Small Force Metrology for AFM, Stylus Instruments, CMM and Nanoindenter via Reference Springs and Sensors

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Abstract — With the increasing spread of soft polymer products the calibration of probing forces of atomic force microscopes, stylus instruments, coordinate measuring machines and nanoindenters becomes more and more important in order not to scratch the surface of these products during quality control. New sensors and reference springs for force calibration and new calibration methods for these devices will be presented along with some comparison measurements revealing the status of force and stiffness calibration. The contribution closes with an outlook on the current status of probing force standardization.

Index Terms — double spring, meander spring, probing force calibration, reference spring, stiffness calibration.

1. INTRODUCTION

The contribution presents the state of the art and new developments in small force metrology for tactile topography measuring instruments and instrumented indenters based on reference springs and sensors. Atomic force microscopes (AFM), stylus instruments, coordinate measuring machines (CMM) and nanoindenters need the traceable calibration of the applied probing force for reliable topography measurements and mechanical parameter measurements.

2. AVAILABLE METHODS FOR FORCE CALIBRATION

Some of the available methods for force calibration of the above-mentioned instruments will be presented, concentrating on the use of passive reference springs and sensors. The latest developments, a meander type spring with integrated sensor and a topographic AFM spring based on Micro-Electro-Mechanical-Systems (MEMS) will be described in detail.

A. Passive reference springs

Passive reference springs in the shape of cantilevers are available for AFMs, stylus instruments (type FS-C from SiMetricS, Germany) and coordinate measuring machines (CMM). For instrumented indentation testing (IIT) machines and especially for nanoindenters, new silicon based double-springs have been developed (type FS-FD from SiMetricS) which allow not only to calibrate forces up to 500 mN but moreover offer a single calibration point for force and deflection [1]. The only drawback of these standards is the silicon surface, which is not hard enough for indenter radii smaller than 25 μ m [2] (can be improved by a local sheet or layer of a hard material).

B. Micro-force sensors

Two new commercially available micro-force sensors with different sensing principles will be presented, one with capacitive sensing and the option of different tip forms and different force ranges from μ N to mN (Type FT-S from FemtoTools, Switzerland) and the other with piezoresistive sensing, a one square millimeter big probing area and an excellent linearity up to 1 mN [3]. Furthermore, a new topographic sensor for AFM was developed. By simply measuring a surface profile on the artefact, the deflection of the compliant shaft can be measured and from the known stiffness the force can be calculated. Additionally, the device allows to create electrostatic forces which increase the stiffness of the artefact in order to measure higher forces up to some ten micro Newtons.

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3. TRACEABLE STIFFNESS CALIBRATION

PTB's primary small force standard with a force resolution of 1.4 pN showed a stiffness uncertainty in AFM cantilever calibration of 0.6 % [4]. Standard traceable stiffness calibration of reference springs with uncertainties down to 4 % is provided by PTB using compensation balances. Two new approaches will be presented.

A. Compensation balance, nanopositioner and laser interferometer

To the standard compensation balance approach a laser interferometer is added, reducing the uncertainty level for stiffness and force to below 1 % [5].

B. Calibrated MEMS and nanopositioner

The second approach is based on a precisely stiffness calibrated MEMS actuators and is aiming at uncertainty levels of less than 4 %, especially for the calibration of the stiffness of AFM cantilevers used for high precision force spectroscopy.

4. COMPARISON MEASUREMENTS

The latest comparison measurements will be presented to demonstrate the state of the art in measurement uncertainty in the different fields. Comparison measurements are available for piezo-resistive force sensors [6], passive silicon reference springs for nanoindenters [2], commercially available capacitive force sensors [7] and AFM cantilevers [5].

5. STANDARDIZATION

For stylus instruments passive reference springs or balances are standardized in Germany [8]. For AFM cantilever spring constant calibration, a lot of different methods are described in ISO 11779. For nanoindenters either a calibrated force sensor, calibrated masses and a lever arm system or using electronic balances are standardized.

6. CONCLUSION

New sensors, passive reference springs for the force calibration of different tactile instruments and nanoindenters including traceable instrumentation for the calibration of these devices have been presented.

REFERENCES

- 1. Frühauf, J.; Gärtner, E.; Herrmann, K.; Menelao, F.; Schwenk, D.; Chudoba, T.; Vollmar, H.-P. Calibration of instruments for hardness testing by use of a standard. *Recent Adv. Theory Pract. Hardness Meas. HARDMEKO* **2007**, 141–145.
- Brand, U.; Chudoba, T.; Griepentrog, M.; Schwenk, D.; Bosch, G.; Scheerer, H.; Gärtner, E. Round robin for testing instrumented indenters with silicon reference springs. *Int. J. Mater. Res.* 2015, *106*, 1215–1223.
- 3. Hamdana, G.; Bertke, M.; Doering, L.; Frank, T.; Brand, U.; Wasisto, H.S.; Peiner, E. Transferable micromachined piezoresistive force sensor with integrated double-meander-spring system. *J. Sens. Sens. Syst.* **2017**, *6*, 121–133.
- Nesterov, V.; Belai, O.; Nies, D.; Buetefisch, S.; Mueller, M.; Ahbe, T.; Naparty, D.; Popadic, R.; Wolff, H. SI-traceable determination of the spring constant of a soft cantilever using the nanonewton force facility based on electrostatic methods. *Metrologia* 2016, *53*, 1031–1044.
- 5. Kühnel, M. National comparison of spring constant measurements of atomic force microscope cantilevers. In Proceedings of the IMEKO TC3, TC5 and TC22 International Conference 2017; 2017.
- 6. Kim, M.-S.; Pratt, J.R.; Brand, U.; Jones, C.W. Report on the first international comparison of small force facilities: a pilot study at the micronewton level. *Metrologia* **2012**, *49*, 70–81.
- 7. Marti, K.; Wuethrich, C.; Russi, S.; Brand, U.; Li, Z. Micro-force measurements: a new instrument at METAS. *Be Publ. MST* **2020**.
- 8. DIN 32567-3:2014-10 Production equipment for microsystems Determination of the influence of materials on the optical and tactile dimensional metrology Derivation of correction values for tactile measuring devices; 2014;

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