

DETERMINATION OF AFM-CANTILEVER SPRING CONSTANTS USING THE TU ILMENAU FORCE DISPLACEMENT MEASUREMENT DEVICE

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Abstract – This paper discusses the determination of spring constants of AFM-cantilevers using a force displacement measurement device based on electromagnetic force compensated (EMFC) load cells.

The EMFC load cell was modified to utilize it as a combined displacement and force measurement device enabling the possibility to determine the spring constant of AFM-cantilevers with one sensor without the need of an additional displacement setting stage.

The paper focuses on the determination of spring constants and the determination of the measurement uncertainty, which is down to 2.75 %.

Keywords: spring constant, AFM-cantilever, force measurement, EMFC load cell

1. INTRODUCTION

The atomic force microscopy (AFM) can deliver data at the atomic-scale and is capable to probe the interaction forces between surfaces gained. However, reliable quantitative measurements are difficult to obtain because a very precise knowledge of the cantilever spring constant is necessary.

Traditionally the AFM-cantilevers are made from Silicon or Silicon-compounds and the displacement is detected by laser-based or piezoresistive readout. Standard sized cantilevers are usually too stiff to image at high speeds with high resolution. Herein, a high sensitivity to small forces is required resulting in an aggressive cantilever-size scaling [1].

Mass fabrication of low-cost cantilevers results in wide tolerances of the force- and spring constant of cantilevers. It is difficult to control the thickness of the cantilever using standard MEMS technology. The non-uniformities in geometry among the fabricated cantilevers have to be taken into account [2].

The determination of AFM-cantilevers spring constants is an upcoming field of investigation in research and industry. There are several measurement setups and methods described in literature [5-17]. The currently lowest measurement uncertainty of approximately 2% is achieved by using a static force calibration described in [15]. This method uses a load cell for force measurement and a displacement setting stage. The spring constant c is then calculated using the measured force F and the displacement z :

$$c = \frac{F}{z} \quad (1)$$

The measurement system developed at TU Ilmenau uses a direct determination of the spring constant by setting a displacement and measuring the acting force. The measurement system was described in [3] and is based on a preliminary system described in [4].

The TU Ilmenau measurement system uses an EMFC load cell with a modified control loop. This modified load cell is used to set the displacement of its load button and measure the acting force simultaneously. This is called measurement mode 1. A second measurement mode uses an additional piezoelectric nanostage to set the displacement separately; the EMFC load cell is then used for force measurement only. This so called measurement mode 2 is used to compare the measurements with the state of the art methods described in literature.

2. MEASUREMENT SETUP

The measurement system consists of several components including the EMFC load cell (1), a triple beam interferometer (2) for traceable displacement measurements at different positions and the piezoelectric nanostage (6). The alignment of the AFM-cantilever (4) towards the load button (5) is done with an x-y-z- stage (3) and controlled with a long distance microscope camera (7). Everything is linked using a metrological frame (8). (Fig. 1)

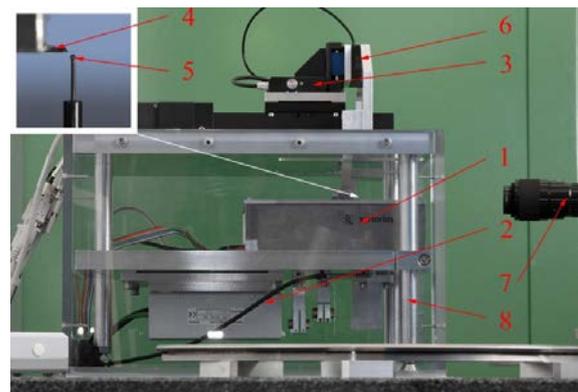


Fig. 1: Measurement system (1 – EMFC load cell, 2 – interferometer, 3 – x-y-z-stage, 4 – cantilever, 5 – load button, 6 – piezoelectric nanostage, 7 – camera, 8 – metrological frame)

The EMFC load cell is a commercial system WZ2P from Sartorius Lab Instruments [18] it uses modified electronics and control loop [4]. This control loop bases on an Agilent multimeter for the measurement of the load cell levers position, a MATLAB code for the calculation of the

compensation current which is supplied by a HP digital current source to the voice coil.

The force measurement range is 20 mN. The load button is a ruby sphere with a diameter of 300 μm and is attached to the weighing pan of the load cell.

The interferometer (SIOS SP-TR [19]) provides a traceable displacement measurement with a resolution of 80 pm.

The cantilever to test is glued to a stiff aluminium carrier. This carrier is clamped on the connector which is fixed on the piezo nanostage.

2.1. Measurement mode 1

In measurement mode 1 the EMFC load cell is used for displacement setting and force measuring simultaneously. Therefore the control loop of the EMFC load cell is modified to set different positions of its conversion lever. Consequently the position of the load button can be set in a range of $\pm 13 \mu\text{m}$. A force is needed to set the position of the load button as the flexure hinges have certain stiffness, resulting in the spring constant c_{EMFC} of the EMFC load cell.

This method is similar to the electrostatic force compensation described in [17].

To determine the spring constant of an AFM-cantilever, the cantilever is held in a static position and the load button is moved. As the load button gets in contact with the cantilever, an additional force acts on the load cell. The determined spring constant c_{meas} is then the sum of the spring constant of the cantilever c_{canti} and of the EMFC load cell c_{EMFC} .

Additionally the EMFC load cell has a static spring constant c_{stat} in static force measurement mode. This spring constant results from the deformation of the conversion lever as well as the parallel beams of the load cell. In our setup this deformation is included within the measurement of the load button's position z_i . Therefore this influence is suppressed in our measurement. This is an advantage compared to [5, 6, 8, 11 and 15].

The stiffness of the cantilevers holder combined with the positioning stage and the piezoelectric nanostage result in the spring constant c_{hold} . All acting spring constants in measurement mode 1 are shown in Fig. 2.

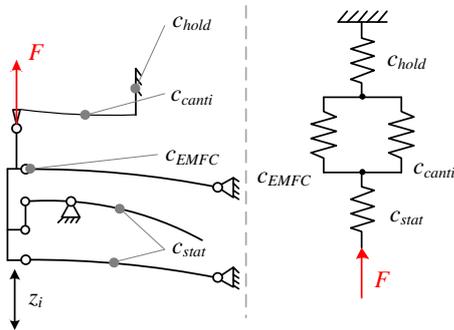


Fig. 2: acting spring constants in measurement mode 1

2.2. Measurement mode 2

The second measurement mode is related to the state of the art as described in [5, 6, 8, 11 and 15]. The EMFC load cell is used for force measurement only, the load button is in a quasi-steady position. As the cantilever touches the load button, the force is measured by the load cell. Additionally

the deformation of the load cell due to its non-infinite static stiffness c_{stat} is measured with the interferometer. Like the state of the art devices, the movement of the cantilever is measured with the internal capacitive displacement measurement of the piezoelectric nanostage PI 621.1 [20].

The deformation of the cantilever is assumed to be the displacement of the nanostage z_{Piezo} compensated by the deformation of the load cell z_i (2).

$$z = z_{Piezo} - z_i \quad (2)$$

As explained above this is an advantage compared to [5, 6, 8, 11 and 15], because the uncertainty contribution of the static stiffness of the load cell c_{stat} is suppressed. The spring constant of the cantilever c_{canti} is determined directly by measuring the force and the displacement. Additionally the spring constant of the cantilevers holder and the piezoelectric nanostage c_{hold} was calibrated prior to the measurement (Fig. 3).

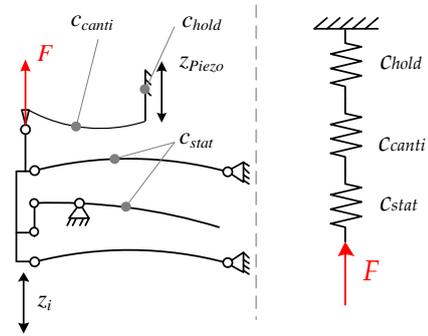


Fig. 3: acting spring constants in measurement mode 2

3. MEASUREMENTS

3.1. Force measurement

The force measurement is performed using a commercial WZ2P EMFC load cell from Sartorius but with modified electronics and controller as described in [4]. The primary output of the load cell is the compensation current, which was calibrated to the force using class E0 weights (1/2 tolerance E1, according OIML-R111 [21]).

The repeatability of the force measurement was determined by loading and unloading a weight (2.65 mg) several times (>1000). The standard deviation of the measured force was determined to 6.2 nN with our setup including the modified control loop. The relative standard deviation is approximately 0.03 %

3.2. Displacement setting and measurement

The position of the EMFC load cells conversion lever can be set to any value in its moving range. Consequently the load button can be set to any position in its moving range of $\pm 13 \mu\text{m}$. The repeatability of the load button's position was determined to 0.8 nm with the interferometer.

The piezoelectric nanostage has a good linearity of 0.02 % and a repeatability of approximately 1 nm [20].

The interferometer linearity deviation was tested to be $< \pm 3 \text{ nm}$. It is periodical and occurs over one interference order or 316.4 nm.

3.3. Cantilever spring constant determination

The determination of the cantilever's spring constant is performed by setting the displacement in discrete steps which are held for 20 s. The displacement is chosen to be approximately 800 nm with 100 nm steps. Due to the specific setting time of the EMFC load cell, only the last 10 s of each step are used for the determination of the mean value of force and displacement at the specific point. The spring constant is then calculated by a linear regression of the measured force and displacement.

The cantilever used for the measurements is a PPP-NCLR-50 non-contact cantilever from Nanosensors with a length of 225 μm and a given spring constant of 21 - 98 Nm^{-1} [22].

3.4 Cantilever measurement - Measurement Mode 1

Fig. 4 depicts the acting force versus the displacement of the cantilever using measurement mode 1.

This curve is a mean value of 50 measurements, the loading curve is depicted in blue and the unloading curve is green. The less steep part of the curve indicates that the cantilever is not in contact.

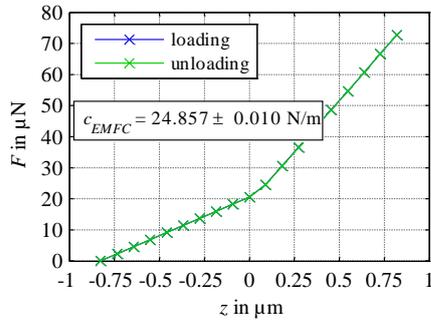


Fig. 4: force displacement curve AFM-cantilever measurement mode 1, raw data

The slope of the less steep part of the measurement curve (below $z = 0$) represents the spring constant of the EMFC load cell c_{EMFC} , which was calculated to be

$$c_{EMFC} = 24.857 \text{ Nm}^{-1} \pm 0.04 \%$$

Subtracting this spring constant from the measured curve leads to the spring constant of the cantilever shown in Fig. 5.

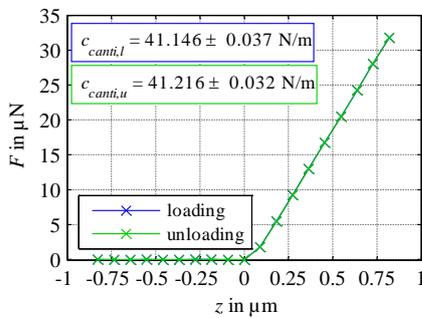


Fig. 5: force displacement curve AFM-cantilever measurement mode 1, corrected spring constant

The spring constant of the test sample was determined to $c_{canti,l} = 41.146 \text{ Nm}^{-1} \pm 0.09 \%$ for the loading and $c_{canti,u} = 41.216 \text{ Nm}^{-1} \pm 0.08 \%$ for the unloading process.

The given standard uncertainty was calculated by using the spring constants determined in every of the 50 loading and unloading cycles according to GUM [23].

3.5. Cantilever measurement - Measurement Mode 2

The same cantilever was measured using the second measurement mode. The force displacement curve is shown in Fig. 6. The spring constant was determined to $c_{canti,l} = 40.816 \text{ Nm}^{-1} \pm 0.06 \%$ for the loading and $c_{canti,u} = 40.695 \text{ Nm}^{-1} \pm 0.06 \%$ for the unloading process. The given measurement uncertainty was calculated the same way as described above.

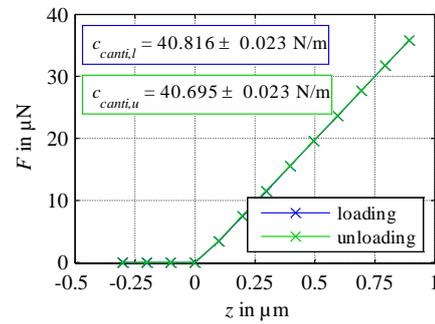


Fig. 6: force displacement curve AFM-cantilever, measurement mode 2

4. MEASUREMENT UNCERTAINTY

There are several systematic influences on the measurement which contributes to the measurement uncertainty. Mostly they were considered in [5-17] in a similar way.

4.1. Alignment of cantilever

The biggest contribution is the alignment of the cantilever towards the load button as shown in fig. 7 and 8.

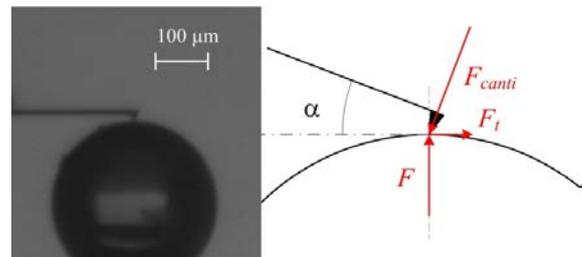


Fig. 7: Alignment of cantilever towards load button

If the cantilever is tilted by the angle α an additional tangential force F_t occurs which is perpendicular to the force F indicated by the measurement system [6].

The spring constant which is determined for a tilted cantilever is c_α (3).

$$c_\alpha = \frac{F}{z} \quad (3)$$

The measured spring constant c_α changes with the tilting angle α of the cantilever and is influenced by the friction coefficient μ (4) [6].

$$c_{\alpha,rel} = \frac{c_\alpha}{c_{canti}} - 1 = \frac{1}{\cos(\alpha) \cdot [\cos(\alpha) - \mu \cdot \sin(\alpha)]} - 1 \quad (4)$$

The alignment of the cantilever towards the load button can be verified using the camera system as shown in Fig. 7 on the left side. Assuming a maximum permissible tilting angle of $\alpha = 2^\circ$ and $\mu = 0.31$ [24] the relative error of the determined spring constant gets 1.2 % ($k = 1$).

An additional contribution is the offset of the cantilever tip towards the load button (fig. 8).

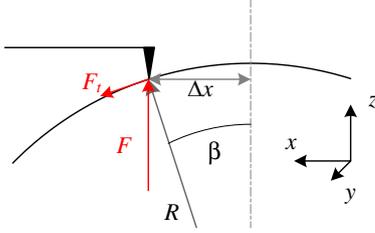


Fig. 8: Alignment if cantilever, offset of cantilever

This offset can occur in x- and y- direction resulting deviations in the measured force. An offset in the x-direction is shown in fig 9. The measured deformation is influenced by an occurring tangential force F_t as well as the friction coefficient μ and the angle β which leads to the relative deviation $c_{\Delta x,rel}$ (5) of the spring constant shown as red curve in fig. 9.

$$c_{\Delta x,rel} = \frac{c_{\Delta x}}{c_{canti}} - 1 = -\mu \cdot \sin(\beta) = -\mu \cdot \frac{\Delta x}{R} \quad (5)$$

Additionally a measurement of the cantilever's spring constant in dependence of the displacement Δx was performed (blue and green curve) and is in good agreement to the theory described above (red curve).

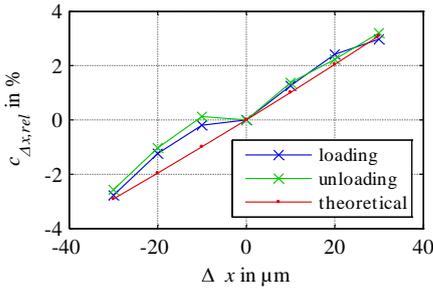


Fig. 9: Offset of cantilever in x-direction and resulting spring constant

The deviation between the measurement and theory might be due to the actual acting friction coefficient. In [5] the acting friction coefficient was estimated to be in a range of $\mu = 0.1...0.5$ for several repeated measurements. This large uncertainty might result from the contact parameters between the sharp cantilever tip and the actual point of contact on the ruby sphere. This is also a contribution to the standard deviation of the measured stiffness. Furthermore, the difference between loading and unloading seems to rise as Δx gets $\neq 0$. This might be an explanation for the measured discrepancy of the loading and unloading stiffness

in 3.1 and 3.2. In further investigations a tilting of the piezoelectric nanostage while its movement (measurement mode 2) was observed to be in the range of $6 \mu\text{rad}$ for pitch and yaw. Such a tilting was not observed for the displacement setting using the load cell (measurement mode 1). Accompanied by the possible influence of the friction coefficient in dependence of the actual contact parameters this might explain the difference among the stiffness measured in mode 1 and mode 2.

A displacement of approximately $\Delta x = 1...2 \mu\text{m}$ can be resolved using the camera system. Assuming a maximum permissible displacement error of $\Delta x = 5 \mu\text{m}$ the relative uncertainty of the spring constant results to 0.52 %.

4.2. Spring constants of nanostage and load cell

Further influences are the spring constants of the measurement setup which are in series to the spring constant of the cantilever. According to Fig. 2 and Fig. 3 the spring constant results to (6) for the measurement mode 1 and (7) for the measurement mode 2 whereby c_{meas} is the actual measured spring constant.

$$c_{Canti,MM1} = \frac{c_{hold} \cdot c_{meas}}{c_{meas} - c_{hold}} - c_{EMFC} \quad (6)$$

$$c_{Canti,MM2} = \frac{c_{hold} \cdot c_{meas}}{c_{meas} - c_{hold}} \quad (7)$$

Therefore the spring constants of the holder including the piezoelectric nanostage c_{hold} and the EMFC load cell c_{EMFC} have to be taken into account. By measuring the position of the load button, the static spring constant of the EMFC load cell c_{stat} can be neglected as described above.

The spring constant of the holder c_{hold} was measured by loading the holder with known masses and measuring the deflection using the interferometer. This spring constant was determined to $c_{hold} = 257.38 \text{ kNm}^{-1} \pm 2.52 \%$ ($k = 1$), which is mainly due to the stiffness of the nanostage [20]. Based on (6) and (7) this leads to a negligible influence on the measured spring constant ($\approx 41 \text{ Nm}^{-1}$) of $< 0.02 \%$.

4.3. Calibration of force measurement

The EMFC load cell uses a voice coil for the compensation of the acting force. This voice coil is characterized by the magnetic field strength B and the length of the wire l . Both factors are combined to the factor Bl . This factor Bl describes the sensitivity of the force measurement towards the compensation current I_{comp} . Consequently deviations of the factor result in deviations of the force measurement. Additionally the factor Bl is influenced by the temperature, as the magnetic field strength of a permanent magnet decreases with rising temperature. This temperature influence is described by the temperature coefficient of factor TK_{Bl} and the change of the temperature:

$$F = I_{comp} \cdot (Bl_{20^\circ\text{C}} + TK_{Bl} \cdot \Delta T_{20}) \quad (8)$$

The factor itself was determined by the calibration of the EMFC load cell using standard mass pieces class E0 and measuring the compensation force. The factor was determined to $Bl = 11.5850 \text{ NA}^{-1}$ at a temperature of 22.15°C .

The relative standard uncertainty of the calibration results from the tolerance of the weights [21] used for the specific calibrated force (fig. 10).

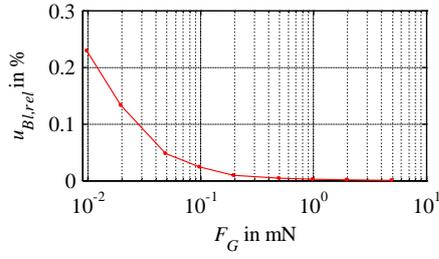


Fig. 10: relative standard uncertainty factor Bl

The force steps during the cantilever measurement were approximately $5 \mu\text{N}$, therefore a relative standard uncertainty of the force calibration of 0.23 % is assumed.

To measure the temperature influence the EMFC load cell was alternating loaded and unloaded with a mass piece, resulting in a variation of the compensation current. Additionally the temperature was changed. The variation of the load divided by the resulting variation of the compensation current leads to the temperature coefficient of the factor Bl , which was determined to:

$$TK_{Bl} = -3.819 \cdot 10^{-3} \frac{\text{N}}{\text{AK}} \pm 0.096 \%$$

The resolution of the temperature measurement (1 mK) as well as the temperature change (0.1 K) as well as the temperature coefficient are very small. Their influence on the uncertainty of the force measurement is less than 0.1 %, therefore it can be neglected.

The spring constant of the load cell is also influenced by the temperature, since the Young's modulus of the load cells material changes with temperature.

This influence can be neglected during measurement, as the spring constant of the EMFC load cell is measured additionally using measurement mode 1.

4.4 Displacement measurement

For the uncertainty determination of the displacement measurements z_i and z_{Piezo} the nonlinearity and the repeatability are considered as the most significant influences.

The repeatability of the displacement z_i set by the nanostage was measured with the interferometer to 0.8 nm.

In our cantilever measurements, the maximum deformation was set to $0.9 \mu\text{m}$, resulting in a relative repeatability of z_{Piezo} of 0.09 %.

Based on the datasheet values, the nonlinearity of the piezo stage is given with 0.02 %.

The repeatability of the displacement z_i set by the load cell was determined the same way with the same result of 0.8 nm ($\pm 0.09 \%$).

The nonlinearity of the interferometer is dependent on the adjustment of the laser beams and was measured to be $< 3 \text{ nm}$ with a 316.2 nm period ($\pm \lambda/2$). This conforms to a relative contribution of the nonlinearity of 0.33 % in the range of $0.9 \mu\text{m}$.

4.5. Uncertainty budget

The calculation of the force measurements uncertainty using measurement mode 2 is the same as in measurement mode 1. The standard uncertainty of the force calibration is given by relative uncertainty of the compensation current (standard deviation of measurement during each displacement step, 0.03 %) and the uncertainty of factor Bl due to the calibrations using the E0 weights.

Concerning the uncertainty of the cantilevers deformation there is a difference in measurement mode 1 and measurement mode 2. In measurement mode 1 the displacement measurement is performed using the interferometer (z_i). In measurement mode 2 the displacement of the cantilever is measured as the difference of position data of the piezoelectric nanostage and the z_{Piezo} and the interferometer z_i (cf. (2)).

The uncertainty budget of both measurement modes are shown in table 1.

5. DISCUSSION

The measurements of the stiffness have shown a good agreement between the results of mode 1 ($c_{Canti,l} = 41.146 \text{ N/m} \pm 2.75 \%$ ($k = 2$)) and mode 2 ($c_{Canti,l} = 40.816 \text{ N/m} \pm 2.75 \%$ ($k = 2$)).

The uncertainty considerations pointed out that the largest contribution to the uncertainty (1.2 % and 0.52 % ($k = 1$)) is the alignment of the cantilever towards the load button, resulting in lateral forces that distort the measured

Table 1: relative measurement uncertainty and uncertainty contribution for both measurement modes

| Component of uncertainty | Relative uncertainty in % ($k = 1$) | | Uncertainty type |
|---|---------------------------------------|--------------------|------------------|
| | Measurement mode 1 | Measurement mode 2 | |
| Force measurement (overall) | 0.23 | 0.23 | Combined |
| Force calibration | 0.23 | 0.23 | Combined |
| Repeatability of force measurement | 0.03 | 0.03 | A |
| Displacement measurement (overall) | 0.34 | 0.35 | Combined |
| Repeatability of piezoelectric nanostage z_{piezo} | negligible | 0.09 | A |
| Linearity of piezoelectric nanostage | negligible | 0.02 | B |
| Repeatability of position load button z_i | 0.09 | 0.09 | A |
| Linearity of interferometer | 0.33 | 0.33 | B |
| Repeatability of spring constant of EMFC load cell c_{EMFC} | 0.04 | negligible | A |
| Repeatability of spring constant c_{canti} | 0.09 | 0.06 | A |
| Tilting of cantilever α | 1.20 | 1.20 | B |
| Displacement of cantilever Δx | 0.52 | 0.52 | B |
| Combined measurement uncertainty $k = 2$ | 2.75 | 2.75 | |

stiffness. The lateral forces are dependent on the friction coefficient μ between the cantilever tip and the ruby sphere. Since μ strongly depends on the contact parameters, its value ($\mu = 0.1 \dots 0.5$ [5]) leads to large uncertainties of the lateral forces. This might explain the difference between the determined spring constant in measurement mode 1 and 2 especially since the movement of the piezoelectric nanostage is affected by tilting (mode 2) in contrast to the movement of the load button (mode 1).

6. CONCLUSION

The current version of the force displacement measurement setup for the determination of AFM cantilever spring constants uses a modified EMFC load cell for force measurement and displacement setting. Related to the state of the art a piezoelectric nanostage can be used for displacement setting additional, the load cell is then used for force measurement only. The measurement results with those two measurement modes correspond to another and are

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in good agreement. The extended measurement uncertainty was calculated to 2.75 % for both measurement modes, which is in good agreement with the uncertainties achieved in [15].

The alignment of the cantilever towards the load button is the most significant contribution. Hence, improvements of this static calibration method for cantilever spring constants or the sensitivity of micro force sensors can only be achieved, if the alignment of the cantilever towards the load button and the contact parameters will be further investigated.

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