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CALIBRATION OF LOW-FORCE STYLUS PROBES

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Abstract: Traceable measurement of micro forces involved during stylus profiling is described. For this purpose two methods based on a transferable cantilever-type force standard were developed using the cantilever deflection and the output voltage of an integrated piezoresistive strain gauge. Force calibration was performed with commercial stylus instruments in the range of 10 - 200 µN.

Keywords: micro force calibration standard, stiffness artefact, load cell, stylus instrument.

1. INTRODUCTION

Traceable micro- and nano metrology is needed for quality control of advanced manufacturing processes in industrial or commercial environment. Independent proof of different measurement methods can be obtained by international comparisons of standards, e.g. depth-setting or roughness standards [1, 2]. Stylus profiling and indentation instruments as well as atomic force microscopes (AFM) are widely used tools for micro- and nano mechanical testing. For proper use they need traceable probing force metrology which extends over the range of nano Newton to milli Newton [1, 3 - 5].

Surface profiling using a stylus probe is proposed for the measurement of bending stiffness, residual stress and fracture limit of MEMS devices [6 - 10]. Commercial stylus instruments, which can be found in most IC fabrication facilities, offer an adjustable static force setting typically within 1 µN and 10 mN. Accurate and reliable mechanical testing requires periodical re-calibration of the probing force as it is common practise for the vertical position read-out of the stylus by profiling a thickness artefact. Consequently, there is a demand for transferable low-force standards, i.e. intermediaries by which the stylus force setting can be compared with a suitable standard, e.g. a calibrated nano balance.

At PTB force-transfer artefacts, AFM probes and silicon cantilevers, have been investigated to calibrate fiber probes and stylus instruments [11]. An electrical nano balance was developed at NPL providing a probing area and a measurement range designed for the calibration of atomic AFM probes [12]. However, owing to their small size of typically a few hundreds of microns in length and a few tens of microns in width AFM probes are not convenient for force calibration of commercial tactile probing instruments. Furthermore, by increasing the cantilever length with respect to typical AFM probes both reliability and accuracy of the load cell can be improved [13]. The described calibration method using stiffness artefacts [11] requires precise definition of the loading position using end marks on the cantilever which is tedious and represents a considerable source of error.

Therefore, silicon microcantilevers with integrated piezoresistive strain gauges have been developed which can be calibrated both as stiffness artefacts and as load cells [14]. In this study we describe a procedure based on these cantilevers to measure the force involved during stylus profiling. For this purpose profiling of the cantilever surface along its axis was performed using commercial stylus instruments at probing forces of 10 - 200 µN (Fig. 1). The aim is to establish a method of micro force measurement based on transferable cantilever-type standards which allows a fast and accurate calibration of the probing force of stylus instruments. For the experiments we used two different stylus instruments (P11, Tencor Corp., Santa Clara, CA, USA and Nanostep 1000, Taylor Hobson, Leicester, UK).

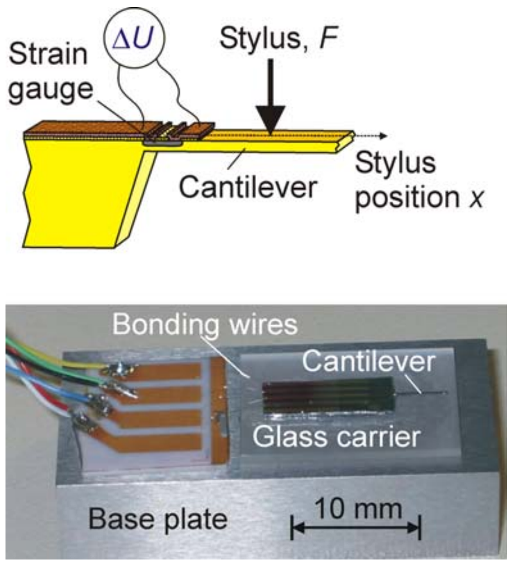

Fig. 1. Transferable cantilever-type force standard with integrated piezoresistive strain gauge in a schematic representation (upper) and in a photograph (lower)

2. FABRICATION OF FORCE STANDARD

Cantilever-type force sensors are realized using a bulk micromachining process based on standard photo-lithography, wet etching and diffusion from a spin-on-source (Fig. 3, [14, 15]). The process is started by etching of an *n*-type silicon wafer (2-5 Ωcm) using TMAH solution (tetra methyl ammonium hydroxide, 20%, 80 °C) through a mask of thermal oxide to obtain a membrane structure. Subsequently, *p*-type resistors are realized by boron diffusion from a spin-on silica emulsion source (Emulsitone Borofilm 100, 1100°C). For improved contact formation p^+-type regions are fabricated in a second diffusion step (1200°C). Compared to other doping techniques like implantation and deposition diffusion neither induces crystal damage nor requires toxic gases or hazardous materials.

Subsequently, a probing tip which ensures defined loading conditions during calibration is realized at the cantilever free end. By undercut etching of a circular oxide mask using TMAH a pyramid is generated exhibiting an octagonal base and sidewalls represented by {133} facets as the fastest etching planes. During this step, simultaneously, the membrane is thinned to its final thickness.

The wafer is oxidized and patterned for contact holes to the p^+-type regions of the piezoresistors. A gold/chromium metallization is deposited by e-beam evaporation. The cantilever is released wet chemically using potassium hydroxide solution (KOH, 30 %, 60°C). We prefer KOH during this final etching step due to its lower mask undercut compared with TMAH. A protection of the connecting lines during this step is not necessary. The resulting cantilevers are nearly 5 mm long, 200 µm wide and 50 µm thick.

3. CALIBRATION PROCEDURE

3.1 Calibration of cantilever sensor

Calibration of micro pipettes using a compensation balance is performed by incrementally moving it with its tip against the weighing pan of the balance (Fig. 2, [16]). The pipette is roughly positioned to the pan using a 2D piezoactuator (Picomotor actuator 8302, Newfocus, San Jose, USA). Subsequently, it is lowered until it is in contact with the pan. Now calibration starts by incremental movement of the pipette against the pan in steps of 125 nm at a resolution of 1 nm and a reproducibility of 5 nm using a piezoactuator with capacitive feedback (P 721, Physik Instrumente (PI), Karlsruhe, Germany). The procedure is stopped when a maximum force of 100 µN is attained as measured using a compensation balance offering a resolution of 1 nN and a reproducibility of 2.5 nN (SC2, Sartorius, Göttingen, Germany). The pipette is tilted to the pan by an angle of 30°. Therefore, the pipette stiffness is given by the slope of the load-deflection curve multiplied by cos(30°) = 0.866. The complete setup is mounted in a thermally isolated box ensuring a temperature drift of less than 10 mK/h. During one calibration run (typically 30 min) a temperature drift within 2-5 mK can be maintained.

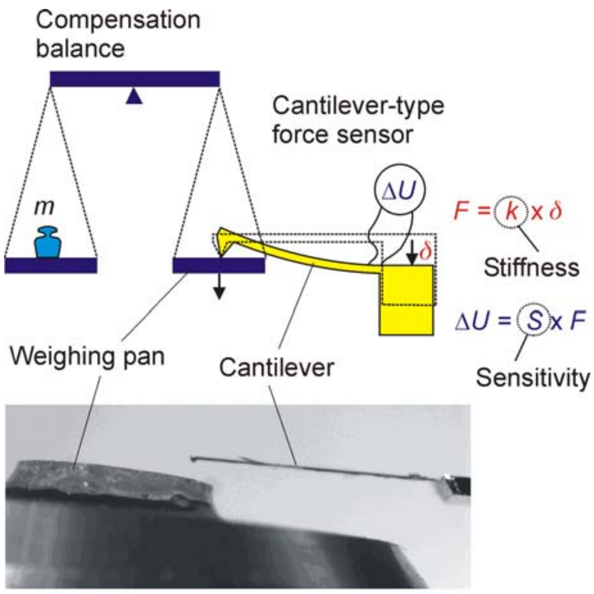

Fig. 2. Setup for cantilever force sensor calibration in a schematic representation (upper) and in a photograph (lower)

For calibration the sensor is mounted into a connector designed for simultaneous load-deflection and load-strain gauge output voltage measurement. Electrical connection is attained by pressed contacts formed between the connector pins and the Au/Cr pads on the sensor chip. Thus wire bonding can be omitted. The bridge output voltage is measured at a supply voltage of 1 V using a d.c. low-noise bridge amplifier (ML10B, HBM Mess- und Systemtechnik, Darmstadt, Germany) operated below a cut-off frequency of 500 Hz.

In Fig. 4a typical force-deflection and strain-gauge-output-voltage characteristics of a cantilever sensor (D47B) are displayed. We observe linear increase of force and simultaneous decrease of output voltage with increasing *z* immediately after the probing area has touched the weighing pan. By least-squares fitting we obtain the slopes which correspond to the cantilever stiffness *k* and the sensor sensitivity *S*, respectively. Respective calibration runs are performed and analyzed repeatedly. By averaging within 15-100 µN we find values of $k = 7.509 \pm 0.09$ N/m and $S = -0.0351 \pm 0.0002$ mV/µN for the stiffness and the sensitivity, respectively.

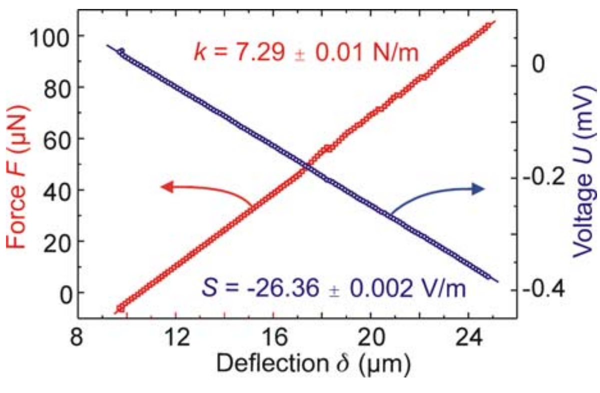

Fig. 3. Typical load-deflection and voltage-deflection curves of cantilever sensor D4

3.2 Calibration of stylus instruments

Figure 1 shows a transferable cantilever-type force standard with integrated piezoresistive strain gauge in a schematic representation (upper part) and in a photograph (lower part). Probing the cantilever along its axis using a stylus instrument yields curves of cantilever deflection and strain- gauge output voltage which depend on the position x along the cantilever as ~ x^3 and ~ x, respectively:

$$\delta = -\frac{F}{k}\left(\frac{x - x_0}{l_{\text{eff}}}\right)^3 + A\left(\frac{x - x_0}{l_{\text{eff}}}\right)^2 \qquad (1)$$

$$\Delta U = -FS\left(\frac{x - x_0}{l_{\text{eff}} - l_g}\right) \qquad (2)$$

with $l_{\text{eff}} = l + x_0$ which accounts for a reduced cantilever bending stiffness owing to a not ideally fixed clamping to the frame and the distance $l_g = 0.1$ mm of the strain gauge with respect to the cantilever clamp. The second term in eq. (1) takes into account cantilever bending caused by a vertical strain gradient.

Figure 4 exhibits the deflection and voltage profiles measured at a calibrated cantilever-type force standard (D4) using a commercial stylus instrument.

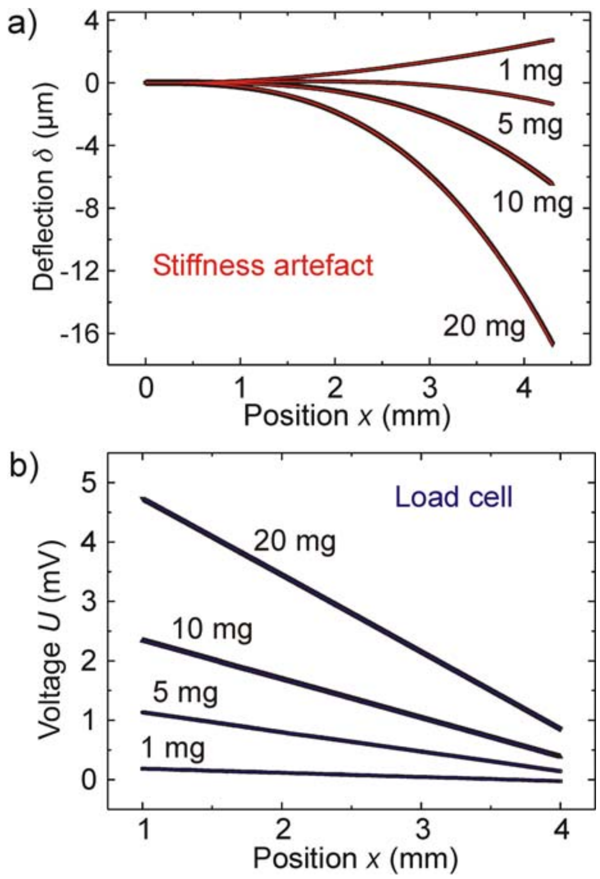

Fig. 4. Deflection (a) and voltage profiles (b) measured with a calibrated cantilever-type force standard (D4) using a stylus instrument

The load setting was varied from 1-20 mg. Excellent agreement with the expectation according to the analytical formulas (eqs. (1) and (2)) is found over the entire scanning range in both figures. For fitting the profiles we use F and x_0 as adjustable parameters in both cases. Fitting of the deflection profile requires A as an additional parameter. The values of F resulting from the fit are shown in Fig. 5 with respect to the setting.

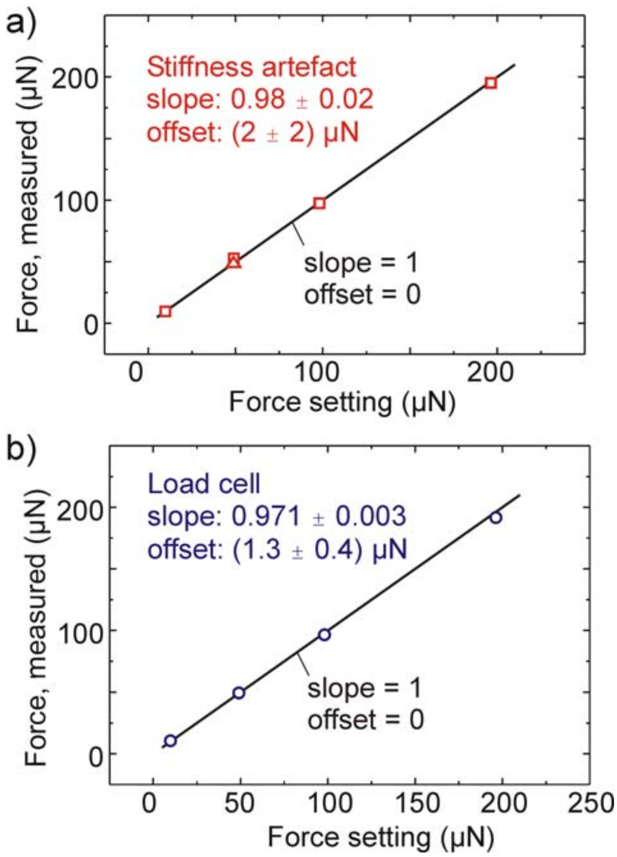

Fig. 5. Measured forces vs. setting forces of a stylus instrument used to profile along a calibrated cantilever-type force standard (D4) . Deflection (a) and voltage read out (b) are analyzed.

We find only small deviations of the measured from setting values of the selected forces as indicated by a comparison with the straight lines representing perfect agreement. The deviation of -(2-3) % compares very well with -4.0 % reported for a indentation machine operated within 50-600 µN [Pra]. Here a potential error in the manufacturer's factory calibration procedures was suggested. In addition we find an offset of 1-2 µN. The non-linearity of the calibration curves determined by the deflection and the voltage read out amounts to 2 % and 0.3 %, respectively.

Using these results the uncertainty of the described calibration methods can be estimated:

$$\Delta F = \sqrt{\left(F_{\text{set}} \times \Delta\text{slope}\right)^2 + \left(\Delta\text{offset}\right)^2} \qquad (3)$$

In the considered force range of 10-200 µN we find uncertainties of 2-4 µN and 0.4-0.8 µN using the deflection and the voltage profile, respectively. In Table 1 the results of both calibration methods are summarized.

Table 1. Paramters of force calibration methods.

Parameter	stiffness artefact	load cell
Range (µN)	10 - 200	10 - 200
Reproducibility (%)	3	0.1 - 0.3
Uncertainty (µN)	2 - 4	0.4 - 0.8

For comparison a second stylus instrument is investigated using the same cantilever force sensor as above (D4). Typical deflection and voltage read out profiles at similar force settings are displayed in Fig. 6. Good agreement with eqs. (1) and (2) is only obtained at small stylus deflection, i.e. $\delta < 1$µm while considerable deviations are visible above. This finding is confirmed by both the deflection (a) and the voltage profiles (b) presented in Fig. 6. We conclude that the probing force decreases at large stylus deflections of more than 1 µm. The setting forces given in the figures are determined by fitting eqs. (1) and (2) to the deflection and voltage profiles, respectively, in the range of $x < 2.5$ µm.

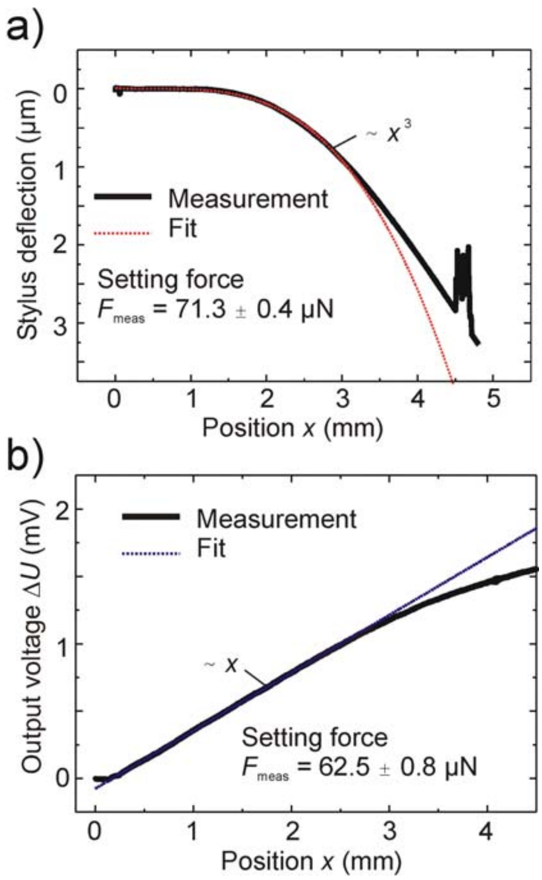

Fig. 6. Deflection (a) and voltage profiles (b) measured with a calibrated cantilever-type force standard (D4) using a stylus instrument

Load-cell output-voltage profiles are measured repeatedly at four different force settings to check the reproducibility of the calibration method with the second stylus instrument. The results of an analysis in the constant-force range of the stylus instrument are given in Table 2 and Fig. 7. They show reproducibilities of 0.2-2 % in a calibration of 6-110 µN which is impaired with respect to the results obtained with the first stylus instrument (cf. Table 1). One setting (1.9) was tested twice, at the beginning and the end of the series of experiments with the second stylus instrument. We find a deviation of almost 20 % between both measurements. Plotting the measured forces against the setting values a non-linear dependence is observed. These results show that force calibration is obligatory before this stylus instrument can be properly used.

Table 2. Force calibration of stylus instrument.

Setting (a.u)	Force (µN)
1.9	52 ± 2
2.5	109.9 ± 0.2
1.0	5.7 ± 0.4
1.5	24 ± 1
1.9	62 ± 2

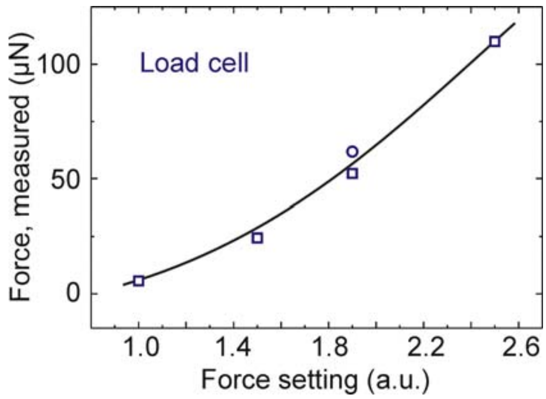

Fig.7. Force applied by the second stylus instrument to a calibrated cantilever-type force standard (D4) operated as a load cell

3. CONCLUSION

In this study traceable measurement of forces involved during stylus profiling was described. Two methods were developed based on a transferable cantilever-type force standards used both as a stiffness artefacts and active load cells. Commercial stylus instruments were calibrated at a reproducibility of few percent and better in the range of up to 200 µN by measuring deflection and load-cell output-voltage profiles. Using the load-cell signal uncertainties well below 1 µN could be achieved.

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