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DEVELOPMENT OF A FORCE DISPLACEMENT MEASUREMENT DEVICE FOR THE DETERMINATION OF SPRING CONSTANTS

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ABSTRACT

This paper discusses a measurement device for the determination of force displacement curves or spring constants respectively. Especially the calibration of spring constants of atomic force microscopes (short: AFM) cantilevers is an important field of investigation and concentration of this work.
The spring constant can be measured with two measurement modes. One mode uses separate force and displacement measurement devices which correspond to the state of the art. The second measurement mode is more sophisticated using one sensor which measures force as well as the displacement simultaneously; a separate nanostage is not need.
The measured sample is an AFM type cantilever, its determined spring constant is in both measurement modes approximately 50 Nm^{-1} with a very good repeatability of 0.02 %.

Index Terms - spring constant, force measurement, AFM cantilever, EMFC load cells

1. INTRODUCTION

The calibration of force displacement curves (spring constants) of AFM cantilevers and the determination of force-signal curves (force sensitivity) of cantilever type micro force sensors is a known field of investigation in metrology. There are several measurement setups and measurement strategies as well as performed calibrations and international comparisons described in literature [1 … 11]. The lowest measurement uncertainty was achieved by using a static force calibration described in [3].
The current investigations at TU Ilmenau are based on a preliminary setup described in [12] and it is also related to the international state of the art, especially described by the international comparison described in [9].
The spring constant of the sample is determined by using an electromagnetic force compensated (short: EMFC) load cell. A modification of the control loop enables the possibility to set the displacement of the weighing pan and measure the acting force simultaneously [12]. Additionally it is also possible to set the displacement with a commercial piezoelectric nanostage and using the EMFC load cell for force measurement only. This is similar to other to measurement setups as described in [2], [3] and [4]. This second measurement mode can be used to verify the results of measurement mode. Both measurement modes are supposed to be used for international comparison in this field of investigation in the future. Especially measurement mode two can be used for direct comparison, as it describes the state of the art.

2. MEASUREMENT SETUP

The measurement setup consists of six main components as depicted in Figure 1. The xyz-stage (1) is a commercial system from Physik Instrument (short: PI) using an M-403.3 translation stage [13] in z- direction and two miniature translation stages M-110.1 [14] in x- and y- direction, enabling the possibility to align the sample (3) in a wide range. The measurement axis of the sample is defined in z-direction.
A piezoelectric nanostage (2) PI P-621.1 is attached to the positioning system. This system has a positioning resolution of 0.1 nm [15] and is used to set the displacement of the sample in the second measurement mode.
The EMFC load cell (WZA215-LC, Sartorius) (5) is used for force measurement as well as a nanopositioning system. It has a force resolution of 100 nN with a measurement range of 2 N [16]. A commercial tactile stylus (Mitutoyo, STU-M2-RU-1-10) (4) with a 1 mm diameter ruby is used as load button.
The movement of the nanostage or the sample respectively as well as the displacement of the load button are measured with two axis of the triple beam interferometer (6) SP-TR from SIOS which has a resolution of 80 pm [17]. The third axis is used as reference measuring against the measurement frame (7).

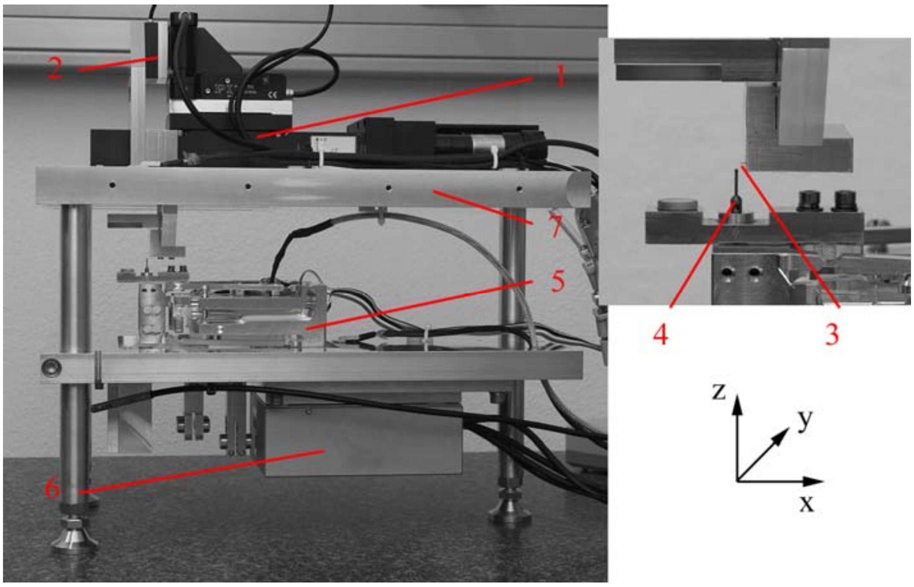

Figure 1 -measurement setup
1 – xyz stage, 2 – piezoelectric nanostage, 3 – sample (cantilever), 4 – load button, 5 – EMFC load cell, 6 – triple beam interferometer, 7 – measurement frame

Not depicted is the long-distance microscope camera (ThorLabs), which is used to align the sample relative to the load button.

3. MEASUREMENT PRINCIPLE

The used sample is currently a non contact AFM cantilever. The cantilever is glued to an aluminum holder in a horizontal position. This alignment differs to the tilted position used in AFM microscopes. In our case the deflection of the cantilever is perpendicular to the invoking force therefore a lateral force on the cantilever as it occurs in AFM microscopes is unlikely and reduces the measurement uncertainty [2]. As the cantilever deflects a spherical load button is needed to prevent the cantilever holder touching the load button. The ruby load button has a diameter of 1 mm. The alignment of the cantilever relative to the load button is

performed with the xyz- stage and controlled with the long distance microscope camera. The long distance microscope has a 12 times magnification and the camera has a resolution of 1200x 1024 pixel. The effective resolution is approximately 2 µm, the software interpolates the picture to an image resolution of 1 µm. Figure 2 shows a picture taken with the long distance microscope camera showing the cantilever touching the load button.
The positioning resolution of the xyz- stage is given with 1 µm, which can barely be resolved by the camera.

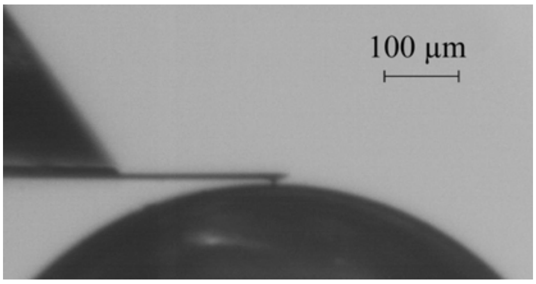

Figure 2- picture of cantilever and load button using the long distance microscope camera

There are two measurement modes as mentioned above. The two measurement modes differ in the setting of the displacement of the cantilever relative to the load button. In the first measurement mode the cantilever is fixed and the load button moves and in the second measurement mode vice versa.

3.1 Measurement mode 1 – combined force and displacement measurement

The measurement mode 1 differs from the state of the art cantilever measurement as described in [1] and [2]. In our case the cantilever is positioned relative to the load button described above in a way that they do not touch each other and afterwards the cantilever is fixed. The displacement of the load button is generated using the EMFC load cell as a nanopositioning system with combined force measurement.

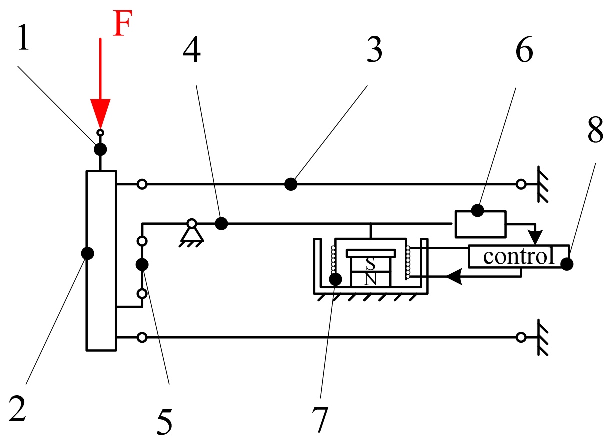

Figure 3 – schematic of an EMFC load cell
1 – load button, 2 – pan carrier, 3 – parallel spring system, 4 – conversion lever, 5 – coupling element, 6 – position sensor, 7 – voice coil, 8 – control loop

Figure 3 depicts a schematic of an EMFC load cell. A force acts on the load button (1) and deflects the pan carrier (2) as well as the parallel spring system (3). The conversion lever (4) which is attached to the pan carrier using a coupling element (5) also deflects. This movement is measured with an optical position sensor (6). Attached to the conversion lever is a voice

coil (7). If the conversion lever deflects a control loop (8) controls its position setting the current of the voice coil in a way that the resulting Lorentz force compensates the force which acts on the load button. The compensation current is a measure for the acting force.
In a commercial load cell the conversion lever is controlled to a fixed position (position signal zero). We modified the control loop in a way that the conversion lever can be set to any position in its moving range [18], [19].
The load button is controlled to its lower position and the cantilever is aligned slightly above the load button using the xyz- stage. The load button is then moved upwards until it touches the cantilever and moved further so that it bends the cantilever. The quotient of the resulting force and the displacement of the load button result to the sum of the spring constants of the cantilever c_{canti} and the EMCF load cell c_{EMFC}. The spring constant of the EMFC load cell is caused by the deflection of the load cell's flexure hinges. The spring constant of the EMFC load cell is known by the measurement without the cantilever and is subtracted. Section 4.3.1 shows the measurement of cantilever with measurement mode 1.

3.2 Measurement mode 2 – decoupled force and displacement measurement

In contrast to the first measurement mode, the second measurement mode uses the EMFC load cell only for force measurement; the conversion lever is controlled to a fixed position.
Again the cantilever is aligned slightly above the load button using the xyz- stage. The xyz-stage is then fixed. The displacement is then set by using a commercial piezoelectric nanostage until the cantilever touches the load button and then moved further, resulting a deflection of the cantilever and a force respectively. The force is measured with the EMFC load cell.
The piezoelectric nanostage has an internal capacitive measurement system with a resolution of 0.1 nm. Additionally the deflection is measured with the interferometer, thus it is traceable to the SI base unit length.

4. MEASUREMENTS

4.1 EMFC load cell

4.1.1 Force

The EMFC load cell's output signal is the compensation current calculated by the control loop, therefore a calibration is needed to get the relation to the force. For the first experiment we use type F1 standard mass pieces according to OIML R-111 [20]. The weight of the mass pieces is calculated with the gravitational acceleration in the laboratory of $g = 9.810131 \pm 0.00041\ \mathrm{ms}^{-1}$ taken from the Schwere Inforamtionssystem of the PTB [21].
The calibration was done with masses ranging from 10 mg up to 5 g and forces of 100 µN up to 50 mN respectively. The force sensitivity is calculated to $E_F = 5.0307 \pm 0.0001\ \mathrm{mA\ N}^{-1}$ (k = 2) (see Figure 4).

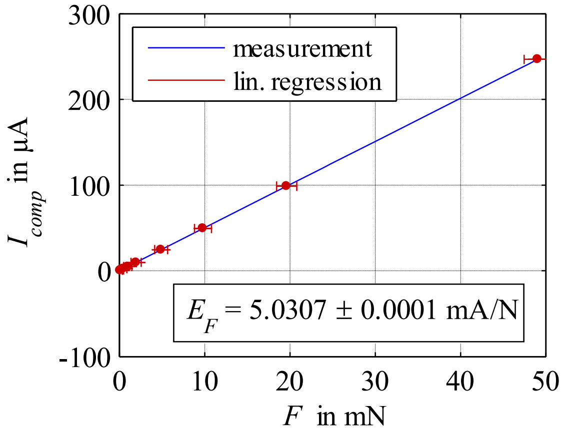

Figure 4 – Force calibration of the EMFC load cell; I_{comp} – compensation current, F – force, E_F – force sensitivity, red dots: measurements, blue curve: linear regression

The repeatability of the force was determined by using a wire weight with a known mass (m_{Wire} = 0.283 g). The repeatability was calculated to s_F = 100 nN (standard deviation), which corresponds to the measurement resolution given by the manufacturer [16].

4.1.2 Displacement

The displacement of the load button is controlled with the signal of the position sensor and measured using one beam of the interferometer. Figure 5 depicts the measured displacement versus the voltage of the position sensor. The measurement was done with the unloaded and loaded EMFC load cell. The load was the same wire weight mentioned above. The position was controlled to voltages in the range of U_{pos} = -6...7.5 V.

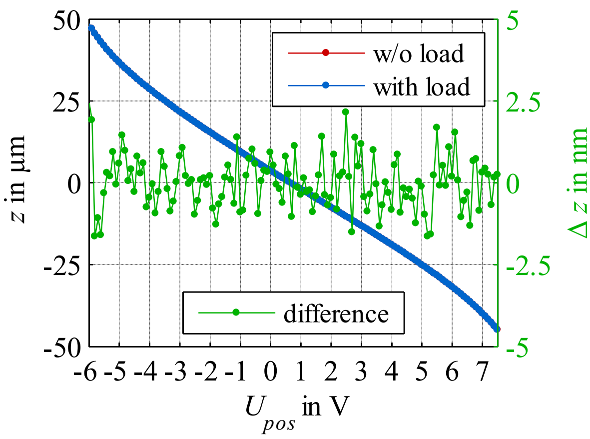

Figure 5 – displacement of the load button z versus signal of position sensor (Upos) – blue and red dots: unloaded and loaded EMFC load cell, green line difference between both

The resulting displacement of the load button is between $z = \pm$ 50 µm. The difference of the position between loaded und unloaded EMFC load cell is less than $\Delta z = \pm$ 2 nm.
The repeatability of the displacement measurement was determined to s_z = 1.4 nm (standard deviation).

4.1.3 Spring constant

With the known current to force behavior of the EMFC load cell the spring constant of that system can be determined using the displacement measurement and the known compensation current. The force is calculated by multiplying the compensation current with the force sensitivity E_F. Figure 6 shows the force of the EMFC load cell versus the position of the weighing pan.

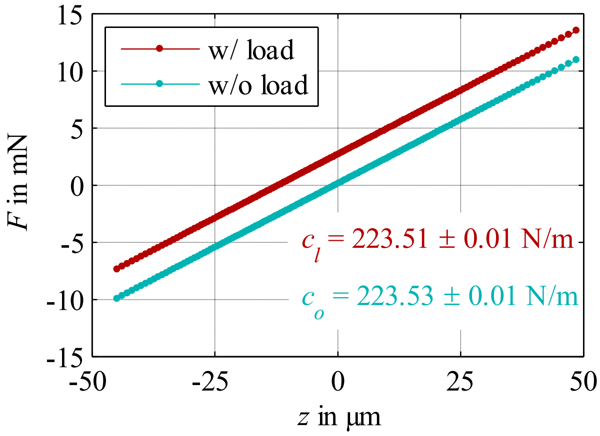

Figure 6 – force versus displacement of weighing pan, blue and red curve: unloaded and loaded EMFC load cell, $c_{l,o}$ – spring constants loaded and unloaded

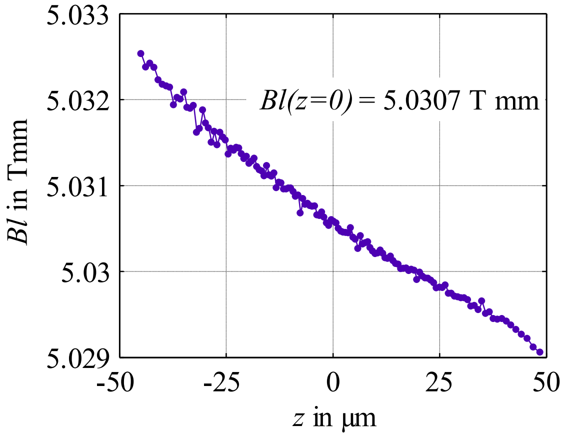

Figure 7 – Bl as characteristic value of the voice coil versus the displacement of the pan carrier

The difference of the forces between loaded and unloaded EMFC load cell divided by the current at position z = 0 results in the product of the magnetic field strength B and the length of the wire l of the coil called Bl. This characteristic value of the voice coil or the EMFC load cell respectively is shown in Figure 7. There is a deviation of the Bl in the moving range of the pan carrier which is caused by the magnetic field distribution of the voice coil's permanent magnet. This behavior has to be taken into account for the determination of the force to displacement behavior when the measurement mode 1 is used.

4.2 Displacement measurements

4.2.1 Nanostage

The piezoelectric nanostage has an internal capacitive displacement measurement; the chosen displacement is controlled using this internal measurement system. The positioning repeatability is referred to 0.1 nm by the manufacturer [15]. However the linearity and repeatability is measured using the interferometer.

Figure 8 shows the linearity of the piezoelectric nanostage measured with the interferometer (red dots) and the linear regression of that measurement (blue line). The green line depicts the difference between the values measured with the piezoelectric nanostage (z_{piezo}) and the displacement measured with the interferometer (z). The difference is the linearity error of the piezoelectric nanostage which was determined to $\Delta z = \pm 10$ nm.

Figure 9 depicts the short term stability of the piezoelectric nanostage measured with the interferometer (red line) and the internal reference measurement system (blue line). The standard deviation of the measurement with the internal capacitive measurement system is $s_{cap} = 6.96$ nm and with the interferometer $s_{int} = 0.84$ nm.

The repeatability of the displacement set with the nanostage was determined by applying displacement steps of 10 µm 20 times. The standard deviation of the steps is 1.9 nm measured with the interferometer und 11.9 nm measured with the internal capacitive measurement system.

The internal measurement system shows a poor repeatability, however the controlled position is much better. We assume that the error is due to the analog digital conversion of the measurement values of the reference system. The nanostage uses the analog signal of its reference measurement system to control the position. The long period deviations in the interferometer signal is probably due to thermal convection in the setup.

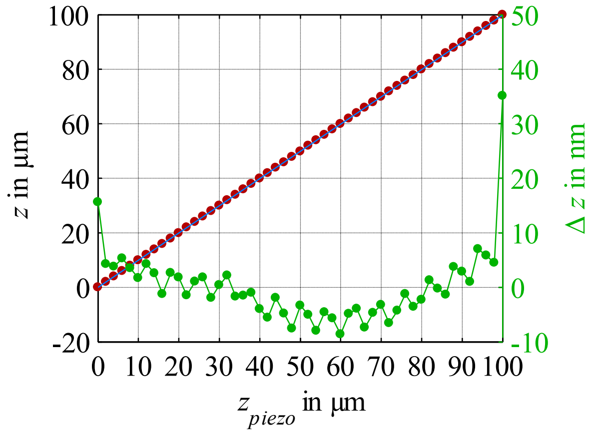

Figure 8 – displacement of nanostage measured with the internal capacitive measurement system versus the displacement measured with the interferometer

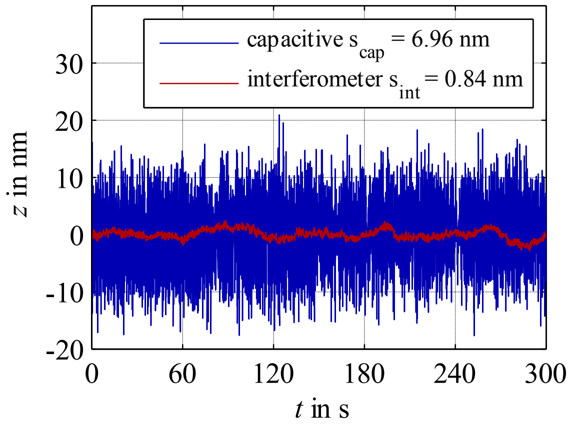

Figure 9 – short term stability of the piezoelectric nanostage measured with the interferometer (red line) and the internal capacitive measurement system (blue line)

4.3 AFM cantilever

The cantilever is a sample of unknown origin. The length of the cantilever is approximately 225 µm.

The measurements were performed for both measurements modes with similar displacement. The displacement was set to $z = \pm 6$ µm whilst the cantilever was in contact in a range of $z_{canti} = 0 \ldots 6$ µm. Step width was $\Delta z = 0.5$ µm.

4.3.1 Measurement mode 1

Figure 10 shows the force to displacement measurement of the cantilever using the first measurement mode.

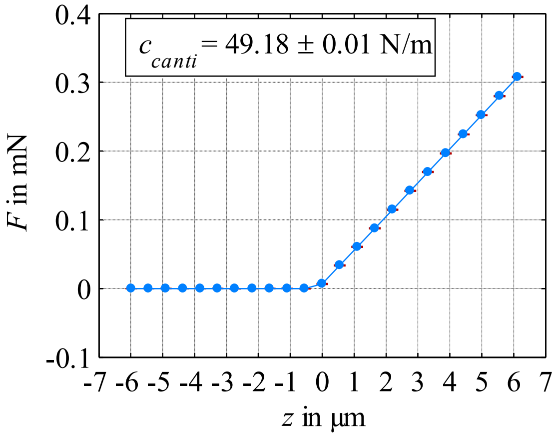

Figure 10 – force displacement measurement of the cantilever using the measurement mode 1, c_{canti} - spring constant

The spring constant was determined to $c_{canti} = 49.18 \pm 0.01$ N m^{-1} (k=2). The mean value and measurement uncertainty was determined using a series of 20 measurements according to GUM [22].

4.3.2 Measurement mode 2

The second measurement mode gives pretty much the same result for the spring constant which was determined to $c_{canti} = 49.04 \pm 0.01$ N m^{-1} (k=2). The measurement uncertainty was determined using a series of 20 measurements. Figure 11 depicts the force displacement measurement for the same cantilever.

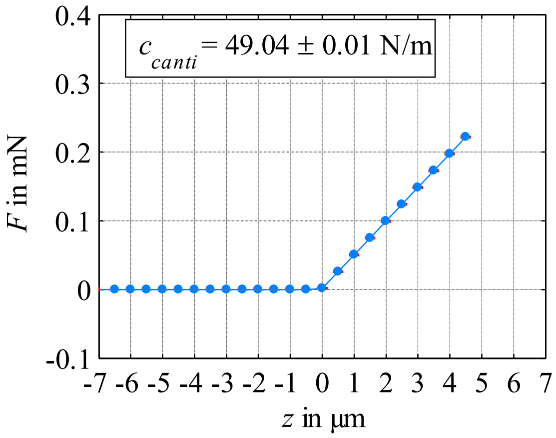

Figure 11 – force displacement measurement of the cantilever using measurement mode 2, c_{canti} – spring constant of the cantilever

5. CONCLUSION

A new measurement system for the determination of force displacement curves and spring constants was build up and investigated. The measurement system has two measurement modes; measurement mode 1 uses the force sensor also for displacement measurement and setting which is unique. The second measurement mode corresponds to the state of the art on this field of investigation using the force sensor for force measurement only and a separate piezoelectric nanostage for displacement setting. It was shown that the measurement system has a good repeatability concerting force and displacement measurement in both measurement modes.

An AFM cantilever was measured using both measurement modes; the determined spring constant of that cantilever was determined to $c_{canti} \approx 49$ N m^{-1}. The relative repeatability is in both measurement modes $u_{c,canti,rel} = 0.02$ % (k = 2). There is still a discrepancy of the determined spring constants mean value. This discrepancy might be caused by thermal influences.

6. OUTLOOK

The measurement setup is still under investigation concerning environmental influences especially temperature.
The spring constant of the EMFC load cell is now much higher than the spring constant of the samples. The measurement uncertainty of the EMFC load cell's spring constant directly affects the measurement uncertainty of the determined spring constant of the investigated samples in measurement mode 1. In future we will investigate an EMFC load cell with a higher force resolution and much lower spring constant by using a system with a maximum force measurement range of 20 mN and a force resolution of 1 nN [23]. However the current EMFC load cell is well suited for big spring constants and high forces.
In future a full measurement uncertainty will be calculated including all known influences such as the measurement uncertainty of the force measurement and its calibration, the displacement measurement and the influence of the cantilever alignment which was determined as biggest contribution to the measurement uncertainty by [2].

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REFERENCES

[1] M.-S. Kim et al., Atomic force microscope cantilever calibration device for quantified force metrology at micro- or nano-scale regime: the nano force calibrator (NFC), Metrologia 43 (2006) 389-395.
[2] M.-S. Kim et al., Si-traceable determination of spring constants of various atomic force microscope cantilevers with a small uncertainty of 1 %, Meas. Sci. Technol. 18 (2007) 3351-3358.
[3] C. Clifford et al., The determination of atomic force microscope cantilever spring constants via dimensional methods for nanomechanical analysis, Nanotechnology 17 (2005) 1666-1680.
[4] L. Doering et al., Micro force transfer standards, Proceedings of IMEKO 2002, TC3, TC5, TC22 Conference, Celle, Germany, 2002.
[5] I. Behrens et al., Piezoresistive cantilever as a portable micro force calibration standard, J. Micromech. Microeng. 13(4) (2003), 171-177.
[6] J. Pratt et al., Progress toward systéme internationale d'unités traceable force metrology for nanomechanics, Mater.Res. 19(1) (2004) 366-379.

[7] L. Doering et al., Calibration of low-force stylus probes, in: Proceedings of XVIII IMEKO World Congress, Rio de Janeiro, Brasil, 2006.
[8] W. Hoffmann et al., Methods of characterizing micro mechanical beams and its calibration for the application in micro force measurement systems, in: Proc. MicroTec, 2000, pp. 819-823.
[9] M.-S. Kim et al., Report on the first international comparison of small force facilities: a pilot study at the micronewton level, Metrologia 49 (2012) 70-81
[10] J. Pratt et al., Review of SI traceable force metrology for instrumented indentation and atomic force microscopy, Meas. Sci. Technol. 16 (11) (2005) 2129-2137.
[11] M.-S. Kim et al., Long and wide cantilever as a microforce transfer artefact, in: Proceedings of XX IMEKO World Congress, Busan, South Korea, 2012.
[12] C. Diethold et al., Determination of force to displacement curves using a nanopositioning system based on electromagnetic force compensated balances, Measurement (2014), doi:10.1016/j.measurement.2014.02.034.
[13] Physik Instrumente (PI) GmbH & Co. KG, Precision translation stage M-403.3 (2009)
[14] Physik Instrumente (PI) GmbH & Co. KG, Compact micro- translation stage M-110.1 (2008)
[15] Physik Instrumente (PI) GmbH & Co. KG, PIHera Piezo linear stage P-621.1 (2008)
[16] Sartorius Weighing Technologies GmbH, Weender Landstraße 94-108, D-37075 Göttingen, Germany, Sartorius Wägezellen Modelle WZA614-NC, WZA215-LC, WZA245-NC, WZA26-NC (Januar 2013).
[17] SIOS Meßtechnik GmbH, Am Vogelherd 46, 98693 Ilmenau, Germany, Dreistrahlinterferometer mit Planspiegelreflektor Serie SP-TR (March 2014).
[18] T. Fröhlich, F. Hilbrunner, C. Diethold, Verfahren und Vorrichtung zur Vorgabe von Kraft-Weg-Kennlinien, Patent DE102011111238 B4, May 2nd 2013
[19] C. Diethold et al., Nanopositioning system with combined force measurement based on electromagnetic force compensated balances, Proceedings of XX IMEKO Wolrd Concress, Busan, South Korea, 2012
[20] Organisation internationale de métrologie légale, OIML R-111, Tech. rep., Paris (2004).
[21] Physikalisch Technische Bundesanstalt, Schwere- Informationssystem SIS, Tech. rep., PTB, Braunschweig, Germany (2007).
[22] BIPM, IEC, IFCC, IUPAC, IUPAP, OIML, Guide to the expression of uncertainty in measurement, Tech. rep., ISO, Geneva (1993).
[23] Sartorius Weighing Technologies GmbH, Weender Landstraße 94-108, D-37075 Göttingen, Germany, Sartorius Wägezelle WZ2P-CW (Dezember 2009)

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