Tutorial of the EMRP-Project MechProNO:

Traceable measurement of mechanical properties of nano-objects

24th March 2015

Technical University Dresden, Institute of Process Engineering and Environmental Technology. Research Group Mechanical Process Engineering
Merkel-Bau, Zi. 118, Helmholtzstrasse 14, Dresden

9:00	**Welcome** Prof. M. Stintz, Head of the Research Group Mechanical Process Engineering, TU Dresden Uwe Brand, Coordinator of MechProNO, PTB, Braunschweig
	Fabrication of nano-objects
9:10	**Preparation methods for nano-objects on substrates** Petra Fiala, Michael Stintz, TU Dresden
9:50	**Combining nano-object fabrication and force-measurements in a FIB/SEM System** Nicole Wollschläger, Werner Österle, BAM, Berlin and Uwe Brand, Sai Gao, PTB, Braunschweig
10:30	*Coffee Break*
	Measurement of dimensions and mechanical properties
10:45	**Investigation of mechanical properties of nano-pillars by indentation techniques** Michael Griepentrog, Philip Reinstädt, BAM, Berlin
11:25	**Metrological atomic force microscopy for the measurement of nanostructures** Andrew Yacoot, Giovanni Mattia Lazzerini, NPL, GB
12:05	**Humidity controlled AFM** Jeremias Seppä, Virpi Korpelainen, MIKES, Finland
12:45	*Lunch*
	Traceable AFM Cantilever Stiffness Calibration
13:45	**Traceable cantilever stiffness calibration method using a MEMS nano-force transducer** Sai Gao, Uwe Brand, PTB, Braunschweig
14:25	**MEMS technology for fabrication of electrostatic actuators and application examples** Karla Hiller, Susann Hahn, Thomas Gessner, TU Chemnitz
	Modelling
15:05	**Atomistic simulations of AFM indentation of Gold nanorods** Bernhard Reischl, Antti Kuronen, Kai Nordlund, Hannu Husu, Virpi Korpelainen, Jeremias Seppä, and Antti Lassila, MIKES, Finland
15:45	**The finite element method** Radek Šlesinger, CMI, Brno
16:25	*Summary, Good Practice Guides and final discussion*
16:45	**End**

Please register for the workshop by sending a brief email to the organisation team:

Petra.Fiala@tu-dresden.de

There is **no** registration fee for the workshop.

Traceable measurement of mechanical properties of nano-objects

The aim of the EMRP project “Traceable measurement of mechanical properties of nano objects” (MechProNO) is to develop and improve methods to measure the mechanical properties of nano-objects traceable to the SI units. Eight partners from European National Metrology Institutes, designated institutes and research institutes cooperated in a joint project to develop corresponding solutions.

The basic idea was to use Atomic Force Microscopy (AFM) for the characterization of the dimensional and mechanical properties of nano-objects.

Different nano-objects have been created and investigated. The Research Group Mechanical Process Engineering of the Institute of Process Engineering and Environmental Technology of Technical University Dresden (TUD) has developed methods to prepare a large variety of nano-objects on substrates. The Federal Institute for Materials Research and Testing **(**BAM) in Berlin has developed a method using the Focussed Ion-Beam (FIB) to create nano-beams from Si-Nitride, Si and SiO_2 membranes. They used a commercially available force measuring system inside the FIB to measure the stiffness of these beams. Nano-pillars were fabricated from Silicon and a photoresist. Indentation measurements on the pillars were carried out and resulted in a measured elastic indentation modulus which was 50 % smaller than the bulk elastic modulus [1] by using typical analysis procedures! However, it was found, that if the compressibility of the pillars is taken into account the resulting elastic modulus of the nano-pillars was still very well comparable to the typical bulk value.

Two different AFMs have been developed in MechProNO. The National Physical Laboratory (NPL) in Teddington has developed a metrological AFM for high-precision dimensional measurements of nano-objects. When modelling the mechanical properties of nano-objects accurate dimensions of the objects are necessary. Results can be affected by AFM scan parameters or by the shape of the AFM tip itself. The tutorial will explore in more detail how to determine the shape of tips from AFM images using the blind tip-reconstruction routines and how to use that information to remove the effect of tip shape partially to receive a more accurate value of the shape of the nano-object.

Humidity of the ambient air could affect dimensional and mechanical measurements of nano-objects, too. The thickness of the water layer covering the sample surface depends on the ambient humidity and sample properties. This water layer naturally affects the AFM probing of sample properties. To characterize these effects experimentally, an humidity controlled AFM chamber has been designed and built at MIKES. The principles and performance of the humidity controlled AFM will be given. Furthermore, the latest progress with e.g. experimental characterization of tip-sample adhesion forces at different humidities at MIKES will be reported.

Reliable force measurements with AFMs are only possible if the measurement of the normal stiffness of cantilever is improved, i.e. more accurate. We present an approach based on the well-known Thermal Noise Method in combination with reference cantilevers with accurate calibrated stiffness (by PTB). PTB will show its new approach to calibrate stiffness by using a calibrated MEMS reference spring actuators. The design and fabrication of these MEMS devices is presented by TU Chemnitz.

The tutorial will also show advantages of two modelling approaches for the mechanical properties of nano-objects to achieve a better understanding of nano-objects: the Finite Element Analysis (FEA) and the Molecular Dynamic Calculation (MDC).

[1] Li Zhi, Gao Sai, Pohlenz Frank, Brand Uwe, Koenders Ludger, Peiner Erwin: Determination of the mechanical properties of nano-pillars using the nanoindentation technique. Nanotechnology and Precision Engineering 3 (2014), 182 - 188

Preparation methods for nano-objects on substrates

Michael Stintz, Petra Fiala

Technical University Dresden, Institute of Process Engineering and Environmental Technology. Research Group Mechanical Process Engineering

The presentation includes a compilation of all investigations regarding to preparation of nano-objects on substrates. It contains an overview of selected materials and substrates, depicts possibilities for size and zeta potential evaluation and characterisation of materials using measurement techniques and describes procedures for substrate cleaning as wet chemical cleaning and dry cleaning methods. Approved preparation methods as drying, rinsing, dip coating and electrostatic precipitation be explained and applied to preparation of selected materials on substrates (60 nm gold spheres, 25x66/77 nm gold nanorods, 100 nm and 304 nm SiO_2 spheres, 50 nm silver spheres, 60 nm diameter silver nanowires, TiO_2 spheres < 150 nm) and their fitness is evaluated. There are formulated guidelines detailing optimised preparation methods.

Combining Nano-object Fabrication and Force-measurements in a FIB/SEM System

Nicole Wollschläger, Werner Österle, BAM, Berlin

Uwe Brand, Sai Gao, PTB, Braunschweig

The smaller, the stiffer! Since this hypothesis came up, lots of scientists tried to confirm or to disclaim this prediction. Up to now no clear result was obtained, because both the fabrication and the mechanical testing of nano-objects are challenging tasks with numerous uncertainties which cannot be fully assessed yet.

Here we present a process combining the fabrication of nano-objects and the mechanical testing in one machine equipped with a focused ion beam and an electron beam. The nano-objects were cut out from freestanding and commercially available silicon nitride membranes with the help of focused Gallium ions in a vacuum chamber. Beams of different dimensions were then deformed elastically while measuring the applied force with a Kleindiek micromanipulator equipped with a force sensor. Using the SEM column of the dual beam instrument (Fei Quanta 3D), the experiment could be observed live at the screen. Finally, force-displacement curves were obtained by combining force and image data. The results will be compared with the other methods available within MechProNO, such as Instrumented Indentation (IIT) and Scanning Force Microscopy (AFM).

Investigation of mechanical properties of nano-pillars by indentation techniques

M. Griepentrog, P. Reinstädt

BAM Federal Institute for Materials Research and Testing
Division 6.7 Surface Modification and Measurement Technique
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A methodology for performing uniaxial compression tests on nano-pillars is presented. Samples are tested in uniaxial compression using a nanoindentation device equipped with a flat tip. The obtained stress–strain curve can be used to examine the plastic response of samples of different sizes that are from the same bulk material.
Using the example of investigations of silicon nano-pillars in the EMRP project MechProNO main aspects of this methodology are described and discussed in detail.

These main aspects are:
- Characterization of shape and size of the pillars before and after the test with different methods
- Definition of the exact position of the indent using high resolution imaging methods
- Choice and characterization of the indenter to be used
- Definition of the test cycle
- Evaluation and correction of the test results using different model conceptions

Metrological Atomic Force Microscopy for the measurement of nanostructures

Andrew Yacoot, Giovanni Mattia Lazzerini
National Physical Laboratory, Hampton Road, Teddington Middlesex TW11 0LW

Optical interferometry is the main route used to achieve traceability for dimensional metrology via the wavelength of frequency-stabilized helium neon lasers. Atomic force microscopy provides a window on the nano world, but AFM instruments are not directly traceable. Over the last few years many national metrology institutes have developed so-called metrological AFMs with optical interferometers to traceably measure the relative movement between the AFM cantilever and the sample. These instruments can be used to calibrate both transfer standards for conventional AFMs and traceably measure, with low uncertainty, samples for research purposes. The latter use is playing a more important role in supporting the rapidly growing area of nanotechnology. In this workshop we will describe the operating principle of metrological AFMs and show how they have been used in the EMRP project MechProNO for accurately measuring the size of nanoparticles. We will illustrate some of the pitfalls that can occur when trying to obtain quantitative data from AFM images. Results can be affected by AFM scan parameters or by the shape of the AFM tip. We will finally explore how to determine the shape of the tip from AFM images using the blind tip reconstruction routines and how to use that information to partially remove the effect of tip shape from AFM measurements.

Humidity controlled AFM

Jeremias Seppä, Virpi Korpelainen

MIKES Metrology, VTT Oy, Espoo, Finland

Humidity of the ambient air affects dimensional and mechanical measurements of nano-objects. The thickness of the layer of water molecules surrounding the sample surface depends on the ambient humidity and the sample properties. This water layer naturally affects the probing of sample properties using e.g. AFM. To experimentally characterize these effects, a humidity controlled AFM has been designed and built at MIKES. The principles and performance of the humidity controlled AFM are described. Furthermore, the latest progress with e.g. experimental characterization of tip-sample adhesion forces at different humidities at MIKES is reported.

Traceable cantilever stiffness calibration method using a MEMS nano-force transducer

Sai Gao[1], Uwe Brand[1], Wolfgang Engl[2], Thomas Sulzbach[2]

[1]Physikalisch-Technische Bundesanstalt, 38116 Braunschweig

[2]Nanoworld Services GmbH, Erlangen

Quantitative nano-mechanical measurements using atomic force microscopy (AFM) require that the mechanical performance of AFM cantilevers in use, especially their normal spring constant, be carefully calibrated. At PTB a reference spring method based on a well-developed MEMS nano-force transducer has been realized. With the well calibrated MEMS nano-force transducer, AFM cantilevers with stiffnesses ranging from 0.1 N/m to 50 N/m can be traceable calibrated with low measurement uncertainty. In the workshop we will describe the details of the method:

- the MEMS design, MEMS actuation and displacement sensing system
- the stiffness calibration of the MEMS using a high precision compensation balance
- the AFM cantilever normal stiffness calibration system

With this method three kinds of cantilevers from the Nanoworld GmbH (Erlangen, Germany) with stiffnesses of about 0.5 N/m, 2.8 N/m and 5 N/m have been calibrated. The calibration results are compared with that of the thermal noise method applied by NanoWorld Services GmbH.

MEMS technology for fabrication of electrostatic actuators and application examples

Karla Hiller, Susann Hahn, Thomas Gessner

Technische Universität Chemnitz

In this paper we present the MEMS technology based on the bonding and deep reactive etching (BDRIE) method, which can be widely used especially for MEMS with in-plane motion direction (1D or 2) and preferably applying the electrostatic drive and capacitive detection. Examples for fabrication of actuators for measurement tips, e.g. in a SPM head, will be shown. As these MEMS need access to the moveable part from one or more sides, the separation of such wafers is a challenging task. Result of our specific method for separation will be shown and discussed. New approaches for protecting such chips from damage by applying a cover wafer will be introduced. Furthermore it will be demonstrated that this technology platform is also open for a large variety of other applications. By adding additional process steps and materials, new drive and detection methods, such as thermal actuators and piezoresistive detectors, may be involved.

Atomistic Simulations of AFM Indentation of Gold Nanorods

Bernhard Reischl[1], Antti Kuronen[2], Kai Nordlund[2],
Hannu Husu[3], Virpi Korpelainen[3], Jeremias Seppä[3], and Antti Lassila[3]

[1] Nanochemistry Research institute, Curtin University, Perth, Western Australia,
[2] Department of Physics, University of Helsinki, Finland,
[3] MIKES Metrology, VTT Oy, Espoo, Finland

Mechanical properties of nanoscale objects can differ significantly from those of macroscopic objects of the same material, leading to many opportunities for new applications in nanotechnology. However, the measurement of these properties at the nanoscale remains in itself a significant challenge.

We performed large-scale atomistic molecular dynamics simulations of AFM indentation experiments on gold nanorods with diamond AFM tips. Emphasis was placed on matching the experimental size and shape of both the nano-objects and the AFM tip apex, and including the substrate. These simulations give insight into the atomic scale processes happening during indentation: dislocation creation, migration, and subsequent annihilation at a free surface, leading to plastic deformation of the sample.

The finite element method

Radek Šlesinger

Czech Metrology Institute, Brno, Czech Republic

The finite element method (FEM) is one of the most common and developed tools for numerical solution of partial differential equations. After a slightly theoretical introduction to its principles, we will discuss the basic components of a free software environment for the complete finite element modelling process.
Using FEniCS and SfePy software packages, we will demonstrate use of the method on solving mechanical problems relevant in MechProNO project: beam bending, nanorods indentation, and surface roughness effects in nanoindentation.

Hotels in Dresden

City centre:
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http://www.ibis-dresden.de/

Close to University:
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