

## Direct thermal noise calibration of colloidal probe cantilevers



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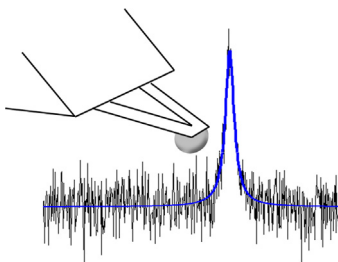
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### HIGHLIGHTS

- The order of “calibration” and “particle attachment” is irrelevant.
- The equipartition theorem is applicable to colloidal probes as well.
- The material and the size of the microparticles used made no difference.
- The cantilevers spring constant, shape or dimensions showed no influence.
- Advice on preparation to increase reliability and reproducibility is suggested.

### GRAPHICAL ABSTRACT



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### ABSTRACT

We show that the spring constant of colloidal probes made by attaching spherical microparticles to atomic force microscope cantilevers can be directly calibrated by the thermal noise method. We show that the energy equipartition theorem used for cantilevers, which is at the origin of the thermal noise method, is applicable to colloidal probes as well. In some close-to-ideal cases the spring constants agree within 5% with the spring constants of the bare cantilevers, given that some technical precautions during preparation of the colloidal probes are taken care of. We found no major influence of materials or sizes of the microparticles used, of absolute values of the used cantilevers' spring constants, shape, or dimensions.

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## 1. Introduction

The invention of the atomic force microscope (AFM) by Binnig, Quate and Gerber in 1986 [1] in the series of Scanning Probe Microscopes allowed not only to image surfaces with “atomic” resolution, but also to measure intra- and intermolecular forces

with unprecedented sensitivity. The force sensitivity comes from using micro machined cantilevers as force sensors with a certain “stiffness” in combination with an optical detection scheme (light lever technique in the majority of cases) capable to measure sub-nanometer displacements of the cantilever sensors. In order to determine the force acting on the cantilever from the measured cantilever's deflection, one needs to know the spring constant. The resulting force can only be known precisely, when this spring constant is known as accurate as possible. Otherwise, the in principal very precise measurement of the cantilevers deflection does not

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lead to precise forces due to uncertainty of the spring constant value. In 1991 the colloidal probe (CP) technique was introduced by Ducker et al. and Butt [2,3]. Here, well defined and mostly spherical colloidal particles replaced the nanometric sharp tips located at the end of the cantilevers. This allowed not only measurements of surface forces between particles and flat surfaces or in between particles [4], but also easy interpretation of the results in terms of known models in order to determine mechanical properties of micron-sized particles [5]. One approach is the so-called Derjaguin approximation [6]. The Derjaguin approximation is valid if the decay length of the intermolecular forces is much smaller than the curvature of the surfaces. This condition is satisfied for CP measurements with particles with radii of curvature in the order of few micrometers, but not for AFM measurements with tips with radii of curvature of few nanometers. Further advantages of the CP approach are the well-defined geometry of the larger contact region due to the spherical shape of the particles, the larger choice of available materials, and the possibility of chemically modifying the surface of the particles. Especially in biology or polymer science particles with modified surface chemistry are increasingly used, since the surface functionalization of spherical particles is of higher quality and of longer lifetime compared to sharp cantilever tips, especially in the contact area region. The commonly used particles range in diameter from below one micrometer to several tens of micrometers and are either glued or sintered [7] to rectangular or triangular tipless cantilevers. Topical reviews on the use of CP technique are found in, e.g. [8,9], and on other methods for fixing particles on cantilevers in, e.g. [10–12]. By now, colloidal probes are also commercially available from a limited number of suppliers.

Quite a number of calibration procedures have been put forward in the past years for characterizing normal spring constants of cantilevers. Reviews on calibration procedures can be found, e.g. in [13,14], while topical papers are, e.g. [15–27]. We limit ourselves to a brief recap of the three most common methods that are applicable to all types of cantilevers, without the need of knowing a priori any of their geometrical or mechanical properties.

The first, published by Cleveland et al. [17] and thus commonly called *Cleveland method*, is based on measuring the shift of the resonance frequency of a cantilever upon loading with different masses. Plotting added mass versus inverse of the resonance frequency squared yields a line, whose slope corresponds to the normal spring constant. The second, published by Gibson et al. and Torii et al. [21,22] is based on loading a “standard” pre-calibrated cantilever with the “unknown” cantilever. By knowing the loading force (N) and measuring the resulting bending or deflection (m), the spring constant can be calculated. This method is commonly referred to as *reference cantilever method* or *reference spring method*. The third, called *thermal noise method*, applies the energy equipartition theorem to relate the Brownian motion of the cantilever to its spring constant. It relies on the acquisition of the cantilever’s thermal power spectrum (square of the fluctuations of its vibration amplitude as a function of frequency). It has been proposed in its original form by Hutter and Bechhoefer [28] and was later slightly modified by Butt and Jaschke [19] to account for energy contributions of higher cantilever oscillation modes.

When using colloidal probes, one common approach is to calibrate the cantilevers by one of the existing calibration procedures [15–20,22–27,29] before attaching a particle at their free end. Alternatively, colloidal probes are calibrated by the *reference cantilever method* after a particle has been attached. When colloidal probes are used in liquids, also other in situ calibration routines have been proposed. They are based on the evaluation of the hydrodynamic force exerted by the liquid on the colloidal probe when it is dragged with a known speed across a liquid or pushed toward a rigid wall [30–33]. Chung et al. considered the possibility to apply the thermal noise method to calibrate spring constants of cantilevers even after

the attachment of particles and compared it with other methods [34]. However, the question whether the attached particle influences the result of this calibration method was not addressed.

The aim of this short communication is to show, that colloidal probes can be directly calibrated by the quick and easy to implement thermal noise method, with measured spring constants being similar to those of the bare cantilevers.

The thermal noise method has the big advantage that no cantilever parameters, like geometrical dimensions or material constants, are involved in the calibration. So, deviations from ideal conditions of shape or material properties – that can never be excluded, do not have any influence. Cook et al. found good agreement in between the thermal noise method and the Sader method for cantilevers with spring constants that vary nearly over two orders of magnitude. They argue, because the methods derive from distinct physics, both could be suggested as practical standards for the field [35].

## 2. Principles of the thermal noise method

The *thermal noise method* applies the equipartition theorem to cantilevers. There is, however, no reason against applying these energy considerations to colloidal probes as well. It states that if a cantilever is in thermal equilibrium, every independent quadratic term of its total energy has a mean value equal to  $1/2k_B T$ , where  $k_B$  is the Boltzmann constant and  $T$  the absolute temperature. The cantilever is considered as an ideal spring having a potential energy equal to  $1/2k\langle z^2 \rangle$ , with  $k$  the spring constant and  $\langle z^2 \rangle$  the mean square cantilever displacement in any vibration mode. This elastic energy is equated to the kinetic energy of the cantilever, i.e. its thermal vibration energy [28]

$$\frac{1}{2}k_B T = \frac{1}{2}k\langle z^2 \rangle \quad (1)$$

This equation holds to a first approximation whether the cantilever is V-shaped or rectangular, made of a single material or a multilayer structure, tipless or a colloidal probe. Following this equation one could easily calculate the spring constant of a bare cantilever or a colloidal probe writing

$$k = \frac{k_B T}{\langle z^2 \rangle} \quad (2)$$

Commercial software, like the one we used, calculates the mean square displacement as integral of the square of the first (or fundamental) mode of the amplitude power spectrum. Kinetic energy, however, is stored in all vibration modes and not only in the first one. Eq. (2) thus overestimates the spring constant and needs to be slightly modified [19]

$$k = \frac{12K_B T}{\alpha_1^4 \langle z_1^2 \rangle} \quad (3)$$

where  $\alpha_1 = 1.875$  is the parameter quantifying the amount of energy stored in the first vibration mode and  $\langle z_1^2 \rangle$  is the mean square cantilever displacement in the first vibration mode.

Another effect to take into consideration is that the optical lever technique does not detect cantilever deflections. The optical lever technique measures the inclination of the cantilever, i.e. an angle, and not the deflection, i.e. a distance. Deflection and inclination are proportional, and so a further correction factor has to be added to Eq. (3) [8,36–38]

$$k = \beta \frac{k_B T}{\langle z_*^2 \rangle} \quad (4)$$

where  $\langle z_*^2 \rangle$  is the mean square effective deflection and  $\beta = 0.817$ . The effective deflection is the deflection we read from the instrument after determining the sensitivity from the contact – or constant

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