APPLICATION NOTE 3

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Evaluation of AFM cantilever spring constant

INTRODUCTION

Atomic Force Microscopy (AFM) is a widely used measurement technique across multiple disciplines and makes use of the interaction between a sharp scanning probe and the sample surface. The probe can act as a highly sensitive force sensor, enabling measurement of tip-sample interaction forces with high spatial resolution. Here, the interaction force is a key parameter for controlling and interpreting the measurement results, so the ability to quantify this force is of critical importance. This can be readily achieved by measuring the deflection of the probe and applying Hooke's law, provided the cantilever spring constant is known. Several different methods have been proposed and used for evaluating the cantilever spring constant, which are summarised in this Application Note. A comparison and evaluation of these methods based on practical and technical considerations is offered.

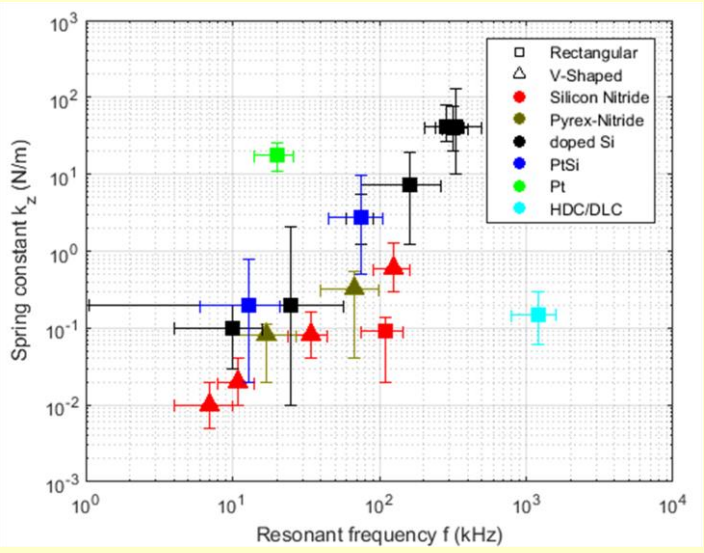

Figure 1 compares the quoted values of spring constants and resonant frequencies for AFM probes used by members of the SPM2.0 consortium. This figure illustrates both the wide range of values for AFM probe characteristics used for various applications, and also highlights the broad uncertainties attached to these parameters according to the manufacturers' nominal specifications.

Figure 1. Bimodal AM-FM scheme. Two excitation signals are combined in order to excite the cantilever in its first and second mode. By scanning the surface, the deflection signal is recorded and processed to finally obtain the bimodal observables. From those observables, a theoretical framework is applied and the nanomechanical parameters are reconstructed.

METHODS OF SPRING CONSTANT CALIBRATION

Methods of cantilever spring constant calibration can be broadly categorised into 3 families: dimensional methods, static methods, and dynamic methods.

Dimensional Methods

Dimensional methods use a mathematical model to describe the geometry of the cantilever to evaluate its spring constant. This approach requires prior knowledge of intrinsic material properties.

For simple geometries, such as rectangular cantilevers, mathematical expressions can be applied (requiring some simplifying assumptions), as illustrated in Figure 2. Similar expressions with varying levels of rigour can be used for more complex cantilever geometries, and to account for coatings of different materials. [1]

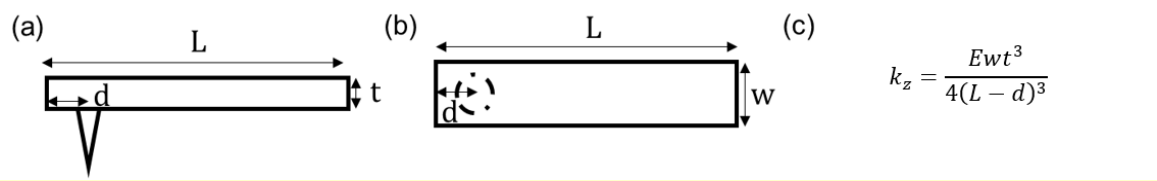

Figure 1. Rectangular cantilever geometries with associated dimensional variables in (a) cross-sectional and (b) top view. (c) Equation for calculating spring constant k_z based on cantilever dimensions and Young's modulus.

The accuracy of these methods is limited by the fidelity of the model in terms of the geometry, dimensions, and material properties. The spring constant is very sensitive to the thickness of the cantilever, which is challenging to measure accurately. The uncertainty associated with dimensional approaches has been estimated at 10-25 % but varies on a case-by-case basis. [2]

Static Methods

Static methods of force constant evaluation measure the static force constant experimentally by applying controlled forces to the cantilever and measuring the resulting displacement. [3]

A conceptually simple approach is to add reference masses to the cantilever as a load, as illustrated in Figure 3. In practice, this is not straightforward and dependent upon the accuracy of the reference mass and accurate placement on the cantilever. A more widely adopted technique uses a nanoindenting unit as the force actuator, which can be calibrated to give well-quantified loads. Uncertainties better than 10 % can be readily achieved and are primarily dependent on the calibration of the nanoindenter. However, it is a laborious measurement and not suitable for very stiff or very compliant cantilevers.

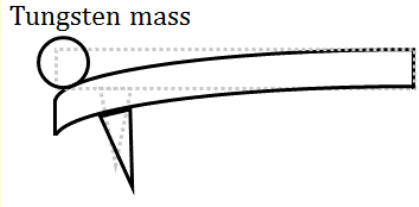

Figure 2. Illustration of cantilever deflection measurement resulting from placement of reference mass.

An alternative strategy is the use of reference cantilevers, which are commercially available and provide a more user-friendly means of cantilever calibration. [4] A limitation is that the uncertainty is large if the cantilever under test has a very different spring constant from the reference cantilever. The measurement also involves direct contact between the two cantilevers and can damage the probe.

Dynamic Methods

Dynamic methods provide a more indirect measurement of the cantilever spring constant using theoretical models to relate experimentally determined dynamic cantilever properties to the cantilever stiffness. Dynamic methods rely on measuring the time-dependent cantilever displacement, which is available in most commercial AFM systems and hence simple to implement. The 'added mass method' measures the shift in resonant frequency of a cantilever arising from the addition of a known mass. [5] The uncertainty achievable using this measurement is limited by the need to precisely position and attach the added mass. The Sader method is based on modelling the hydrodynamics of the surrounding medium. [6] For a simple cantilever geometry, the hydrodynamic function $\Gamma(\omega)$ can be calculated, leading to the following expression for the cantilever spring constant

$$k_z = 0.1906\rho_f W^2 L Q_f \Gamma_i(\omega_f)\omega_f^2.$$

Here, ρ_f is the fluid density, W and L are the cantilever width and length respectively, Q_f is the resonance quality factor of the cantilever immersed in fluid, ω_f the resonance frequency in fluid and $\Gamma_i(\omega_f)$ the imaginary component of the hydrodynamic function. The resonant frequency and quality factor are obtained by fitting a simple harmonic oscillator model to the resonant spectrum of the cantilever. This method applies only to rectangular cantilevers and requires knowledge of the surrounding fluid properties (typically air or vacuum). Extension of the Sader method to arbitrary cantilever geometries has been demonstrated but requires experimental determination of the hydrodynamic function. Uncertainty in spring constant calibration down to ± 6 % has been achieved using state-of-the-art dimensional measurements but under typical conditions an uncertainty of 15 % to 20 % is expected [2].

A further group of methods are the 'thermal methods', which use energetic considerations rather than mechanical ones. [7] The underlying concept is that a harmonic oscillator in thermal equilibrium with its surroundings vibrates due to thermal energy. Thermal energy is be most effectively converted into cantilever oscillation at its eigenfrequencies. When considering only the fundamental frequency, the thermal vibration amplitude is integrated in the frequency domain to give the area of the power spectral density, PSD, which leads to the expression:

$$k_z = \frac{k_b T}{PSD}$$

where k_b is the Boltzmann constant and T the absolute temperature. When the higher order modes are ignored, errors of up to 30 % can be introduced. Consideration of all modes is required for a more accurate measurement, with further consideration of how the real deflection of the cantilever relates to the apparent deflection given by the readout of the specific AFM instrument. Butt and Jaschke formulated these corrections, resulting in achievable uncertainties of around 15 %. [8]

A further extension of these methods is the Global Calibration Initiative, which aims to standardise AFM based force measurements around the world. [9] Users report the model of cantilever used and their results obtained with the thermal method, and the spring constant value is refined with the hybrid Sader method. By using a wider base of experimental data the uncertainty in the spring constant is reduced but it is unclear how to quantify this from a metrological point of view.

EVALUATION OF METHODS

AFM cantilever calibration lacks a 'gold standard' which allows one to reliably assess the achievable accuracy of each calibration method against a recognised and verified method. [10] Hence, when uncertainties are quantified in literature, they are obtained by evaluating the achieved uncertainty using the discussed method against either another method or some form of numerical simulation.

Whilst the accuracy of the methods is an important factor in choosing which method to employ for cantilever spring constant calibration, consultation of the SPM2.0 consortium found that the practical considerations of convenience and simplicity must also be considered. For this reason, the dynamic methods (thermal method and Sader method) are particularly attractive in spite of the higher accuracies that might be achieved using static methods.
From a metrological perspective, the static methods are attractive as they do not intrinsically rely on assumptions made on the working cantilever and thus are not subject to the accuracy related challenges that were previously discussed. Hence, static methods are the most suitable candidates when high precision quantitative data is required, or for metrological calibration with traceability to the SI. While the nanoindenter method is unpractical and relies on additional equipment, the reference spring method is moderately user friendly. However, at this time the commercial availability of traceable reference springs is limited, and it is understood that when using reference springs that are not traceable this method loses its main advantage and falls behind due to reduced practicality. Further information on the standardised application of these methods is provided in ISO 11775:2015. [1]

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