Methods for transferring the SI unit of force from millinewtons to piconewtons

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ABSTRACT

The establishment of standards for small force measurement requires a link to an absolute measurement of force traceable to the international system of units (SI). To this end, a host of different means are being employed by the NIST small force measurement project to realize and transfer small forces between 5 millinewtons and 5 piconewtons. Realizations based on deadweights and electrostatic forces, as well as transfer artifacts based on the mechanical properties of single DNA molecules, will be discussed. The application of each of these approaches will also be discussed as a calibration method for different kinds of instruments requiring the measurement of small forces.

INTRODUCTION

Beginning with the Meter Convention in 1875, the international system of units (SI) has evolved into the primary basis for scientific measurement. Within the U.S., the National Institute of Standards and Technology (NIST) is charged with maintaining this coherent set of units. This includes the dissemination of mass and force using standards based on the international standard kilogram. More recently, the establishment of a small force metrology project within NIST has expanded the range of forces that can be measured within the SI toward ranges most useful for microelectromechanical systems (MEMS) and nanotechnology. Methods based on deadweights, and electrostatics are used at NIST for traceably measuring forces from millinewtons to hundreds of piconewtons, and each is appropriate for use in specific types of scientific experiments. Both of these traceable small force measurement methods will be briefly described in the context of their application to commonly used methods for small force measurement.

The newton is an example of a so-called derived unit in that it is composed of several other SI base units: the meter, kilogram, and second. As such there is more than one way in which a force can be realized as a physical measurement consistent within the definitions of the SI. By far the most common is the use of deadweights [1]. In this case, a force is realized through the application of Newtonian mechanics, so that the magnitude of the force is determined by multiplying a known mass value determined through (usually indirect) comparison with the international standard kilogram by the acceleration of local gravity determined, for example, by an absolute gravimeter measurement [2], as is illustrated in Figure 1. Any transfer of these forces (e.g., a calibration of a force transducer) must be done carefully so that the sensor is aligned with local gravitational acceleration. Deadweights can certainly be used for SI-traceable small force measurement as long as masses small enough for the desired calibration are available.

Currently, the smallest traceable masses available are 0.5 mg. Although prototype masses as small as 100 μg have been recently created, the uncertainty in the value of mass increases as deadweights get smaller [3].

Electrostatics provides an alternative to deadweight force. The attractive force between two oppositely charged conductors can be calculated as

$$F = \tfrac{1}{2}\,(dC/dz)\,V^2 \qquad (1)$$

where dC/dz is the gradient in capacitance along a linear coordinate, z, assuming capacitance gradients in other directions are negligible, and V is electrical potential. Using this fundamental measurement equation, an electrostatic force can be realized through a traceable measurement of voltage, capacitance, and distance [4]. Since each of these quantities can themselves be measured using methods based on fundamental physical invariants, the Josephson junction for voltage, the calculable capacitor for capacitance, and the speed of light for distance [5], this provides a somewhat less direct traceability path than deadweights. Easier scalability to small force is the principle advantage of the electrostatic approach, since a variable capacitor can be engineered to provide a desired capacitance gradient, and therefore a desired force. In addition, these electrical quantities can be traceably determined with high accuracy using commercially available equipment.

TRACEABLE SMALL FORCE USING DEADWEIGHTS

A small force measurement system calibrated using deadweights has been developed at NIST for measuring forces between 5 µN and 5000 µN. The system consists of a capacitive force transducer, and a carousel used to place deadweights on the transducer. By measuring the transducer's capacitance as a function of deadweight force and fitting a polynomial to the force-capacitance curve, a traceable calibration can be obtained in the force range above. If a separate calibration is performed for each decade in force, a combined standard uncertainty of 0.2 % can be obtained for the calibration for forces greater than 10 μN [6]. Representative data from this type of calibration are shown in Figure 2.

This traceable calibration can then be transferred to a force-measuring scientific instrument simply by placing the capacitive load cell in the measurement platform of interest. In the force range from 5 µN to 5000 µN, the main application is use in instrumented indentation machines. These materials characterization tools use a sharp diamond indenter tip to press against a surface of interest to determine near-surface mechanical properties, including Young's modulus and hardness through analysis of the force-displacement curves generated during the indentation [7]. The use of the deadweight-calibrated force cell ultimately allows calibration of the instrumented indenter's force measurement with sub-percent accuracy [6]. The relative uncertainty of the calibration increases substantially as force decreases, however.

TRACEABLE ELECTROSTATIC FORCES

Electrostatics provides an alternative to deadweights for traceable force calibration. The primary realization of small force at NIST is the electrostatic force balance (EFB). This instrument has been described previously in the literature [8], and will not be discussed further in this article. Recently, advances have been made that allow a similar approach to be used to directly calibrate small force measuring instruments in a traceable fashion using electrostatics. Realization of electrostatic force requires designing a capacitor with a well-defined capacitance gradient to constrain the direction of the force. One approach to defining a desired capacitance gradient is to exploit radial symmetry. The capacitance between a conductive sphere and flat plane provides such a geometry. The electrical force applied to the sphere is distributed equally about its surface, so that the net force between the sphere and flat acts perpendicular to the flat, and through the center of the sphere [9]. Provided a capacitance gradient can be measured between a sphere and flat, the application of a voltage between them will produce a force of known magnitude and direction.

Initially, this type of measurement was performed on an instrumented indentation force transducer [10]. The sphere and flat capacitor was formed using a stainless steel ball at the end of the indenter tip, and a polished semi-rigid coaxial cable as is shown in Figure 3. The position of the indenter tip was monitored at its opposite end using a laser interferometer to provide a traceable measurement of displacement. The measured capacitance gradient was nonlinear with distance, as expected for this geometry, so a piecewise linear fit was employed to determine the capacitance gradient over short intervals of displacement. Voltages were then applied between the sphere and flat to displace the center plate of the indentation transducer. The design of the transducer used in this work used a mobile plate attached to the indenter tip which was supported by an internal flexure spring. Since the force-displacement relationship was approximately linear over the range tested, this allowed the calculation of an indenter spring constant. This spring constant was also measured by applying deadweight forces to the indenter. A third measurement was also performed using the calibrated indentation transducer discussed in the previous section while measuring the indenter's internal spring deflection interferometrically to cross-check the electrostatic force determination with other traceable forces. The spring constant determined using electrostatics had a relative expanded uncertainty of 2.5 %, and agreed with the deadweight calibration methods within this uncertainty.

Another type of instrument commonly used to measure small forces is the atomic force microscope (AFM). The AFM monitors the flexing of a microfabricated cantilever spring to measure force. The spring constants of these sensors can also be calibrated in a procedure similar to that used for the instrumented indentation sensors. In this case, however, the dimensions of the system are much smaller; a conductive sphere as small as 30 µm is attached to the end of an AFM cantilever. Subsequently, the capacitance gradient between the sphere and a custom-fabricated micro-coaxial cable is measured, as shown in Figure 3. As a voltage, and hence electrostatic force, is applied, the deflection of the cantilever is monitored, providing a direct measurement of the cantilever spring constant and the force sensitivity of the AFM with a relative combined standard uncertainty of less than 5 % [11].

INTRINSIC FORCES

Currently, the traceable small force metrology described above is being used to develop intrinsic force standards. These are natural phenomena that have a well-defined force value associated with them. A traceable measurement of the force that causes the phenomenon will provide a reference force value for calibrations performed outside of NIST. An example of such a phenomenon is the overstretch transition of DNA [12, 13]. During single-molecule force measurements, a transition occurs at approximately 65 piconewtons in which the DNA molecule elongates significantly with very little extra applied force. The force-displacement curve of a single molecule of DNA measured with a force-calibrated AFM is shown in Figure 4. This particular molecule was amplified from a segment of the plasmid vector pBR322, and was measured in Tris/sodium chloride buffer. The transformation can be seen as a plateau in the curve. The methods developed for small force measurement at NIST are being used to calibrate the force at which this plateau occurs. The DNA itself then will become a force reference that will allow the calibration of a wide variety of force measuring instruments such as optical, magnetic, and dielectrophoretic tweezers.

APPLICATION

Currently, the NIST small force measurement project has enabled the development of traceable force measurement methods applicable to measurements from 5 mN to 200 pN, and is working to extend the range of traceable force measurements to even smaller forces. Within this force range, several different approaches can be used for force measurement. However, specific measurement techniques may be easier to implement with specific instruments. Table 1 shows a matrix of traceable force measurement techniques along with the force measuring instruments they have been applied to, and the operating range of the forces measured. Although not a focus of this paper, the use of reference cantilevers as transfer standards for small force measurement has been employed at NIST fairly extensively [8, 15-17]. The use of traceable force measurement is already required by standards for instrumented indentation [14], and is in development for AFM. In the future, standards for smaller forces can be developed as needed to meet the needs of emerging applications requiring accurate force measurement.

Table 1. Summary of traceable force measurement methods at NIST.

Traceable force measurement method	**Force range (N)**	**Instruments used**	**References**
Deadweights	$5 \times 10^{-3} – 5 \times 10^{-6}$	Instrumented indentation	6, 15
Electrostatics	$2 \times 10^{-4} – 2 \times 10^{-10}$	Instrumented indentation, AFM	8, 10, 11, 16
Reference cantilevers	$4 \times 10^{-6} – 1 \times 10^{-9}$	AFM, instrumented indentation	8, 15, 16, 17
Intrinsic forces	$1 \times 10^{-8} – 1 \times 10^{-12}$	AFM, optical tweezers	In development

CONCLUSION

The techniques available for small force measurement at NIST have been outlined. Although each of these measurements has a traceability path to the SI through established physical models, they are appropriate for different kinds of calibrations. The deadweight approach is suited to the 5 µN to 5000 µN forces of interest for instrumented indentation, and electrostatics accesses the force range from hundreds of piconewtons to 200 µN commonly used in AFM. Work is also progressing on the traceable calibration of intrinsic standards that will allow the calibration of a wide variety of other types of small force measurement instrumentation in the regime from nanonewtons to piconewtons.

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