

Book of Abstracts



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PROGRAM OVERVIEW

SPM Workshop 2019

Wednesday 27^{th}

12:00 - 13:00		Registration
13:00 - 13:10		Opening
13:10 - 13:50	Fernando Araujo	Towards 3D nanoscale imaging of complex thin films
	de Castro	using scanning probe microscopy
13:50 - 14:30	Denis Vasyukov	Introduction to Scanning Microwave Microscopy
14:30 - 14:50	Anna Charvátová	Nanoindentor tip analysis by AFM
	Campbell	
14:50 - 15:10	Dušan Novotný	Měřicí technika Morava s.r.o. company presentation
15:10 - 15:30		Coffee break
15:30 - 15:50	Teodor Gotszalk	Array of electromagnetically cantilevers for force-distance
		spectroscopy metrological investigations
15:50 - 16:10	Daniel Haško	Measurement of the elastic properties of soft samples in fluids
16:10 - 16:30	Vilma Buršíková	Characterisation of plasma-polymer organosilicon composite
		thin films deposited under dusty plasma conditions
16:30 - 16:50	Jiří Buršík	Local mechanical properties of advanced thermoelectrics
16:50 - 17:10	Egor Ukraintsev	Peak Force AFM study of a chiral helicene-based macrocycles
		assembly on HOPG
17:10 - 17:30	David Nečas	Tip dilation artefacts & roughness measurement – parametric
		approach
17:30 - 17:50	Filip Jakeš	RMI company presentation
18:00 - 19:00		Dinner

Thursday $\mathbf{28}^{th}$

9:00 - 9:40	Volker Neu	Probing magnetic textures on the nanoscale
9:40 - 10:00	Matěj Hývl	Contact force in C-AFM
10:00 - 10:20	Jaroslav Kuliček	Microscopic surface potential study for understanding ionic
		migration in perovskites
10:20 - 10:40	Dušan Novotný	Měřicí technika Morava s.r.o. company presentation
10:40 - 11:00		Coffee break
11:00 - 11:40	Yannick De Wilde	Near-field investigation of plasmonic materials and
		devices at infrared wavelengths
11:40 - 12:00	Petr Klapetek	FDTD calculations of infrared SNOM probes
12:00 - 12:20	Jan Vávra	Scattering-Type Scanning Near-field Optical Microscopy and
		Spectroscopy for Nanoscale Chemical Analysis – company
		presentation
12:30 - 14:50		Lunch & Group photo
14:50 - 15:10	Radovan Vraník	Mechanical response of helicene molecules on $Ag(111)$ sub-
		strate to RF pulses
15:10 - 15:30	Mihai-George	AFM measurements of laser-induced damaged sites
	Mureşan	
15:30 - 15:50	Bohuslav Rezek	Nano-analysis of composition and photovoltaic properties of
		nanodiamonds with polypyrrole
15:50 - 16:10	Łukasz Zarodkiewicz	MSA System company presentation
16:10 - 16:30		Coffee break

16:30 - 16:50	Ivan Oštádal	Cleaning tungsten STM tips for UHV measurements
16:50 - 17:10	Pavel Kocan	Electric field of an STM tip as a tool for probing stability of
		molecular layers
17:10 - 17:30	Jiří Doležal	Metastability of a copper phthalocyanine molecule on a thin
		insulating NaCl layer
17:30 - 17:50	Taras Chutora	On-surface synthesis of ethynylene bridged anthracene poly-
		mers
17:50 - 18:10	Benjamin Mallada	Atomic-scale study of impact of Boron and Nitrogen dopants
		in graphene chemical reactivity by STM/AFM/KPFM and
		DFT calculations.
18:10 - 18:30	Zdeněk Nováček	LiteScope TM AFM-in-SEM: New applications and tools for
		in-situ sample analysis, company presentation
19:00 - 23:00		Social evening

Friday 29th

		-
9:20 - 9:30	Lukáš Fojt	Characterization of carbon-based electrodes using AFM and
		Raman spectroscopy techniques
9:30 - 9:50	Marek Havlíček	Polymer spheres calibration for use in particle counters done
		by AFM
9:50 - 10:10	Jan Martinek	SThM and infrared microscope calibration method
10:10 - 10:30	Jan Vaniš	Characterization of ZnO nanorod heterostructures
10:30 - 10:50	Marek Černík	alpha300Ri Inverted 3D Confocal Raman Microscopy – com-
		pany presentation
10:50 - 11:10		Coffee break
11:10 - 11:30	Dušan Hemzal	Noble metal nanoparticles as template for successful charac-
		terization of biomolecules
11:30 - 11:50	Jan Přibyl	AFM based spectroscopy for nanomechanical mapping of li-
		ving cells, biomolecules and biomaterials
11:50 - 12:10	Tomáš Finsterle	Microscopic study change of adhesion after drug exposure on
		surface coating with diamond and gold nanoparticles
12:10 - 12:30	Dušan Novotný	Měřicí technika Morava s.r.o. company presentation
12:30		End of the workshop, lunch

ABSTRACTS

SPM Workshop 2019

Wed 13:10 – 13:50 Fernando Araujo de Castro

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Towards 3D nanoscale imaging of complex thin films using scanning probe microscopy

Scanning probe microscopy (SPM) is a versatile method to obtain nanoscale resolution imaging of materials properties. It uses a physical probe in contact or at close distance from the surface to probe a wide range of sample properties depending on the measurement mode and specific characteristics of the measurement system. These can include topographical, mechanical, optical, electrical, magnetic, and thermal, among other properties. Identifying and controlling the depth of probe (or probe volume) of a SPM method allows subsurface information to be extracted from 2D maps and continues to be a hot topic of research. Despite that, more often than not, SPM is still seen as a surface technique (2D). For the characterisation of thin films, the depth of information obtained by different SPM modes can be of similar dimension to the thickness of the sample, opening the possibility of 3D nanoscale imaging with SPM. In this presentation, I'll discuss two approaches we are taking to develop SPM based methods for 3D nanoscale characterisation. The first example makes use of hybrid data analysis of different SPM measurement channels: topography, photoconductive AFM and tip-enhanced optical spectroscopy. The second example is based on conductive-AFM tomography, where we demonstrate the possibility of using commercial conductive probes for both slicing and measuring the samples and we discuss challenges in data processing to create a 3D data volume reconstruction. The need for further work on calibration samples and procedures will be highlighted and ongoing activities in this direction will be discussed.

Acknowledgement

This work has been funded by the EMPIR 16ENG03 HyMET project and by the UK Department for Business, Energy and Industrial Strategy (BEIS) through the National Measurement System. The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States.

Wed 13:50 – 14:30 **Denis Vasyukov**

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Introduction to Scanning Microwave Microscopy

The scanning microwave microscopy (SMM) is a still rather new member of the family of scanning probe techniques. It has attracted attention due to its ability to characterize various electrical properties of samples, like dopant density, sheet conductance and dielectric properties. The basic working principle is to send a microwave signal to the scanning tip, where it is reflected depending on the sample underneath. The material parameters at the tip-sample contact determine the measured reflection coefficient (S₁₁) in amplitude and phase. As demonstrated recently in a report of SMM measurements on n-doped GaAs films [1], authors are able to extract quantitatively carrier densities of unknown GaAs samples after calibrating their SMM using S₁₁ measured on three samples with known dopant density. The method is based on the same principle as the short-open-load (SOL) algorithm used for the one-port calibration of vector network analyzers. In my talk, I will discuss the applications of SMM and new developments in the metrology of SMM measurements.

References

[1] Arne Buchter, et al. (2018), Scanning microwave microscopy applied to semiconducting GaAs structures, *Rev. Sci. Instrum.*, 89, 023704.

Wed 14:30 – 14:50 Anna Charvátová Campbell

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Nanoindentor tip analysis by AFM

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Nanoindentation is a popular method for the measurement of hardness and elastic modulus of nanoscale materials, such as nanocomposites, thin films and other nanostructured materials. The correct evaluation depends crucially on the correct evaluation of the so-called area function, which describes the shape of the indenter tip. The direct measurement of the area function by atomic force microscopy (AFM) is a less frequent approach. It has however its advantages such as full information about the shape, the possibility to distinguish contamination, and last but not least traceability. In this contribution we present approaches to the evaluation of the measured tip shape. Experimental settings, corrections of experimental data and data processing will be addressed. Sources of uncertainties will be analyzed and evaluated, the uncertainty budget will be modelled using Monte Carlo simulations.

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Wed 14:50 - 15:10 **Dušan Novotný**

Měřicí technika Morava s.r.o., company presentation

Wed 15:30 – 15:50 Teodor Gotszalk

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Array of electromagnetically cantilevers for force-distance spectroscopy metrological investigations

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Figure 1. Arrays of electromagnetically cantilevers: a) Type I b) Type II-for microsphere probes integration

Various actuation methods of cantilever deflection have been proposed including thermomechanical and electromagnetic schemes. In the electromagnetic technique the magnetic field interacts with the current flowing through a conductive loop exciting the Lorentz force [1,2]. In our investigations we fabricated, measured and applied 1×4 arrays of electromagnetic cantilevers. The designed and fabricated array cantilevers are applied in force-distance (F-z) spectroscopy investigations of molecular forces acting between



Figure 2. Deflection sensitivity of electromagnetically actuated cantilevers a) Type I, b) Type II

the functionalized flat cantilevers and a microspheres on the substrates [3]. The array setup and deflection control of every cantilever increases the investigation throughput and improves the reliability and repeatability of the conducted experiments. The arrays of electromagnetically cantilevers were fabricated in silicon on insulator (SOI) technology, which ensured that the beam geometry (beam thickness in particular) was the same within one array. As an input substrate the SOI 4" wafers with 1 and 1.5 micron thick buried oxide and device layer respectively was used. The handle and device layers are n-type conductivity and 3–5 Ohm cm resistivity. Boron doped layers deposition was applied to overcompensate the device layer for the p+ type. A 100 nm thick gold layer was sputtered on the wafer and the metal mirrors and the contact pads were defined in a photolithography process. Plasma etching was applied to define the final shape of cantilever. After the mask definition in a back side photolithography, in two separate plasma dry etching processes the silicon (handle wafer 400 μ m) and silicon dioxide (box 1 μ m) were etched. Application of permanent magnets and Helmholtz coils enabled a metrological cantilever characterization. Various arrays were designed which made it possible to fabricate structures of various resonance frequency (from 5 to 20 kHz) and stiffness (from 0.01 N/m to 0.1 N/m) - Fig. 1. The actuation sensitivity was characterized for static and resonance experiments, which confirmed that the fabricated sensors are of use in metrological force-distance spectroscopy - Fig. 2. Results of model chemical interactions between functionalized cantilevers and microspheres will be presented and discussed.

References

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[2] B. Lee, C. B. Prater, and W. P. King, *Nanotechnology* 23, 055709 (2012).

[3] D. Kopiec, P. Pałetko, K. Nieradka, W. Majstrzyk, P. Kunicki, A. Sierakowski, G. Jóźwiak, T. Gotszalk, Sensors and Actuators, B: Chemical, 213, 566-573, 2015.

Wed 15:50 – 16:10 **Daniel Haško**

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Measurement of the elastic properties of soft samples in fluids

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The reliable determination of the mechanical properties of soft samples by atomic force microscopy (AFM) is useful in biological research. AFM offers the ability to map local heterogeneities in elasticity relevant to the dimensions of the cells. Nowadays three dimensional (3D) cell cultures based on extracellular matrices begin to be used more frequently in biomedical research because they better mimic the natural arrangement of cells in native tissues. Except of synthetic artificial polymers the matrix may be natural, based on Matrigel or collagen type I. It was shown that the cellular shape, adhesion, viability, motility, proliferation, function and differentiation depend on extracellular matrix mechanical properties. In the present study two modes of AFM operating in fluids were used for measurement of elastic properties of Matrigel and collagen samples immersed in phosphate buffer solution in Petri dishes. From the recorded F-D curves the Young's modulus of the extracellular matrices has been calculated. The gained knowledge will be used in experiments utilizing 3D cell cultures based on extracellular matrices for development of clinically relevant in vitro tumour models.

Acknowledgement

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Wed 16:10 – 16:30 Vilma Buršíková

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Characterisation of plasma-polymer organosilicon composite thin films deposited under dusty plasma conditions

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During the past decades nanocomposite polymer coatings have attracted increasing attention because of their unique optical, mechanical, magnetic and optoelectronic properties arising from the combination of organic matrix and inorganic nanoparticles. Combinations of the attractive functionalities of both components at the nanolevel acquired from organic polymers and inorganic nanoparticles, are expected to exhibit synergistically improved material properties. Among the numerous nanocomposite preparation methods, the deposition under dusty plasma condition is one of the most attractive tools, which could be successfully applied in many industrial and technological applications, ranging from microelectronic industry to aerospace industry and biomedical applications. In the present work plasma-polymer nanocomposite thin films were prepared under dusty plasma conditions. Due to their nanocomposite structure, the films showed very interesting mechanical properties, for example high elastic recovery resulting in behaviour similar to superelasticity. Variation of the deposition conditions enabled to vary the surface composition and structure of the deposited films. The surface structure of the films influenced their surface free energy in a wide range so it was possible to prepare films with hydrophilic as well as hydrophobic properties. The mechanical properties of the films were studied using nanoindentation technique and the surface structure was studied using atomic force microscopy.

Wed 16:30 – 16:50 Jiří Buršík

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J. Buršík¹, V. Buršíková², G. Rogl³, P. Rogl³

Local mechanical properties of advanced thermoelectrics

¹Institute of Physics of Materials, Czech Academy of Sciences, Žižkova 22, Brno, Czech Republic ²Dept. of Physical Electronics, Faculty of Science, Masaryk University, Kotlářská 2, Brno, Czech Republic ³Institute of Materials Chemistry and Research, University of Vienna, Währingerstr. 42, Wien, Austria Skutterudites are known as excellent thermoelectric materials with a good efficiency for the conversion of heat to electricity. Researchers are trying to further enhance their thermoelectric properties e.g. by doping, filling, by dispersing nanoparticles or by nanostructuring. Various routes of production (consisting of various ways of powdering, compacting, deformation, annealing etc.) strongly influence the final properties. In this work we use an industrial p-type DDyFe₃CoSb₁2 skutterudite powder processed by several routes. We study the microstructure and local mechanical properties by means of analytical electron microscopy, instrumented depth sensing nanoindentation tests and AFM.

The work is supported by the Czech Science Foundation as project 17-12844S.

Wed 16:50 – 17:10 Egor Ukraintsev

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Peak Force AFM study of a chiral helicene-based macrocycles assembly on HOPG

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Large non-planar π -conjugated macrocycles have long been a target of intense research for their challenging synthesis and promising applications in material science [1]. We used olefin metathesis reaction to prepare triangular macrocycles bearing helicene building blocks. These molecules combine rigidity of π -conjugated structures with inherent chirality of the helicenes. The high molecular weight of the macrocycles does not allow their evaporative deposition and subsequent UHV AFM/STM imaging, ambient AFM technique was therefore employed.

The microscopic study of the helicene macrocycles was performed using ultrasharp SNL-B cantilevers (radius d = 2 nm) on ICON AFM in Peak Force QNM mode at low setpoint (20 - 100 pN). Freshly prepared macrocycle solutions with concentration 10 - 7 g/ml in dichloromethane were deposited by drop-casting on a freshly cleaved HOPG. On such samples, AFM was able to resolve the structure of individual 3 nm molecules. The experimentally observed distorted hexagonal structure is in agreement with theoretical calculations. Our AFM images showed existence of stripes with different directions. The 5.4° angle between the stripes might indicate that the stripes are formed by the macrocycles of opposite chirality. Similar pattern formation was observed earlier [2].

Annealing of the samples at 30 °C caused movement of the macrocycle molecules on the HOPG surface and their self-assembly into monolayers. More complex zigzag structures and higher multilayer nanocrystals were found on the annealed sample as well. In addition, even soft PFQNM measurements by AFM tip caused the movement of helicene molecules on the surface of HOPG, their alignment into patterns and removal of the higher multilayer nanocrystals. This research was supported by ERDF in the frame of the project "Centre of Advanced Applied Sciences", No. CZ.02.1.01/0.0/0.0/16_019/0000778 and Czech Science Foundation (reg. No. 18-20433S).

References

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Wed 17:10 – 17:30 **David Nečas**

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Tip dilation artefacts & roughness measurement – parametric approach

Morphological dilation of surface with AFM tip (tip convolution) is a more or less unavoidable effect which distorts the measured topography. Hence many reconstruction methods based on Legendre transformation or Villarrubia's morphological algorithms were proposed and studied as well as various other corrections for specific measured geometrical shapes. In characterisation of randomly rough surfaces we are usually only interested in statistical quantities, such as mean square roughness or autocorrelation length. Therefore, a different approach can be taken. Instead of attempting to reconstruct the surface, we find the map (true roughness parameters; tip parameters) \rightarrow (measured parameters) for a specific class of rough surfaces. This allows a simple and fast estimation of tip convolution influence and, to a certain degree, also correction. We explore this avenue for Gaussian surfaces, show how composite parameters allow reducing the dimensionality of the problem and elucidate some interesting related phenomena, such as the increase of measured roughness with tip wear.

Wed 17:30 – 17:50 **Filip Jakeš**

RMI, company presentation

Thu 9:00 – 9:40 Volker Neu

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Probing magnetic textures on the nanoscale

Magnetic force microscopy (MFM) has established its place as an extremely valuable method for the investigation of magnetic microstructures on the nanometer scale. Beyond being a purely qualitative imaging technique, quantitative MFM has the capability to locally measure the stray fields and eventually provide quantitative input data for a reconstruction of the underlying magnetization structure. This requires a full calibration of the imaging properties of the MFM tip and the most general approach is through the determination of the so-called tip transfer function (TTF) in Fourier space [1-3]. A calibrated tip transforms MFM signals into true stray field values on the nanometer scale. Furthermore, the field profile is corrected for the tip broadening and thus allows a true size determination of isolated magnetic objects. Reconstructing the magnetization texture from the stray field landscape will need additional knowledge on the sample. Here, micromagnetic simulations can help by providing valid initial magnetization models.

I will report on our activities within a current European metrology project [4] to establish materials, measurement protocols and analysis procedures for a routine application of qMFM [5] and will present various examples, which demonstrate the large benefit of treating MFM data quantitatively. These examples range from vortex states at nanowires to magnetic vortex textures in thin films with perpendicular anisotropy and to optically written domains in magnetic data storage media [6].

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References

[1] Hug et al. JAP 83 (1998)

[2] Vock et al. APL 105 (2014)

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[4] EMPIR 15SIB06 NanoMag [5] Nečas et al. Sci.Rep. 9 (2019)

[6] John et al. Sci. Rep. 7 (2017)

Thu 9:40 – 10:00 Matěj Hývl

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Matěj Hývl, Martin Ledinský, Antonín Fejfar

Contact force in C-AFM

In recent past, conductive AFM (C-AFM) has seen an increase usage as a unique tool for characterization of electrical properties of nano-electronic and nano-photonic devices. Despite this, there is still no universal method to obtain quantitative results via these measurements. So far, the only technique capable of such results is scanning spreading resistance microscopy (SSRM) using very high contact forces [1,2].

However, applying high forces is not viable for every sample or application and some experiments therefore need to be performed in low contact force regime. In our work, we will first present force-current curves obtained with C-AFM, demonstrating changes in the current detected on the gold sample based on the changes of the contact force. From the experiments performed on Bruker's Dimension ICON AFM it is apparent that small changes in the contact force lead to the significant changes in the detected current. We will also discuss artifacts created by changes in the contact force caused by topography of the sample.

References

[1] P. De Wolf, M. Geva, T. Hantschel, W. Vandervorst, and R. B. Bylsma, 'Two-dimensional carrier profiling of InP structures using scanning spreading resistance microscopy', Appl. Phys. Lett., vol. 73, no. 15, pp. 2155–2157, Oct. 1998.

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Thu 10:00 – 10:20 Jaroslav Kuliček

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Microscopic surface potential study for understanding ionic migration in perovskites

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Perovskites are one of the most intensively studied photovoltaic materials nowadays due a strong optical absorption, an adjustable band gap, long diffusion lengths, ambipolar charge transport, high carrier mobility, and a high tolerance of defects [1]. They are applied in various opto-electronic devices, such as solar cells, light-emitting diode, photodetectors, lasers [2]. Microscopic studies of photovoltaic materials can provide useful information about material roughness, conductivity, structure, mechanical and opto-electronic properties as well as about kinetic effects from short to long time scale. These characteristics are important for understanding and improving the photovoltaic performance. The microscopic study of organic perovskites with respect to their opto-electronic properties and ionic migration is presented in this work. The morphology of samples was studied by Atomic force microscopy (AFM). The first experiments were

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were performed on WITec alpha300 RAS combining AFM, KPFM, Raman and PL measurements. This work is a part of a wider study focused on the understanding of ionic migration in the perovskites. The work was supported by the project CZ.02.1.01/0.0/15_003/0000464 – Centre of Advanced Photovoltaics.

focused on finding optimal parameters for topography measurements. One of the goals was to correctly set parameters to remove the noise and instabilities occurring during the measurement due to various reasons. Surface potential of samples was measured by Kelvin probe force microscopy (KPFM). In this part, we also focused on optimizing the measurement parameters for reliable surface potential measurement. The photoluminescence (PL) of the samples was measured by Confocal Raman Microscopy. All measurements

References

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Thu 10:20 – 10:40 **Dušan Novotný**

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Měřicí technika Morava s.r.o., company presentation

Thu 11:00 – 11:40 Yannick De Wilde

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Near-field investigation of plasmonic materials and devices at infrared wavelengths

Classical optical microscopy allows one to detect propagating electromagnetic fields and is limited in spatial resolution to approximately half the wavelength of observation. This diffraction limit prevents one in principle to perform nano-optical observations in the infrared, where the wavelength is typically in the range of tens of micrometers. For the same reason, Fourier transform infrared spectroscopy cannot achieve a spatial resolution better than tens of micrometers, which often restricts the technique to global investigations of large size samples.

In this presentation, we will show how the use of scattering type near-field scanning optical microscopy (sNSOM) in the infrared allows one to overcome the diffraction limit and to capture physical phenomena involving purely evanescent electromagnetic fields. We will discuss the adaptation of the sNSOM to the detection of the sole thermal radiation of a sample in the near-field zone in a mode called thermal radiation scanning tunneling microscopy (TRSTM). The capabilities of the techniques will be illustrated by several examples like the near-field investigation of semiconductor laser sources, and the thermal radiation associated to surface polaritons and infrared plasmonic antennas.

Thu 11:40 - 12:00 Petr Klapetek

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FDTD calculations of infrared SNOM probes

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Infrared scattering Scanning Near Field Optical Microscopy is a promising way how to get chemical resolution within the family of Scanning Probe Microscopy techniques. Similarly to other optical techniques in SPM the method is highly dependent on existence of suitable probes. In this contribution, numerical tools for calculating the probe response will be presented, based on the open source Finite Difference in Time Domain method solver GSvit. Use of numerical computing allows better understanding of the phenomena that lead to possibility of obtaining high resolution results and allows optimization of the probes for best possible performance for particula wavelengths or materials. Moreover, attempts to create novel probes experimentally and first measurement results will be presented.

Thu 12:00 – 12:20 **Jan Vávra**

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Neaspec, company presentation Scattering-Type Scanning Near-field Optical Microscopy and Spectroscopy for Nanoscale Chemical Analysis

Adrian Cernescu, Sergiu Amarie, Jan Vávra

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Scattering-type Scanning Near-field Optical Microscopy (s-SNOM) is an optical microscopy and spectroscopy approach based on scanning probe technology, bypassing the ubiquitous diffraction limit of light to achieve a spatial resolution below 20nanometers. s-SNOM employs the strong confinement of light at the apex of a sharp metallic AFM tip to create a nanoscale optical hot-spot. Analyzing the scattered light from the tip enables the extraction of the optical properties absorption, reflectivity) of the sample directly below the tip and yields nanoscale resolved images and nanoscale spectroscopy (hyperspectral nano-FTIR) information simultaneous to topography. This presentation we will introduce the basic principle of near-field microscopy and hyperspectral nano-FTIR for imaging and spectroscopy with 10 nanometer spatial resolution. In addition we will summarize the latest achievements in the field of near-field microscopy and spectroscopy on polymers, biomaterials and 2D materials and will focus on applications in chemical analysis and material identification at the nanoscale.

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Thu 14:50 – 15:10 Radovan Vraník

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Radio-frequency scanning tunneling spectroscopy of helicene molecules on Ag(111)

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²Linz Institute of Technology (LIT), Johannes Kepler University Linz, Altenbergerstrasse 69, Linz, Austria Radio frequency scanning tunneling spectroscopy (RF-STS) is an advanced experimental technique which already lead to successful investigation of single molecules by exciting not only their mechanical [1,2], but also spin [3,4] and plasmonic [5] degrees of freedom. This very unique technique provides a synergy of the high spatial resolution of low-temperature (LT) STM and the possibility to investigate mechanical resonance effects of single molecules. As a demonstration of what RF-STS is capable of, we report on excitation of mechanical eigenmodes related to helicene derivatives adsorbed on Ag(111) observed at frequencies between 150 and 450 MHz. References

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- [5] G. Serrano et al., Sci. Rep. 7, 9708 (2017).

Thu 15:10 – 15:30 Mihai-George Mureşan

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AFM measurements of laser-induced damaged sites

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²Czech Metrology Institute, Department of Primary Nanometrology and Technical Length, Brno, Czechia Moving toward high-power laser systems requires a better understanding of the laser beam transport limitations. The major one is related to the maximum fluence of certain optical elements – AR/HR (antireflecting/high-reflecting) coatings can withstand without being damaged. Laser-induced damage threshold testing (as described by ISO21254-1-5) is used to evaluate the surface resistance of an optical element. Surface characterization is usually done using optical microscopes but employment of smaller laser sources for testing requires a different approach in order to correctly quantify the damage. AFM seems particularly interesting, as it is capable of performing calibration for optical recognition of the damage sites. The surface damage measured using tapping mode AFM were compared with laser confocal microscope scans.

Thu 15:30 – 15:50 **Bohuslav Rezek**

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Nano-analysis of composition and photovoltaic properties of nanodiamonds with polypyrrole

Inexpensive, unobtrusive, and safe renewable energy sources are nowadays increasing in importance. We will present and discuss experimental methodology for studying interactions of organic dye (polypyrrole) with diamond nanoparticles by comparing atomic force microscopy, microscopic and microscopic Kelvin probe and infrared spectroscopy on pristine and composite materials. We also analyze influence of illumination and correlate the data with theoretical DFT simulations.

Thu 15:50 – 16:10 Łukasz Zarodkiewicz

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MSA System, company presentation

Thu 16:30 – 16:50 Ivan Ošťádal

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Ivan Oštádal, Pavel Sobotík, Pavel Kocán

Cleaning tungsten STM tips for UHV measurements

Tungsten tips prepared by electrochemical etching have to be cleaned and stabilized for successful STM measurements under UHV conditions. A layer of a natural oxide and other residual adsorbates on a tip apex result in unstable tunneling barrier and random fluctuations of tunneling current at a fixed position above investigated surface. Additionally an undesired interaction between the clean tip and surface during

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scanning may cause collecting of adsorbate atoms on the tip apex and changing electronic properties of the tip. The STM measurements in UHV require a presence of an effective in-situ procedure for cleaning tips. Especially reliable scanning tunneling spectroscopy measurements need the tip cleaning.

Mostly used methods for the tip cleaning are compared and discussed with respect of manipulation comfort and consequences for the tip quality. Finally a design of a new "ready to use" solution is introduced and the final form implemented at an UHV STM head developed in our laboratory is presented.

Thu 16:50 – 17:10 **Pavel Kocán**

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Pavel Kocán¹, Peter Matvija¹, Pavel Sobotík¹, Barbara Pieczyrak², Leszek Jurczyszyn², Filip Rozbořil¹, Ivan Oštádal¹

Electric field of an STM tip as a tool for probing stability of molecular layers

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Ordering of planar organic molecules adsorbed on metal-passivated surfaces is influenced by two factors: First, surface periodicity represents a grid for the molecules and thus dictates possible separations between them. Second, atomic structure of the molecules, especially chemical properties of molecular periphery, drives interaction between the molecules. While fluorinated copper phthalocyanines ($F_{16}CuPC$) on Si(111)-Tl(1×1) do not tend to self-order and instead form a 2D gas [1], non-fluorinated copper phthalocyanines ($F_{0}CuPC$) form on the same surface ordered structures but only at close-to-monolayer coverage or in a strong field of an STM tip [2]. For a detailed comparison we extend the study to molecular mixtures of $F_{16}CuPC$ and $_{0}CuPC$ and to the molecules fluorinated just partially ($F_{8}CuPC$).

The STM technique is used not only to visualize the molecular structures, but also to probe their stability. The high-gradient electric field of the STM tip interacts with dipole-carrying molecules on the surface and the resulting forces stabilize or destabilize the molecular structures depending on applied voltage.

The home-made room-temperature STM is used to study ordering of the phthalocyanines on the Si(111)- $Tl(1\times 1)$ surface and stability of the ordered islands with respect to the field of the STM tip. Ab-initio calculations show the lowest-energy arrangements and the charge transfers induced by adsorption of the molecules. Kinetic Monte Carlo simulations are used to study in detail kinetic aspects of molecular behavior in the strong electric field.

References

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 P. Matvija, F. Rozbořil, P. Sobotík, I. Ošt'ádal, B. Pieczyrak, L. Jurczyszyn, Scientific Reports 7, 7357 (2017).

Thu 17:10 – 17:30 **Jiří Doležal**

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Jiří Doležal^{1,2}, Jesus Redondo^{1,2}, Aleš Cahlík¹, Martin Švec¹

Metastability of a copper phthalocyanine molecule on a thin insulating NaCl layer

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We studied the origin of the metastability of single copper phthalocyanine (CuPc) molecules on a NaCl bilayer on Cu(100) by a combination of scanning tunneling microscopy (STM) and non-contact atomic

force microscopy (nc-AFM) with CO-functionalized tips at 5 K. It was proposed that the mechanism of reversible switching of the molecule is injection or removal of an electron to/from its LUMO, resulting in a metastable neutral or anion charge state of the CuPc [1].

By dI/dV mapping with Cu- and CO-tips and their comparison with the theory, we first identified the



Figure 1: Simultaneously acquired constant height STM and AFM image of CuPc with CO tip. The molecule state was spontaneously switched during the scanning (bottom-up) from the "anion" to the "neutral" state. Sample bias = 25 mV.

electronic levels possibly playing role in the charging process. Subsequently we used the methodology of electrostatic charge mapping to evaluate the charge redistribution within the CuPc, when it is switched between the two states [2]. The frequency-shift vs. distance spectroscopy was employed to measure a vertical relaxation and distortion of the CuPc due to the switching.

We find a negligible charge redistribution, but a consistent vertical shift and bending of the CuPc. These findings challenge the proposed origin of metastability as caused by charging of the molecular level and point toward a conformational switching of the CuPc/NaCl system.

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Thu 17:30 – 17:50 **Taras Chutora**

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On-surface synthesis of ethynylene bridged anthracene polymers

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Engineering and producing low band gap π -conjugated polymers [1] is a burgeoning area in basic and applied research. We report an unprecedented on-surface synthesis protocol to design poly(p-anthracene ethynylene) molecular wires on an Au(111) surface.

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Figure 1: Representation of the thermal reaction steps which enable polymerization.

Here we report on RT deposition of a quinoid anthracene precursor on Au(111) substrate and inspection with non-contact atomic force microscopy (NC-AFM) and scanning tunnelling microscopy (STM). Subsequent annealing to 400 K resulting in anthracene-based polymers, with a measured electronic bandgap of 1.5 eV. High-resolution NC-AFM with a CO functionalized tip [2] unambiguously corroborates the nature of the ethynylene bridge bond between anthracene moieties. Complementary theoretical simulations of the reaction pathways illustrate the mechanism of the chemical reaction, highlighting three major steps (see Figure 1).

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Thu 17:50 – 18:10 Benjamin Mallada

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Atomic-scale study of impact of Boron and Nitrogen dopants in graphene chemical reactivity by STM/AFM/KPFM and DFT calculations.

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Controllable incorporation of nitrogen and boron heteroatoms in graphene provides donor and acceptors centers tuning its electronic, magnetic properties and chemical reactivity with potential application on storage energy and molecular sensors [1]. Despite the enormous interest developed, an atomistic investigation of the doped graphene chemical reactivity is missing. We report our combined experimental Scanning Tunneling Microscopy (STM), Atomic Force Microscopy (AFM) and Kelvin Probe Force Microscopy (KPFM) measurements at 5K in Ultra High Vacuum (UHV) of Nitrogen and Boron co-doped graphene on SiC(0001) [2]. Our atomically resolved AFM images with CO-functionalized tip, reveal a very distinct contrast for graphitic N and B dopants attributed to electron density variations. This finding is corroborated by both experimental and calculated interaction energy at the dopant sites which is dominated by the electrostatic potential. Additionally, a shift in the local work function as a result of lend-accommodation of electrons from the graphene to the dopants is observed by means of KPFM measurements [3]. Remarkably, we map the in- and out- of the plane electrostatic potential of the dopants highlighting the relevance of the polarization for tuning chemical reactivity of graphene.

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Thu 18:10 – 18:30 Zdeněk Nováček

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LiteScopeTM AFM-in-SEM: New applications and tools for in-situ sample analysis, company presentation

Precise analysis via multiple techniques is desired for a successful development of various materials and applications in many scientific fields. The combination of two widely used techniques such as scanning electron microscopy (SEM) and atomic force microscopy (AFM) is not only very useful but necessary in many applications.

LiteScopeTM AFM produced by NenoVision is carefully designed for direct integration into many different SEM microscopes. It extends the instrument's capabilities and offers several benefits like 3D surface characterization, height/depth profiling, surface roughness calculation, precise tip navigation, local electric conductivity estimation, spectroscopic regimes, other electric and magnetic measurements, etc. all at the same ambient conditions during simultaneous measurement. LiteScopeTM is not only easy to install but also easy to use. Moreover, it has unique and patented technology for true correlative imaging, so-called Correlative Probe and Electron MicroscopyTM (CPEM). The secret behind this method is that neither the electron beam nor the AFM probe tip is scanning. The movement is done by the piezo scanner with the sample while the AFM tip and the electron beam have a constant shift of known value. CPEM can accommodate several signals from different detectors (SE, BSE) or related techniques like FIB, EBIC or CL. LiteScopeTM offers also a new approach to conductive measurements, so-called conductive CPEM (c-CPEM). This regime can provide contrast on conductive particles in a nonconductive matrix. Capabilities of this device including new applications will be presented on various samples and materials.

Fri 9:20 – 9:30 Lukáš Fojt

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Characterization of carbon-based electrodes using AFM and Raman spectroscopy techniques

We have used AFM and Raman spectroscopy for characterization of different carbon-based electrodes used in electrochemical research of boron cluster compounds. We have studied glassy carbon electrodes (with different surface treatments), pyrolytic graphite electrodes (in basal and edge orientation), screen printed graphite electrodes (with different working electrodes material composition) and as a last one pencil graphite electrodes. For our purposes, the polished glassy carbon electrode was selected as the best one material and polishing by 1 μ m diamond particles as the best surface treatment.

This work was supported by the Grant No. 19-04630S from Czech Science Foundation.

Fri 9:30 – 9:50 Marek Havlíček

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Marek Havlíček $^{1,2},$ Jiří Šperka¹, Petr Klapetek 1,2

Polymer spheres calibration for use in particle counters done by AFM

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Calibration of the particle counters is typically done using monodisperse reference particles such as polymer spheres. Correct determination of the particle sizes is a key factor for the precise and repeatable measurements. The diameter and the size distribution of the reference particles can be determined in a traceable way using atomic force microscope (AFM). The height of the particles corresponding to their diameter can be automatically evaluated using SPM software such as Gwyddion using built-in functions. The reference surface roughness, tip-particle/substrate interaction and other possible deviations will be discussed.

Fri 9:50 – 10:10 Jan Martinek

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SThM and infrared microscope calibration method

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In today's highly integrated microelectronic systems there is a need for high resolution spatial temperature measurement on chips. The resolution requirements are higher than the infrared imaging system are capable of and the investigated areas of the chips are often being rather large for the most common Scanning Thermal Microscope (SThM).

In the presentation, we describe two quantitative methods to acquire the thermal map with high resolution on large area – noncontact method based on infrared radiation and Scanning Thermal Microscopy (SThM). In both methods the expected thermal properties of the sample were thoroughly calculated and the prediction was in agreement with the experimental results. For the study of infrared radiation the composition of the sample together with the spectral sensitivity of the sensor were taken into account. In the SThM part, there were discrepancies based on unequal conditions during calibration and subsequent measurement. Using the finite element method (FEM) simulation of thermal field the problem has been solved and successfully experimentally verified.

For both methods a special sample with embedded thermometer capable of being heated internally or externally was used.

Fri 10:10 – 10:30 **Jan Vaniš**

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J. Vaniš, S. Tiagulskyi, J. Zelinka, Š. Kučerová, H. Faitová and J. Grym

Characterization of ZnO nanorod heterostructures

We report on the characterization of ZnO nanorod heterostructures by conductive atomic force microscopy (CAFM) and by tungsten nanomanipulator needle in FIB-SEM-GIS-SIMS apparatus. We compare both methods and discuss the influence of the contact geometry and of the sample surface treatment on the

electrical characteristics. Moreover, the time evolution of the measuring system with its subsequent degradation is reported. The understanding of the electric charge transport and Schottky barrier formation at the nanoscale is essential for the application of semiconductor nanostructures in electronic and photonic devices.

This work was supported by the Czech Science Foundation project 17-00355S and 19-02804S.

Fri 10:30 – 10:50 **Marek Černík**

Uni-Export Instruments, company presentation Alpha300Ri Inverted 3D Confocal Raman Microscopy

Fri 11:10 – 11:30 **Dušan Hemzal**

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Noble metal nanoparticles as template for successful characterization of biomolecules

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Figure 1: Room ambient SPM measurements over SiO_2/Au substrate. Left: STM topography map of the bare substrate with high-resolution inset (scale bar is 5 nm). Right: AFM phase-image of Rtt103 protein immobilized over the SiO2/Au substrate.

Characteristic size of biologically relevant organic molecules such as proteins or DNA poses constraints on substrates on which these molecules need to be immobilized for successful SPM (ie. STM or AFM) characterization. As a matter of fact, substrates such as mica or HOPG with atomically flat surfaces are required. While these substrates are widely accessible, their surface modification can be somewhat tedious and, of course, the non-conducting nature of mica is contradicting STM measurements.

As a remedy to the above complications we turn to metallic surfaces. Luckily, even a simple PVD gold surface over well polished glass slide is capable of providing well resolved room-ambient AFM images (see Fig. 1). Further decrease of surface roughness can be reached using template- stripping approach [1].

However, to reach pure crystallographic surfaces we present our running work results on using noble metal nanoparticles in role of an SPM substrate. There are many reliable synthesis protocols [2] that allow



Figure 2: SEM image of mixture of silver nanoparticles with absorbance measurement shown in the inset (left) and STM of Rtt103 protein over atomically resolved gold surface (right), the scale bar is 1 nm.



Figure 3: Tunable-excitation surface enhanced resonance Raman spectra (CWT-SERRS) spectra of 1uM solution of rhodamine 6G in Ag NP colloid (with absorption maximum at 450 nm). The excitation wavelengths are given in legend, the vertical axes share absolute units (normalised to 50 s acquisition at 100 uW). The inset shows conventional Raman spectrum of 100 uM R6G (acquisition 100 s by 30 mW at 633 nm). As the optical path for both samples was same, the SERRS enhancement can be easily calculated, close to $EF=10^6$ at 1510/cm peak.

controlled formation of flat prisms – with thickness in the range 5-10 nm and lateral dimension 20-100 nm (see Fig. 2). In the same time, the anti-aggregation protection of the nanoparticles obtained during their synthesis serves as active surface modification and can be altered using simple tools.

Finally, size-tuning of the nanoparticles can be used to obtain collocalized plasmonic enhanced Raman spectra (SERS) of the (bio)molecules (see Fig. 3).

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Fri 11:30 – 11:50 **Jan Přibyl**

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AFM based spectroscopy for nanomechanical mapping of living cells, biomolecules and biomaterials

Atomic Force Microscope (AFM) is a three-dimensional high-resolution topographic technique. It is suitable for biological applications in native conditions with the ability to measure bending of the cantilever probe with extremely high precision. This allows AFM to be used as a mechanical nanosensor, in cell based biosensing of drug effects and in study of growth factor effects on cells. Among the multitude of methods applied to measure the stiffness of cells and tissues, micro-indentation using an Atomic Force Microscopy (AFM) provides a way to reliably measure the stiffness of living cells.

Among the other devices for microindentation the Atomic Force Microscope (AFM) is commercially available and has been widely applied to characterize mechanical properties of living cells and tissues. Moreover, AFM allows to scan living cell topography under nearly physiological conditions (liquid medium, elevated temperature), and offers a force spectroscopy mode. In this mode, cell is indented at many sites and its complete elastic response is recorded which enables to reconstruct its stiffness map. However not only living cell cultures can be investigated by this method, but also a variety of soft bio samples such as plant tissues, extracellular matrix and hydrogels can be studied. Combination of the stiffness mapping with confocal microscopy would bring better understanding to the cellular processes, when the cytoskeleton remodelling is related to the mechanical properties of the cell.



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Fri 11:50 – 12:10 Tomáš Finsterle

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Tomáš Finsterle¹, Ivana Pilarčíková¹, Ilona Ali Bláhová¹, Egor Ukraintsev^{1,2}, Štěpán Potocký², Štěpán Stehlík², Alexander Kromka², Eugenie Nepovimová³, Kamil Musílek³, Kamil Kuča³, Bohuslav Rezek¹

Microscopic study change of adhesion after drug exposure on surface coating with diamond and gold nanoparticles

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Figure 1a) Detail of adhesion map, b) Distribution of adhesion force between Si cantilever and sensors with various nanoparticles prior and after drug exposure, c) Model showing the drug configuration on H-DND and O-DND with embedded Au nanoparticles.

The easy and fast detection of drug content and concentration levels is demanded in biological research as well as in clinical practice. Therefore, label-free platforms based on electrical detection are intensively developed. Nanomaterials and structures are often introduced to provide a fast and sensitive response. Nanodiamonds have unique properties for use in biosensors.

Here we study on microscopic level how nanodiamonds and gold nanoparticles interact with drug molecules. The sensors were made of interdigitated Au electrodes coated by nanodiamonds. Both types of sensors were also further combined with Au colloidal nanoparticles (AuNP, size 20 nm) for providing nanoscale binding sites (spacing 100 nm). The morphological differences and chemical compositions and molecular vibrations on the prepared sensors were characterized by scanning electron microscopy (SEM). Atomic force microscopy with CF4 treated tips was employed to measure local tip-surface adhesion forces and surface topography. Correlation of the EDX and FTIR spectroscopic data with the obtained AFM adhesion maps shows that the drug binds to all nanoparticle materials (H-DND, O-DND, AuNP) be their standalone or combined in a composite, although the drug molecules are in different configurations.

Fri 12:10 – 12:30 **Dušan Novotný**

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Měřicí technika Morava s.r.o., company presentation

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