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Thursday 20.4.

10:00 – 10:15		*Welcome*
10:15 – 10:45	**Bruno de la Torre**	**Real-space imaging σ-holes and π-holes with Kelvin Force Probe Microscopy**
10:45 – 11:00	Martin Švec	Light, tips and molecules: SPM on the path to direct nano-optical measurements
11:00 – 11:15	Bohuslav Rezek	AFM and simulations of 2D/3D molecular assembly via ZnO dipoles
11:15 – 11:30	Martin Setvín	Tracking single polarons by STM/AFM
11:30 – 11:45	Pavel Kocán	Tunneling of single charges to Transition Metal Oxides – kinetic Monte Carlo Simulations
11:45 – 12:00	Pavel Jelínek	Nickelocene molecule as SPM magnetic sensor
12:00 – 12:15	Llorenç Albons Caldentey	Surface characterization of the ferroelectric perovskite $BaTiO_3$ by noncontact AFM
12:15 – 12:30	Jakub Horák	Chemistry at the nanoscale – AFM meets IR Spectroscopy (company presentation)
12:30 – 14:00		*Lunch*
14:00 – 14:30	**Andrew Yacoot**	**Traceability for SPM through the lattice parameter of silicon and bringing metrology to high speed AFM**
14:30 – 14:45	David Nečas	You are measuring it wrong again
14:45 – 15:00	Gaoliang Dai	Overview of 3D nanometrology at the PTB based on the SPM and TEM techniques
15:00 – 15:15	Ján Šoltýs	MFM tip with a ferromagnetic disk-shaped apex
15:15 – 15:30	Viktor Witkovský	On computing GUM-compliant uncertainty matrix for the parameters of the specific nonlinear EIV models
15:30 – 15:45	Matěj Hývl	Contact Force in Current-Detecting Atomic Force Microscopy – Lessons for C-AFM Tomography in Photovoltaic Research
15:45 – 16:00	Swarnendu Banerjee	3D Tomography on advanced photovoltaic (PV) structures – Examples of good practice
16:00 – 16:15	Šárka Kučerová	Piezo Force Microscopy as a powerful tool to investigate the polarity of crystallites in ZnO seed layers
16:15 – 16:30	Karel Šec	FTIR-SNOM spectroscopy and imaging with single widely tunable laser (company presentation)
16:30 – 17:00		*Coffee break*
17:00 – 17:30	**Małgorzata Lekka**	**The use of AFM to detect cancer-related changes in cells**
17:30 – 17:45	David Rutherford	Correlative Atomic Force Microscopy and Scanning Electron Microscopy analysis of a Bacteria-Diamond-Metal nanocomposite
17:45 – 18:00	Jan Přibyl	Atomic Force Microscopy in Biological Research
18:00 – 18:15	Šimon Klimovič	Novel uses of Atomic Force Microscopy (AFM) to study contractile properties of cardiac cells.
18:15 – 18:30	Radka Obořilová	Investigation of microbial lysis on the sensor surfaces
18:30 – 18:45	Naďa Labajová	Clostridioides difficile DivIVA protein-lipid interactions study by AFM and Cryo-EM
18:45 – 19:00	Vladimíra Tomečková	Graphical analysis of tear fluid by using atomic force microscopy
19:45 – 23:00		*Social evening*

Friday 21.4.

9:00 – 9:30	**Virpi Korpelainen**	**MetExSPM project: Development of traceable methods for high speed and large range SPM**
9:30 – 9:45	Jan Thiesler	A novel high-dynamic large-range hybrid SPM scanner
9:45 – 10:00	Dominik Badura	Calibration of active piezoresistive cantilevers
10:00 – 10:15	Petr Klapetek	GwyScope: open hardware playground for adaptive SPM scanning
10:15 – 10:30	Ondřej Novotný	New generation of AFM in SEM – company presentation
10:30 – 10:45	Jaroslav Kuliček	AFM/photo-KPFM/micro-Raman correlation on G/h-BN edges
10:45 – 11:00	Marek Černík	Correlative Raman-AFM Imaging – Techniques and Applications (company presentation)
11:00 – 11:30		*Coffee break*
11:30 – 11:45	Egor Ukraintsev	Non-contact non-resonant atomic force microscopy method for measurements of highly mobile molecules and nanoparticles
11:45 – 12:00	Bartosz Pruchnik ...	Scanning thermal microscope with transformer brigde input electronics
12:00 – 12:15	Wiktor Połacik	Atomic force microscopy in single-specimen measurements of nanowires
12:15 – 12:30	Daniel Haško	AFM and 3D optical microscopy study of ablation craters created by laser-induced breakdown spectroscopy
12:30 – 12:45	Dušan Novotný	Měřicí technika Morava s.r.o. (company presentation)
12:45 – 14:00		*End of the workshop, lunch*

Thu 10:15 – 10:45 **Bruno de la Torre** `bruno.de@upol.cz`

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Real-space imaging σ-holes and π-holes with Kelvin Force Probe Microscopy

Noncovalent interactions between molecules play an important role in supramolecular chemistry, molecular biology, and materials science. For instance, natural machinery often has a significant non-covalent component (e.g. protein folding, recognition) and rational interference in such 'living' devices can have pharmacological implications. For the rational design/tweaking of supramolecular systems it is helpful to understand the forces that make these systems stick to one another.
In this contribution, I will focus on the real-space visualization of the σ-hole [1] and π-hole [2]. By a set of Atomic Force Microscopy (AFM), Kelvin Probe Force Microscopy (KPFM), and Scanning Tunneling Microscopy experiments we discriminate the emerge of both σ-hole and π-hole on a single molecule with unprecedented submolecular resolution. Our findings are fully supported by an atomistic model obtained from DFT calculations which allow us to simulate both AFM and KPFM images. These results may potentially open a new way to characterize biological and chemical systems in which anisotropic charges play a decisive role.

[1] B. Mallada et al. Science 2021, 374, 863– 867

[2] B. Mallada et al. In preparation

Thu 10:45 – 11:00 **Martin Švec** `svec@fzu.cz`

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J. Doležal[a], R. C. de Campos Ferreira[a], S. Canola[a], P. Hapala[a], P. Merino[b], M. Švec[a]

Light, tips and molecules: SPM on the path to direct nano-optical measurements

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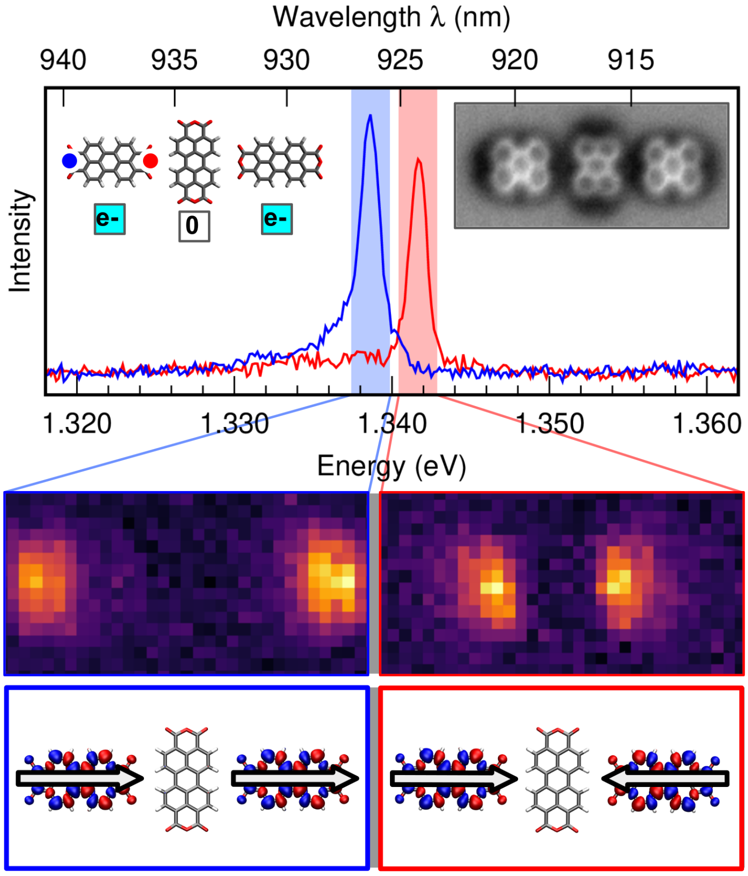
Exploration of essential photophysical phenomena at the level of individual quantum objects requires highly specialized optical spectroscopies working at the very limit of sensitivity or implies the use of plasmonic nanostructures to overcome the fundamental resolution limits achievable with visible and infrared light. The recent developments emerging in the field of Scanning Probe Light Microscopy bring the unique opportunity to pursue intriguing, often hard-to-access interactions of light and matter at the scale of single molecules and beyond, with the finesse characteristic for the technique. The plasmonic nanocavity formed naturally between the tip and sample enables vast enhancement of the light coupling to the studied systems, and visualization of the fascinating photophysics within nanoscopic emitters. The modes of operation can be switched between the electroluminescence, photoluminescence and tip-enhanced Raman spectroscopy. Complemented with the today standard toolbox of cryogenic STM, STS and AFM provides a reliable support in questions of the system geometry and electronic configuration[1]. As the emitted photons and their spectra carry a rich information about the eigenmodes, charges, vibronics and temporal evolution of the transient states, we can build a detailed picture of the cycles comprising the process of single-photon generation[2], relevant energy conversion mechanisms, quasiparticle delocalization over excitonic nanoclusters[3], interaction with electrical fields and vibronic coupling to electronic transitions[4].



Exciton delocalization in a PTCDA molecular cluster

References:

[1] Charge carrier injection electroluminescence with CO functionalized tips on single molecular emitters. J Doležal, P Merino, J Redondo, L Ondič, A Cahlík and M Švec, Nano Lett. 19, 8605-861 (2019)

[2] Exciton-Trion Conversion Dynamics in a Single Molecule. J Doležal, S Canola, P Merino and M Švec, ACS Nano 15, 7694–7699 (2021)

[3] Real space visualization of entangled excitonic states in charged molecular assemblies. J Doležal, S Canola, P Hapala, R. Ferreira, P Merino and M Švec, ACS Nano 16, 1082-1088 (2022)

[4] Evidence of trion-libron coupling in chirally adsorbed single molecules. J Doležal, S Canola, P Hapala, RCC Ferreira, P Merino, M Švec, Nature Comm. 13, 6008 (2022)

Thu 11:00 – 11:15 **Bohuslav Rezek** `rezekboh@fel.cvut.cz`

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AFM and simulations of 2D/3D molecular assembly via ZnO dipoles

The impact of surface polarity of different ZnO facets on thiorphan adsorption is studied both experimentally by atomic force microscopy and theoretically by force field molecular dynamics (FFMD) and density functional tight binding simulations (DFTB) [10.1021/acs.langmuir.2c02393]. Polar ZnO surfaces cause formation of thiorphan nanodots, where size of nanodots depends on the direction of dipoles: small (4 nm) nanodots are formed of Zn-faced ZnO while large (25 nm) nanodots are formed on O-faced ZnO. Non-polar ZnO surfaces cause self-assembly into layered nanoislands with characteristic 4 nm layer thickness, which subsequently merge into rigid nanolayers. The self-assembly is shown to be controlled solely by the effect of surface dipoles electric field orientation and magnitude whereas effects of surface chemistry or solution are negligible. The results thus also show a way for controlling assembly of thiorphan and other molecular nanomaterials for diverse applications.

Thu 11:15 – 11:30 **Martin Setvín** `martin.setvin@mff.cuni.cz`

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J. Redondo, I. Sokolovic, D. Wrana, A. Alexander, L. Albons, P. Kocan, M. Setvin

Tracking single polarons by STM/AFM

Polarons [1] are quasiparticles created by the coupling of excess electrons or holes with lattice distortions in ionic crystals: Electron-phonon interactions lead to a partial or complete localization of the charge carrier. Even though the concept is known for almost a century, polarons attract increasing attention due to their impact on materials properties, such as electrical transport, optical properties, surface reactivity or magnetoresistance. This talk will demonstrate the possibility to probe and manipulate single polarons by combined scanning tunnelling microscopy/noncontact atomic force microscopy. The methodology will be illustrated on photoexcited hole-polarons on bulk-terminated $SrTiO_3$ (001) and on tip-induced electron- and hole-polarons on Fe_2O_3 (1-102).
References:

[1] Franchini, C., Reticcioli, M., Setvin, M., Diebold, U. Polarons in Materials. Nature Reviews Materials 6, 560-586 (2021).

Thu 11:30 – 11:45 **Pavel Kocán** `pavel.kocan@mff.cuni.cz`

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P. Kocan, V. Gabriel, J. Redondo, D. Wrana, A. Alexander, L. Albons, M. Setvin

Tunneling of single charges to Transition Metal Oxides – kinetic Monte Carlo Simulations

Electrons or holes stabilized in potential wells created by the distortion of the surrounding lattice of a polarizable material are known as polarons [1]. These quasiparticles are responsible for the electrical conductivity [2] of dielectrics, which is crucial for the chemical activity of photo- or electrocatalysts [3]. Transition metal oxides, such as hematite ($\alpha - Fe_2O_3$) or potassium tantalate (KTaO3) studied here, are materials with promising photoelectrochemical activity towards the water-splitting reactions [4,5].
It will be shown that electrons and holes can be injected into the Fe_2O_3(1-102) or the $KTaO_3$(001) surface by the Q-Plus sensor with single-charge precision. Here we will focus on the tunneling process and the kinetics of the injected charges in the form of polarons, interacting electrostatically with each other and with the field of the tip. We have developed a kinetic Monte Carlo (KMC) model consistent with experimental data. In the presentation we will provide a strategy to use the KMC simulations to extract information about the polaronic injection and diffusion from experimental data on a single-polaron level.

This work was supported by the Czech Science Foundation (GACR 20-21727X)
and by GAUK/Primus/20/SCI/009.

References:

[1] C. Franchini, M. Reticcioli, M. Setvin, U. Diebold, Nat. Rev. Mater. 0123456789 (2021).

[2] K. M. Rosso, D. M. A. Smith, M. Dupuis, J. Chem. Phys. 118, 6455–6466 (2003).

[3] C. Lohaus, A. Klein, W. Jaegermann, Nat. Commun. 9, 1–7 (2018).

[4] C. Li, Z. Luo, T. Wang, J. Gong, Adv. Mater. 30, (2018).

[5] E. Grabowska, Applied Catalysis B: Environmenta, 186, 97 (2016).

Thu 11:45 – 12:00 **Pavel Jelínek** `jelinekp@fzu.cz`

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Andrés Pinar Solé, Oleksandr Stetsovych, Manish Kumar, Diego Soler Polo, Pavel Jelínek

Nickelocene molecule as SPM magnetic sensor

The invention of tip functionalization with a single molecule [1] enabled scanning probe microscopy to achieve unprecedented spatial resolution, including the chemical resolution of single molecules [2] or imaging of anisotropic charge distribution on a single atom [3].
Recently, it has been demonstrated that probe functionalization with a single nickelocene molecule enables us to detect spin states of individual atoms [4] and molecules [5]. Here, as a magnetic sensor, we used a nickelocene molecule ($NiCp_2$), consisting of a Ni atom sandwiched between two cyclopentadienyl rings. As an S=1 system, it presents magnetic-induced spectral features due to the inelastic electron spin-flip.
In this presentation, we will present several examples of measurements with nickelocene tips as well as a simulation tool kit that enables us to interpret experimental spectra.

References

[1] P. Jelinek JPCM, 29, 343002 (2017)

[2] L. Gross et al, Science 324, 1428 (2009)

[3] B. Mallada et al, Science 374, 863 (2021)

[4] B. Verlhac et al., Science 366, 623–627 (2019)

[5] Ch. Wackerlin et al, ACS Nano 16, 16402(2022)

Thu 12:00 – 12:15 **Llorenç Albons Caldentey** `llorensalbonscaldentey@gmail.com`

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Llorenç Albons, Dominik Wrana, Igor Sokolovic, Aji Alexander, Martin Setvin

Surface characterization of the ferroelectric perovskite $BaTiO_3$ by noncontact AFM

Characterization of $BaTiO_3$ is an interesting field due to its ferroelectricity and surface chemistry. Specifically, ferroelectric polarization may be used to separate and trap charges at the surface of the crystal, as well as to alter the surface chemistry. In this work we show that we can prepare a pristine $BaTiO_3$ surface by cleaving in UHV and characterize it with atomic resolution using the qPlus nc-AFM/STM. In addition, we demonstrate a reversible ferroelectric polarization of the material at the atomic scale, by application of the tip-sample bias voltage.

Thu 12:15 – 12:30 **Jakub Horák** `jakub.horak@mt-m.eu`

Měřící technika Morava, s.r.o

Chemistry at the nanoscale – AFM meets IR Spectroscopy (company presentation)

Thu 14:00 – 14:30 **Andrew Yacoot** `andrew.yacoot@npl.co.uk`

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Traceability for SPM through the lattice parameter of silicon and bringing metrology to high speed AFM

The 2019 revision to the International System of units (SI) included a secondary realisation of the metre based on the lattice parameter of silicon. The talk will explain how this can be used to provide a traceability route for height measurements using scanning probe microscopes. Algorithms developed for the processing of atomic step height measurements will also be described.
In recent years there has been considerable developments in the area of high-speed atomic force microscopes as an imaging technique. This talk will show the NPL metrological high-speed AFM that has an average scanning of 9 mm/second and can be used for quantitative measurements of nanoscale structures.

Thu 14:30 – 14:45 **David Nečas** `David.Necas@ceitec.vutbr.cz`

CEITEC Brno University of Technology
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You are measuring it wrong again

Choosing correctly the interval/set of independent variable values is crucial for reaching reasonable accuracy in many measurements. In scanning probe microscopy the theoretically good choices are frequently impractical as scanning a tiny and more or less arbitrary part of the sample surface may be the only option we really have. Repetition and statistical analysis helps to establish representativeness, but is not a panacea. In particular, if we consistently (and misguidedly) choose to measure regions which provide too little or skewed information about the measurand, there is only so much repetition can do. And, unfortunately, a region on the sample which 'feels right' to measure intuitively does not automatically mean an actually good choice. This contribution demonstrates the problem in a few selected examples where a good measurement strategy can either be seen as rather counterintuitive or – even when it can be perhaps guessed correctly – one would not guess the explanation why it works so much better than the alternatives. We focus mainly on the analysis of two dimensional measurements, both important for nanometrology. One is the height of mono atomic steps on silicon, now adopted as a secondary realisation of metre. The other is a lateral measurement of a grating period/pitch.

Thu 14:45 – 15:00 **Gaoliang Dai** `gaoliang.dai@ptb.de`

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Gaoliang Dai, Jan Thiesler, Johannes Degenhardt, Kai Hahm, Rainer Tutsch

Overview of 3D nanometrology at the PTB based on the SPM and TEM techniques

True-3D metrology of complex micro- and nanostructures becomes increasingly important for advanced manufacturing, for instance, the development of next generation integrated circuits (IC) with gate-all-around field-effect transistors (GAAFET) and/or 3D stacked (3D-SIC) architectures. This paper aims to give an overview on the recent research progress achieved at the PTB for traceable 3D nanometrology. Several fundamental aspects are focused: novel true-3D probing sensor, measurement strategy, tip geometry characterisation and issues of structure/tip deformation.

PTB has developed a novel and patented true-3D AFM probe, referred to as 3D-Nanoprobe. Such a probe can be realized by introducing flexure hinge structures to the cantilever of a conventional CD-AFM probe. It has quasi-isotropic stiffness in three directions and is thus more powerful for detecting 3D tip-sample interaction forces in AFM measurements.

AFM measurements are typically realized by scanning a tip/sample with a quasi-constant speed along lateral axes while keeping the tip-sample distance constant using a servo controller along the z axis. This measurement strategy is, however, no more appropriate for true-3D measurements. To overcome this problem, PTB has developed a new measurement strategy referred to as the vector approach probing (VAP) strategy, where the tip probes the structure surfaces along their normal direction point-by-point. The VAP strategy offers advantages such as higher 3D probing sensitivity, more measurement flexibility and lower tip wear.

From the morphological point of view, the measured image by an AFM tip is the dilated result of the measured structures by the geometry of its tip. Consequently, the tip geometry is usually (one of) the most important error sources in true-3D metrology. To overcome this problem, PTB has developed a new method for characterizing and correcting AFM tip geometry based on a bottom-up traceability approach with a measurement uncertainty of approx. 1 nm.

AFM measurements are based on tip-sample interaction forces, which will inevitably result in structure/tip deformation and thus impact measurement results. Our recent investigation indicates that such deformation could be a significant error source, particularly, in measurements of thin and tall structures and/or using sharp and tall tips.

Detailed research concepts, implementation and results will be introduced in the talk.

Thu 15:00 – 15:15 **Ján Šoltýs** `jan.soltys@savba.sk`

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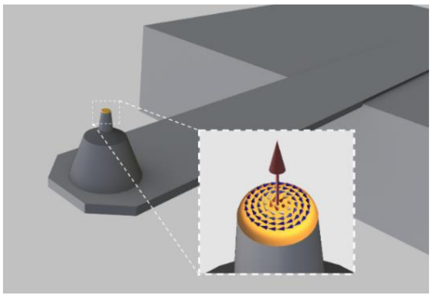
Ján Šoltýs, Iuliia Vetrova, Juraj Feilhauer, Sergei Krylov, Ján Fedor, Vladimír Cambel

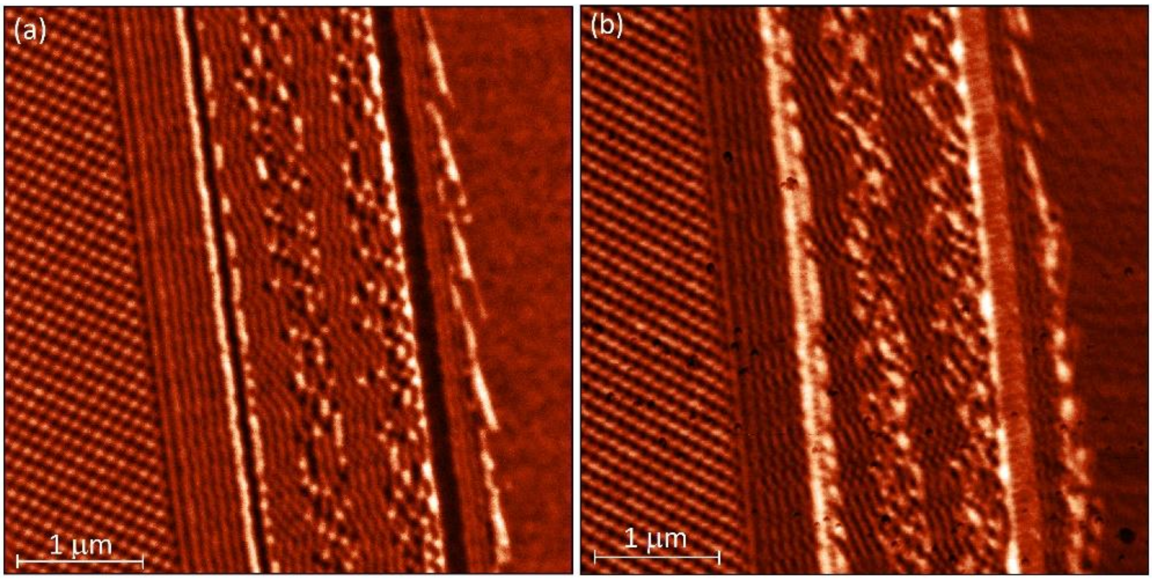
MFM tip with a ferromagnetic disk-shaped apex

We have developed magnetic force microscopy tip with a ferromagnetic disk-shaped apex (FMD tip). The fabrication process of the FMD tip is based on the modification of commercial AFM tips by focused ion beam and subsequent deposition of permalloy (Py). This process leads to a cylindrical tip with the Py disk located on top. The unique feature of such a tip is that its mechanical and magnetic properties are disentangled. The ground state magnetic texture of the Py disk is a magnetic vortex where the magnetization circulates in-plane around the disk center with a small region of out-of-plane magnetization in the vicinity of the center called the vortex core [1, 2]. The circular arrangement of the magnetic dipoles creates a closure domain state generating no stray field, leaving the vortex core as the only source of the stray field of the tip. The vortex core acts like a magnetically sharp nanoscale probe while the tip apex is quite large (100 - 300 nm), i.e. robust and wear-resistant.

Here we present our theoretical and experimental study of the FMD tip performance and limitations. We verified its ability by scanning various magnetic structures, including sample with sub-micron-sized domains (bits on HDD) and magnetically soft sample with large domains. Tests on a high-density magnetic recording medium showed that it provides a similar spatial resolution to a commercial MFM probe. It is thanks to the fact that vortex-core plays main role in the tip-sample magnetic interaction. However, when scanning large domains, the situation is different. The obtained MFM scans provide a different magnetic image compared to commercial CoCr tip. The image highlights the boundaries between domains with opposite magnetization. The reason for such sensing is due to the induced magnetization of the tip by the sample stray field. This has the consequence that the MFM contrast is mostly generated by the interaction between the sample and the induced charge rather than with the vortex-core.



Sketch of the vortex-core tip. The ferromagnetic disk is placed at the tip apex. Vortex core is represented by the out-of-plane brown arrow.



Comparison of MFM scans measured by the commercial low-moment tip (a), and by the FMD tip (b).

References:

[1] Šoltýs J., Feilhauer J., Vetrova Iu., Tóbik J., Bublikov K., Ščepka T., Fedor J., Dérer J., Cambel V. (2020) "Magnetic-field imaging using vortex-core MFM tip", Appl. Phys. Lett. 116, 242406

[2] S. Krylov , I. Vetrova , J. Feilhauer , J. Fedor , J. Derer , J. Soltýs and V. Cambel, (2022) "Improved durable vortex core MFM tip,"Journal of Magnetism and Magnetic Materials, 555, 169357

Thu 15:15 – 15:30 **Viktor Witkovský** `witkovsky@savba.sk`

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On computing GUM-compliant uncertainty matrix for the parameters of the specific nonlinear EIV models

We address the problem of computing uncertainty matrices for the parameters in non-linear EIV models, specifically focusing on straight-line calibration models with errors in both variables. We explain used approaches for determining the uncertainty matrices of these models and establish a clear relationship between the approximate uncertainty matrices based on ISO technical specification 28037:2010 and the law of propagation of uncertainty (LPU) as specified in JCGM 100:2008 (GUM) and its supplements. Our method begins with the concept of straight-line calibration outlined in ISO technical specification and utilizes an iterative algorithm to obtain weighted total least squares (WTLS) estimates of the model parameters, followed by the associated uncertainty matrix which is related to the covariance matrix of the best linear unbiased estimator (BLUE) in the corresponding linearized regression model. We refer to this matrix as the ISO/TS uncertainty matrix. Additionally, we derive the GUM-compliant uncertainty matrix from the implicit measurement model according to JCGM 100:2008, which we refer to as the GUM/LPU uncertainty matrix. Our analysis establishes a unique relationship between the two uncertainty matrices, providing a robust approach for estimating the uncertainty associated with non-linear EIV models.

Thu 15:30 – 15:45 **Matěj Hývl** `hyvl@fzu.cz`

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M. Hývl, M. Ledinský, A. Fejfar

Contact Force in Current-Detecting Atomic Force Microscopy – Lessons for C-AFM Tomography in Photovoltaic Research

With the rise of nanostructured solar cells came the need for nano-characterisation of photovoltaic properties. The best candidate to fulfil this need is, in many cases, scanning probe microscopy (SPM). For electrical characterization, conductive AFM (C-AFM) is the natural candidate of choice.

During any current-detecting AFM measurement, we detect current flowing through a circuit that consists of the microscope, the probe tip, cantilever and chip, sample and all the interfaces between them. Aside from spreading resistance of the sample, the measured signal is therefore dependent on many other parameters. Most problematic part of the measurement circuit is, however, the local electrical contact between the tip and the sample, characterized by R_{cont}.

In my talk, I would like to focus on the tip-sample contact resistance and its changes caused by sample topography and practically demonstrate how to out-smart these and other inherently present shortcomings of current-detecting AFM in regard to characterization of solar cell electronic properties. Application of two new techniques, Scalpel C-AFM and C-AFM Tomography [1] will be demonstrated on the solar cell samples [2].

References:

[1] U. Celano et al., ‘Conductive-AFM tomography for 3D filament observation in resistive switching devices’, in 2013 IEEE International Electron Devices Meeting, Dec. 2013, p. 21.6.1-21.6.4.

[2] M. Hývl et al., ‘Nanoscale Study of the Hole-Selective Passivating Contacts with High Thermal Budget Using C-AFM Tomography’, ACS Appl. Mater. Interfaces, vol. 13, no. 8, pp. 9994–10000, Mar. 2021

Thu 15:45 – 16:00 **Swarnendu Banerjee** `banerjee@fzu.cz`

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Swarnendu Banerjee, Matěj Hývl, Mykhailo Khytko, Martin Ledinský, Antonín Fejfar

3D Tomography on advanced photovoltaic (PV) structures – Examples of good practice

The use of doped microcrystalline silicon (µc-Si:H) thin films as carrier selective contacts in the back side of silicon heterojunction (SHJ) solar cells, allows this technology to achieve high world record conversion efficiencies (> 26%).[1] A study of surface-dependent growth of such thin films is crucial to understand the phenomenon of ‘interband tunneling’ of carriers, present in tunnel interdigitated back-contacted technology (tunnel-IBC)[2]. To study the structural and electrical properties of such films, through the evolution of electrical resistances on the nanoscale, 3D tomography using C-AFM in contact mode (also called scalpel AFM) can be a promising technique.[3] During this method, we can remove sample materials layer by layer as thin slices while parallelly measuring the conductivity.

In this work, we report an approach for a reliable scalpel AFM, based on our experiences through a series of measurements. Finding a low and stable removal rate during each scan, using either a full boron-doped diamond probe or a probe with proficient doped diamond coating, is one of the main objectives of such an experiment. Scan parameters like normal force applied on the tip, scan rate, number of points/line, direction of the scanning etc. can alter the removal rate significantly. Proper cleaning of a contaminated tip is found to be beneficial to have good process consistency. Optimizing all the above-described procedures, it's possible to generate a definitive 3D tomogram.

In addition to the results achieved on microcrystalline silicon thin films, we will represent a few results from our previous work on hole-selective passivating contacts, consisting of an interfacial layer of silicon oxide (SiO_x) and a layer of boron-doped SiC_x(p) using tomographic AFM.[4]

References:

[1] Yoshikawa, K., Kawasaki, H., Yoshida, W. et al. Silicon heterojunction solar cell with interdigitated back contacts for a photoconversion efficiency over 26%. Nat Energy 2, 17032 (2017).

[2] Tomasi, A., Paviet-Salomon, B., Jeangros, Q. et al. Simple processing of back-contacted silicon heterojunction solar cells using selective-area crystalline growth. Nat Energy 2, 17062 (2017).

[3] Luria, J., Kutes, Y., Moore, A. et al. Charge transport in CdTe solar cells revealed by conductive tomographic atomic force microscopy. Nat Energy 1, 16150 (2016).

[4] ACS Appl. Mater. Interfaces 2021, 13, 8, 9994–10000.

Thu 16:00 – 16:15 **Šárka Kučerová** `kucerova@ufe.cz`

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Piezo Force Microscopy as a powerful tool to investigate the polarity of crystallites in ZnO seed layers

Properties of the ZnO seed layers are fundamental for the growth of ZnO nanorods. The crystalline structure of the ZnO seed layer can affect the nucleation and growth of ZnO nanorods in a number of ways, which differ significantly from the nucleation and growth of the ZnO nanorods on ZnO bulk substrates or heteroepitaxial ZnO layers. The size, shape, and preferential alignment along the c-axis as well as the in-plane orientation of the crystallites within the ZnO seed layer are crucial for successful nucleation, good alignment, and crystallinity of the ZnO nanorods. The size, shape, preferential alignment along the c-axis, and also optical and electrical properties can be influenced and enhanced by heat treatment [1, 2]. However, there is another property of the ZnO crystallites that changes and evolves during heat treatment along with the preferential alignment along the c-axis, the polarity of the crystallites. The spontaneous polarization in the ZnO wurtzite structure is present due to the missing center of symmetry, therefore the [0001] direction and [0001] direction are not equivalent. Moreover, the polarity of the crystallite surfaces in the seed layer not only influences the optical and electronic properties but also impacts the nucleation and growth of the ZnO nanorods. To investigate the polarity of crystallite surfaces within the ZnO seed layer, we used Piezo Force Microscopy (PFM), a powerful tool from the SPM family. PFM is one of the AFM modes that can map the piezoelectric and ferroelectric properties of materials. We present our PFM investigation of the crystallite polarity evolution during heat treatment of the ZnO seed layers. Coupled with XRD, TEM, and SEM results, we compare the polarity evolution with the preferential alignment along the c-axis of the crystallites within the ZnO seed layer. We also present ZnO nanorod arrays prepared by patterned CBD growth on the investigated seed layers, where the impact of the ZnO seed layer (SL) on the growth of the ZnO nanorods is even more significant and visible. Furthermore, we present and discuss the advantages and challenges of PFM, which we have encountered during our experiments.

References:

[1] Bašinová, N., Černohorský, O., Grym, J., Kučerová, S., Faitová, H., Yatskiv, R., … Maixner, J. (2019). Highly Textured Seed Layers for the Growth of Vertically Oriented ZnO Nanorods. Crystals, 9(11), 566.

[2] Yatskiv, R., Grym, J., Kučerová, Š., Tiagulskyi, S., Černohorský, O., Bašinová, N., Veselý, J. (2022). Tunable visible emission in nanostructured thin films and bulk ZnO. Journal of Sol-Gel Science and Technology, 102(2), 447–453.

Thu 16:15 – 16:30 **Karel Šec** `info@nicoletcz.cz`

Nicolet CZ, s.r.o

FTIR-SNOM spectroscopy and imaging with single widely tunable laser (company presentation)

Thu 17:00 – 17:30 **Małgorzata Lekka** `malgorzata.lekka@ifj.edu.pl`

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The use of AFM to detect cancer-related changes in cells

The determination of the mechanical and rheological properties of living cells as an indicator of cancer progression has become possible with the development of local measurement techniques such as atomic force microscopy (AFM). Its most important advantage is a nanoscopic character, implying that very local alterations can be quantified. The results gathered from AFM measurements of various cancers show that, for most cancers, individual cells are characterized by the lower apparent Young's modulus, denoting higher cell deformability. Simultaneously, cancer cells are more viscous. The measured moduli depends on various factors, like the properties of substrates used for cell growth, force loading rate, or indentation depth, revealing their relative character. Despite this, the obtained results proved the AFM capability to recognize mechanically and/or rheologically altered cells. This can significantly impact the development of methodological approaches that precisely identify pathological cells and their response to various treatments.

Thu 17:30 – 17:45 **David Rutherford** `ruthedav@fel.cvut.cz`

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Technická 2, Praha 6, Czech Republic, 160 00

David Rutherford, Kateřina Kolářová, Jaroslav Čech, Petr Haušild, Jaroslav Kuliček, Egor Ukraintsev, Štěpán Stehlík, Radek Dao, Jan Neuman, Bohuslav Rezek

Correlative Atomic Force Microscopy and Scanning Electron Microscopy analysis of a Bacteria-Diamond-Metal nanocomposite

Research investigating the interface between bacteria and nanomaterials requires a multi-faceted microscopic approach to fully elucidate the mechanism of interaction. Herein, we correlate data obtained using an atomic force microscope that operated inside a scanning electron microscope (AFM-in-SEM) from a novel, multi-component sample relevant for life science research. Topographical information (AFM) was correlated with chemical and morphological information (SEM-SE) from the same region of interest analyzed under the same conditions to create a 3-D correlative probe electron microscopy (CPEM) image. It was possible to simultaneously obtain data since neither technique interfered with the other during data acquisition. Further chemical characterisation of the same region of interest was obtained after AFM-in-SEM analysis using *in situ* (EDS) and *ex situ* (Raman) spectroscopic techniques. We also detail the creation of a 3-D red, green, blue (RGB) image that merged three grayscale SEM images with topographical information (AFM) from the same region of interest to provide information regarding the precise location of the diamond-metal nanocomposite on the bacteria surface prior to analysis.

Thu 17:45 – 18:00 **Jan Přibyl** `jan.pribyl@ceitec.muni.cz`

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Kamenice 5, CZ-62500 Brno, Czech Republic

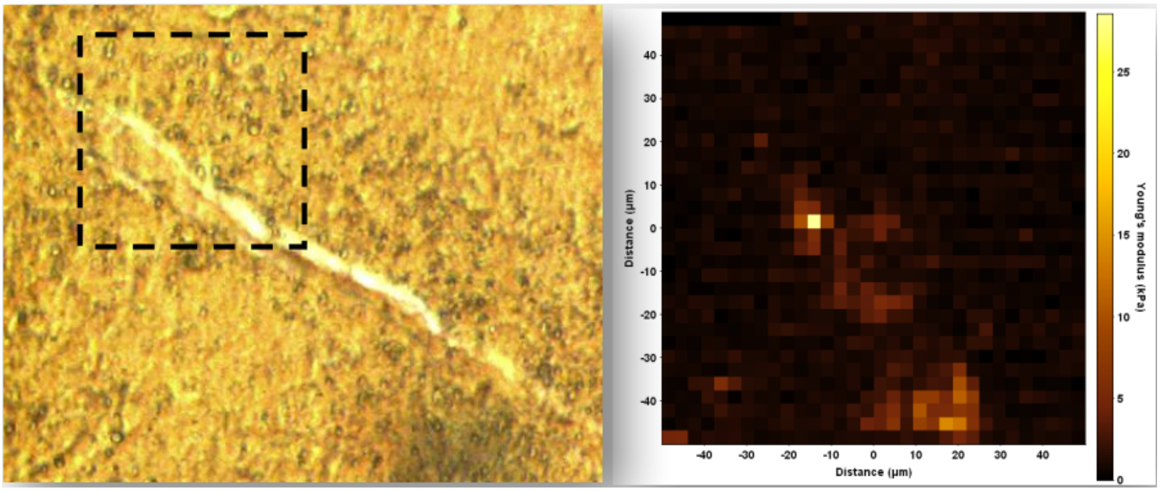
Jan Přibyl, Radka Obořilová, Šimon Klimovič, Daniil Kabanov

Atomic Force Microscopy in Biological Research

Atomic Force Microscopy (AFM) is a high-resolution microscopy technique (Horňáková et al., 2016) highly suitable for working with biospecimens, as it can operate under semi-physiological conditions. Moreover, the AFM may provide nano-localized information about the sample's elastic (Golan et al., 2018) and other mechanical properties (Ojha et al., 2022). However, there are some characteristics that AFM can not provide, such as chemical composition, optical properties, and/or information about sample electrical properties (Caluori et al., 2019).

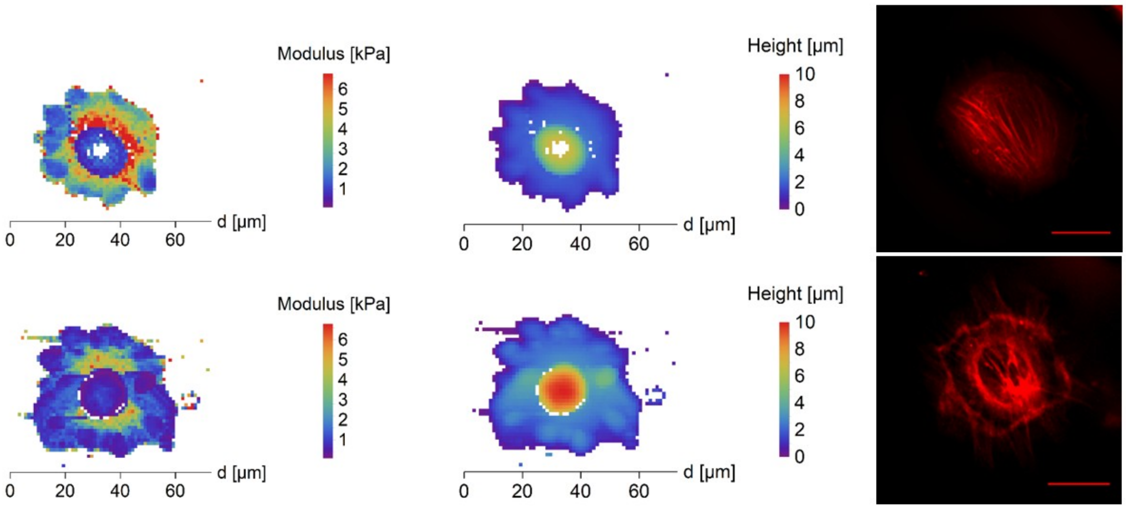
A complex description of the sample is available by a combination of AFM with one or more correlative techniques. AFM with Raman microscopy enriches the topographic and mechanical mapping with information about the sample's chemical composition. Polarizing microscopy can help, for example, in localizing collagen fibers. Fluorescence microscopy then adds the cell cytoskeleton structure to the information from elastic mapping. Another example of a correlative approach could be a combination of mechanical monitoring of cardiac contractions and electrical potential monitoring using a multielectrode array.

These and other examples of the correlative approach of combining scanning probe microscopy with other techniques will be discussed in the presentation, including possibilities for further development and optimization.



Co-localization of collagen fibers in liver samples by polarization microscopy combined with AFM nanoindentation (sample property: M. Gregor, UMG CAS)

Using the values of amplitudes including the noise floor of the pivot value in meters (vibrometer) and volts (author's electronic circuit), the measurement sensitivity of the active piezoresistive cantilever was calculated as demonstrated in figure 3.



Stiffness map and topography profile obtained by AFM nanoindentation correlated with actin filaments structure (fluorescence microscopy), fibroblast live cell study.

Acknowledgement
CIISB, Instruct-CZ Centre of Instruct-ERIC EU consortium, funded by MEYS CR infrastructure project LM2023042 and European Regional Development Fund-Project „UP CIISB“
(No. CZ.02.1.01/0.0/0.0/18_046/0015974), is gratefully acknowledged for the financial support of the measurements at the CF Nanobiotechnology.

References

[1] Caluori, G., Pribyl, J., Pesl, M., Jelinkova, S., Rotrekl, V., Skladal, P., & Raiteri, R. (2019). Non-invasive electromechanical cell-based biosensors for improved investigation of 3D cardiac models. Biosensors and Bioelectronics, 124–125.

[2] Golan, M., Jelinkova, S., Kratochvílová, I., Skládal, P., Pešl, M., Rotrekl, V., & Pribyl, J. (2018). AFM monitoring the influence of selected cryoprotectants on regeneration of cryopreserved cells mechanical properties. Frontiers in Physiology, 9(JUN).

[3] Horňáková, V., Přibyl, J., & Skládal, P. (2016). Study of DNA immobilization on mica surface by atomic force microscopy. Monatshefte Fur Chemie, 147(5).

[4] Ojha, S., Pribyl, J., Klimovic, S., Hadraba, D., Jirouskova, M., & Gregor, M. (2022). Measurement of Liver Stiffness using Atomic Force Microscopy Coupled with Polarization Microscopy. Journal of Visualized Experiments: JoVE, 2022(185).

Thu 18:00 – 18:15 **Šimon Klimovič** `simon.klimovic@ceitec.muni.cz`

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Šimon Klimovič, Deborah Beckerová, Daniil Kabanov, Vladimir Rotrekl, Martin Pešl, Jan Přibyl

Novel uses of Atomic Force Microscopy (AFM) to study contractile properties of cardiac cells.

Atomic force microscopy (AFM) is typically used as a high-resolution imaging tool to study the morphology and mechanical properties of the sample surface by moving the sharp tip mounted on a flexible cantilever. Robust feedback system and micromechanical transducers are responsible for the constant adjustment of tip-sample interaction; however, they can be utilized to monitor the contraction dynamics of cardiomyocytes. This AFM-based biosensor consists of cantilever landed on hPSC-induced cardiomyocyte cluster and recording vertical deflection in time (mechanocardiograms) in physiological conditions. Beating rate, relaxation time, contraction force, and other biomechanical properties can subtract from vertical deflection recording to study the effects of cardiomyocyte-contraction affecting drugs. More recently, a combination of vertical and lateral deflection data of two spontaneously coupled clusters was also utilized to study arrhythmia-inducing drugs in more depth. In the presentation, we will introduce how the AFM can be used in phenotyping cardiomyocytes with emphasis on methodology, analysis of data, and how this approach can be further combined with other techniques such as multi-electrode array (MEA).

Thu 18:15 – 18:30 **Radka Obořilová** `radka.oborilova@ceitec.muni.cz`

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Radka Obořilová, Hana Šimečková, Matěj Pastucha, Ivana Mašlaňová, Jan Přibyl, Petr Skládal, Zdeněk Farka

Investigation of microbial lysis on the sensor surfaces

Due to the increasing incidence of antimicrobial resistance, alternative approaches are being sought to combat superinfections. A suitable alternative to antibiotics are phages and enzybiotics, including their synergistic effect with antibiotics. Because it is necessary to set the synergy of phage and antibiotic specifically and thus a large number of different combinations of lytic agents are created, their selection has to be evaluated quickly and objectively. Monitoring the lytic action over time can speed up the collection and evaluation of the data for further applications of alternative drugs to treat fast-spreading and highly persistent bacterial strains in our immediate environment. Techniques based on biosensors enable rapid detection of pathogens in real time, verification of bacteria sensitivity to commonly used antimicrobial agents, but also the selection of suitable lytic agents. The detection of lysis takes place on the surface of a biosensor with immobilized bacteria, which has the potential to be used for the study of biofilms. An example of such a biosensor is surface plasmon resonance (SPR), which records the kinetics of bacterial lysis based on the change in resonance angle. In our approach the bacteria are immobilized on the surface of the SPR chip and phage action is monitored as mass loss after a typical delay in the lytic cycle. Atomic force microscopy (AFM) is used to image lysis directly in the native conditions of the growth medium. Here, Staphylococcus aureus was lysed using the enzyme lysostaphin and phage P68 from the Podoviridae family at 37°C using SPR and AFM. In addition to visualization, AFM was used to study changes in the mechanical properties of the bacterial wall during lysis, resulting in a decrease in Young's modulus (E). These advanced methods can provide deeper insight into bacterial lysis and may aid in future efforts to automatically detect the appropriate lytic agent for personalized treatment [1].

[1] Obořilová R., Šimečková H., Pastucha M., Klimovič Š., Víšová I., Přibyl J., Vaisocherová-Lísalová H., Pantůček R., Skládal P., Mašlaňová I., Farka Z. Nanoscale, 2021,13, 13538-13549

CIISB, Instruct-CZ Centre of Instruct-ERIC EU consortium, funded by MEYS CR infrastructure project LM2023042 and European Regional Development Fund-Project „UP CIISB“
(No. CZ.02.1.01/0.0/0.0/18_046/0015974), is gratefully acknowledged for the financial support of the measurements at the CF Nanobiotechnology.

Thu 18:30 – 18:45 **Naďa Labajová** nada.pavlendova@savba.sk

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Clostridioides difficile DivIVA protein-lipid interactions study by AFM and Cryo-EM

Accurate division septum positioning is important for effective bacterial cell division and spreading. Min system is a negative regulator of the cell division. Its presence at specific cell sites blocks division septum formation [1]. Min system has been most extensively studied in Gram-negative Escherichia coli and Gram-positive Bacillus subtilis. In E. coli the Min system acts very dynamically and undergoes pole-to-pole oscillation [2]. It comprises of MinC, MinD and MinE proteins. B. subtilis Min system does not oscillate, localizes at the cell poles more stably and consists of MinC, MinD, MinJ and DivIVA [3]. MinC and MinD constitute a complex, which is an actual division inhibitor. MinE and DivIVA are topological markers of Min system in E. coli and B. subtilis, respectively. While MinE acts very dynamically, and is essential for Min system oscillation [4], DivIVA attaches the Min system to areas with high negative membrane curvature, cell poles and division site [5]. However, Gram-positive human pathogen Clostridioides difficile possesses unique Min system composed of MinC, MinD, MinE but also DivIVA protein. We have shown previously that C. difficile DivIVA can bind to curved but also to planar membranes containing negatively charged phospholipids, especially cardiolipin. Upon binding, DivIVA changes the cardiolipin distribution and alters morphology of these lipid membranes [6]. To better characterise the DivIVA protein-membrane complex we employed atomic force microscopy, force spectroscopy, transmission electron and cryo-electron microscopy.

This work was supported by VEGA – Grant No. 2/0033/22 from the Slovak Academy of Sciences, a Grant APVV-18-0104 from the Slovak Research and Development Agency and European Regional Development Fund-Project „UP CIISB“ (No. CZ.02.1.01/0.0/0.0/18_046/0015974).

References:

[1] Errington & Wu (2017) In Prokaryotic Cytoskeletons. (Löwe & Amos, eds), 1. ed, pp. 67–101. Springer International Publishing, Switzerland.

[2] Ramm et al. (2019) Cellular and Molecular Life Sciences 76, 4245–4273.

[3] Feddersen et al. (2021) mBio 12.

[4] Raskin & De Boer (1999) Journal of Bacteriology 181, 6419–6424.

[5] Lenarcic et al. (2009) The EMBO Journal 28, 2272–2282.

[6] Labajová et al. (2021) International Journal of Molecular Sciences 22, 8350.

Thu 18:45 – 19:00 **Vladimíra Tomečková** vladimira.tomeckova@upjs.sk

Department of Medical and Clinical Biochemistry, Pavol Jozef Šafárik University in Košice
Park Angelinum 9, Košice 041 54, Slovakia

Daria Kondrakhová, Vladimír Komanický, Katarína Zakutanská, Natália Tomašovičová, Vladimíra Tomečková

Graphical analysis of tear fluid by using atomic force microscopy

Tear fluid is nontraditional body fluid with the same composition as blood but without the presence of hemoglobin. Tear fluid of healthy subjects as well as patients with various ocular (syndrome of dry eye, glaucoma) and systemic diseases (diabetes mellitus, multiple sclerosis, major psychiatric disorder) were collected from dogs only by saline solution, from people also by glass microcapillary. Each sample (2–10 μl) was placed and analyzed on microscopic slide as a whole complex without separation by atomic force microcopy (AFM). Our results showed AFM fingerprinting of human tear fluid can discriminate subjects with particular disease from healthy controls with high sensitivity and specificity and confirmed the merit of proteomic profiling. Moreover, data obtained from AFM surface images could serve as input data for the computational model in diagnostics. The integration of data obtained by the above-mentioned method could become a valuable aid in the predictive, preventive and personalized medicine.

Experimental work at Safarik University has been supported by the grant of the Slovak Research and Development Agency under the contract No APVV-20-0324.

Fri 9:00 – 9:30 **Virpi Korpelainen** `virpi.korpelainen@vtt.fi`

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MetExSPM project: Development of traceable methods for high speed and large range SPM

Today, complex nanostructures and nanodevices are used in photonics, quantum technology and nanoelectronics, and increasingly in healthcare and in novel materials research. Fast, accurate and traceable High-Speed Scanning Probe Microscopy (HS-SPM) has great potential for use in identifying faulty nanoproducts across multistage production processes and offers the benefits of increased productivity and reduced wastage. Conventional scanning probe microscopy (SPM) is either too slow to cover large sample areas or if fast, it lacks positioning accuracy. This project is developing essential scanning probe microscope components and ultimately a validated and traceable prototype HS-SPM measurement system suitable for use in industrial measurements.

Industry, universities, and research institutes perform many high-resolution measurements; however, high resolution or high precision does not necessarily mean high accuracy. Without proper calibration and a good understanding of probe sample interactions, dimensional/property measurement errors may be as large as 30%. At present, in the nanomanufacturing, semiconductor, nanometrology and quantum technology fields, a lack of measurement traceability to the SI metre, the associated uncertainties, and the effects of speed on measurement accuracy do not meet user requirements for higher speed (>1 mm/s) combined with larger scanning area ($>1\,\mathrm{mm}^2$) and better accuracy (∼nm).

To turn high-speed SPMs from qualitative imaging instruments to high accuracy measurement instruments suitable for industrial quality control applications, requires the development of scanning microscope stages with far greater stability, improved probing systems and advanced measurement strategies that combine high-speed scanning with the possibility to collect local electrical or mechanical properties.

The overall objective of this project is to design and develop technologies for transforming HS-SPM (∼10 mm/s) metrology instruments for use in industrial high-speed quantitative multi-sensing metrology with a target traceable position measurement uncertainty of 1 nm. The developed instrument will be suitable for the industrial characterisation of functional nanostructure property combinations (electrical, chemical, mechanical, dimensional).

The specific objectives of the project are:

1. To design, develop, manufacture and characterise the frequency response, and noise level of multifunctional high-frequency (resonance frequency ω_0=1 MHz) self-sensing and/or selfactuating probes and control electronics that will form a sub system suitable for a compact HS-SPM prototype designed for use in industrial environments.

2. To develop a new generation of scanning stages for HS-SPMs, which will be capable of high-speed motion (∼10 mm/s) and large stroke (∼10 mm) with inherent metrological traceability to the SI

metre. The scanning stages should include high-speed 6-DoF interferometry sensors, which will enable real-time measurements in industry without dynamic position errors. The target position noise is 0.5 nm.

3. To develop open source experimental control and data processing software tools, for adaptive scanning and compressive sampling suitable for high speed SPM (∼10 mm/s) surface nanometrology measurements over large areas (1 cm^2), using hybrid stage combinations (piezo-electric and/or MagLev). This will enable real-time traceable quantitative multi-sensing measurements (topography combined with electrical or mechanical properties).

4. To incorporate the probes, scanning stages and software tools developed in objectives 1–3 into at least one new fully assembled and characterised custom-designed prototype HS-SPM which will be capable of multi-sensing measurements for the industrial characterisation of functional nanostructure property combinations (topography combined with electrical or mechanical properties). In addition, to demonstrate its performance against other HS-AFM and/or HS-SPM by comparative measurements of reference samples and by measurements of industrially relevant samples (e.g. – an optical industry ultra-smooth optical surface, rectangular gratings, silicon samples relevant for the semiconductor industry, etc.).

5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (accredited laboratories, instrumentation manufacturers), standards developing organisations (CEN, ISO) and end users (SPM manufacturers).

The current state of the project will be presented.

Fri 9:30 – 9:45 **Jan Thiesler** `Gaoliang.Dai@ptb.de`

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Jan Thiesler, Rudolf Krueger, Rainer Gloess, Marcel Mohr, Harry Marth, Gaoliang Dai

A novel high-dynamic large-range hybrid SPM scanner

Conventional SPM scanners typically apply polycrystalline piezo material such as lead zirconate titanate (PZT) to achieve fast and small-area scanning. Although being low-cost and highly dynamic, such PZT scanners have (very) poor positioning accuracy owing to the behaviour of the piezo material. Moreover, such scanners have non-flat motion trajectories, resulting in the so-called "bow" artefact in measured images. "High-end" AFMs usually apply flexure hinge piezo stages as scanners. For better motion accuracy such scanners are typically operated in closed-loop control by applying accurate position sensors as feedback. However, the phase lag of the closed-loop system limits the measurement speed of SPMs. Typically, PZT scanners have a motion stroke of (far) less than 100 µm. To achieve long- stroke measurements, micro positioning stages equipped mechanical guiding and driven by spindle or linear motors are usually applied. However, they suffer from poor dynamic properties and motion errors due to the mechanical guiding systems.

Within the scope of a Joint Research Project within the European Metrology Research Programme (EMPIR) entitled "Traceability of localised functional properties of nanostructures with high speed scanning probe microscopy (MetExSPM)", a novel kind of SPM scanner based on a hybrid combination of three stages: a 3-axes monocrystalline piezo stage, a 6-axes polycrystalline piezo stage and a 6-axes large-stroke magnetic levitation (MagLev) stage are being jointly developed by the PTB and the company PI.

Two hybrid design concepts are proposed. The first concerns the hybrid combination of the piezo stages with the MagLev stage. It thus combines high motion dynamics and high positioning resolution of the piezo stages with the long stroke of the MagLev stage. The second concerns the hybrid application of a 3-axes monocrystalline piezo stage and a 6-axes flexure-hinge based closed-loop polycrystalline piezo stage, where the monocrystalline piezo stage is applied to compensate the limited dynamics of the polycrystalline piezo stage. The hybrid design concepts mentioned above offer significant advantages by complementarily combing the strengths of different stages, while compensating and mitigating their weakness with each other.

All stages (MagLev and piezo stages) move sample and/or tip in parallel under real-time servo control in a coordinated manner, thus realising long-stroke, high-speed and highly accurate SPM scanning. The main advantages of a MagLev stage are its smooth and controllable accuracy and perfect cleanness during scan movements in relation to conventional mechanically guided or air bearing systems. The stability and tracking performance of a MagLev stage depend only on the sensors, the drivers, and the control algorithms. The Maglev stage will feature (near) power-less levitation, which results in near-zero energy demand and heat dissipation when the stage is kept stationary or moved with constant speeds. Driving power is only needed to accelerate or decelerate the stage. This innovative design concept is crucial for dramatically reducing the heat dissipation, which is an important factor for achieving low measurement drift and high measurement accuracy.

Design concept and implementation of the scanner will be detailed in this talk.

Fri 9:45 – 10:00 **Dominik Badura** `dominik.badura@pwr.edu.pl`

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Dominik Badura, Bartosz Pruchnik, Teodor Gotszalk, Ivo Rangelow

Calibration of active piezoresistive cantilevers

In the classical design of an atomic force microscope, it takes several minutes to measure a surface because it requires at least 512 lines for a high-resolution image. The scanning time of one line is about 1-2 seconds. In the HS -AFM (High Speed Atomic Force Microsopy) method, the minimum speed is 12 - 15 lines per second. To enable high-resolution imaging in the shortest possible time, a micro-electro-mechanical system -MEMS was used, which is characterized by a high resonant frequency (>300kHz), an integrated cantilever deflection detector system and a static and resonant tip deflection actuator. The purpose of this work is to demonstrate how to determine the sensitivity of an active piezoresistive cantilever, that is, the value of the electrical signal with respect to the deflection of the cantilever by 1 nm.

The test methodology used a vibrometer and proprietary electronics to record the signal from the deflection detector. This paper presents a method for determining the resonant frequency from thermomechanical noise. Three separate methods of actuating the lever were also demonstrated, along with a discussion of their advantages and disadvantages. The paper also demonstrates a way to increase the sensitivity of an active piezoresistive cantilever.

Figure 1 shows an active piezoresistive cantilever. The most important components have been signed, namely the thermomechanical actuator, which is responsible for deflecting the lever under the influence of a flowing current of the appropriate frequency. The deflection detector, or piezoresisitive Wheatstone bridge, from which an electrical signal proportional to the deflection is read.

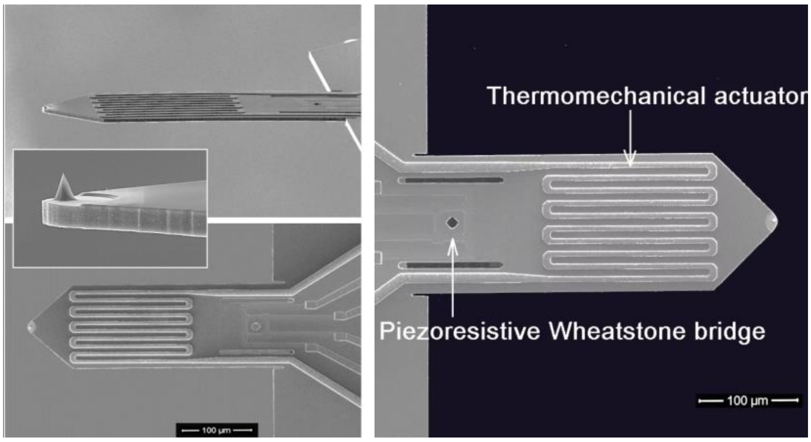

Fig. 1 – Active piezoresistive microcantilever

After a series of measurements, it was shown for which actuation mode and for which modulus the measurement sensitivity is highest. Figure 2 shows the measurement of thermomechanical noise on the vibrometer and the author's electronic system.

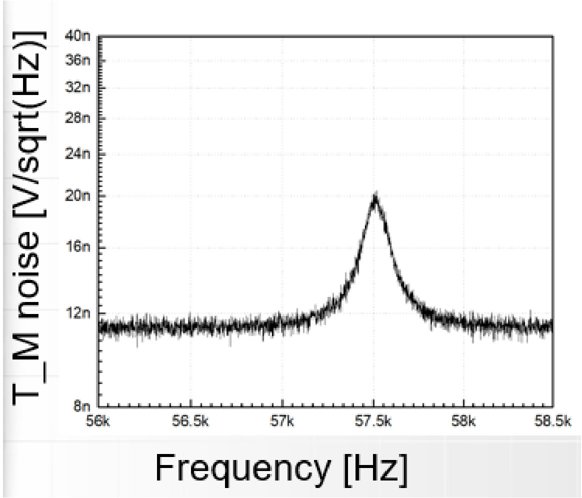

Fig. 2 – Thermomechanical noise. (a) Value read using a vibrometer. (b) Value read using proprietary electronics from a swing detector.

Using the values of amplitudes including the noise floor of the pivot value in meters (vibrometer) and volts (author's electronic circuit), the measurement sensitivity of the active piezoresistive cantilever was calculated as demonstrated in figure 3.

Fig. 3 - Visualize the free vibration of the cantilever. The deflection value is determined by a vibrometer, while the detector signal is measured by a proprietary electronic system.

Conclusions:
The paper presents an alternative solution for atomic force microscopy. The system does not use optoelectronic components, which results in virtually no calibration of the system. In addition, it allows very fast scanning of surfaces with 18-bit resolution. Because of the possibility of precise control of the cantilever and the possibility of depositing various materials on the cantilever, such as diamond, it is also possible to modify surfaces (nanoscratching). This paper demonstrates several ways to calibrate an active piezoresistive cantilever, ways to compensate for manufacturing defects or unfavorable physical phenomena (e.g., bimetal), and to improve the measurement sensitivity of the piezoresistive cantilever.

Fri 10:00 – 10:15 **Petr Klapetek** `pklapetek@cmi.cz`

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GwyScope: open hardware playground for adaptive SPM scanning

A low-cost Digital Signal Processor (DSP) unit for advanced Scanning Probe Microscopy measurements will be presented. It is based on Red Pitaya board and custom built electronic boards with additional high bit depth AD and DA converters. By providing all the necessary information (position and time) with each data point collected it can be used for any scan path, using either existing libraries for scan path generation or creating adaptive scan paths using Lua scripting interface. The DSP is also capable of performing statistical calculations, that can be used for decision making during scan or for the scan path optimisation on the DSP level. Examples of using this DSP with a high-speed Scanning Probe Microscope developed at CMI will be presented as well.

Fri 10:15 – 10:30 **Ondřej Novotný** `info@nenovision.com`

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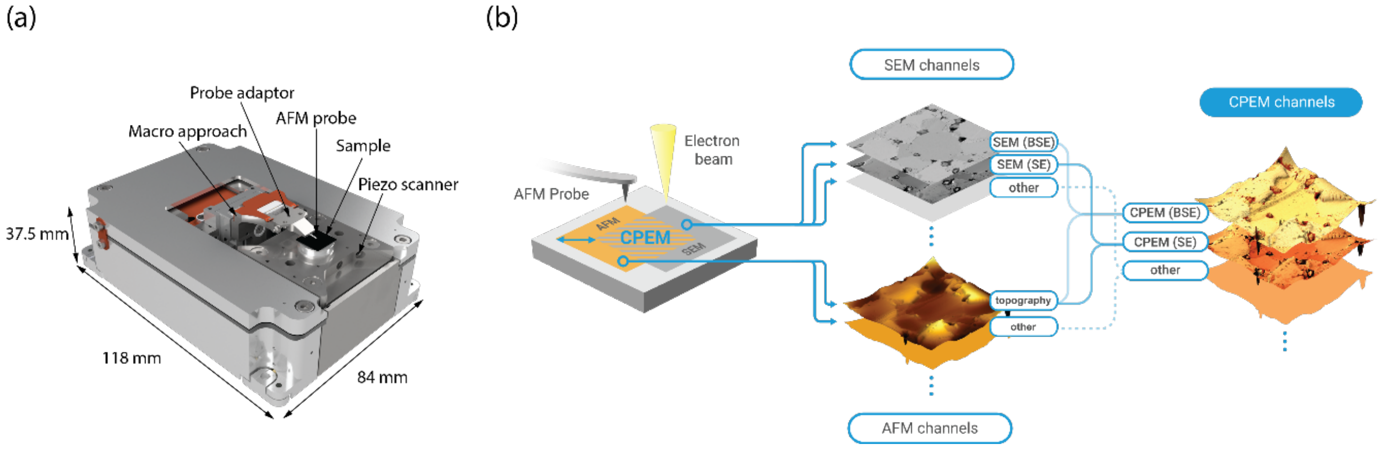
New generation of AFM in SEM – company presentation

Correlative in-situ microscopy which combines the benefits of different imaging systems has become very important tool helping us to understand the complexity of the sample properties. For these reasons, the correlative microscopy is one of the hot topics of nowadays research. When we imagine combination of two complementary techniques, atomic force microscopy (AFM) and scanning electron microscopy (SEM), there are several advantages of this setup, such as complexity of the measurement, in in-situ conditions, and with precise localization to the area of interest.

To be able to combine these techniques, NenoVision company has developed a unique Atomic Force Microscope (AFM), LiteScope™, for easy „plug & play“ integration into the SEMs. The connection of AFM and SEM enables to merge the strengths of both techniques, resulting in effective workflow and possibilities of complex sample analysis that was difficult or readily impossible by conventional, separate AFM and SEM instrumentation.

Recently, we launched our new generation of AFM-in-SEM, LiteScope 2.5, which is built on a completely new control unit NenoBox with Open Hardware DSP controller GwyScope. From our key innovations, we have integrated second fast lock-in amplifier with multiple high-frequency inputs and outputs, improving the connectivity to a variety of external devices, pushing the limits for advanced non-standard experiments involved in mechanical, electrical and magnetic in-situ analyses. Furthermore, we have simplified the user experience by implementing several automated features, such as probe auto-tuning, predefined workflows, and enhanced AI computing power. These improvements are designed to support our customers' expertise in both simple and complex tasks, making their experience with LiteScope 2.5 seamless and efficient.

Additionally, NenoVision's proprietary Correlative Probe and Electron Microscopy, shortly CPEM, is a unique method allowing for nanometer precise AFM and SEM data correlation thanks to the measurement setup. During the imaging, electron beam and AFM tip keep a constant offset and remain static. The scanning movement is conducted by a piezo scanner that carries the sample. This ensures simultaneous SEM and AFM data collection in the same coordinate system and conditions, and with identical pixel size. The resulting 3D CPEM view can combine multiple channels, both from AFM and SEM, enabling thorough sample analysis and clear data interpretation for specific applications.



In conclusion, the AFM-in-SEM strategy benefits from the complementarity of both techniques alongside significant savings both in time and resources. Also, it opens completely new possibilities for complex analyses and advanced data correlation and measurements thanks to optimized effective workflows in fields like Material science, Semiconductors, Batteries, Life science, and other areas of research and industry.

Company introduction

NenoVision is a technology company based in Brno, Czech Republic. We were the first spin-off from the Brno University of Technology and the Central European Institute of Technology (CEITEC). Over 30% of world production of electron microscopes is manufactured in Brno. The city has a long tradition in the development of scientific instruments and is also referred to as the world center of electron microscopy. Our goal is to move microscopy to the next level by continuing with the established tradition and expertise within the field and bringing innovative Correlative Probe and Electron Microscopy Technology to the market.

Fri 10:30 – 10:45 **Jaroslav Kuliček** kulicjar@fel.cvut.cz

Faculty of Electrical Engineering, Czech Technical University in Prague
Prague, Czech Republic

Jaroslav Kuliček, Takatoshi Yamada, Takashi Taniguchi, Bohuslav Rezek

AFM/photo-KPFM/micro-Raman correlation on G/h-BN edges

Graphene is a carbon material interesting for electronic applications due to its high quality, electronic properties, transparency, thermal stability, easy preparation, and surface modification[1]. Using graphene in photovoltaics is limited by intrinsic zero-bandgap, and encapsulation in the hexagonal Boron Nitride (hBN) overcomes the graphene limits and improves charge carrier mobility, making G/hBN heterostructure interesting for optoelectronic applications [2].

In this work, we studied the properties of the Graphene/hexagonal Boron Nitride (G/hBN) edges between the heterostructure flakes and substrate. Heterostructures were prepared by adding high-pressure, high-temperature (HPHT) hexagonal Boron Nitride on SiO_2 or Si substrate. The chemical vapour deposition (CVD) graphene was laid over the whole substrate using the PMMA transfer method. After graphene transfer on substrates, the prepared heterostructures were cleaned by annealing at 400°C in Ar/H_2. By Force Microscopy has been determined the morphology and thickness of edges. The optoelectronic properties and composition of the G/h- BN heterostructures edges have been studied by Kelvin Probe Force Microscopy (KPFM) and Raman micro-spectroscopy and mapping. The surface potential increased on the

G/hBN heterostructures edges and gradually decreased on the SiO_2 substrate, while surface potential on the Si substrate was uniform. Enhanced Raman intensity from Si and Graphene along the G/hBN edge on the Si substrate under visible light was observed. Enhanced Raman intensity along the G/hBN edge is probably related to localised electrons concentration and suitable perpendicular orientation of plasmonic vibrations. An enhancement of the Raman signal was not observed at the G/hBN edge on the SiO_2 substrate.

Acknowledgements:

This work was partly supported by JSPS KAKENHI Grant Numbers JP 20H02191 and 20H00354 and by the ERDF and MEYS project CZ.02.1.01/0.0/15_003/0000464 (CAP).

References:

[1] Nandanapalli, K. R.; Mudusu, D.; Lee, S. Functionalization of Graphene Layers and Advancements in Device Applications. Carbon 2019, 152, 954–985. https://doi.org/10.1016/j.carbon.2019.06.081.

[2] Dean, C. R.; Young, A. F.; Meric, I.; Lee, C.; Wang, L.; Sorgenfrei, S.; Watanabe, K.; Taniguchi, T.; Kim, P.; Shepard, K. L.; Hone, J. Boron Nitride Substrates for High-Quality Graphene Electronics. Nature Nanotech 2010, 5 (10), 722–726. https://doi.org/10.1038/nnano.2010.172.

Fri 10:45 – 11:00 **Marek Černík** `uniexport@uniexport.co.cz`

Uni-Export Instruments, s.r.o

Correlative Raman-AFM Imaging – Techniques and Applications (company presentation)

Fri 11:30 – 11:45 **Egor Ukraintsev** `ukraints@fzu.cz`

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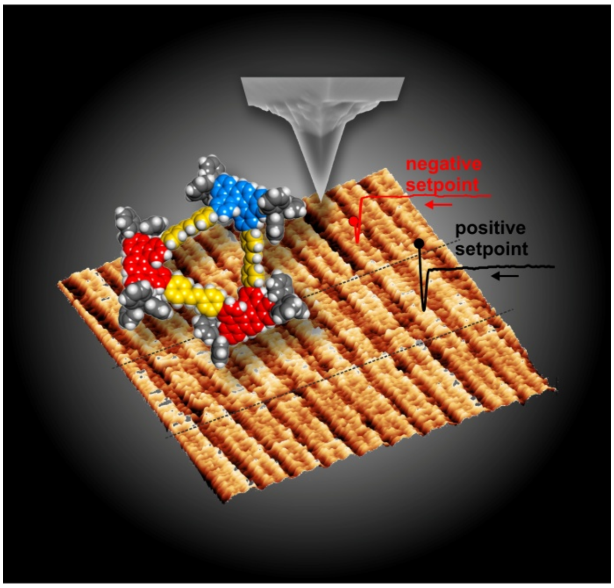
Egor Ukraintsev, Bohuslav Rezek

Non-contact non-resonant atomic force microscopy method for measurements of highly mobile molecules and nanoparticles

Attractive tip-to-sample interactions are used in non-contact non-resonant (NCNR) AFM mode to measure surface morphology. Atomic force microscopy (AFM) is nowadays indispensable versatile scanning probe method widely employed for fundamental and applied research in physics, chemistry, biology as well as industrial metrology. Here we show how the attractive forces in AFM under ambient conditions can be used with advantage to probe surface properties in a very sensitive way even on highly mobile molecules and nanoparticles. We introduce a stable non-contact non-resonant (NCNR) AFM method which enables to reliably perform measurements in the attractive force regime even in air by controlling the tip position in the intimate surface vicinity without touching it. We demonstrate proof-of-concept results using sine-wave oscillations with negative setpoint value on helicene-based macrocycles and nanodiamonds on SiO_2. We compare the results with other conventional AFM regimes, showing NCNR advantages such as higher spatial resolution, reduced tip contamination, and negligible sample modification. We analyze principle physical and chemical mechanisms influencing the measurements, discuss issues of stability and various possible method implementations. We explain how the NCNR method can be applied in any AFM system by a mere software modification. The method thus opens a new research field for measurements of highly sensitive and mobile nanoscale objects under air and other environments.



Fri 11:45 – 12:00 **Bartosz Pruchnik** `bartosz.pruchnik@pwr.edu.pl`

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Scanning thermal microscope with transformer brigde input electronics

We describe novel method for precise measurement of temperature in the scanning thermal microscope (SThM). We introduce technical description of construction of transformer-bridge setup along with signal acquisition. We present measurements performed with introduced setup and provide discussion about the limits of resolution of transformer-bridge-based SThM.

Transformer bridge is comparative setup, in which measurement signals from two resistors: probing resistor and reference resistor are being subtracted. Primary coils are the source of the signals in branches of the bridge, while secondary coil is source of an output sum signal. Bridge is being supplied with sinusoidal signals of equal frequencies and amplitudes chosen specifically to cancel out. Signals are voltage supplies, which induce currents in branches respectively to loads. Resistances of thermoresistor and reference resistor are attuned in such a way, that amplitudes of signals are of similar values. Change in value of resistance in one branch leads to change in branch current and imbalance of the bridge. The output signal is filtered, amplified and directed onto the lock-in amplifier. Lock-in radius signal is then fed into ADC card of the measurement system.

Detector of the SThM is thermally sensitive microcantilever. Probe used in experiments was KNT-SThM-2an by Kelvin Nanotechnology. According to manufacturer, probe has temperature resolution of 0.1 K, with resistances in the proximity of 350 Ω. Cantilevers have stiffness' not exceeding 0.4 N/m and tip radius not greater than 100 nm. In the ARMScope system [1] cantilever deflection is monitored within OBD setup with resolution down to 5.6 pm. Total system resolution in measurements performed with KNT-SThM-2an cantilevers is in range of 0.2 nm.

Resistance of thermoresistor embedded in cantilever is measured with alternating signal, therefore actuation occurs. Deflection of the cantilever can be observed at doubled actuation frequency. Amplitude of lowest signal at which temperature contrast was clearly visible was 200 μA, so power dissipated was approximately 15 μW. Amplitude of vibrations in such case is equal 0.2 nm. If to treat cantilever vibrations as a source of noise, then definitive resolution of the system equals 0.244 nm.

Measurements were done in thermoresistor functioning as a heat source. Temperature of the sensor, being equilibrium of heat power and heat conductivity of the cantilever and the substrate, depends solely on the thermal conductivity of the substrate material. What is observed is therefore contrast in thermal conductances, visible as a temperature difference. Therefore sensitivity of the setup depends heavily on power dissipated by the heater-sensor and material differences in the setup as well as integration time of lock-in amplifier and external thermal noises. Preliminary measurements were conducted on a Brüker SThM calibration sample with carbon fibers embedded in epoxy resin. Material contrast observed on the sample can be used to calculate potential resolution of the SThM setup.

References:

[1] Kopiec D., Majstrzyk W., Gacka E., Pruchnik B., Badura D., Sierakowski A., Janus P., Gotszalk T.: „Metrology and control of electromagnetically actuated cantilevers using optical beam deflection method", Metrology And Measurement Systems 4/2021 vol. 28

Fri 12:00 – 12:15 **Wiktor Połacik** `bartosz.pruchnik@pwr.edu.pl`

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Bartosz Pruchnik, Janusz Fidelus, Ewelina Gacka, Tomasz Piasecki, Teodor Gotszalk

Atomic force microscopy in single-specimen measurements of nanowires

We present approach for measurements of single nanowires (NW) specimens. With appropriate preparatory practices it is possible to measure single NWs in method based on specifically designed substrates. Mechanical, electrical and thermal properties of NWs can be measured on such samples, giving insight into properties of material in high aspect ratio object as well as properties of NW itself. Approach covers usage of scanning thermal microscope (SThM) and conductive substrates for measurement of mechanical, thermal properties and resistivity of single NW. Setup enables as well measurements of interrelated properties such as piezoresistivity or thermomechanical deformation.

Nanowires can be selectively picked from complex aggregates and delivered onto any place on substrate. To determine mechanical properties it is best to leave NW with one degree of freedom orthogonal to the plane of cantilever [1]. For measurement of electrical properties however it is necessary to provide NW with electrical contacts. For that purpose specialised substrates were produced in few types. For mechanical measurement substrates with mechanical features were chosen; for mechanical and thermal conductivity measurement substrates with elevated, thermally isolated islands were designed; finally for electrical and thermal measurements substrates with outside-connected pads were made.

NW were placed on selected substrates with EasyLift nanomanipulator operations. Chosen NW was picked up, transferred to the right substrate and bound to the surface with material delivered with focused electron beam induced deposition (FEBID). NWs were then mounted mechanically and connected electrically. In measurement, depending on the parameter looked after, NWs were mounted straight or slightly bent to induce preliminary strain.

Mechanical properties were determined by changing force indenting the NW while the deformation was measured. Instead of regular F-z spectroscopy, whole length of the NW was measured along with substrate in the proximity, what allowed real differential estimation [2]. In the same setup, thermal conductivity of the NW can be measured, as the thermal contrast is dependent solely on the conductivity of the NW. With conductivity measurements, SThM makes also possible mapping the Joule heat field.

We present results of measurements of NWs transferred to various substrates. We determine properties of ZnO material and show contrast to the bulk form. We present results of conductivity measurements in 4-wire setup. Finally we show results of thermomechanical measurements. We conclude presented family of methods as a complex approach for singular NWs examination.

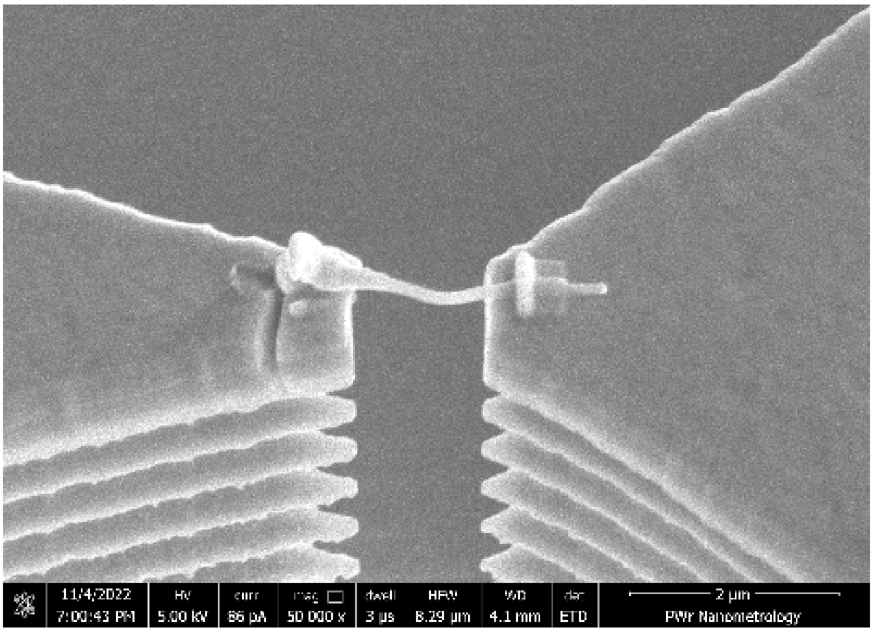

Figure 1 Exemplary NW bound to the surface over the gap

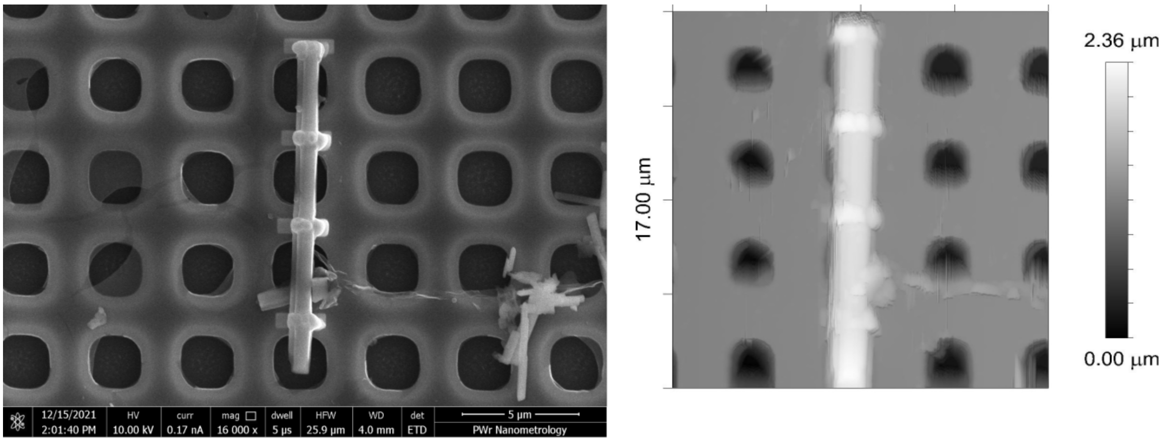

Figure 2 Single NW observed with scanning electron microscope (left) and atomic force microscope (right)

References

[1] A. Kulik, A. Kis, G. Gremaud, S. Hengsberger, G. Luengo, P. Zysset, L. Forró, Nanoscale Mechanical Properties – Measuring Techniques and Applications, in: B. Bhushan (Ed.), Springer Handb. Nanotechnol., Springer Berlin Heidelberg, Berlin, Heidelberg, 2007: pp. 1107–1136. https://doi.org/10.1007/978-3-540-29857-1_36

[2] J.-P. Salvetat, G.A.D. Briggs, J.-M. Bonard, R.R. Bacsa, A.J. Kulik, T. Stöckli, N.A. Burnham, L. Forró, Elastic and shear moduli of single-walled carbon nanotube ropes, Phys. Rev. Lett. 82 (1999) 944–947. https://doi.org/10.1103/PhysRevLett.82.944

[3] Bartosz C. Pruchnik, Janusz D. Fidelus, Ewelina Gacka, Krystyna Mika, Leszek Zaraska, Grzegorz D. Sulka, Teodor P. Gotszalk, Relationship between the Structural Properties of Zno Nanowires and Their Modulus of Elasticity, Available at SSRN 4363727

Fri 12:15 – 12:30 **Daniel Haško** `daniel.hasko@cvtisr.sk`

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Ilkovičova 3, 841 04 Bratislava, Slovak Republic

AFM and 3D optical microscopy study of ablation craters created by laser-induced breakdown spectroscopy

For qualitative and quantitative analysis of different types of samples (solid, liquid, gaseous) nowadays the spectroscopic methods are widely used. Among others more sophisticated and expensive methods the laser-induced breakdown spectroscopy (LIBS) is characteristics by its speed, simplicity, and robustness as well as relatively low cost. Despite of its relative low detection limits, which are however improved every year, in some cases is LIBS the only possible diagnostic method (e.g. analysis of the Mars surface or diagnostics of trapped radioactive fuel in the walls of the thermonuclear reactor). For removing the less material possible from analyzed sample, the laser exposure needs to be small. We can achieve this by using short laser pulses or partial de-focusing of the laser beam but with the energies needed to achieve the laser ablation. For direct analysis of the surfaces of layers or subsurface changes in materials and for determining the dimensions of ablation craters or detection of the presence of "bumps"at the edges of created craters the atomic force and 3D optical microscopy (AFM and 3D OM) methods were employed.

Acknowledgement
This work was financially supported by Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic No. VEGA-1/0803/21.

Fri 12:30 – 12:45 **Dušan Novotný** `dusan.novotny@mt-m.eu`

Měřící technika Morava, s.r.o

Měřicí technika Morava s.r.o. (company presentation)

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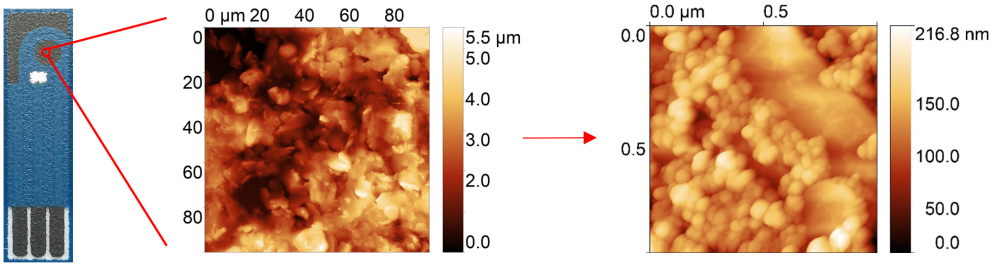
Surface characterization of home-made screen printed electrodes as a biosensors

We have used AFM and Raman spectroscopy for characterization of home-made screen printed electrodes used as disposable electrochemical biosensors. The screen printed electrodes were utilized for use as the electrochemical sensor for DNA and/or RNA fragments detection. The above mentioned methods helped us to resolve the problem of electrochemical parasitic peaks coming from the electrolyte solution used for the detection of biomolecules. We used heat treatment to remove the source of these interfering background peaks.



Visualization of the home-made carbon screen printed electrode.

This research has been supported by the Czech Science Foundation, project No. 21-46325L.