NEW WAYS TO DETERMINE VERY SMALL MASSES AND FORCES

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ABSTRACT

The goal of this paper is to draw attention to mass determinations at nominal values well below 1 µg. Traceability to the International System of Units (SI) is challenging because mass standards having nominal values less than 1 mg are difficult to produce and thus not commonly available. We limit this burgeoning subject to only three examples: the calibration of atomic force microscopes with respect to calibrated dead-weights; the confirmation that a NEMS device has attogram resolution; and the direct determination of the gravitational mass of the neutron. We do not mention the important topic of magnetic resonance force microscopy because the applications to mass measurement are less direct than in the examples that have been chosen.

1. INTRODUCTION

In a real sense, the vast and growing field of nanometrology was already foreseen by Richard Feynman in his 1959 address “There’s plenty of room at the bottom” [1]. Mass metrology has not been immune to this revolution [2].

The smallest mass standard commonly used in metrology is 1 mg. The smallest resolution of mass comparators commonly used in mass metrology is 0.1 µg. However, recent advances in technology have created new fields and opportunities for mass measurements far smaller than these limits.

We start with the atomic force microscope (AFM) and its “calibration”. This device is based on a flexible cantilever, typically 100 µm long. The free end is attracted to (or repelled by) the object under study and the deflection is measured to a precision of picometres. The cantilever has a spring constant that is typically about 0.1 N/m. The challenge is to determine this constant, either by calibration or by other means. The problem is analogous to determining the sensitivity of a sensitive balance. In fact the calibration of AFMs has been discussed at recent meetings of IMEKO TC3 in the context of force measurements. The focus here will be on traceability to the kilogram of AFM calibrations.

The next type of device to be considered is that recently reported by Craighead and colleagues at Cornell University (USA) [3,4]. It is also a cantilever but, in this case, it is only 4 µm long and is used to determine the inertial mass of microscopic objects. The developers have demonstrated that their device is suitable for mass determinations in the attogram range (1 attogram = 10^{-18} g). The Cornell group have now configured their device to detect a change in mass due to the presence of a specific virus. A second group at Cornell has recently reported a different type of force/mass transducer based on the vibration of a carbon nanotube.

Finally, we recall W. Paul’s elegant determination of the gravitational mass of the neutron [5]. Paul was able to create a magnetic “spring” within a neutron storage ring and determine how far this spring stretches due to the added weight of a neutron. The sensitivity of the measurement was 0.1 yg (1 yoctogram = 10^{-24} g). His result is consistent with the accepted value of the inertial mass of the neutron. Although this work was done about 25 years ago, it does not seem to be well known to mass metrologists.

2. CANTILEVER SYSTEMS

The AFM and the most well-known Craighead devices are cantilevers. In its operational mode, the AFM works by deflection. If a horizontal cantilever is fixed at one end, the free end will deflect in the vertical direction when a vertical force *F* is applied. If the deflection, Δz is small, then the cantilever can be described as a simple spring with constant *k* :

$$F = k\Delta z \tag{1}$$

Assuming that Δz can be accurately measured, calibration of the device consists of determining the spring constant *k*. There is in fact no requirement that the cantilever be horizontal. However, if it is horizontal, we have the possibility that *F* can be produced by a deadweight.

Cantilevers can also operate in a dynamic mode whereby the free end of the cantilever is made to oscillate at its resonant frequency f_0, giving us a second equation:

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{m}} \tag{2}$$

where *m* is the mass at the tip of a "massless" cantilever. If the cantilever cannot be considered to be massless, then a change in mass of Δm will lead to a new resonant frequency f_1 so that

$$\Delta m = \frac{k}{(2\pi)^2}\left[\frac{1}{f_1^2} - \frac{1}{f_0^2}\right] \ . \tag{3}$$

Note that the same spring constant *k* appears in (1), (2) and (3).

3. CALIBRATION OF AFMs BY MEANS OF DEADWEIGHTS

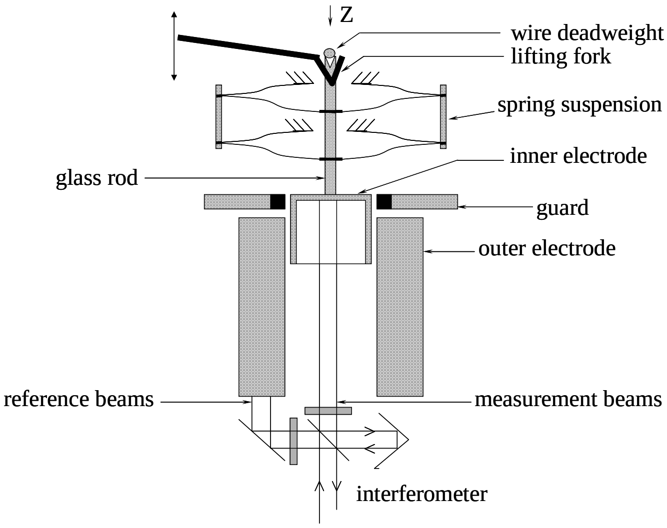
In his review of the calibration of AFMs [6], Sader identifies three main strategies for determining *k*:

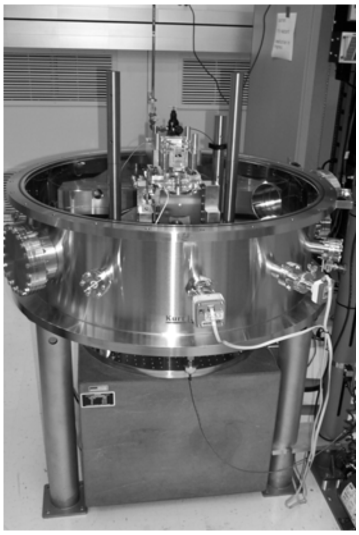
1) Theoretical calculation. For instance, a finite element analysis can determine *k* based on the dimensions of the cantilever and the physical properties of the material(s) from which it is made.
2) Static deflection. A known force can be applied to the tip of the cantilever and *k* can be determined from (1).
3) Dynamic vibrational response. The value of *k* can be determined from (2) or (3).

In this brief paper, we take as our only example the second strategy and, in particular, the most recent results from Pratt and his colleagues at NIST [7,8]. Their apparatus is shown in Fig. 1. From the schematic diagram, it can be seen that a force along the vertical axis can be applied in two ways. The first is by adding a deadweight of either 20 µN or 200 µN at the top of the vertical column. The force is determined from the calibrated mass of the deadweight and the local acceleration of gravity. The second method is based on a capacitive transducer whose inner and outer electrodes are shown in Fig. 1, can be calibrated from first principles. Recall that the electrostatic potential energy *U* that is stored in a capacitor is given by

$$U = \frac{1}{2}CV^2 \tag{4}$$

Figure 1: Schematic diagram of NIST apparatus (left) and photograph (right). The diameter of the inner electrode is 15 mm. (Provided by the Small Force Metrology Laboratory, NIST)





where V is the voltage across the capacitor plates. The vertical force of this system is given by the gradient of U in the Z direction and, finally, a change in force is given by

$$\Delta F = \frac{1}{2}\frac{\partial C}{\partial Z}\left(V_1^2 - V_2^2\right) \quad . \tag{5}$$

The derivative of C must be determined in a separate experiment. The authors argue plausibly that, if the electrostatic force deduced from (5) agrees with the deadweights, then this indicates that the transducer is working properly. This approach is analagous to earlier attempts to determine the SI volt in terms of the base units [9]. The transducer is then used to calibrate AFMs. After considerable effort the NIST apparatus has demonstrated agreement with deadweights to better than 0.1 percent when operating with amplitude-modulated a.c. voltage. At d.c. one sees the effect of surface or “patch” fields on the capacitor electrodes. Patch fields of voltage V_s modify the voltage term in (5) so that it becomes ($V_1^2 - V_2^2 + V_s(V_1 - V_2)$). In [8], the voltage V_s is shown to be of order 0.1 V. The effect of V_s is eliminated at d.c. by reversing the polarity of the two electrodes and averaging the the two results. The NIST group plans to use the capacitive transducer to extrapolate to much smaller forces (< 1 nN) than can be realized with deadweights.

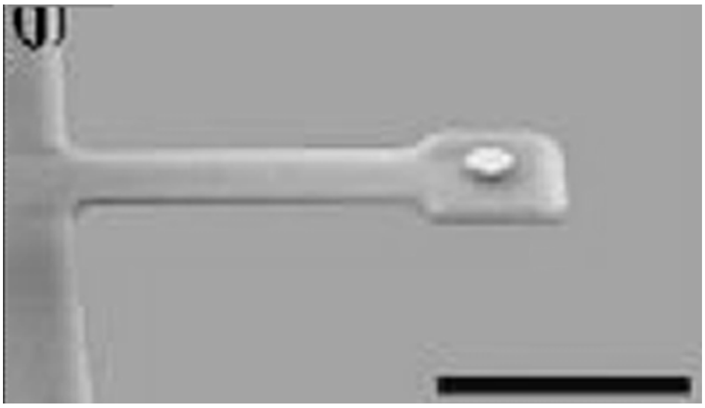
For a complete picture, [6] should be consulted along with subsequent developments in this rapidly-advancing area.

4. OSCILLATING CANTILEVERS

A general introduction to this subject has been given by Lavrik and Datskos [10,11]. These authors point out that, since the weight of objects of molecular size is too small to be measured, one determines the inertial mass as, for example, in (3). Of course the gravitational mass (used to calculate weight) and inertial mass are known to be identical for the purposes of mass metrology. According to [11], a general rule is that the smallest detectable mass at room temperature is roughly 10^{-6} times the mass of the cantilever. This limit is determined by thermal noise in the oscillation frequency of the cantilever. Therefore the cantilever dimensions must be reduced to achieve greater precision. However, reduction in size below optical wavelengths makes detection of the oscillation frequency difficult.

It is arguable that the performance of oscillating cantilevers has been revolutionized by the Craighead Group and Cornell University (USA) [3]. Their devices are referred to as NEMS (nanoelectromechanical systems). In a paper published in 2004 [4], Craighead and his colleagues reported a cantilever system with a resolution of better than 1 ag. The cantilevers, 4 µm long, 500 nm wide and 160 nm thick are fabricated from a silicon/silicon nitride wafer (Figure 2). The paddle at the free end is 1 µm by 1 µm. The resonant frequency of an oscillator ranges from 1 MHz to 15 MHz.

Figure 2 : Cantilever capable of detecting changes in mass of 1 ag. (Craighead Group/Cornell University(USA))



How does one determine the sensitivity of such a device? Note the gold dot fused to the free end of the cantilever. First, the resonant frequency of the system shown in Fig. 2 was measured. Then Craighead and his colleagues then exposed the cantilever to a sulphur-based organic compound, thiolate. Thiolate is known to form a self-organized monolayer on gold surfaces. A calculation showed that, in one instance, this contamination increased the mass at the end of the cantilever by only 6 ag, which was easily detected. Cantilevers having gold dots with diameters from 50 nm to 400 nm were studied. A careful calculation estimated the sensitivity of the device to be about 0.4 ag. It was also possible to measure the effect of the gold dots themselves on the resonant frequency of the cantilevers.

A different group at Cornell has made a similarly sensitive device based on the “guitar-string-like” oscillations of a nanotube clamped at both ends [12].

The Craighead Group intends to extend their work to the zeptogram range (1 zg = 10^{-21} g), in which case the devices could be configured to identify DNA and other biological molecules [3,4].

A recent paper estimates the ultimate limits to NEMs devices [13].

5. GRAVITATIONAL MASS OF A NEUTRON

The remark that masses of molecular size and smaller are too small to be weighed [11] has at least one exception. At the end of his Nobel lecture, Wolfgang Paul describes how the mass of a neutron, m_n , may be determined by weighing [5]. The preceding section of his lecture describes a storage ring for confining neutrons and he shows that the vertical force produced by this device can be described by (1).

The force confining the neutrons in the vertical (Z) direction is created by the sextupole magnetic field produced an electrical current I flowing in six toroidal rings (Fig. 3). The vertical force on any neutron in the beam is given by

$$F = \mu \frac{\partial B}{\partial Z} \tag{6}$$

where μ is the magnetic moment of the neutron and B is the magnetic induction in the vertical direction. In the storage ring,

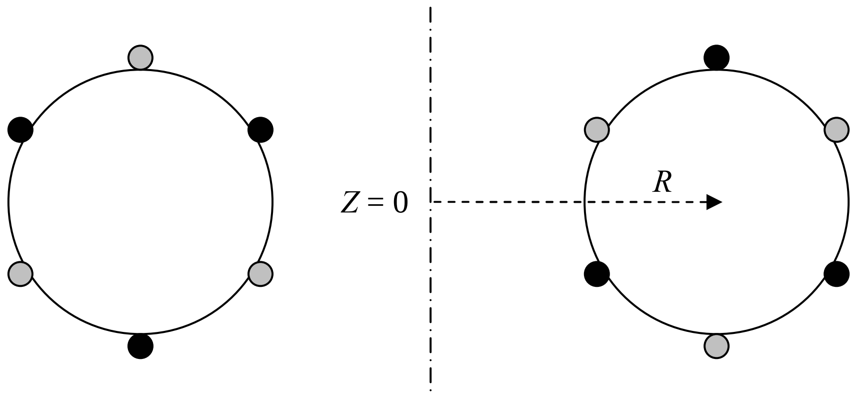
$$\frac{\partial B}{\partial Z} \propto IZ \,.$$

Therefore,

$$\Delta F = \left(C\mu I\right) \cdot \Delta Z \tag{7}$$

where C is calculable from the geometry of the storage ring. It is now evident that the force equation looks like (1), where the “spring” constant is proportional to I.

Figure 3 : The storage ring is a toroid of radius R. A sextupole magnetic field is produced when a current I flows in the six toroidal wires shown in the figure. In the cross-section shown here, grey represents current flowing into the page and black represents the current flowing out of the page. The vertical line is the axis of cylindrical symmetry.



It is remarkable that the spring constant is so weak that the weight of the neutron lengthens the “spring” by a measurable amount. Setting $\Delta F = m_n g$, we can use (7) to predict that gradient of 1.7 T/m is required to balance the weight $m_n g$ of a neutron.

In the absence of the earth’s gravitational field, the neutron beam would be centred about Z=0. For the apparatus described by Paul, the neutron beam was displaced downward by 4.8 mm when I=50 A. We would therefore expect that the downward displacement would be only 1.2 mm at I=200 A and that was indeed the case. Based on a fit to the data of displacement versus current, Paul and his colleagues inferred that the gravitational mass of the neutron is

$$m_n = (1.63 \pm 0.06) \times 10^{-24}\ \text{g}.$$

This agrees with the inertial mass of the neutron to within 3%. The inertial mass is now known to a relative uncertainty of 1.7×10^{-7} [14] but it is remarkable that the gravitational mass can be measured at all. As Paul notes, the measurement of the gravitational mass is only possible because electrostatic forces are nonexistent in this case.

REFERENCES

[1] Feynman R.P., "There's plenty of room at the bottom", 1959. Available on internet at http://www.its.caltech.edu/~feynman/

[2] Roukes M., "Plenty of room indeed", *Scientific American*, Sept. 2001, pp. 48-57.

[3] The Craighead Research Group: http://www.hgc.cornell.edu

[4] Ilic R., Craighead H.G., Krylov S., Senartne W., Ober C., Neuzil P, "Attogram detection using nanoelectomechanical oscillators", *J. Appl. Phys.*, **95**, 2004, pp. 3694-3703.

[5] Paul W., "Electromagnetic traps for charged and neutral particles", *Rev. Mod. Phys.*, **62**, 1990, pp. 531-540.

[6] Sader J.E., "Calibration of Atomic Force Microscope Cantilevers", *Encyclopedia of Surface and Colloid Science,* P. Somassundaran, Ed., Marcel Dekker (2002), pp. 846-856.

[7] Pratt J.R., Smith D.T., Newell D.B., Kramar J.A., Whitenton E., "Progress toward Système International d'Unités traceable force metrology for nanomechanics", *J. Mater. Res.*, **19**, 2004, pp. 366-379.

[8] Pratt J.R., Newell D.B., Kramar J.A., Seugling R.M., "Realizing and disseminating the SI micronewton with the next generation NIST electrostatic force balance", *Proc. 19th Annual Meeting of American Society for Precision Engineering*, Orlando FL (USA), 24-29 October 2004, to be published.

[9] Sienknecht V., Funck T., "Realization of the SI volt by means of a voltage balance", *Metrologia*, **22,** 1986, pp. 209-212.

[10] Lavrik N.V., Datskos, P.G., "Femtogram mass detection using photothermally actuated nanomechanical resonators", *Appl. Phys. Lett.*, **82**, 2003, pp. 2697-2699.

[11] Lavrik N.V., Datskos P.G., "Nanomechanics weighs in", *Physics World*, April 2004, p. 19.

[12] Sazonova, V., Yaish Y., Ustünel H., Roundy D., Arias T.A., McEuen P.L., "A tunable carbon nanotube electromechanical oscillator", *Nature*, **431**, 2004, pp. 284-287.

[13] Ekinci K.L, Yang Y.T., Roukes M.L., "Ultimate limits to inertial mass sensing based upon nanoelectromechanical systems", *J. Appl. Phys.*, **95**, 2004, pp. 2682-2689.

[14] Mohr, P.J., Taylor B.N., *2002 CODATA Recommended Values*, http://physics.nist.gov/cuu/constants/index.html

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