessary to rely on these nanonewton force measurements for the control of manufacturing processes, the evaluation of device performance, and the characterization of material behavior. Correspondingly, a desire for accurate, traceable, small force measurement is emerging within ISO task groups and American Society for Testing and Materials (ASTM) committees that work on instrumented indentation standards [5] and the fatigue and fracture of structural films. However, no methods for establishing force measurement traceability at these levels are currently available. It is within this context that the NIST Microforce competence is being developed, with the purpose of creating a facility and instruments capable of providing a viable primary force standard below 10⁻⁵ N, and with the goal of realizing force in this range at a relative uncertainty of parts in 10⁴. This new project complements a body of existing work at NIST to develop standards and methods for the instrumented indentation community, with the two combining to provide a metrological basis for manufacturers seeking traceable characterization of thin film mechanical properties. In what follows, we highlight our recent advances, outline possible future directions, and summarize ongoing work towards the establishment of small force and instrumented indentation standards.

A PRIMARY SMALL FORCE STANDARD

Conceptually, the most straightforward approach to force realization is to use a calibrated mass as a dead weight in a known gravitational field; however, it is not possible to maintain high levels of relative precision with this approach as the masses are subdivided to achieve smaller forces. The smallest calibrated mass available from NIST is 1 mg (approx. 10 µN dead weight) having a relative uncertainty at the level of a few parts in 10⁴. In principle smaller masses could be calibrated, but they would be difficult to handle, and the trend is for the relative uncertainty to increase in inverse proportion to the decrease in mass [6]. If this trend continues, the uncertainty would reach the same magnitude as the force at a dead weight producing 1 nN.

Alternatively, forces can be realized in this range via the electrical units defined in the International System of Units (SI), and linked to the Josephson and quantized Hall effects. The SI unit of length is also required. This realization can be done using electromagnetic forces (e.g., the NIST Watt Balance Experiment [7]) or using electrostatic forces [8]. We chose the latter because the required metrology seems somewhat simpler to execute, and the forces generated, although generally less than those feasible electromagnetically, are appropriate for the force range of interest.
Figure 1. The relative magnitude of small force phenomena and the instruments employed in their measure.

The mechanical work required to change the separation between two electrodes of a capacitor while maintaining constant voltage is

$$dW = F \, dz = \frac{1}{2} \, V^2 \, dC$$

where $dW$ is the change in energy, $F$ is the force, $dz$ is the change in separation, $V$ is the electric potential across the capacitor, and $dC$ is the change in capacitance. Thus force can be realized from electrical units by measuring $V$ and the capacitance gradient, $dC/dz$:

$$F = \frac{1}{2} \left( \frac{dC}{dz} \right) \, V^2.$$  \hspace{1cm} (2)

As a validation of this electrostatic force realization, we desire to cross check with dead weight forces, at least in the higher force range where the uncertainty achievable mechanically is still competitive. For this reason we have designed our force generator to operate along the vertical axis as part of an electromechanical null balance, as in Figure 2.

Recent results with this instrument [9,10], referred to as the NIST Electrostatic Force Balance, demonstrated a relative standard uncertainty of a few parts in $10^4$ in the comparison of gravitational and electrostatic forces ranging between $10 \, \mu N$ and $100 \, \mu N$. The experiment consisted of operating the force generator as a null displacement force balance, where the force generator comprised a pair of concentric cylinders. In this arrangement, the outer cylinder of the resulting capacitor is fixed while the inner is free to move along an axis constrained by a rectilinear spring mechanism, as in Figure 2. The relative position of the electrodes is maintained using servo control. The voltage required to maintain the null geometry of the capacitor when a load is applied to the suspended electrode can thus be directly interpreted as an SI force from previous measurements of the capacitance gradient. This gradient is obtained by displacing the moving electrode while recording both the resulting change in displacement and capacitance. These measurements are made using traceable methods: an interferometer for displacement and a calibrated ac bridge for capacitance.

At present, the resolution of our prototype balance is on the order of $10^{-8} \, \text{N}$ due to limits on the resolution of null using an interferometer to detect the deflection of the comparatively stiff balance suspension (approximately $10^{-8} \, \text{m}$ and $13.4 \, \text{N/m}$, respectively). To extend the resolution of the device, a new suspension system has been designed [11] and plans have been
made to move the entire system into a vacuum chamber. The new suspension system has demonstrated the ability to produce an equivalent spring constant below 0.05 N/m using a stiffness compensation scheme. The mechanism also makes use of a counter balanced parallelogram linkage, so the center of gravity may be located on the central pivot axis. This helps minimize the sensitivity to seismic disturbances. The move to vacuum will eliminate variations in the index of refraction that affect both the capacitance and displacement metrology, eliminate air currents and acoustic vibrations that perturb the balance, and it will increase the breakdown voltage, extending the useful range of the balance. Resolution on the order of $10^{-11}$ N is expected due to these combined improvements. At this level, we hope to explore molecular and optical forces. For instance, as indicated in Figure 1, the pressure exerted by light can be computed from determinations of the incident optical power (W) and reflectivity ($r$) using the speed of light (c). These measurements can be made independently and used to verify the resolving power of the balance system. We also observe in Figure 1 that most covalent bonds have strengths of a few nanonewtons. The next generation balance should allow us to measure such forces with percent level accuracy in a fashion consistent with the SI. For this work, we intend to use the electrostatic force balance as a surface forces apparatus, and could conceivably perform a Casimir force experiment, paving the way for a quantum based force standard linked to the SI. Eventually, it is also our purpose to design transfer artifacts, i.e., calibrated load cells or force generators, through which we can disseminate this realized force standard to users in industry and academia. Two systems we have considered to date are commercially available piezoresistive cantilevers and capacitance based micro-load cells, custom fabricated at NIST.

FORCE TRANSFER STANDARDS

The typical macroscopic force transfer standard is a high-quality strain-gage load cell capable of reproducing changes of load within its operating range with accuracy on the order of parts in $10^6$ of its full scale value (e.g., kilonewtons). At the level of micro and perhaps nanonewton forces, we would like to transfer the unit of force with a more modest accuracy of a few tenths of a percent. This is the accuracy sought in draft measurement standards for instrumented indentation, and is representative of the growing metrology needs in the measurement of small force.

The notion of transferring the SI unit of force through an artifact is well developed at the macroscale and is characterized by two fundamental approaches. As an example of the first approach, consider a typical loading apparatus, such as a materials testing machine, that is equipped with a load cell to measure the magnitude of the tensile or compressive forces that it applies along its loading axis. This load cell can be calibrated against a primary standard of force, such as a deadweight. In this fashion, the unit of force is disseminated from the primary standard. In the second approach, for this example, the machine is calibrated indirectly using a calibrated specimen. This calibrated specimen is, in fact, another load cell that has been calibrated against a deadweight machine. Once again, the unit of force is disseminated from a primary standard to the testing machine, this time through calibration of an intermediary load cell. The accuracy of this second approach is inferior to the first due to the added complexity of interfacing the two load cells. However, the use of a secondary force standard has advantages when a large number of machines of varying geometry must be calibrated on a continuous basis, since only one load cell needs to be calibrated against a primary standard.

What we seek in a microscale load cell transfer standard is, by analogy to these conventional force practices, a device with a well-defined loading point, responsive to loads only along a well-defined axis, and possessing its own sensor for converting the load to a usable readout. This readout is preferably a voltage that is repeatable to parts in $10^5$. We further desire this load cell to be capable of use in either of the two transfer approaches, which implies, in the case of scanning probe instruments, that it must be compatible with both commercial AFM sensor and specimen holders. This last requirement suggests that there will be instances when the load cell should fit within a 3.6 x 1.6 x 0.1 mm volume typical of the chips employed by commercial AFM’s.
Piezoresistive cantilever calibration experiment

The sensitivity of a piezoresistive cantilever was determined in a proof of concept experiment [12] to begin examining how the unit of force can be disseminated from the primary electrostatic force balance. The cantilever was selected as an example of a device that could function as a calibrated transfer artifact for scanning probe instruments.

The electrostatic force balance was probed using a commercially available piezoresistive cantilever, as illustrated in Figure 3. In the proof of concept experiment, the cantilever had a nominal resistance of \(2 \, \text{k}\Omega\), length of approximately \(3 \times 10^{-4}\) m, and spring constant of \(1\) N/m, all according to the manufacturer's specifications. Changes in resistance were recorded using an a.c. Wheatstone bridge configuration and a two-wire connection. The bridge was excited with a \(1\) kHz sinusoidal voltage of \(1\) V peak amplitude, and the bridge output was monitored using a lock-in amplifier. The cantilever chip came mounted on a ceramic base with gold contacts for making electrical connections to which we soldered the two wire leads.

An obvious issue to be addressed in the development of a standard load cell for AFM will be a methodology for fixing the orientation of the device with respect to the primary force balance. We chose to use the ceramic base as a reference surface, gluing the cantilever assembly to a glass microscope slide, which was then clamped in a fixture that held the assembly at an angle approximately \(14\) degrees from the horizontal defined by the balance weighing pan. The angle was selected as representative of the nominal angle used in typical AFM instruments, though, to our knowledge, no standard exists. The fixture was attached to a combination coarse and fine adjustment three-axis scanning stage for probing the balance. In these initial experiments, no attempt was made to align the vertical stage axis to the balance primary axis, though this too will be important in developing a standard procedure.

The cantilever was brought into contact with the top of the balance by manually turning a micrometer screw on the vertical axis of the three-axis stage. The contact region was defined using a \(3 \times 10^{-4}\) m polished glass cube affixed to the weighing pan. The physical contact point was observed optically to be near the edge of this cube using a long standoff microscope. Upon contact, the force was sensed by the bridge output. A nominal preload of \(10^{-6}\) N was established by monitoring the balance output before re-zeroing the bridge. An automated fine motion scan was next executed using an electrostrictive actuator to drive the stage, pushing the cantilever into the balance pan. The stage was displaced a fixed increment, the balance allowed to settle for about one minute, and then the servo voltage and bridge output were sampled 150 times at nominally \(3\) Hz, with the average of these values being recorded as a single load point. The stage was scanned in and out through six such increments. This load and unload sequence was repeated between 30 and 40 times to yield a complete set of measurements over a period of about \(3\) hours. Each scan was fit using a least squares straight line to determine the slope and hence the sensitivity for a given scan. Each scan was normalized about the initial load and bridge output in an attempt to account for drift. The estimated sensitivity for the cantilever is reported as the average of the fits, with the standard deviation of the measurements indicating the repeatability of the setup. The cantilever was then retracted from the balance along the vertical axis and then parked off to the side. This entire experiment was repeated to check for reproducibility. Results are graphed in Figure 4, where we see that scan (a) yields a sensitivity of \(4.95\) with a deviation of \(0.06\) \(\mu\)N/m/V/V (1 %) while scan (b) yields a sensitivity of \(4.67\) with a deviation of \(0.03\) \(\mu\)N/m/V/V (0.6 %).

The data obtained from the proof of concept experiment reveal a discrepancy between measurements, since the deviation between the two measurement groups was greater than the repeatability within a setup. Subsequently, we have modified the apparatus and the procedure. The glass cube has been replaced, since closer inspection revealed chips and inconsistencies in the appearance of its surface. Now a ruby sphere (3 mm diameter, 0.64 \(\mu\)m sphericity) acts as the contact. This sphere is a precision optical component possessing a polished surface, and is clearly devoid of topology within the resolution of our microscope (on the order of a few \(\mu\)m). Instead of the ac bridge detection technique, we now simply use a precision resistance meter, allowing direct measurement of the cantilever sensitivity in terms of Newtons/Ohm.
The revisions appear to have eliminated the discrepancy, with the variation between recent measurement groups being consistent with the variation within a measurement ensemble, presently around 0.7% of the nominal sensitivity. However, much remains to be examined regarding the influence of the contact surface on the measured sensitivity. For instance, we do not know if the meniscus forces present at the contact will have a measurable influence on sensitivity. This influence might be revealed by a dependence on humidity, for example. The influence of errors in alignment (are we touching the top of the sphere?) also remains to be explored. We plan to examine these and other issues in the future as we begin the process of refining the technique and developing a complete uncertainty analysis necessary to produce a traceable calibration procedure.

Capacitance based micro-load cells
The load elements of instrumented indentation systems vary widely and do not fit conveniently into the previous analogies. In the case of indentation machines, the load application and sensing element are most often one and the same, and they are often electromechanical force generators functionally akin to the Electrostatic Force Balance. An indirect calibration scheme, where the indenter probes a load cell of known sensitivity, is the most likely scenario for calibration of these instruments. The forces produced and measured by such systems can vary from a few nanonewtons to tens of millinewtons. Direct application of deadweight load is thus a viable approach for calibration in the upper end of the operating range. However, from our experience we know that it is important to apply the calibration load in the same fashion as the loads to be measured. Distortions of the frame or suspension due to off axis loading will affect the measured sensitivity. Typically, deadweight calibration of the force generator is accomplished by applying a tensile load. In contrast, most indentation is performed in compression. To begin examining these issues, we have attempted to construct a cantilever capacitor load cell demonstrated by Howard and Smith [13], as shown schematically in Figure 5. The load cell was constructed in collaboration with Dr. Stuart Smith of the University of North Carolina at Charlotte and Mr. Shane Woody, of Insitu Tec, Inc. The design has a flat profile and a volume that is compatible with specimen stages typical of instrumented indentation equipment. A parallelogram flexure constrains the motion axis, so the cell should be less sensitive to placement of load than cantilever designs. Load is detected as a change in capacitance, measured using an ac transformer bridge and lock-in amplifier.

The sensitivity is a nonlinear function of the nominal separation of the parallel plate electrodes and drift has been a problem. Sensitivities have been measured using an instrumented indentation machine, rather than the primary balance, for speed and convenience. The indentation machine applies a 350 µN load in ten ascending steps, then reverses the sequence. Following standard practice [14], the load cell is cycled three times before data is collected. A linear fit of the sensor output voltage versus applied load yields an average sensitivity of 75.79 µN/mV with a standard deviation of 0.09 µN/mV after nine calibration sequences. Clearly, the uncertainty associated with the linear fit is much greater than might be inferred from the standard deviation of sensitivities, since a rather large hysteresis of 3 µN is apparent in Figure 6 (b). A second order regression of only the ascending force response produces residuals far more random in structure, possessing a standard deviation of 0.05 µN as shown in Figure 6 (d). This deviation is near the resolution of the indentation machine. We have yet to repeat these measurements to assess reproducibility.

Commercial equipment and materials are identified in order to adequately specify certain procedures. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.
Figure 5. Parallelogram load element with integral capacitance displacement sensor

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y = 75.787x - 3.4925 \\
R^2 = 0.9996
\]

Figure 6. Capacitance force sensor calibration results (a) linear regression of all ascending and descending load data (b) residuals from linear fit of all data points (c) residuals from linear fit of only ascending force data (d) residuals from second degree polynomial curve fit to ascending force data
**INSTRUMENTED INDENTATION STANDARDS**

Indentation methods have traditionally been used to measure the hardness of materials, using applied forces ranging from 0.1 N to 1500 N. Standard test methods typically required only a knowledge of the maximum force applied (and perhaps the value of a small preload) and the maximum depth achieved or the dimensions of the resulting residual impression. In recent years, the technique has been extended to significantly smaller applied forces, with force and displacement data collected throughout the loading-unloading cycle. Analyses of these force-displacement data permit the determination of both the specimen hardness and elastic modulus [1]. This type of measurement, commonly referred to as instrumented or depth-sensing indentation, can now be performed with a variety of commercially available indentation machines. Several of these have displacement resolution better than 1 nanometer and force resolution better than 1 micronewton, and can obtain meaningful mechanical property information at indentation depths as small as 10 nanometers. When used at small forces and depths, the technique is referred to as nanoindentation.

Improvements in instrumentation and data analysis have made nanoindentation the method of choice for measuring the mechanical properties of very small volumes of material. In particular, nanoindentation is now used routinely to characterize the mechanical properties of thin films and coatings as diverse as auto paint, TiN coatings for extending the life of cutting tools, and surface films on computer hard disk drive surfaces. Problems arise in the marketplace, however, as soon as a manufacturer tries to make properties measured by nanoindentation part of a product specification, as there currently are no U.S. or international standard test methods for performing nanoindentation tests, and no procedure for verify the performance of a testing machine. There is a Draft International Standard (DIS) in ISO (ISO/DIS 14577 –1,2,3) entitled “Metallic materials – Instrumented indentation test for hardness and materials parameters,” but it has not yet received final approval, and it deals solely with the testing of bulk materials. A fourth part for the document (ISO/DIS 14577-4), which deals specifically with the testing of thin films, is under development. In the U.S., an ASTM Task Group (E28.06.11) is developing both a Standard Practice and a Test Method for instrumented indentation, but these documents have not yet been submitted for sub-committee ballot.

In all of the standards draft documents, the issue of traceable calibration and verification of force and displacement data produced by the testing machine is a primary concern. In order to obtain values for hardness and elastic modulus with an accuracy on the order of 5 %, force and displacement data must be accurate to 1 %, and must therefore be calibrated and verified with devices that are accurate at the level of 0.25 % to 0.5 %. Heterodyne interferometry can provide the necessary length metrology but, as noted above, there is at present no traceable force standard below 10 micronewtons. Current standards documents simply state that the devices used for force verification should be traceable to national standards “if available” [5].

**NIST MICROFORCE RESEARCH FACILITY**

NIST has developed an extensive laboratory capability around the Electrostatic Force Balance experiment to support research on quantitative small-force measurement and standards. The NIST Microforce laboratory is well equipped with instruments representing the two general classes of small force measurement devices, namely instrumented indentation machines and atomic force microscopes. The laboratory also maintains a small class 100 clean room for sample preparation. The sample prep room acts as a pressurized buffer between a general purpose lab and a temperature-controlled isolation room. This isolation room is designed to provide shielding from both acoustic and electrical noise, temperature control, and clean, low-velocity laminar air flow within the room. A vibration isolated optical table is housed in this isolation room along with a bell jar type vacuum chamber. The NIST Electrostatic Force Balance will ultimately be housed in the vacuum chamber within the isolation room.

**CONCLUSION**

The quantitative measurement of small force is of increasing importance to both research and commerce. Recognizing this, the National Institute of Standards and Technology has embarked on an effort to establish a metrological basis for these measurements. The focus of NIST efforts is on the technically challenging regime below 10^-6 N, where reliance on traditional deadweight realizations of force becomes problematic. Here we have reviewed NIST research into the problem of developing a primary realization of force through the SI electrical and length units, the dissemination of this force through calibrated artifacts, such as piezoresistive micro-cantilevers and compact capacitive load cells, and the ultimate role such calibrated artifacts may come to play within the context of international standards for the measurement of materials properties using instrumented indentation equipment. To the best of our knowledge, NIST is the pioneering laboratory in the world in the determination of SI forces in this regime, and hopes this review of its nascent efforts will stimulate others to embrace this technical challenge.

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