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NATIONAL COMPARISON OF SPRING CONSTANT MEASUREMENTS OF ATOMIC FORCE MICROSCOPE CANTILEVERS

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Abstract: This paper illustrates a comparison of spring constant measurements between Technische Universität Ilmenau (TU Ilmenau) and Physikalisch-Technische Bundesanstalt (PTB). For the traceable comparison, an atomic force microscope (AFM) cantilever with a nominal stiffness of 40 N/m was chosen as the transfer artefact. The determined spring constants differ less than 1 %. The measurement uncertainty was ≤ 4 % (k = 2) and hence conforms to the state of the art of AFM cantilever spring constant measurements. The main common uncertainty contribution of TU Ilmenau and PTB is due to the adjustment of the cantilever towards the calibration force.

Keywords: spring constant, force, deformation, cantilever, micro force sensors

1. INTRODUCTION

The application of special micro force sensors for the measurement of forces in the subnano- and nanonewton range is strongly growing in different fields of science, such as biology, biophysics, medical- or materials science: Measurements of force interactions between living cells [1-6] or nanomechanical investigations of cancer cells [7, 8] are impressive examples of those applications. A further instance is nanoindentation, where surface and layer properties, such as Young's modulus or hardness, are determined in the nanonewton scale [9-12].
Beside other sensor principles, AFM cantilevers are often used for those force measurements. If the cantilever spring constant c_{Canti} is calibrated, a force F can be determined based on a measured deflection z, cf. equation (1).
Furthermore, cantilevers equipped with strain gauges wired to a Wheatstone bridge are available. In this case, forces can be detected by measuring the bridge voltage.

$$F = c_{Canti} \cdot z \tag{1}$$

Due to the growing application of micro force sensors for force measurements in the nano newton range, the national metrology institutes NIST, NPL, PTB and KRISS have developed force facilities to supply a traceable calibration of those kind of force sensors [13-19]. A first international comparison of micro force sensor calibrations was carried out by NIST, NPL PTB and KRISS [19].

The calibration of the spring constant is performed by pushing the cantilever or the micro force sensor on a reference force sensor. Thereby, the acting force F and the deformation z are measured simultaneously. Based on equation (1) the spring constant c_{Canti} is determined.
The force facilities of PTB and KRISS apply commercial electromagnetic force compensated load cells (EMFC) for force measurement and piezo actuators to push the cantilever on the load cell. The deflection z is assumed to be the displacement that is set by the piezo. Currently a measurement uncertainty of approximately 2 % (k = 2) for c_{Canti} can be achieved [18].
NIST developed their own electrostatic force compensated load cell whereas the cantilever to be calibrated can be probed (deflected) with the load cell itself by moving the "weighing pan" of the load cell with the internal electrostatic actuator [15, 16]. The highest force resolution is achieved by the NIST system. A repeatability of the measured force of 0.7 nN was proven [16]. As disadvantages of the NIST system high nonlinearities as well as creeping and hysteresis are reported [15, 16, 19].
Beside the work of the national metrology institutes, a nano newton force facility was developed at TU Ilmenau. It is used for research and development in small force metrology at the Institute of Process Measurement and Sensor Technology. Furthermore, it is applied for investigations in the field of microelectromechanical systems such as AFM cantilevers at the TU Ilmenau.
The TU Ilmenau force facility works based on the principle described above and uses a commercial EMFC load cell with a self-developed digital control loop [20, 21]. Additional to the systems of PTB and KRISS, an Interferometer is applied for a traceable deflection measurement. Former studies showed that the TU Ilmenau facility is suitable for traceable calibrations of AFM cantilevers with uncertainties of spring constants in the range of 2 - 3% (k = 2) [20, 21].
The aim of the spring constant measurements presented in this paper is the comparison of the TU Ilmenau results with those of the PTB and thus a verification of the performance of the TU Ilmenau system. We are aware that an AFM cantilever with its simple bending beam geometry is not the best available transfer artefact for this kind of comparison. Nevertheless, we used an AFM cantilever to link our results to the results of the international comparison between the NMIs [19]. AFM Cantilevers were used here as transfer artefacts as well. The deviation between the spring constants determined by KRISS, NIST and PTB was 3.2 % (2 σ) for artefact #5 (c_{Canti} ~ 78 N/m).

2. TRANSFER ARTEFACT (AFM CANTILEVER)

As transfer artefact for the spring constant measurements a Nanosensors™ PPP-NCLR50 non-contact and tapping mode AFM cantilever was used. Its nominal length, width and thickness is listed with 225 µm, 38 µm and 7 µm respectively. From these dimensions a nominal spring constant of 48 N/m results. Due to the fabrication tolerances the specified range of the spring constant is indicated with 21-98 N/m.

For a better handling, the cantilever was glued on an aluminium carrier. The carrier can be fixed in the force facilities by two M 2.5 screws.

3. FORCE FACILITIES OF TU ILMENAU AND PTB

The PTB force facility is described in detail elsewhere [19]. A Mettler SAG 245 compensation balance is used for the precise force measurements. A stylus with a 200 µm ruby sphere is fixed on the weighing pan. It can be replaced by a conical-shaped diamond stylus, where the tip of the cone was grinded off to a flat with 200 µm diameter. The cantilever to calibrate is fixed on a piezo actuator (PI Pifoc with 100 µm travel) and can be pushed on the stylus by the piezo. The deflection of the piezo is measured with the internal capacitive deflection sensor. When the cantilever probes the stylus, this deflection is assumed to be the deformation of the cantilever. Simultaneously the force is measured.

The TU Ilmenau facility has been published earlier [20, 21]. It mainly consists of a Sartorius WZ2P compensation balance [22], a SIOS SP TR 2000 Triple beam interferometer [23] and a piezo actuator (PI Hera P-621.1CL with 100 µm travel [24]). The balance is driven with a self-developed digital control loop. It offers the possibility to move (deflect) the weighing pan with the balance internal voice coil and probe the fixed cantilever with the balance itself. Thus, the deflection is generated and the force is measured with the balance simultaneously. The physical weighing pan is removed from the monolithic balance mechanism. Instead of the weighing pan a stylus with a 300 µm ruby sphere (load button) is fixed on the movable part of the balance to probe the cantilever. The piezo actuator is held in a static position. This procedure is called "Measurement Mode 1" (MM1). The deflection of the weighing pan and possible deviations of the piezo actuator null position are detected with the interferometer. The difference of both deflections is assumed to be the deflection of the cantilever. Deviations of the piezo actuator null position can be due to thermal expansion or elastic deformations due to the applied force of the measurement loop (including the frame).

Additionally to MM1, the TU Ilmenau facility can be operated in „Measurement Mode 2“ (MM2) which conforms to the measurement principle of PTB and KRISS. Here, the weighing pan (the load button) is kept in a static position and the cantilever is moved by the piezo actuator. The deflection of the cantilever is assumed to be the difference of the deviations of the null-deflection of the weighing pan and the movement of the piezo. Deviations of the null-deflection of the weighing pan can be due to thermal expansion or elastic deformations as well.

PTB and TU Ilmenau adjust the cantilever with respect to the load button by the help of a microscope camera with micrometer resolution.

4. MEASUREMENTS

The cantilever was first measured in January 2015 at TU Ilmenau with MM1 and MM2 probing it with the ruby sphere. Then it was sent to PTB and measured in September 2015 by probing it with the ruby sphere and the diamond flat. Afterwards it was brought back to TU Ilmenau and measured there again in June 2016 with MM1 and MM2.

4.1. Measurement procedure

TU Ilmenau MM2 and PTB

The cantilever is pushed on the load button (weighing pan) and is deflected by a stepwise increase of the piezo actuators deflection. After the maximum deflection was reached and measured, the deflection is decreased in the same way. Each step is held for 15 s (TU Ilmenau). The force and the deformation of each step is determined as the mean value of the signals between 10 .. 15 s after each deformation change. The cantilever spring constant is determined by linear regression of the force-deflection curves for increasing and decreasing deflections. In the first measurement series TU Ilmenau applied four steps with 700 nm step-size resulting in a maximum force of ~ 118 µN. In the second measurement series eight 160 nm steps with a resulting maximum measurement force of ~54 µN were chosen.

PTB uses a waiting time of 6 s and then took one balance value and applied 30 steps with a maximum force of 250 µN. This loading and unloading procedure was repeated N = 4 times (first measurement series at TU Ilmenau) or N = 140 times respectively (at PTB and second measurement series at TU Ilmenau). Afterwards the mean values of the spring constants were computed for the increasing (loading) as well as the decreasing (unloading) curves.

TU Ilmenau MM1

In measurement mode 1 (MM1) the cantilever is probed by setting defined deflections of the weighing pan. This is done by moving the mechanical lever mechanism of the balance with the internal electromechanical actuator. Due to the internal spring constant c_{EMFC} of the mechanical mechanism, the sum c_{Sum} of this spring constant and the spring constant of the cantilever c_{Canti} is measured when the cantilever is probed.

$$c_{Sum} = c_{Canti} + c_{EMFC} \quad (2)$$

To determine the spring constant c_{Canti}, c_{EMFC} is measured before and after each probing of the cantilever and subtracted from c_{Sum}. This procedure has already been described in detail [20, 21].

Table 1: Parameters and results of the spring constant measurements

	TU Ilmenau first measurement series		PTB		TU Ilmenau second measurement series	
	MM1	MM2	sphere	flat	MM1	MM2
			Measurement parameters			
N (Number of load cycles)	4	4	10	150	140	150
Deflection steps	4	4	30	30	8	8
mean temperature in °C	22.37	22.38	22.46	22,30	23,47	23,37
mean humidity in % r.H.	28.52	28.70	49.9	41.1	38,61	36,63
F_{max} in µN	118	118	250	250	54	54
			Spring constant *c* ± standard uncertainty Typ A in N/m			
$c_{loading}$ in N/m	42,344 ± 0,030	42,431 ± 0,036	42,6 ± 0.048	41,976 ± 0.0011	42,6097 ± 0,0016	42,3961 ± 0,0016
$c_{unloading}$ in N/m	42,429 ± 0,035	42,669 ± 0,039	41,9 ± 0.048	41,974 ± 0.0011	42,6302 ± 0,0016	42,4010 ± 0,0015
c_{mean} in N/m	42,387 ± 0,020	42,550 ± 0,019	42,3 ± 0.048	41,975 ± 0.0011	42,6200 ± 0,0015	42,3985 ± 0,0013

Table 2: Uncertainty budget for the spring constants, given in relative values

	TU Ilmenau		**PTB**		**TU Ilmenau**	
Uncertainty source	MM1	MM2	sphere	flat	MM1	MM2
Repeatability c_{mean}	$4{,}8 \cdot 10^{-4}$	$4{,}8 \cdot 10^{-4}$	$1.14 \cdot 10^{-3}$	$2{,}6 \cdot 10^{-5}$	$3{,}6 \cdot 10^{-5}$	$3{,}1 \cdot 10^{-5}$
Force measurement	$1.8 \cdot 10^{-4}$	$1.8 \cdot 10^{-4}$	$1.0 \cdot 10^{-2}$	$1.0 \cdot 10^{-2}$	$1.8 \cdot 10^{-4}$	$1.8 \cdot 10^{-4}$
Deflection measurement	$1.6 \cdot 10^{-6}$	$2.3 \cdot 10^{-4}$	$1.75 \cdot 10^{-2}$	$1.75 \cdot 10^{-2}$	$1.6 \cdot 10^{-6}$	$2.3 \cdot 10^{-4}$
Nonlinearity of *c*	$3.3 \cdot 10^{-3}$	$3.7 \cdot 10^{-3}$	$1.0 \cdot 10^{-3}$	$1.0 \cdot 10^{-3}$	$2.1 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$
Stiffness of measurement loop	-	-	$2.5 \cdot 10^{-4}$	$2.5 \cdot 10^{-4}$	-	-
Angular alignment of the cantilever	$6.0 \cdot 10^{-4}$	$6.0 \cdot 10^{-4}$	$6.0 \cdot 10^{-4}$	$6.0 \cdot 10^{-4}$	$6.0 \cdot 10^{-4}$	$6.0 \cdot 10^{-4}$
Lateral alignment of the cantilever	$5.2 \cdot 10^{-3}$	$5.2 \cdot 10^{-3}$	$6.6 \cdot 10^{-3}$	-	$5.2 \cdot 10^{-3}$	$5.2 \cdot 10^{-3}$
Combined rel. uncertainty	$6.3 \cdot 10^{-3}$	$6.5 \cdot 10^{-3}$	$21.3 \cdot 10^{-3}$	$20.1 \cdot 10^{-3}$	$5.3 \cdot 10^{-3}$	$5.3 \cdot 10^{-2}$
c_{mean} with combinded uncertainty in N/m, $k = 2$	**42,39 ± 0.53**	**42,55 ± 0.55**	**42,3 ± 1.8**	**41.98 ± 1.70**	**42,62 ± 0.45**	**42,40 ± 0.45**

As in MM2, c_{Sum} and c_{EMFC} are measured with four (1. measurement series) or eight (2. measurement series) deformation steps each, which are applied for 15 s. The measurements are carried out for loading and unloading again. As in MM2, the measurements where repeated $N = 4$ times (first measurement series at TU Ilmenau) up to $N = 140$ times (second measurement series at TU Ilmenau) as well.

4.2. Measurement results

The results of the comparison measurements between PTB and TU Ilmenau are listed in Table 1 and are depicted in Figure 1. Each displayed spring constant conforms to the mean value of the spring constants determined for loading and unloading.

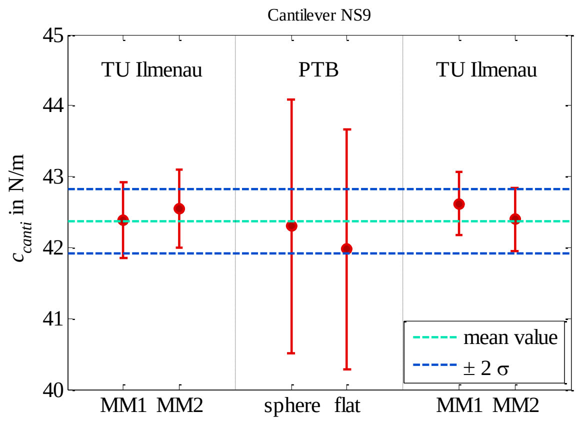

Fig. 1: Determined mean spring constant of the cantilever (red), mean value of all spring constants (cyan) and the twofold standard deviation (2 σ) of those six mean values (blue), ($k = 2$)

4.3 Measurement uncertainty budget

The uncertainty budget is listed in Table 2. The uncertainty contributions are given as relative values. Based on equation (1), we assume that the relative contributions of force F and deflection measurement z are combined as square root of the sums:

$$\frac{u_c}{c} = \sqrt{\left(\frac{\Delta F}{F}\right)^2 + \left(\frac{\Delta z}{z}\right)^2} \quad (3)$$

The contributions are discussed in the following.

Repeatability

Typ A uncertainty of the mean value of N measurements.

Force measurement

TU Ilmenau

Uncertainty of the calibration factor of the balance. The balance was calibrated using E0 weights of 5 mg and 10 mg. Due to their tolerance (rectangular distribution) we achieve an uncertainty of the calibration of $1.8 \cdot 10^{-4}$.

PTB

Uncertainty of the calibration factor of the balance. The balance was calibrated using E1 weights of 5 mg and 20 mg. Due to their tolerance we achieve an uncertainty of the calibration of $1.0 \cdot 10^{-3}$. Nevertheless, for the uncertainty budget the PTB assumes the force measurement with $1.0 \cdot 10^{-2}$ very conservatively.

Deflection measurement

TU Ilmenau

The deflection of the cantilever is assumed to be the difference of the weighing pan deflection and the piezo deflection (see above) which are both measured with the interferometer.

MM1

In MM1 the piezo deflection is quasi zero. Therefore the uncertainty of the piezo deflection measurement can be neglected.

The deflection of the weighing pan (the load button) is measured without an abbe offset (the stylus axis/ load button and the laser beam are congruent). Furthermore we were not able to resolve a significant tilting of the “weighing pan” in former experiments when the weighing pan was deflected with the internal voice coil (angle was < 0.1 µrad). Consequently, first order abbe errors are neglected in MM1.

The possible angle between the direction of the weighing pan deflection and the laser beam is estimated to be $\gamma < 0.1°$ (rectangle distribution). This results in an second order abbe error of $< 1.6 \cdot 10^{-6}$. The small angle is due to the low fabrication tolerances of the monolithic balance mechanism. The mirror which reflects the laser beam is glued directly on the moving part of the monolithic balance. The interferometer itself would only tolerate an angle of < 0.02° between the mirror and the laser beam [25].

Because contributions of the stabilized laser frequency and the refractive index of air are at least one magnitude smaller, we assume $1.6 \cdot 10^{-6}$ to be the uncertainty of the deflection measurement in MM1.

TU MM2

In MM2 the weighing pan (load button) deflection is quasi zero. Therefore its measurement uncertainty can be neglected.

The piezo deflection is measured with an abbe offset of 12 mm. The tilting of the piezo when traveling through its 100 µm range is given in the datasheet with ± 3 µrad (rectangular distribution). This results in an first order abbe error of 36 nm (rectangular distribution) or a relative uncertainty of $2.1 \cdot 10^{-4}$.

The angle between the laser beam and the direction of the piezo deflection must be estimated with higher tolerances. This is due to the tolerances of the adjustment of the piezo actuators direction and the adjustment of the mirror which is mounted on the piezo. We estimate a value of $\gamma < 1°$ which leads to a uncertainty contribution of the second order abbe error of $< 0.9 \cdot 10^{-4}$.

Combining both abbe error contributions leads to a uncertainty of the deflection measurement in case of MM2 of $2.3 \cdot 10^{-4}$.

To publish a smaller uncertainty of the deflection measurement (second order abbe error) in MM2 we have to determine (and adjust) the angle γ more precisely.

PTB

The uncertainty contribution of the capacitive deflection measurement is estimated by a standard measurement uncertainty of $1.75 \cdot 10^{-3}$.

Nonlinearity

The nonlinearity is the deviation of the measured force-deflection curve from its linear fit.

TU Ilmenau

As value for the nonlinearity the confidence interval of the linear regression coefficient is determined.

PTB

The average nonlinearity was ± 0.25 µN at maximum forces of 250 µN.

Stiffness of the measurement loop

The balance, the piezo and the measurement frame do have a finite stiffness. When the cantilever probes the load button (weighing pan) the resulting force also deforms the balance as well as the piezo and the measurement frame where the balance and the piezo is mounted.

TU Ilmenau

As explained above, the deformation (and deflection) of the balance and the piezo is measured with the interferometer. This length measurement is done with respect to a fixed point in the measurement frame. Therefor those deformations are considered when the deflection of the cantilever is determined. Hence, the deformation of the balance, the frame and the piezo does not lead to significant uncertainty contributions for the TU Ilmenau facility.

PTB

The stiffness of the complete measurement setup has been measured by probing against a stiff silicon sample of known Young's modulus. It amounts to 166 kN/m. Based on this stiffness a uncertainty contribution to the spring constant measurements of $2.5 \cdot 10^{-4}$ was derived.

Angular alignment of the cantilever

An angular misalignment $\Delta\alpha$ from the intended 90° between the beam axis of the cantilever and the introduced calibration force leads to a deviation of the bending force introduced in the cantilever. The relative deviation can be described with:

$$\frac{u_{F\alpha}}{F} = 1 - \cos(\Delta\alpha) \qquad (4)$$

PTB and TU Ilmenau estimates the maximum angular misalignment to be $\Delta\alpha < 2°$, leading to an uncertainty contribution of $6.1 \cdot 10^{-3}$.

In earlier work others [18, 19] published the hypothesis that friction and angle depended lateral forces cause a deviation of the measured spring constants. The authors later also used this assumption [21] for uncertainty determination. The lateral force was assumed to be proportional to the static (lateral) friction force $\mu s \cdot F_N$, which is the maximum force before the cantilever tip starts sliding on the load button. Based on further theoretical investigations and measurements we come to the opinion, that the true lateral forces are much smaller. Therefore we do not consider friction and angle depended lateral forces in our uncertainty determination. Nevertheless the influence must be further investigated in future work.

Lateral alignment of the cantilever

A lateral misalignment of the cantilever towards the highest point of the spherical load button generates lateral forces and leads to an uncertainty contribution as well.

TU Ilmenau

With an uncertainty of the lateral alignment of 5 µm (rectangular distribution) we estimate this uncertainty contribution with $0.52 \cdot 10^{-2}$ [21] when the cantilever is probed with the 300 µm ruby sphere. This assumption bases on measured values. However, the geometrical model describing this deviation used friction depended lateral forces as well and needs to be improved in future work.

PTB

With a lateral alignment uncertainty of 5 µm (rectangular distribution) we estimate this uncertainty contribution with $6.6 \cdot 10^{-3}$ when the cantilever is probed with the 200 µm ruby sphere. This assumption bases on measured values. When probing with the diamond flat this contribution is assumed to be zero.

5. DISCUSSION

The spring constants c_{mean} determined with different measurements modes (TU Ilmenau) and different probing styli (PTB) are in a good agreement within 1 % (2 σ). As described above, the international comparison achieved deviations of 3.2 % (2 σ) between KRISS, NIST and PTB measurements for a cantilever with a spring constant in the same magnitude (78 N/m). This verifies the performance of the spring constant calibration device developed by TU Ilmenau.

Due to the traceable deflection measurement with the triple beam interferometer, and the smaller uncertainty of the calibration force the measurement uncertainty achieved with the TU Ilmenau facility is lower than the uncertainty achieved with the PTB setup.

The performance of the comparison is limited by the transfer artefact itself. Due to the simple bending beam shape, the measured cantilever spring constant is highly sensitive to the adjustment of the cantilever towards the load button (probing point). Furthermore, the simple beam shape of a cantilever shows a nonlinear deformation behavior.

6. OUTLOOK

A future goal is the reduction of the uncertainty of spring constant measurements.

This requires the investigation of the angular- and lateral alignment contributions. Furthermore an interferometric and traceable deflection measurement within the PTB facility as well as an improved calibration of the balance would significantly reduce the uncertainty. A minimization of the Abbe offset would lead to a lower deflection measurement uncertainty of the TU Ilmenau measurements.

Spring constant measurements at lower forces and deflections could lead to lower nonlinearity and would also reduce the deterioration of the cantilever tip.

For future comparisons other transfer artefacts than AFM cantilevers might be used. Artefacts with a lower sensitivity to lateral forces, the probing point and a lower nonlinearity as they are described in [26, 27] are potential candidates.

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