

Probe-force calibration experiments using the NIST electrostatic force balance

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The sensitivity of a piezoresistive cantilever force sensor has been determined by probing the weighing pan of the National Institute of Standards and Technology's prototype electrostatic force balance. In this experiment, micronewton contact forces between a force probe and the balance's weighing pan are determined from the voltage required to maintain the null position of the force balance. The force balance incorporates length and electrical metrology so that an electrical realization of force consistent with the International System of Units may be computed from measurements of the voltage and capacitance gradient. Here, the balance serves as a primary realization of force in a continuous range between 0 and 6×10^{-5} N, allowing calibration of the sensor against a traceable standard.

Introduction

Originally conceived as a means to image insulators [1], the atomic force microscope (AFM) has quickly distinguished itself as a useful tool for the measurement of surface forces [2] and, in more recent studies, of intermolecular forces of single macromolecules, such as DNA and proteins [3,4]. The problem of calibrating the force sensitivity of these devices is well known, and a variety of approaches have been considered in the literature [5,6].

Recently, NIST developed an electronic null balance for comparing weak forces derived from mechanical, electrical, optical, or molecular sources as part of our work to link small force measurement to the International System of Units [7]. The goal is to realize and disseminate small force through an electrostatic force balance similar to, but on a smaller scale than, voltage balances used in fundamental electrical metrology [8]. Such a balance might serve as a primary standard for probe-type force measuring instruments in the regime below 10^{-5} N. Eventually, the plan is to design transfer artifacts, i.e., calibrated load cells or force generators, through which we can disseminate this realized force to users in industry or academia.

In this paper, we briefly review the working principles of the electrostatic force balance and describe how it can be employed to provide a force calibration of a piezoresistive AFM cantilever. We suggest that such a cantilever can be employed directly to achieve calibrated force measurements in AFM style instruments, or indirectly by acting as a calibration specimen, or transfer artifact, that can be probed with another cantilever. As a first step in our effort to explore these possibilities, we attempt to calibrate the force sensitivity of a commercially available cantilever. The results of this proof-of-concept experiment are presented and discussed. We find encouraging repeatability in the determination of force sensitivity, with deviations below a percent for a specific contact condition; however, our ability to reproduce the measurement is limited at the level of a few percent, presumably due to inconsistencies at the contact interface. Finally, we conclude with suggestions for improvements in the procedure and apparatus that might result in higher levels of reproducibility.

Force balance and load cell calibration

We have designed and built a prototype electrostatic force balance for realizing forces in the micronewton range, as illustrated schematically in Figure 1(a). The active electrodes are concentric cylinders, the outer serving as the reference and the inner suspended and guided by a rectilinear flexure mechanism. The geometry has been designed such that a near-linear capacitance gradient of 1pF/mm is achieved at a working overlap of 5 mm. Thus, an electrical potential across the cylinders produces an electronic realization of force along the suspension axis

$$F = \frac{1}{2} \frac{dC}{dz} V^2 \quad (1)$$

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where dC is the measured change in capacitance for a given change in electrode overlap dz , and V is the applied electric potential.

Previously, we have used this force balance in an automated null-displacement mode to compare an electric realization of force with the force generated by calibrated deadweights of nominal mass 1 mg, 10 mg, and 20 mg. The forces involved in this comparison were calibrated independently via traceable techniques. SI traceable determinations of capacitance, length, and voltage were used to establish the value of the electronic force, with corrections for the alignment of the interferometer, balance, and electrical axis with gravity. The mass of the deadweights was determined by comparison to the NIST working mass standards with corrections made for buoyancy. Gravity was extrapolated from an absolute gravimeter measurement taken elsewhere on campus using knowledge of the relative elevation between the two sites. Comparisons between the electrostatic and deadweight forces agreed to within a few parts in 10^4 after including all known correction factors [9].

Now we seek to compare probe forces to the calibrated electrostatic forces by once again maintaining a null displacement. In this experiment, we probe the balance pan with a piezoresistive cantilever force sensor, as illustrated in Figure 1(b). The voltage on the electrodes is adjusted by a closed loop feedback servo that produces a compensating force to maintain the null position of the balance as the cantilever is pressed into the weighing pan. In principle, measurement of the change in voltage required to maintain null, combined with prior measurements of the capacitance gradient of the electrodes, allows direct computation of the force existing between the balance stamp and cantilever under test, within the accuracy with which null is maintained. By stepwise variation of the degree to which the sensor presses against the balance, one can map the force sensitivity of the device under test by recording its sensor output as a function of the computed null force. An example taken from our experiments with a piezoresistive cantilever is shown graphically in Figure 1(c).

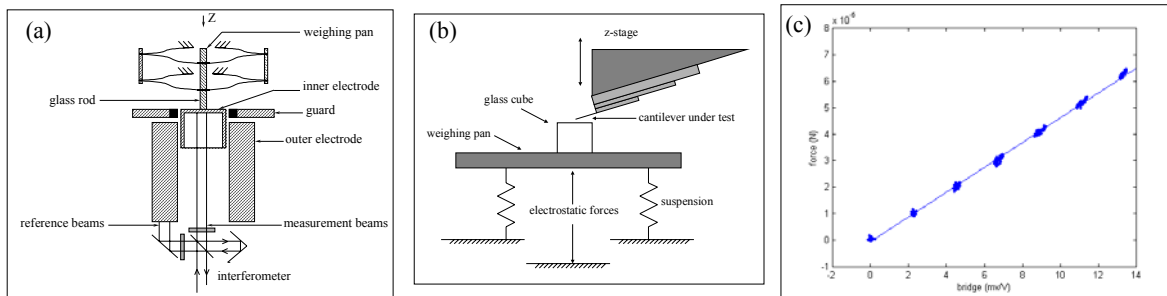


Figure 1. (a) Schematic of the NIST electrostatic force balance, (b) illustration depicting the probing of the balance with a cantilever load element, (c) typical sensitivity data for a piezoresistive type cantilever

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 removed and calibrated against a primary standard of force, such as a deadweight-loading machine. In this fashion, the unit of force is disseminated from the primary standard to the materials testing machine via direct calibration of its load cell.

In the second approach, our example 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 10^4 . We further desire this load cell to be capable of use in either of the two transfer approaches, which implies that it must be compatible with both commercial AFM sensor and specimen holders. This last requirement suggests the load cell should fit within the $3.6 \times 1.6 \times 0.1$ mm volume typical of the chips employed by commercial AFM's.

Survey of scanning probe sensors

A review of the literature reveals a fairly limited selection of sensors in the desired micro to nanonewton force range, with the vast majority based on a cantilever elastic load element that deflects in response to the applied load. Such cantilever sensors usually have an "atomically" sharp tip that provides the requisite well-defined loading point; however, from a metrology standpoint, this arrangement fails to define a single measurement axis, since a cantilever will also respond to moments occurring at the point of contact. Ideally, a constrained load element, such as a parallelogram flexure, is preferable, though few examples of such devices exist on this scale, and none that are directly compatible with AFM instruments are available from commercial sources to our knowledge.

The detection schemes used to measure the elastic deformation of scanning-probe cantilevers, such as capacitive, interferometric, and the well-known optical lever arm techniques [10] present another set of problems for accurate force metrology. For instance, all of these techniques rely on an externally supplied metrology frame in order to monitor the deflection and produce the force readout. In particular, the popular optical lever arm requires a large housing separate from the cantilever to hold the laser light source and quadrant photo detector. Clearly, the displacement calibration of these instruments can vary greatly with set up and alignment of the cantilever with respect to the external frame of reference. Direct calibration of such devices is thus conceivable, but awkward. And it seems we cannot use these devices as an indirect calibration specimen. The only available approach for indirect calibration is to resort to calibrating the spring constant of the load element, since this can be accomplished independent of the detection hardware. However, to embrace this approach requires that the AFM's detection scheme subsequently be calibrated as a displacement consistent with the displacement measured during the primary calibration of the spring constant, a difficult task.

No existing sensor satisfies all our requirements. However, there is a cantilever sensor that avoids many of the above complications, at least as regards the detection scheme, and might be readily adapted to the problem of force transfer. This is the well-known piezoresistance approach, which we review in the following.

Piezoresistive cantilever load cell

The variation of bulk resistivity of a material with applied stress is known as the piezoresistive effect. It has been used in various pressure detection schemes since its discovery, and has been applied successfully to the AFM to achieve atomic resolution imagery [11]. As applied in AFM, a resistor is typically doped into a region of material at the base of a silicon cantilever. The nominal resistance is a function of dopant concentration (typically boron) and can be fabricated using known relationships about semiconductor properties.

Deflection of the cantilever by an applied force causes the resistance of the doped region of the cantilever to vary about its nominal value, the sign and magnitude of deviation depending on the net change in stress experienced within the doped region. The change in resistance can be observed using a simple Wheatstone bridge or Ohm meter of sufficient resolution. Hence, this method of load detection has much in common with traditional strain-gage based load cells used as force transfer standards, albeit at a much smaller scale.

Cantilevers composed of piezoresistive material as described above are available commercially, and a photo of the type of cantilever purchased for use in our experiments is shown in Figure 2, accompanying a plot of the typical "open circuit" terminal voltage of the device. The force sensitivity is nominally $2 \text{ m}\Omega/\text{nN}$, with a low frequency force noise of 0.4 nN (integrated between 10 and 1000 Hz) according to the manufacturer's specification. In practice, low-frequency force measurements made with these sensors seldom resolve better than 10^{-8} N , since the signal below 10 Hz is often dominated by $1/f$ noise in the detection electronics or piezoresistor itself. The plot of

“open-circuit” voltage as a function of frequency shown in Figure 2 reveals this trend. In this plot we distinguish between regimes where the noise has a predominantly $1/f$ character and where the noise has a flat spectrum typical of Johnson noise. So far, the low-frequency noise content has limited the application of these devices in force microscopy, with the vast majority of systems still choosing optical detection. However, there is recent evidence that careful design can manage these noise problems and produce sensors with resolutions that meet or exceed that achieved using optical detection schemes, with demonstrated low frequency resolution at or below 3×10^{-12} N [12]. Finally, we observe that both the nominal resistance and stress sensitivity of a piezoresistor may vary with temperature [13]. This temperature dependence must be quantified if such devices are to be developed as force artifacts, though it will not be addressed here.

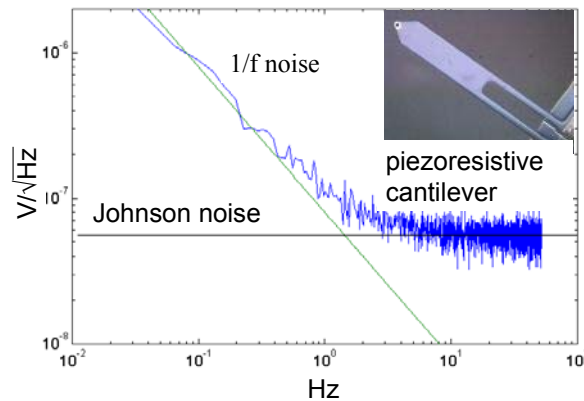


Figure 2. Typical noise signature of piezoresistive cantilever

Proof-of-concept Experiment

The electrostatic force balance was probed using a commercially available piezoresistive cantilever with a nominal resistance of $2 \text{ k}\Omega$, length of approximately $3 \times 10^{-4} \text{ m}$, 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 at 1 Volt 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 $3 \times 10^{-4} \text{ m}$ 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 3 and tabulated in Table 1.

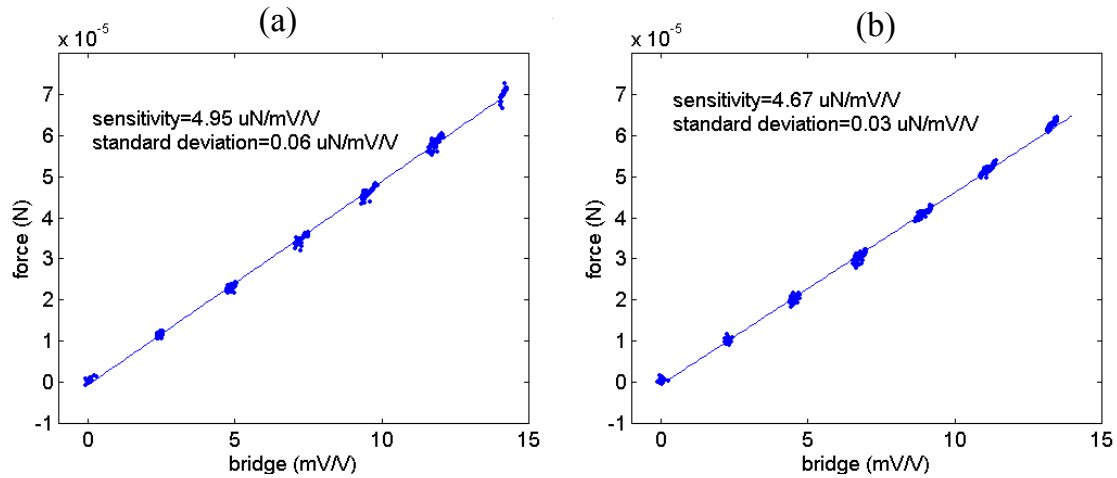


Figure 3. Null electrostatic force versus bridge output. Dots correspond to measured data. A single scan begins and ends at zero and is repeated 40 times in measurement (a) and 36 times in measurement (b). A sensitivity is determined for each scan from a least squares linear regression. The line corresponds to the mean value of sensitivities determined using a complete set of scans.

Table 1. Piezoresistive cantilever sensitivity

Experiment	Number of force scans	Mean sensitivity ($\mu\text{N}/\text{mV}/\text{V}$)	Standard deviation ($\mu\text{N}/\text{mV}/\text{V}$, %)
a	40	4.95	0.06 , 1
b	36	4.67	0.03 , 0.6

Discussion

The repeatability within a given experiment was encouraging. These results, taken alone, suggest the potential to realize a traceable calibration of a microforce artifact at or below the percent level. Comparison between the two experiments, however, shows reproducibility is far more limited, with a discrepancy between experiments on the order of 6%. A variety of factors may contribute to this discrepancy.

Interface uncertainty

The actual contact condition was unobserved. The optical microscope employed lacked the resolution to monitor the fine tip of the AFM cantilever and its contact with the polished glass cube that served as the interface to the balance. For instance, we cannot say if the tip slipped along the interface due to the load. Contaminants and adsorbed water layers present at the interface may also cause variation of the surface contact angle, and this will affect the degree to which the cantilever twists out of plane in response to the applied load.

Misalignment

We have observed that loading the electrostatic force balance with a probe can distort the alignment of the electrodes leading to a variation of the capacitance gradient, and a change in the calibration constant of the balance. Previous investigations indicate that this effect is small (on the order of parts in 10^4 [9]), and we expect this effect should have been fairly consistent between the two experiments. Nevertheless, the orientation of the cantilever load axis with respect to the balance is unknown, and it is possible that the tangential component of force due to misalignment differs in some regard between the two experiments.

Temperature effects

The bridge, piezoresistor, and balance are known to drift with temperature. Temperature near the experiment was monitored and observed to drift as a function of time in a nearly linear fashion from 22° C to 21.2° C. However, the mean temperature differed by only 0.1° C between the experiments and seems an unlikely source of discrepancy.

Zero drift

Finally, we note that the nominal load drifted monotonically on the order of 10×10^{-6} N in the time it took to complete a set of scans. Likewise, the sensitivities computed for each scan drift over the course of an experiment, revealing a slight dependence on preload. Unfortunately, the relative preload state of the cantilever from one experiment to the next was not accurately recorded, since the bridge was adjusted to zero after manual application of the initial preload in each case. It is always possible that an error was made in the initial setup, and that the two experiments were performed at significantly different absolute preloads of the cantilever.

Conclusions

We have demonstrated the calibration of a piezoresistive cantilever force sensor using a specially designed null-force balance that provides null forces that are calibrated in a fashion traceable to the SI. The sensitivity of the cantilever load cell was found to be repeatable at or below the percent level in a given configuration, demonstrating its potential utility as a microforce transfer standard. The experiment was reproduced, and a systematic discrepancy on the order of 6% of the measured cantilever sensitivity was observed. The discrepancy is not surprising, since much work remains to establish standard fixtures, methods, and procedures to ensure consistent contact conditions between load cell and balance. Even given the discrepancy, this calibration technique appears to be a factor of two more precise than most previous techniques, all aimed at determining the spring constant rather than a direct force calibration (see [5]). We submit that such an approach, based on a sensor with an integrated detector, is superior because it avoids the problems inherent in calibrating displacement metrology that is external to the load element.

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