Force constant determination of AFM cantilevers with calibrated thermal tune method

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1. Introduction

Atomic force microscopy (AFM) is quite often used for force measurement applications. The principle of these force measurements is based on measuring the deflection of a cantilever (x) and calculating the force (F), which is applied via a tip at the end of the cantilever to the investigated sample, by the Hooke's law, F= - k * x , with the force constant k. Whereas the cantilever deflection can be measured with very high accuracy in an AFM the force constant has to be known before the force experiment. Therefore, for a precise force measurement the force constant of each cantilever has to be determined. There are many methods to determine the force constant but in general they all had a lack of accuracy. In aspects of speed, non-destruction and reproducibility the thermal tune method according to Hutter & Bechhoefer [1] has many advantages. Therefore, we used this method and calibrated it with a self-made force standard chip that was certified by the German national metrology institute.

2. Theory

The basic principle of the thermal tune method is the equipartition theorem describing the relationship between spring constant and thermal energy [1].

$$1 \quad 2 \; k \; z^2 \; = 1 \quad 2 \; k_B T$$

with the spring constant k, the mean square displacement of the thermal cantilever oscillation z^2 , the Boltzmann constant k_B and the absolute temperature *T*. For determination of the mean square displacement a power spectral density (PSD) analysis of the measured cantilever deflection signal has to be performed and then the area beneath the resonance peak has to be integrated [2]. Due to the fact that the oscillation energy of a cantilever is split into different bending modes the formula has to be corrected by a factor for the fundamental resonance bending mode [3].

$$k = 0.971 \; k_B T \quad z^2$$

This correction factor is valid only for a rectangular cantilever, for triangular cantilever a different correction factor has to be applied.

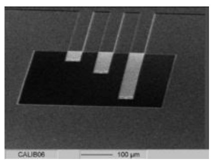
3. Experimental

A SIOS Nano Vibration Analyzer (*Fig.1*) was used for the measurement of the thermal cantilever oscillation. This laser vibrometer has a stabilized HeNe-laser and uses the interferometric principle to detect very small amplitudes (amplitude of the thermal oscillation is about $5 \cdot 10^{-12}$m to $50 \cdot 10^{-12}$m). Due to the interferometer detection principle a calibration of the deflection detection is not necessary and, in contrast to the optical lever method used in AFM's, no angle error occurs. The system measures the amplitude spectrum of cantilevers at room temperature. Then this spectrum is fed into a LabVIEW™ program and converted into the power spectral density (PSD). Force constant, resonance frequency and quality factor are determined by fitting a simple harmonic oscillator function to the resonance peak curve in the PSD.

Fig.1: Laser-vibrometer set-up for thermal tune mesurement (left), NanoWorld AFM probe (PNP DB) with silicon nitride cantilever for reproducibility test (right).

The fabricated silicon force standard has three cantilevers whereas only the long and the middle cantilever were used for the calibration because the stiffness of the shortest one is too high for the thermal tune method (*Fig.2*). At the free end of the cantilevers reference marks are located to ensure a defined measurement position.

Cantilever	long	middle
length	450 µm	200 µm
width	50 µm	50 µm
thickness	2.15 µm	2.15 µm
nominal force constant	0.23 N/m	2.6 N/m



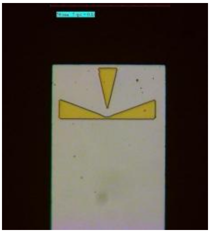


Fig.2: Dimensions of the cantilever (left) - force calibration chip with cantilevers (mid) – reference mark on the cantilever(right).

To be sure about the absolute value of the force constants of cantilevers the reference chip has been sent to the Physikalisch-Technische Bundesanstalt (PTB) for a certified measurement of the force constants. PTB measured force-distance curves by pushing the cantilever with a stylus at the reference mark with forces ranging from 20 to 250 nN for the long cantilever and from 30 to 650 nN for the middle cantilever. Forces and travelling of the stylus were measured simultaneously. The small forces were controlled accurately with a compensation balance. Afterwards, we applied the thermal tune method to the cantilevers of the force standard and deviations from the measured force constants measured by PTB were used to calculate a correction factor for the thermal tune analysis software. Additionally the force constants of the reference cantilevers were also determined by the dimensional-method (calculation from geometric dimensions and material properties) and the Sader-method (calculation from measured frequency and quality factor) [4].

4. Results

The force constants determined by the PTB measurements were 0.209 N/m for the long and 1.888 N/m for the short cantilever. The extended uncertainty in measurement U95 [5] are 0.014 N/m and 0.155 N/m, respectively. In the comparison with the other methods (*Fig.3*) the Sader value is in both cases out of the error ranges, and drift far away for the stiffer cantilever. The dimensional method (with the highest estimated error due to uncertainties in the cantilever thickness measurement) result in a good agreement with the PTB value for the long cantilever but not for middle cantilever. The thermal tune show a slightly lower force constants than the certified PTB values for both cantilever. The error bars of the thermal tune and PTB values are overlapping which indicates that the thermal tune method is good. Nevertheless, the agreement could be improved by introducing a mean calibration factor (1.15) for the two cantilevers which is multiplied with the measured thermal tune value (called "thermal tune calibrated" in *Fig.3* and *4*). Numerous of thermal tune measurements with negligible deviation between determined and certified force constant confirm that this technique eliminates systematic measurement errors. The reproducibility of the thermal tune method is shown in *Fig.4* with the stiffness measurement at a NanoWorld nitrid cantilever. The standard deviation was 4.1% for 20 measurements with new positioning of the laser at the cantilever for each measurement and 2% without new positioning. Long term investigations over a period of one year (*Fig.5*) showed values inside the errors ranges of the certified force standard, only. This demonstrates the validity and the reproducibility of the measurement which is now applicable for all AFM cantilevers in the same stiffness range.

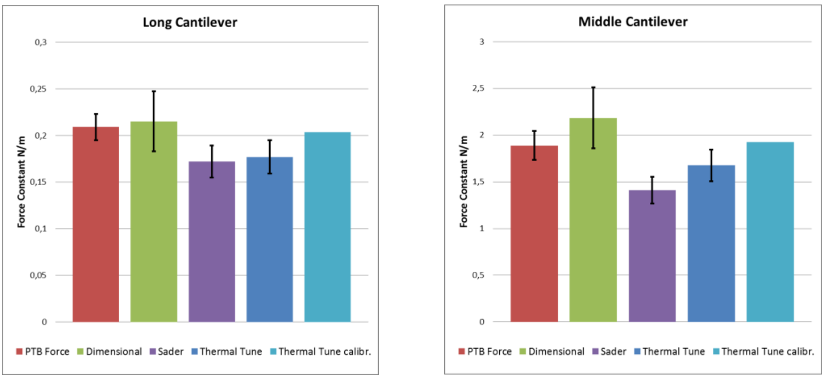


Fig.3: Comparision of the force constants measured with different methods

Method	Long [N/m]	Middle [N/m]	Estimated error
PTB	0.209	1,888	
Dimensional	0.215	2.184	15%
Sader	0,172	1,41	10%
Thermal Tune	0.177	1,675	10%
Thermal Tune calibr.	0,204	1,926	

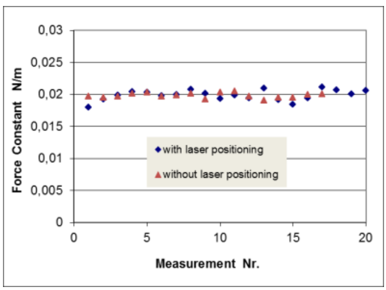


Fig.4: force constants measured with different methods (left) and reproducibility of the thermal tune method, measured at NanoWorld PNP DB Afm probe (k=0,02 N/m)

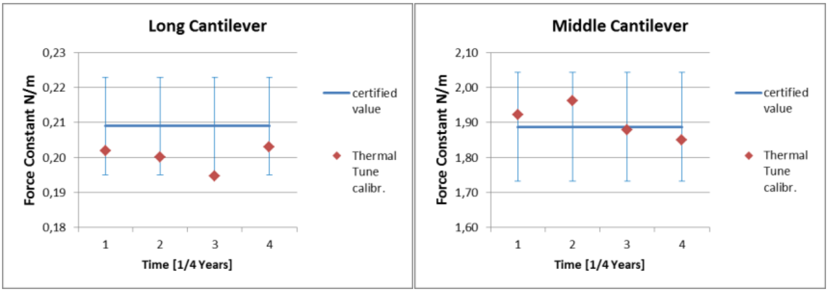


Fig. 5: Calibrated thermal tune force constant measurement in long term experiment

5. Conclusion

Determination of the spring constants of AFM cantilevers are extensively discussed in literature including comparisons of different methods. Here, we present a traceable technique combining speed, convenience and non-destruction of the thermal tune method for reliable determination of force constants of micro cantilevers.

6. References:

[1] J. L. Hutter and J. Bechhoefer, „Calibration of atomic force microscope tips“, Review of Scientific Instruments 64 (1993): 1868.

[2] B. Ohler, "Cantilever spring constant calibration using laser Doppler vibrometry" Review of Scientific Instruments 78 (2007): 63701.

[3] H. Butt and M. Jaschke, „Calculation of thermal noise in atomic force microscopy“, Nanotechnology 6 (1995): 1.

[4] J. E. Sader et al., "Calibration of rectangular atomic force microscope cantilever", Review of Scientific Instruments 70 (1999):3967.

[5] "Guide to the expression of Uncertainty in Measurement", GUM:1995, www.iso.org