Electrochemistry on 2D transition metal dichalcogenides

Ass. Prof. Kim McKelvey
School of Chemistry, Trinity College Dublin - Microelectronics and Electrical Engineering, College St, Dublin, Ireland

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

The layered structure of transition metal dichalcogenides (TMDCs) enables the synthesis of single- and few-layer two-dimensional (2D) materials with unique electronic, chemical, and mechanical properties. However, the lateral 2D feature size has limited efforts to quantify the electrochemical response on these types of materials. Using a scanning electrochemical cell microscopy approach, we investigate the effect of layer thickness on electrochemical response on 2D TMDCs, isolating the response from individual layers and quantifying how the electrochemical activity varies with electronic structure.

Hybrid electrochemical scanning probe microscopy: Physical and electrochemical characterization of interfaces

Prof. Dr. Christine Kranz
Institute of Analytical and Bioanalytical Chemistry, University of Ulm, 89069 Ulm, Germany

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

Hybrid scanning probe techniques such as scanning ion conductance microscopy (SICM) – scanning electrochemical microscopy (SECM) and atomic force microscopy (AFM)-SECM have gained tremendous importance in physical and electrochemical characterization of interfaces [1]. Our research team has pioneered combined AFM-SECM using AFM-tip integrated electrodes, which are not in direct contact with the sample surface [2], and therefore allow simultaneous mapping of physical and electrochemical properties at electrified interfaces in contrast to other approaches based on metallic/metallized AFM tips [3,4]. Recently, we introduced a new type of electrochemical scanning probe tip for combined AFM-SECM measurements providing instead of a conventional AFM tip, a conductive colloid sphere attached to an otherwise insulated cantilever [5]. Such conductive spherical probes may consist of boron-doped diamond [6], a highly attractive electrode material, or may be electrochemically modified, e.g., with polymers, highly suitable for force spectroscopic measurements such as adhesion measurements under potential control.
Within this contribution, selected examples of combined electrochemical and nanomechanical mapping will be presented and discussed along with experimental challenges. In addition, electrochemical force spectroscopic measurements with polydopamine (PDA)-modified colloidal probes will be presented taking advantage of the fact that the surface functionality of PDA and hence the force interaction can be altered by oxidation or reduction of functional groups.

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KPFM for electrochemical studies: From corrosion to battery materials

Dr. Reynier I. Revilla
SURF Electrochemical and Surface Engineering, Vrije Universiteit Brussel, Pleinlaan 2, 1050 Brussel, Belgium

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

Among the different AFM modes, Kelvin probe force microscopy (KPFM) is one of the most used in the field of electrochemistry. Its popularity has greatly increased in recent years. This technique, first applied in the field of corrosion science about 20 years ago, allows direct measurement of the local Volta potential differences resulting from microstructural inhomogeneities in metals and alloys [1]. The measured surface potential by KPFM is directly related to the work function and, therefore, to the chemical potential of the surface. Due to its good sensitivity and spatial resolution, KPFM is an excellent technique for mechanistic studies on the relation between microstructure and the corrosion behavior of metals and alloys. It allows the identification of potentially (electrochemically) active zones and galvanic interactions between the microstructural features of metals, which could play an important role in the pitting corrosion process of these materials.
Besides its application for corrosion studies, KPFM has recently also gained attention in the study of battery materials. KPFM surface potential mappings have been used, among others, to gain insights into the aging mechanism of battery cathode materials.
In this talk, three case studies relevant for the field of electrochemistry are presented. The first one highlights the link between microstructure and corrosion behavior [1-3]. Here, a common misunderstanding concerning the link between surface potential difference and galvanic interaction between different materials is also discussed. The second example shows a KPFM-based approach to study the growth kinetics of the surface native oxide film of metals in air [5]. Finally, the last example provides an analysis of the direct correlation between the local surface potential measured by KPFM and the electrochemical potential of a Li ion-based cathode, and its application to study the cycling aging mechanism of this material [6,7]. These three case studies are part of research lines currently active in the Research Group Electrochemical and Surface Engineering (SURF) of the Vrije Universiteit Brussel (VUB).

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Correlative Electrochemical Multi-Microscopy: Building an Understanding of Electrochemical Interfaces From Local to Global.

Prof. Patrick R. Unwin
Department of Chemistry, University of Warwick, Gibbet Hill, Coventry, CV4 7AL, United Kingdom

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

Electrodes used in electrochemistry, with applications in electrocatalysis, energy storage, sensor technologies and corrosion, are heterogeneous and complex on a range of lengthscales. We advocate new approaches to study electrochemical and electrocatalytic phenomena, whereby the activity of an electrode is visualized by electrochemical microscopy in the form of "activity maps" and "potentiodynamic movies". These quantitative data are then related to co-located electrode structure from complementary high-resolution microscopy and spectroscopy techniques applied in the same area of the electrode. This correlative electrochemical multi-microscopy approach seeks to relate electrode structure to activity clearly and unambiguously. In our work, scanning electrochemical cell microscopy (SECCM) and scanning ion conductance microscopy (SICM) are key techniques that are used for the acquisition of both electrochemical activity maps/movies and topography – synchronously and with nanoscale spatial resolution. These techniques use nanopipette electrochemical (half) cells as probes, which can be made easily in a variety of formats, characterised in detail, and their response modelled. By overlaying data from these measurements with images from co-located microscopy techniques, e.g. electron microscopy and electron backscatter diffraction, micro-Raman spectroscopy and atomic force microscopy, we obtain a deep view of electrochemical interfaces and processes.
This is a general platform for investigating electrochemical interfaces, and illustrative examples of this approach will be

described, including the study of: layered materials, structurally and/or compositionally heterogeneous surfaces such as polycrystalline metals, screen printed electrodes and conducting polymers, and ensemble electrodes comprising of nanoparticles on an electrode support surface, with applications in electrocatalysis and energy storage (batteries). We will also touch on how SECCM and SICM can be used for controlled delivery and to pattern and fabricate structures at interfaces.

A key thesis of our work is that complex electrode surfaces can be broken down and studied as set of simpler "single entities" (e.g., individual steps, terraces, defects, crystal facets, grain boundaries, single particles). The resulting nanoscale understanding of electrochemical reactivity can then be used to create scalable models for electrochemical interfaces that will ultimately facilitate the rational design of functional (electro)materials.

Many talented people at Warwick and elsewhere have contributed enormously to our work in this area over the past decade. I am indebted to them for their significant contributions and they will be acknowledged throughout.

Nanorheological AFM for Basic Polymer Science

Prof. Ken Nakajima
Department of Chemical Science and Engineering, Tokyo Institute of Technology, Tokyo, Japan

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

Nanorheological measurement based on AFM is reviewed with showing several examples of polymeric materials. Dynamic modulus determination, such as storage (E'), loss (E") moduli and loss tangent with wide-frequency range is now realized by this technique. [1, 2]

The first example uses its capability of wide frequency measurement and its temperature control function to validate the time-temperature superposition (TTS) principle. We used styrene-butadiene rubber (SBR) vulcanizate of which glass-rubber transition temperature, Tg is –27°C. [3] It was confirmed that the viscoelastic values of a pure SBR vulcanizate were quantitatively equivalent to bulk DMA results. TTS holds even at nanoscale if just viewing "average," while some breakdown occurs due to heterogeneous nature of GR transition.

As the second example, we applied this technique to silica-filled SBR to investigate the nature of the interfacial rubber region. [4] Its dynamics properties were different from those of matrix rubber regions. The master curve obtained by this technique perfectly coincided with that by bulk DMA. However, by examining the frequency-dependent change of E", we found that this transition does not occur uniformly depending on spatially heterogeneous polymer segmental dynamics.

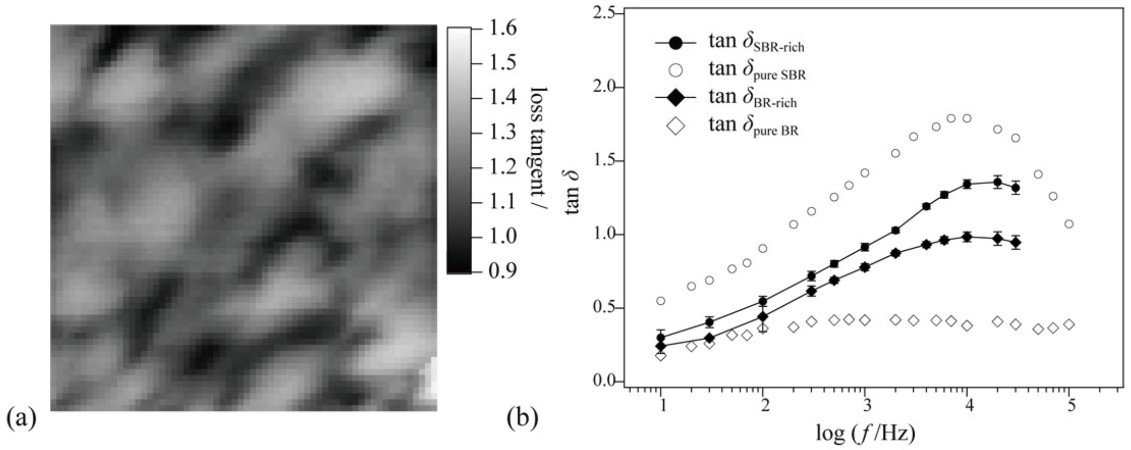

Figure 1. (a) Loss tangent map of SBR/BR blend by nanorheological AFM (f =20 kHz). The scan size is 2.0 μm. (b) Frequency dependence of loss tangent for both SBR/BR blend and each homopolymer.

The miscibility between two polymer species is the third example. Nanorheological AFM was utilized to investigate the nature of a SBR/butadiene rubber (BR) partially miscible blend. The blend seemed to be immiscible according to SEM and conventional AFM techniques. However, when both E' and E" for the blends were compared with those for the homopolymers, values for both the SBR-rich and BR-rich regions did not coincide with those for the pure components as shown in Fig. 1. In particular, the BR-rich region, exhibited a dynamic heterogeneity, where the frequency responses were much more complicated than those of the BR homopolymer, though DSC could not detect it as the change of T_g.

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Characterization of metallo-DNA nanostructures properties with AFM

Dr. Fátima Linares Ordóñez
Centro de Instrumentación Científica, University of Granada, Paseo Profesor Juan Osorio S/N
18071 Granada, Spain

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

Nanotechnology applications rely on the capability to prepare and control functional materials at the nanoscale with high precision [1–3]. The development of novel methodologies that enhance this control has attracted the attention of scientists worldwide. In particular, the development of new strategies towards the fabrication of nanowires with tailored electronic and optical properties has been the subject of extensive efforts due to the practical applications they may offer for the miniaturization of electronic circuits. Importantly, in order to design new nanocircuits the correct organization of the nanowires must be precisely controlled. In this context, DNA has become an important tool for scientists, since intricate functional nanostructures can be prepared based on its sequence programmability, which offers potential applications in nanomachinery, sensing devices, drug delivery, and information processing [4–6]. However, the concept of exploiting the self-organizing properties of DNA to create nanocircuits remains a great challenge and the source of much contentious discussion. This is due to the debatable conducting properties in natural DNA molecules, with some reports pointing towards the insulating behavior of long DNA molecules, whilst other studies have demonstrated charge transfer in short DNA molecules. The electronic properties of DNA molecules have been enhanced using various strategies, including the covalent attachment of functional groups [7,8] or the deposition of a conducting material [9] including different metal atoms (i.e. Ag, Pd, Cu, Au, and Rh). Metallization of DNA molecules has proved to afford DNAnanowires with improved conductivity [10–14].

In this work, we show results for three different helical silver complexes whose molecular structure revealed the formation of helixes consisting of stacked mC–Ag(I)–mC homobase pairs, with the presence of contiguous Ag(I) chains running through the centre of the helixes and building nanowires structures. Their morphology and conductivity behavior were studied by means of AFM, EFM and c-AFM.

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AFM tip-induced strain effects in BiFeO3 films: from structural phase changes to polarization switching and nanofabrication

Prof. Brian Rodriguez
University College Dublin, School of Physics, Conway Institute Belfield Dublin 4, Ireland

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

Recent studies have emphasized the critical role that local mechanical stress can play on the functionality of ferroelectric materials. For instance, stress applied from an atomic force microscope (AFM) tip has been shown to induce polarization reversal through the flexoelectric effect in ultrathin films, where the strain gradients can permeate through the film thickness [1]. In addition, local stress can alter electric field-induced switching behavior, affecting hysteresis loop shape, coercive field, and remnant polarization [2]. The mixed phase form of epitaxially-strained BiFeO3 thin films, having a coexistence of rhombohedral-like R phase, comprising needle-like structures, within a tetragonal-like T phase matrix, meanwhile show a particular susceptibility to applied stress. The effects include transitions between phases, with the resulting boundaries demonstrating enhanced conductivity [3], and stress-induced alterations to electric field-induced hysteresis [4]. In this work, a loading-force dependent evolution of strain effects is reported, from structural phase changes at low forces (< 1 μN) to nanomechanical machining at higher forces (~ 1 µN). Switching behavior and switching currents are also found to be affected by force, with coercive fields decreasing with increasing loading force. Notably, mechanically-induced currents increase with applied force and may enable polarization state determination in ferroelectric memory applications. Lastly, at high forces > 10 µN, a moving tip can be used to remove material. Through a systematic investigation of a range of AFM parameters, including force, we demonstrate that AFM-based machining is an effective tool for rapid and precise modification of local regions of the film, as well as for the fabrication of several different nanostructures, including nanocapacitor arrays with individually addressable ferroelectric elements. AFM-based machining of ferroelectric nanostructures offers advantages over established techniques, such as bottom-up approaches and focused ion beam milling, in select cases where low damage and low-cost modification of already-fabricated thin films are required.

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Quantification of nanoscale electromechanical responses

Dr. Neus Domingo
Institut Català de Nanociencia i Nanotecnologia, CSIC and The Barcelona Institute of Science and Technology, Campus UAB, Bellaterra, Barcelona E-08193, Spain

Session Thursday, 24 September: [Advanced research on ferroelectric materials](#) I [Join the session](#)

Surface electromechanics at the nanoscale are typically studied by Piezoresponse Force Microscopy (PFM), based on the inverse piezoelectric effect. As a first approach, generally only homogeneous responses are taken into account, but it has been realized that the effect of gradients in electromechanical phenomena at the nanoscale can become dominating: the generation of electrical signals after the application of mechanical strain gradients with an AFM tip has been proved, and it has been shown that it is possible to write ferroelectric domains [1] or to move oxygen vacancies and charges.

In this talk, I will review several different phenomena that directly affect quantification of piezoelectric response at the nanoscale, that is, the determination of the d33 effective piezoelectric coefficient. First, I will show how gradient-based electromechanical effects couples and interferes with PFM measurements. I will start by demonstrating the asymmetry in mechanical properties induced by the coupling of flexoelectricity to ferroelectricity leading to ferroelectrics as smart mechanical materials [2], and opening new opportunities to mechanically read ferroelectric polarization states in both, thin films and single crystals, on the base of Contact Resonance Frequency AFM mode. Then, I will put the light in another new aspect: converse flexoelectric effect [3] due to the presence of strong local electric field gradients at the tip end can induce a mechanical strain of the sample in dielectric centrosymmetric materials with magnitudes comparable to piezoelectric d33 coefficient. Finally, I will go over the effect of Schottky barriers on the determination of the d33 coefficient of piezoelectric semiconductors, and show how the metal-insulator junctions can induce non-linear and multiharmonic electromechanical responses [4].

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Reconfigurable 2D Electron Gases in Ferroelectric Domain Walls

Prof. Dr. Lukas Eng
Institute of Applied Physics, TU Dresden, Nöthnitzerstr. 61, 01187 Dresden, Germany, ct.qmat:
Würzburg-Dresden Cluster of Excellence - EXC 2147, TU Dresden, Germany
Nöthnitzer Straße 61, 01187 Dresden, Germanz

Session Thursday, 24 September: [Advanced research on ferroelectric materials](#) I [Join the session](#)

For the last 10 years, domain wall conductivity (DWC) in ferroelectrics has become a very attractive and prospective field of research. We intensively explore DWs in bulk and thin-film LiNbO3 (LNO) single crystals, a material that is known for its large bandgap of ~4 eV, optical endurance, and excellent CMOS compatibility, with the help of dedicated scanning force-microscopy (SFM) methods (i.e. AFM, cAFM, PFM, KPFM, etc.). Nevertheless, when engineering domains across such crystals, domain walls (DWs), i.e. the tiny region that separates adjacent areas of opposite or non-collinear dielectric polarization, show, to our great surprise, excellent metallic-like properties. When applying a small voltage of ±10 V across such a wall, extremely large DWCs of up to ±1 mA per DW are recorded, and this even at room temperature. Mandatory for this to happen, however, are low injection barriers as well as optimized electron (hopping) transport properties along such DWs, as is experimentally mediated by slightly inclining DWs with respect to the polar LNO axes.
In this talk, I will provide clear proof for the existence of this 2-dimensional electron gas (2DEG) in these DWs by exploring Hall-transport properties, excited both with and without external photo-illumination. In addition, I will show how to follow and track the effective trajectory of electrons when flowing along the DW; the latter is achieved by applying a novel type of non-linear optical microscopy, so-called Cherenkov Second Harmonic Microscopy, a method that then reveals a 3-dimensional profile of individual domain walls across the bulk, excellently complementing our SFM surface data. Most interestingly, these large DWCs allow for fabricating nanoelectronics devices, making use of the reconfigurable 2DEG in these DWs.

Advanced functionality in ferroelectric oxides - creating building blocks for nanoscale circuitry

Prof. Dennis Meier
Department of Materials Science and Engineering, Norwegian University of Science and Technology (NTNU), 7034 Trondheim, Norway

Session Thursday, 24 September: [Advanced research on ferroelectric materials](#) I [Join the session](#)

Oxide materials exhibit a broad range of tunable phenomena, including magnetism, multiferroicity, and superconductivity. Oxide interfaces are particularly intriguing. Their low local symmetry combined with the sensitivity to electrostatics and strain leads to unusual physical properties beyond the bulk properties. Recently, ferroelectric domain walls have attracted attention as a novel type of oxide interface; the walls are spatially mobile and allow controlling electronic signals at the atomic scale, holding great potential as multifunctional 2D systems for future nanoelectronics.

In my talk, I will present unique features that occur at improper ferroelectric domain walls in hexagonal manganites and discuss how these walls can be used to emulate the behavior of key electronic components. For our studies, we choose the p-type semiconductor ErMnO3 as it naturally develops all fundamental types of ferroelectric domain wall at room temperature, namely neutral (side-by-side) as well as negatively (tail-to-tail) and positively charged (head-to-head) wall configurations. The walls are explicitly robust and, hence, represent an ideal template onto which the desired electronic behavior can be imposed. I will show how the electronic properties can be optimized and controlled, and discuss the possibility to use such walls for designing 2D digital switches and half-wave rectifiers [1,2], bringing us one step closer to domain-wall based devices and networks for next-generation nanotechnology.

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Nanoscale characterization across several length scales by AFM and fluorescence microscopy

Prof. Thorsten Hugel
Institute of Physical Chemistry and Cluster of Excellence livMatS @ FIT
University of Freiburg, Germany

Session Friday 25 September: Correlative microscopy techniques for comprehensive material characterization | Join the session

The AFM is a versatile tool to investigate a large and still growing number of systems, in particular in combination with fluorescence microscopy.
Here I will first show how unique insights into polymer physics can be obtained by single molecule force spectroscopy. Temperature dependent experiments combined with molecular dynamics simulations could quantify entropic and energetic contributions to the elasticity of polymers and showed that neither PEG nor PNIPAM are pure entropic chains [1]. Then I will show how the mobility within polymer coatings can be determined with AFM-based scratching experiments. This mobility can be surprisingly high in crosslinked coatings.
Next, I will show how a combination of AFM based indentation and fluorescence microscopy is used to correlate nanomechanical properties with the organization of chondrocytes in osteoarthritic cartilage [2]. This correlation is further extended towards the organization of the collagen networks using AFM based fast force-mapping.
Finally, I will present an outlook on the characterization of 3D-printed micrometer scale auxetic elastic structures, which underlines the great versatility of today's AFM-based research.

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Three-dimensional nano-architectures in energy, bio-medical, electronic and sensing applications – optimization based on correlative microscopy and spectroscopy and machine learning

Prof. Dr. Silke Christiansen
Innovation-Institute for Technology and correlative Microscopy, Forchheim, Germany

Session Friday 25 September: Correlative microscopy techniques for comprehensive material characterization | Join the session

Complex nano-architectures of various material combinations (e.g. Si-nanostructures such as wires (NW) and cones (NC), GaN nanostructures, transparent conductive oxides e.g. Al doped ZnO, coinage metal nanoparticles e.g. wires, spheres, graphene and other 2D materials) integrated on Si wafer platforms will be presented for light absorption, light emission and sensing applications.
In this context, we will show nano-material choices e.g. for surface enhanced Raman spectroscopy (SERS) and chemically functionalized SiNWs, SiNCs, GaN NWs with distinct resonances for optical sensing or distinct electrical performance in e.g. electronic nose devices based on resistors or field-effect transistors.
Materials and device optimization will rely on advanced, scale bridging correlated electron- ion-, optical- and x-ray microscopy and spectroscopy (CORRMIC) including atom probe techniques. The application of nanoGPS to enable true correlation with nanoscale precision between different techniques in different machines is essential and respective workflows will be demonstrated. In addition, we will show the application of machine learning strategies to aforementioned heterogeneous data to further exploit optimization of materials and device performance.

Introduction of recent SPM hybrid technology development

Dr. Sang-Joon Cho
Park Systems Corp., KANC 4F, Gwanggyo-ro 109, Suwon 16229, Korea
Center at Advanced Institute of Convergence Technology, SNU, South Korea

Session Friday 25 September: Correlative microscopy techniques for comprehensive material characterization | Join the session

Over the past three decades, AFM (Atomic Force Microscopy) has evolved into an ideal methodology for non-destructive sample scan with longer tip life, higher accuracy, repeatability, and automation. AFM is improving steadily so that it can be widely adopted like other microscopes, such as optics and scanning electron microscopes (SEM). In addition to the recent advances in AFM technology, it further expands the AFM application area by combining with other metrological technologies such as white interferometer (WLI) and photo-induced force microscopy (PiFM). By utilizing the vibration-isolated platform and the low noise z scanner of AFM, the performance of WLI has been greatly improved achieving unprecedented high z resolution.

The Photo-induced Force Microscopy (PiFM) is also integrated into 300mm AFM stage. PiFM can obtain chemical-specific nanoscale images and IR spectra by illuminating IR beam on the sample point of interest. I will also introduce preliminary data of various optical hybrid results.

Mysteries of the chemical bond revealed by atomic force microscopy

Prof. Dr. Franz J. Gießibl
Institute of Experimental and Applied Physics, Faculty of Physics, University of Regensburg, Universitätsstrasse 31, 93053 Regensburg, Germany

Session Friday 25 September: Overcoming barriers in AFM | Join the session

The imaging process in AFM rests on the temporary chemical bonds that are created and broken during the imaging process. CO terminated AFM tips have been shown to provide outstanding spatial resolution on organic molecules,[1]metallic clusters [2] and other samples. Experimental evidence and calculations show that the CO tip is chemically inert and probes organic molecules mainly by Pauli repulsion. [3] Thus, images of organic molecules, graphene etc. observed with a CO tip can be interpreted as a map of the absolute charge density of the sample. The total charge density of a single adatom is approximately given by a Gaussian peak. While single silicon adatoms appear similar to a Gaussian peak when imaged by AFM with a CO terminated tip, copper and iron adatoms adsorbed on Cu(111) and Cu(110) appear as tori. Initially, we explained these images by spz hybridization of the valence electrons of the adatoms. [2] Recent experiments and DFT calculations show that the total charge density of Cu and Fe adatoms is approximately Gaussian – in contrast to the hybridization theory. [4] The bonding strength between the AFM tip and the atoms of the sample depends not only on the chemical identity but also on the coordination - corner atoms in clusters are more reactive than center atoms. [5]

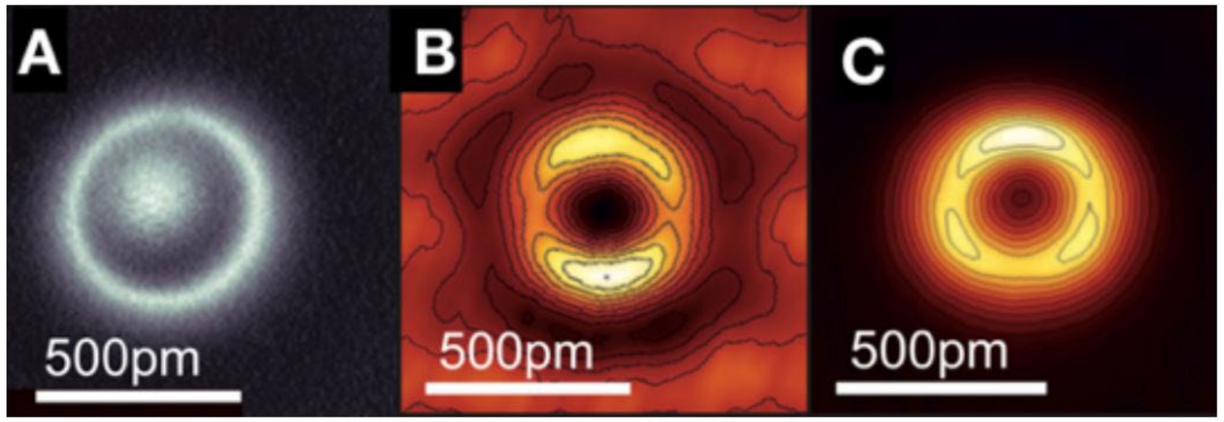

Fig.1 AFM images of various metallic adatoms using a CO terminated tip. A Copper adatom on Cu(111). B Copper adatom on Cu(110). C Iron adatom on Cu(111).

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Traceably quantitative magnetic field measurements with magnetic force microscopy

Dr. Sibylle Sievers
PTB - The National Metrology Institute of Germany, Bundesallee 100, 38116 Braunschweig, Germany

Session Friday 25 September: *Overcoming barriers in AFM* I *Join the session*

Magnetic force microscopy (MFM) can be considered the standard tool for measuring magnetic stray fields with a spatial resolution down to 10 nm. When measuring nano-scale structures, the size of the MFM probe, a magnetically coated tip, usually becomes comparable to the length scale of the field variations and must hence be considered. This is possible by a transfer function (TF)-based calibration approach that correctly considers the non-punctiform character of the magnetic tip. The MFM tip interacts with the sample via its stray field gradient distribution. The TF approach calculates an effective tip stray field gradient distribution, the tip transfer function (TTF), by means of a calibration measurement on a reference sample with well-defined magnetic field distribution. This allows quantitative MFM (qMFM), since the knowledge of the TTF together with the mechanical properties of the tip cantilever facilitates the calculation of the magnetic field distribution of an unknown sample from a constant height MFM measurement by a deconvolution.

In this talk, we will introduce the TF-based calibration concept and discuss its scope of application as well as the limitations. Reference samples are discussed and the results of an intercomparison are presented. As an outlook, first results on a validation of a TTF using NV centre magnetometry are shown, as a first step towards quantum traceability of qMFM.

Instrumental aspects of in situ AFM imaging of solid electrolyte interfaces on battery grade powder electrode materials

Dr. Sergey Luchkin
Skolkovo Institute of Science and Technology, Moscow, Russia

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

The state of the art atomic force microscopy (AFM) is widely used to study dynamic processes at solid-liquid interfaces in electrochemical systems, including secondary metal-ion batteries. Particularly important is in situ investigation of a solid electrolyte interface (SEI) formation – the key component for battery performance and durability 1. The SEI formation is routinely investigated by in situ AFM on a basal plane of highly oriented pyrolytic graphite (HOPG) – a standard model sample 2. Its flat geometry and atomically smooth and clean surface after peeling off the top layer is ideal for AFM measurements. However, it does not represent surface of the battery grade carbon, where the edge to basal plane ratio is much higher than that of HOPG. In a recent study 3 we performed in situ AFM imaging of SEI formation on the battery grade powder particles exposed on cross sections of battery electrodes. Significant difference in SEI nucleation voltage, thickness and mechanical properties between HOPG and battery grade powders was demonstrated. The present work details on instrumental aspects of the in situ AFM measurements of powder samples including proper experimental setup, sample preparation, choice of the appropriate cantilever and scanning parameters. Details of ex situ measurements of the formed SEI such and mechanical and electrical are also presented.

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A comprehensive study of electrochemical HOPG intercalation with $HClO_4$ and H_2SO_4 electrolytes by photoemission spectroscopy and atomic force microscopy

Prof. Gianlorenzo Bussetti
Polytechnic University of Milan, Italy

Session Wednesday 23 September: *Resolving Electrochemical Properties on Functional, Sustainable Materials* I *Join session*

Graphitic materials are employed in many technological applications, from the realization of electrodes in batteries to the production of graphene foils by electrochemical (EC) procedures [1]. In particular, the processes characterizing graphite oxidation have attracted researches in view of optimizing battery duration and quality-rate ratio in graphene production protocols [2]. In this context, highly oriented pyrolytic graphite (HOPG), with its well-defined layered structure, is generally considered a model system to elucidate the mechanisms of the EC oxidation of graphite electrodes, and in particular the intercalation process that occurs at oxidative EC potential ranges [3].

Recently, we demonstrated that anion intercalation can be detected not only at high (within 1.9 and 2.1 V vs SHE) EC potential values, i.e. above literature-reported intercalation stages, but also at lower potentials and allowing for a faster dynamics [4]. Here, we compare the results obtained on HOPG by using two different EC techniques for ion intercalation, namely cyclic voltammetry (CV) and normal pulsed voltammetry (NPV), with two different aqueous electrolytes (i.e. perchloric and sulfuric acid). The samples were analysed in-situ by atomic force microscopy (EC-AFM) and ex-situ by X-ray photoemission spectroscopy (XPS), where ion intercalation results in a shift of the XPS core levels towards lower binding energies, as previously highlighted by one of us [5].

We detect comparable XPS lineshapes after intercalation at high potentials, both with CV and NPV and for both electrolytes. Intercalation of perchlorate ions at lower potentials (namely, at the oxygen evolution potential) surprisingly shows a similar spectral evolution, where much stronger modifications are obtained by applying NPV, compared to CV. Finally, we investigated the effect of increasing the CV scan rate on both sample morphology and chemical surface condition. Our results show that, in spite of a similar XPS behaviour, fast CV scans prevent the development of extended surface defects (such as blisters) usually observed on HOPG due to the formation of gases seeded by ion intercalation.

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Revealing local ionic conductivity and chemical distribution in solid state electrolytes by Electrochemical Strain Microscopy

Prof. Dr. Florian Hausen
RWTH Aachen University, Aachen, Germany, Forschungszentrum Jülich, Germany

Session Wednesday 23 September: Resolving Electrochemical Properties on Functional, Sustainable Materials I Join session

All Solid State Lithium Ion Batteries (ASS-LIB) are of great economic as well as scientific interest. Substituting the flammable liquid organic electrolyte by a lithium ion conductive ceramic is a promising route. Lithium Aluminum Titanium Phosphate, Li1.3Al0.3Ti1.7(PO4)3, is a typical representative of a solid state electrolyte with NASICON type structure, exhibiting superior lithium ion conductivity. [1] The appearance of secondary phases of aluminum phosphate under certain manufacturing conditions [2] demonstrate the need for understanding the correlation of lithium ion mobility and microstructure on sufficiently small scales in solid state electrolytes. Electrochemical Strain Microscopy (ESM) is employed to investigate the influence of the microstructure of the solid state electrolyte LATP. A clear contrast between LATP and the secondary phase is observed, exhibiting significantly reduced Li-ion mobilities in secondary phase areas. In order to correlate chemical information with the ESM signal, Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX) is utilized in a correlative approach. [3] The contribution of grain boundaries to the overall ionic conductivity is discussed in view of recently published impedance spectroscopy results. [4] Additionally, a correlation between the tip-sample capacitance in contact and the local chemical distribution of LATP is observed, leading to new insights into the complex signal formation process in ESM. [5] The applicability on other solid state electrolytes will be critically discussed.

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Kinetics of oxidative etching and Mo oxides produced during oxidation of single microscale 2H MoS2 flakes in air and high relative humidity

Ass. Prof. Robert Szoszkiewicz
University of Warsaw, Poland

Session Wednesday 23 September: 2D materials and nanoelectronics I Join session

For numerous applications of microscopic MoS2 crystals, one must understand their most expected surface reactions. Ubiquitous examples of these are oxidation reactions in air and water. In this talk, we show how we studied the kinetics of microscopic heat-induced oxidative etching in the case of thick, mechanically exfoliated, geological MoS2 crystals in air. Next, based on the literature we discuss particular reaction mechanisms of oxidative etching as well as mention other competitive processes. In the second part of the talk, we show how to differentiate MoO3 oxides and their derivatives on thermally oxidized microscopic 2H MoS2 flakes. In order to do so, we combined XPS and AFM measurements such as topography, friction, creation of nanoscale ripples and scratches on the MoS2 flakes.

Morphological and electrical characterization of coordination polymers containing rhodium paddle wheels

Daniel Steinbach
TU Bergakademie Freiberg, Germany

Session Wednesday 23 September: 2D materials and nanoelectronics I Join session

The electric conductivity of MOFs is generally considered as a prerequisite for many potential applications such as sensors, transducers, electrodes and others.[1,2] To overcome the disadvantages of most MOFs being insulators, conjugated coordination polymers are investigated regarding their electrical conductivity. Therefore, paddle-wheel structures with a documented metal-metal bond, here rhodium derivates, were linked via conjugated organic molecules like pyrazine.[3] Coordination polymers of this type were first synthesized as bulk materials, characterized using XRD, BET and XPS and then deposited as coatings on gold surfaces. Subsequently, the topography of the deposited layers was measured. Different morphologies mainly determined by AFM were obtained by using spin coating and dip coating. Depending on the composition of the coordination polymer well distributed crystals or network like structures were found. The surface layers were further investigated by XPS and XRD. Direct current measurments were carried out on such systems by conductive AFM.
The results point out, that $[Rh_2(ac)_4pyz]_n$ forms orientated nano crystallites on a gold coated surface (figure 1), which are insulating. Changing the axial bond ligand to acetamide causes a conductive coordination polymer with recognisable currents (figure 2).

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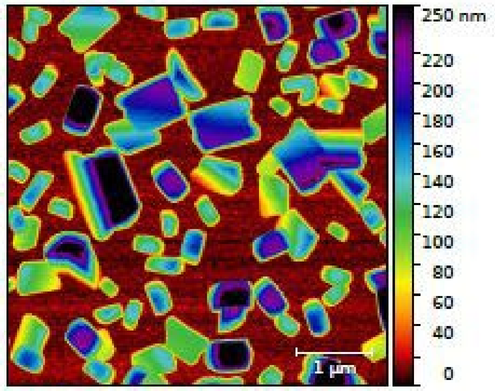

Figure 1: Height image of via dip coating deposited $[Rh_2(ac)_4pyz]_n$ (100 cycles).

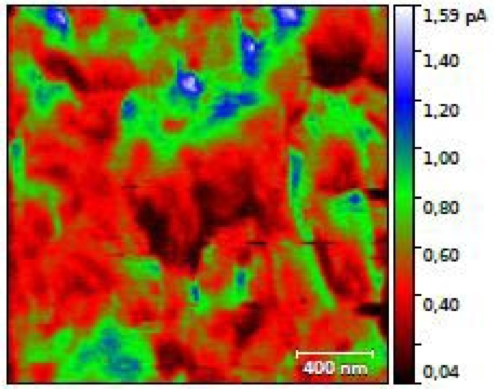

Figure 2: Current image (C-AFM) of via spin coating deposited $[Rh_2(acam)_4pyz]_n$ (40 cycles).

Indentation of metallic thin film on soft substrate: insights for stretchable conductors

Giorgio Cortelli
University of Bologna, Italy

Session Wednesday 23 September: 2D materials and nanoelectronics I Join session

Microstructured gold thin films on poly(dimethylsiloxane) (PDMS) are used as stretchable metallic conductors in low-invasive bioelectronic interfaces [1,2], electronic skin [3], or dielectric elastomer actuators (DEAs). Understanding of the micromechanical properties in such metallic hard-on-soft systems is of crucial importance to ensure device functionality and to optimize device design.
Here we perform AFM nanoindentation experiments on thin films of gold on PDMS. In the experiments, we vary systematically film thickness and tip curvature. Force – indentation curves show two regimes. In the first regime, the response is completely elastic, without plastic deformation and fracture. Above a critical force, a second regime sets in, where fracture of the thin

film is observed, and conductivity changes occur as confirmed by conducting AFM. In the elastic regime, force-indentation curves differ significantly from the one predicted by the Hertz model. The results depend on the metallic film thickness, while they are found to be independent of the tip curvature.

To interpret the experimental data, we perform FEM simulations of the indentation process. The simulations are consistent with the bending of a thin plate on an elastic medium that can be analytically described by a so-called Winkler soil. The model allows extracting the effective thickness and the elastic modulus of the nanoscale thin film from the experimental data.

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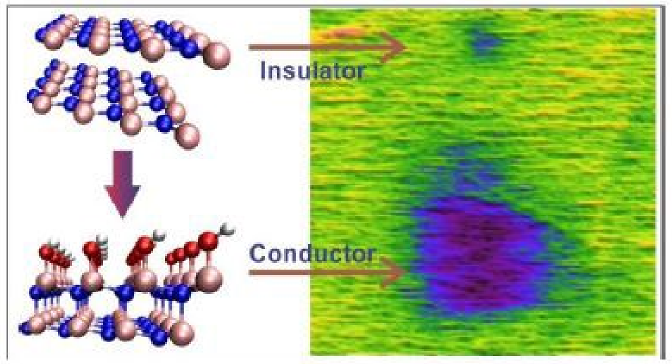
Compression induced modification of boron nitride layers: a conductive two-dimensional BN compound

Prof. Dr. Ana Paula Barboza
Federal University of Ouro Preto, Brazil

Session Wednesday 23 September: [2D materials and nanoelectronics](#) I [Join session](#)

The ability to create new materials with improved properties upon transformation processes applied to conventional materials is the keystone of materials science. Here, hexagonal boron nitride (h-BN), a large bandgap insulator, is transformed into a conductive two-dimensional (2D) material – bonitrol – that is stable at ambient conditions. The process, which requires compression of at least two h-BN layers and hydroxyl ions, is characterized via scanning probe microscopy experiments and ab initio calculations. This new material and its creation mechanism represent a new strategy on the transformation of known 2D materials into artificial advanced materials with novel properties.



Kelvin probe force microscopy-based direct measurements of contact resistance in 2D semiconductor thin film transistors

Dr. Alexandar Matkovic
Montanuniversität Leoben, Leoben, Austria

Sesssion Wednesday 23 September: [2D materials and nanoelectronics](#) I [Join session](#)

This study aims at direct imaging of contact resistance in MoS2-based thin film transistors (TFTs). Exfoliated single-crystal flakes of MoS2 have been used in a bottom-contact TFT configuration. Pyrimidine-containing self-assembled monolayers (SAMs) were employed to tune the work function of gold electrodes. Kelvin probe force microscopy measurements were carried out during operation of the devices in order to directly image potential drops across the channel and to study the influence of different SAM treatments on the contact resistance. By independently imaging potential drops at both carrier injection and extraction points, we demonstrate asymmetry of contact resistances in MoS2-based TFTs, as well as their non-linear and bias-dependent behavior [1].

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Molecular decoration of two-dimensional materials: unveiling crystallographic orientation and grain boundaries

Prof. Mariana Prado
Federal University of Ouro Preto, Brazil

Session Wednesday 23 September: [2D materials and nanoelectronics](#) I [Join session](#)

Following the realization of single-layer graphene atop silicon oxide, much attention has been drawn towards two-dimensional materials. Graphene is a semi-metal, hexagonal boron nitride (hBN) is an insulator and single-layer molybdenum disulfide (MoS2) is a semiconductor with a direct bandgap. Besides those, there are numerous other two-dimensions nanosheets of a variety of materials including other transition metal dichalcogenides and phyllosilicates, minerals composed of parallel sheets of Si_2O_5 tetrahedra. An example of the latter group is talc [1], a solid lubricant with many industrial applications.

Characterizing these 2D materials is a challenge that must be addressed so they can fulfill their potential to revolutionize the industry with their outstanding mechanical, optical, and electrical properties. We present a simple way to reveal the crystallographic orientations of graphene, MoS2, and hBN without the need of acquiring atomic resolution microscopy images. Octadecylphosphonic acid (OPA) is a linear alkyl chain organic molecule that forms a self-assembled monolayer (SAM) on top of the cited materials and that keeps registry with the crystallographic substrate beneath it. An atomic force microscopy (AFM) image easily detects the period of the 2D molecular crystal that forms atop the flake, revealing the substrate's crystallographic orientation. Ab initio calculations reveal that the molecular crystal's ripples (detected in the AFM image) are aligned with MoS2 [2], hBN [3], and graphene's [4] armchair direction.

For MoS2, we also found that our samples, purchased from SPI supplies, presented ripples of the molecular crystal-forming 12° angles that did not reflect the substrate's hexagonal symmetry. Interestingly, Stone-Wales-type defects have been predicted to cause 12°-angled grain boundaries in MoS2 and WS2. Thus, the SAM deposition followed by standard AFM analysis on ambient conditions proposed in this work enables the direct localization of MoS2 grain boundaries. For graphene, ab initio calculations and Raman spectroscopy measurements also show that OPA induces p-type doping of graphene of ~1013 cm-2. Finally, OPA has a different behavior when deposited on top of talc, forming the organized 2D crystal or vertical bilayers, depending on the deposition conditions.

Summing it up, spin coating OPA ethanolic solution on 2D materials is a facile way of discovering flake orientation, characterizing grain boundaries of MoS2 samples, and achieving chemical doping of graphene.

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Wet Graphene Membranes on a Microfluidic Platform

Prof. Ive Almeida
Federal University of Ouro Preto, Brazil

Session Wednesday 23 September: [2D materials and nanoelectronics](#) I [Join session](#)

Graphene is regarded as the toughest two-dimensional material (highest in-plane elastic properties) and, as a consequence, it has been employed/proposed as an ultrathin membrane in a myriad of microfluidic devices. Yet, an experimental investigation of eventual variations on the apparent elastic properties of a suspended graphene membrane in contact with air, water and other liquids is still missing. In this work, the mechanical response of suspended monolayer graphene membranes on a microfluidic platform is investigated via scanning probe microscopy experiments. A high elastic modulus is measured for the membrane when the platform is filled with air, as expected. However, a significant apparent softening/hardening of graphene is observed when water/oleic acid fills the microfluidic system. Through molecular dynamics simulations and a phenomenological model, we tried to understand these behaviors. These results may bring substantial modifications on the design and operation of microfluidic devices which exploit pressure application on graphene membranes.

Molecular recognition of protein receptors through quantitative force maps

Dr. Carlos Marcuello
CSIC-University of Zaragoza, Spain

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

Since its discovery in 1986, atomic force microscopy (AFM) has become a powerful tool to address the physical-chemical properties of matter. In the last decades, strong efforts have been made to analyze the intermolecular interactions between biomolecules by AFM through dynamic force spectroscopy (DFS) under near-physiological conditions. DFS is an operation mode which enables not only the acquisition of single force-distance curves but also, combined with scanning to record tip-sample adhesion maps. Adhesion imaging collects with the simultaneous acquisition of topography in force operational modes. When adhesion maps are produced with functionalized tips scanning on samples substrates covered by affine receptors, it is possible to find the conditions to locate the receptors. Several methods were proposed to locate receptors using different modes and strategies, including dynamic modes (e.g. TREC [1]). In this work, we present an intermittent force method able to give adhesion force maps that not only locate the receptors through rupture events but also provide the rupture forces in each pixel of the image at the pN range. These experiments open new possibilities not only for fast analysis of rupture forces but for developing procedures for detection at the single molecule level.

In this work, we show two alternative studies: i) the analysis of the interaction between the flavoenzyme ferredoxin NADP+ reductase and its redox partners, ferredoxin and flavodoxin. We protected the binding site of the enzyme against the tagging molecules to prevent the loss of enzymatic activity evidenced by molecular recognition imaging [2]. Moreover, different loading rates were tested to obtain the energy landscape parameters for both complexes [3]. In the adhesion imaging maps it was possible to distinguish the enzyme molecules and the rupture forces with both protein partners at the tip in the simultaneous images; ii) the analysis of the adhesion forces between strept(avidin) and biotin. The adhesion maps in implemented certain conditions, indicate that rupture forces of the streptavidin:biotin complexes are 3-fold higher respect to the avidin:biotin complexes (40-80 and 120-170 pN, respectively). This method was probed using mixed protein samples scanned with biotinylated AFM tips, being capable of discriminate among avidin and streptavidin molecules by this molecular recognition imaging method [4,5].

We expect that these alternative protein bioconjugation strategies to attach proteins to solid surfaces/AFM cantilevers preserving their enzymatic activity by one side, together with the intermittent force operating conditions allow to get reproducible adhesion force results that achieve characteristically the identification of protein receptors in mixtures through rupture forces, necessary to design throughput detection devices based on forces.

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The role of lectins in surface adhesion and virulence of Pseudomonas aeruginosa studied with single molecule force spectroscopy

Joanna Zemła
Polish Academy of Sciences, Krakow, Poland

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

On surfaces, bacteria form communities called biofilms, in which a self-produced extracellular matrix acts as a glue and shields bacteria from the environment. In healthcare, biofilm formation plays a crucial role in the development of antibiotic resistance [1]. The biofilm-former Pseudomonas aeruginosa is part of a family of microorganisms exhibiting multi-drug resistant virulence, responsible for most hospital-acquired infections [2]. P. aeruginosa's matrix is mainly composed of exopolysaccharides (EPS), but extracellular DNA and some specific proteins have been shown to be structurally important [3-6]. Although two main EPS (Pel and Psl) have been identified, the precise composition of the matrix remains elusive. Moreover, its distribution within the biofilm and the temporality of its production are not clear.

Bacterial lectins are sugar-binding proteins that mediate adhesion to host surfaces, and are also important for biofilm formation. In the widespread pathogen Pseudomonas aeruginosa, lectins LecA and LecB are known to be key pathogenicity factors, yet their exact role in surface colonization and their implication in virulence pathways are unclear. LecA and LecB structures suggest that they function as tetramers, and could create cell-bacteria, matrix-bacteria, and bacteria-bacteria interactions, which could explain why they are important for adhesion as well as biofilm cohesion.

We have used single molecule force spectroscopy (SMFS) to investigate the composition and structure of the EPS of P. aeruginosa with LecA and LecB molecules covalently attached to the scanning probe. Our recent experiments have shown that both lectins interact specifically with early matrix deposits. Our analysis points to a mannose and galactose-rich matrix, and suggests that lectins could play a role as building blocks and crosslinkers in the matrix. Additionally, Freely Jointed Chain (FJC) model indicates that both Pel and Psl contain galactose units.

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Further development of AFM-based imaging techniques for tracking fenestrae in living Liver Sinusoidal Endothelial Cells

Dr. Bartlomiej Zapotoczny
Polish Academy of Sciences, Krakow, Poland

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

Atomic Force Microscopy (AFM) imaging technique, based on force-distance curve acquisition in time of a few milliseconds each allowed for the first visualization of fenestrae in living Liver Sinusoidal Endothelial Cells (LSECs)(1). Fenestrae – transcellular pores of the size in a range of 50-300 nm, remained undisclosed in living LSECs for nearly 50 years(2). They play a prominent role in maintaining liver homeostasis and integrity. Variations in size and diameter can be used as morphological indicators for LSEC health status. Fenestrae were reported to respond to a variety of external stimuli becoming a great marker for the functional responsiveness of LSECs(3). Understanding the processes of their formation, lifespan, and closing would allow for the therapy enabling restoration of the proper porosity of LSECs.

With the AFM, high-resolution imaging of fenestrae became possible. Single frames collected in the time of tens of seconds allowed us to repetitively collect images in the same area and - as a result - tracking changes in the actin-spectrin structures forming fenestrae. Our recent advances in AFM imaging provided new insights into the dynamic processes of fenestrae formation, migration, disappearance, and closing(4). We succeeded to estimate the mean fenestrae lifespan, describe three different ways of closing of fenestrae, and showed that individual fenestrae can be in an open or closed state(4, 5).

Here, we show that elasticity (described as Young modulus) and porosity can be simultaneously tracked in living LSECs. Together, these two parameters ensure even more complete image of the functional versus the pathological condition of LSEC(6). Initial results of the ongoing project and future outlook for research of LSECs will be presented.

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Single Molecule Atomic Force Microscopy to Unravel Protein Misfolding and Aggregation

Dr. Francesco Simone Ruggeri
University of Cambridge, United Kingdom

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

The presence in human biopsies of polymorphic protein assemblies is associated with the onset of several neurodegenerative diseases. On the other hand, several organisms exploit amyloid fibrils functionally, for instance as bio-adhesives or as scaffolding material. Taking inspiration from nature, ordered amyloid-based materials pave the wave for the development of robust and biocompatible materials for materials science and biomedical applications.
Here we show that atomic force microscopy and force spectroscopy provides a unique window into the morphology, mechanics, and structure of proteins and amyloids on the nanoscale. Furthermore, the combination of high-resolution AFM with microfluidic methods enables to investigate amyloids thermodynamic equilibrium, ultrastructure and formation at the single molecule scale with angstroms resolution. We first demonstrate the identification and characterisation of the smallest elementary unit in the hierarchical assembly of amyloid fibrils, which we termed single-strand protofilaments. Remarkably, these aggregates are present in blood serum, cerebral spinal fluid and brain biopsies obtained from patients affected by Alzheimer's and Parkinson's diseases. Then, in parallel, we demonstrate the capability to tune the biophysical properties of these highly biocompatible aggregates for the development of innovative biomaterials that in contrast enable the enhancement of neuronal regeneration in animal models of nerve damage.
Unraveling the biophysical properties of amyloidogenic structures at the nanoscale is central to understand how proteins misfold and aggregate for understanding the molecular processes underlying the onset of neurodegenerative disorders, as well as to produce a new class of functional materials for biomedical and materials science application.

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High-Aspect Ratio Polymeric Nanoneedle Arrays

James McCormack
University College Dublin, Ireland

Session Thursday, 24 September: Polymer- and Biotechnology I Join the session

High aspect ratio (HAR) nanoneedle arrays are metastructures that can tune the intrinsic properties of substrates such as wettability, reflectivity and biocompatibility by virtue of their physical properties regardless of the substrate's chemical properties. However, engineering dense arrays of HAR nanoneedles using polymeric substrates remains a challenge in soft lithography research as vertical freestanding nanostructures are prone to ground collapse and/or lateral collapse when fabricated using low modulus materials. Innovative patterning processes are required to broaden the scope of suitable materials which can be patterned with these structures. To this end, replica moulding of Si master for nanoneedle arrays using UV-curable polymers was assessed as a viable method for polymeric nanoneedle array production. Two distinct sets of Si nanoneedle arrays were produced using two PS-b-P2VP templates of different molecular weights shown in Figure 1; the first being a closely-packed array with average needle base diameters of 58 nm, pitches of 120 nm and a mean height of 300 nm; the second with larger features with base diameters of 110 nm, pitches of 350 nm with a mean height of 530 nm. FESEM and AFM analysis were used to assess the fidelity of the polyethylene glycol (PEG) features relative to their Si master. Sub-40 nm diameter features decreased in population following replication however, PEG nanoneedles up to aspect ratio 4.6 were produced using our replication method. This presents replica moulding as a simple, low-cost process, capable of producing a library of polymeric nanoneedle arrays with dimensions within a range of current nanostructures being researched for cellular drug delivery and optical applications.

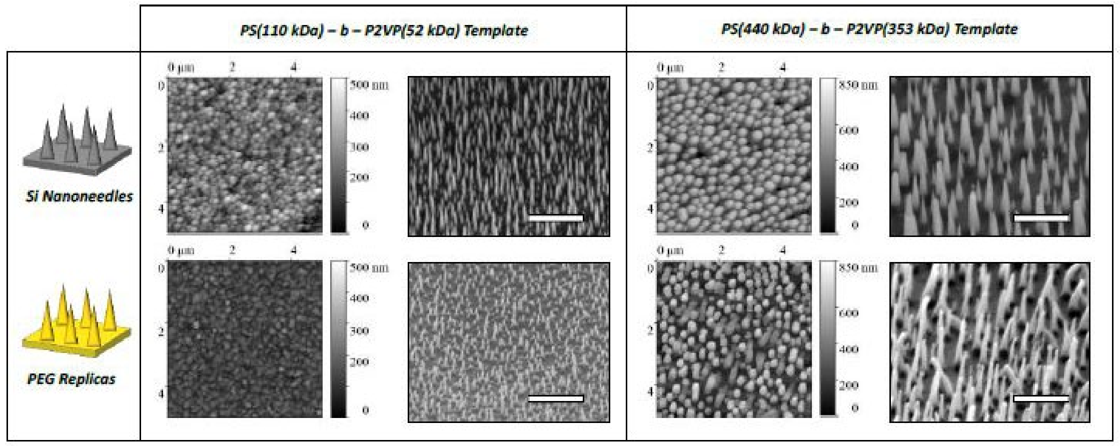

Figure 1: AFM topography scans and tilted FESEM images of Si nanoneedle arrays (Top row) and their PEG replicas (Bottom row). The PS-b-P2VP template used to define the Si nanoneedle array is indicated. All tilted images were obtained at 45o. (Scale bar: 1 μm)deposited [Rh$_2$(ac)$_4$pyz]$_n$ (100 cycles).

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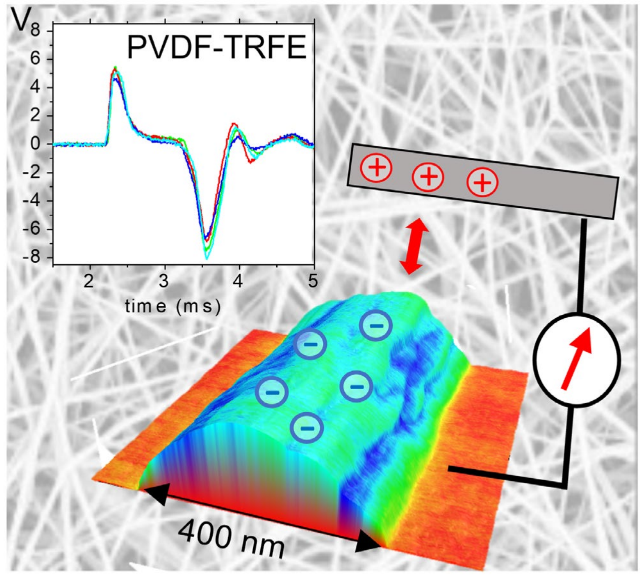
Piezoelectric and electrostatic properties of electrospun PVDF-TrFE nanofibers and their role in electromechanical transduction in nano-generators and strain sensors

Ass. Prof. Tobias Cramer
University of Bologna, Italy

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

Piezo- and ferroelectric nanofibers of the polymer poly(vinylidenefluoride) (PVDF) have been widely employed in strain and pressure sensors as well as nanogenerators for energy harvesting. Despite this interest, the mechanism of electromechanical transduction is under debate and a deeper knowledge about relevant piezoelectric or electrostatic properties of nanofibers is crucial to optimize transduction efficiency. Here we prepare PVDF-TrFE nanofibers at different electrospinning conditions. We compare macroscopic electromechanical response of fiber mats with microscopic analysis of single nanofibers performed by piezoelectric and electrostatic force microscopy. Our results show that electrospinning favors the formation of the piezoelectric beta-phase in the polymer and leads directly to piezoelectric properties that are comparable to annealed thin films. However, during electrospinning the electric field is not strong enough to induce direct ferroelectric domain polarization. Instead, we observe accumulation of triboelectric surface charges and trapped space charge in the polymer that explain the electret like macroscopic electromechanical response.



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Giant electron and hole conduction coexisting in non-oxide ferroelectric domain walls

Dr. Lukas Kuerten
ETH Zurich, Switzerland

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

Further acceleration of electronics requires new flexible concepts for on-demand-devices. In ferroic materials, atomically sharp domain walls (DWs) may offer an ideal platform to realize such versatile microelectronic building blocks because they can be created, manipulated and erased by external fields.
However, conducting DWs have been exclusively studied in ferroic oxides so far, in which domain-wall conductivity usually requires a specific strain configuration of the crystal, an improper character of the ferroelectricity or other unusual properties which render the walls immobile and thus curtail their usefulness and flexibility. Additionally, oxide materials are prone to defects, significantly hampering the utility of their conducting DWs.
In this study, we present a non-oxide material, GaV4S8. This multiferroic material combines crucial domain-wall functionalities which are not found combined in any oxide material. For example, eight orders of magnitude conductivity contrast between walls and bulk lets the walls dominate the overall conductivity behavior. Furthermore, a change from hole-like to electron-like conductivity over smallest length scales occurs in GaV4S8 DWs, unprecedented in oxide materials and possibly allowing for p-n junctions inside domain walls. Most importantly, GaV4S8 is a proper ferroelectric in which conductive DWs emerge in the unstrained crystal spontaneously, such that they retain their flexibility and can be manipulated by electric and magnetic fields.

Probing the behaviour of surface water and ferroelectric PbTiO3 thin films as a function of relative humidity and temperature

Loïc Musy
University of Geneva, Switzerland

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

Surface adsorbates play an essential role in the stability of as-grown and written ferroelectric domains, particularly in thin films, since they provide the screening necessary to stabilise polarization bound charge. The study and control of the ferroelectric-adsorbate interaction is, therefore, key to the engineering of complex ferroelectric patterns which could be used for electromechanical actuation and sensing applications as well as catalytic processes [1,2]. Previous studies have demonstrated that polar adsorbates such as water are strongly influenced by both the direction of the polarisation and the switching history of the ferroelectric [3], with screening strongly dependent on the relative humidity [4].
Here, we report on our investigation of the behaviour of water on ferroelectric PbTiO3 thin film surfaces as a function of relative humidity and temperature at the nanoscale using Kelvin probe force microscopy, piezoelectric force microscopy and high resolution non-contact resonant imaging, and a home-built humidity control system [5]. We observe extremely low charge dissipation, and high localisation of both positive and negative screening charge on patterned domain structures in films with varying as-grown polarisation states, which we attribute to the extremely high quality and low surface roughness of the films, as compared with previous studies where much more extensive charge dynamics were observed [6]. Local measurements of the surface water layer during temperature cycling at low and high humidity will be presented, where the change between a purely solid-like molecular arrangement and the formation of a liquid-like surface layer [7] strongly affects screening charge dynamics.

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Quantifying the Impact of Varying Defect Landscapes on Domain Wall Motion

Ralph Bulanadi
University of Geneva, Switzerland

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

Ferroelectric materials, such as lead titanate, show differently oriented domains of spontaneous electric polarisation. The walls between domains have been modelled as a disordered elastic system, both statically and during displacement under an applied electric field [1,2], where their motion has been observed to follow avalanche statistics via the detection of Barkhausen noise [3]. The universality of these classifications allow for modelling of domain walls at larger scales than typically possible via ab initio methods, and integrating the effects of material defects, which can present a complex and heterogeneous disorder landscape, particularly in thin film ferroelectric samples
Here we report scanning probe microscopy studies into the switching dynamics of lead titanate thin films with varied and controlled defect disorder. The films, grown on strontium titanate substrates, present strain-relieving a-domains that extend through the film, in-plane, along the crystallographic axes. Varying point defect densities have also been introduced via bombardment with He2+ ions. The gradual motion of 180° domain walls in these films under incrementally increasing bias applied by a scanning probe tip was imaged using piezoresponse force microscopy. Computational tools were also developed to access information pertaining to the rate of switching at the nanoscale, as a function of both point defect density and a-domain proximity.
We observe that increasing point defect density significantly increases the average voltage required for switching, as well as the voltage range over which domain wall motion is observed. Domain nucleation also appears preferentially in more heavily bombarded samples. The a-domains appear to act as extended strong pinning sites, imposing directional constraints on the domain wall motion along the film crystallographic axes. In the non-irradiated sample, the effect of these a-domains appear to dominate, leading to large scale domain wall jumps to configurations determined by a-domain position. In contrast, all samples have been observed to express Barkhausen behaviour with similar critical exponents, regardless of defect implantation. In this complex disorder landscape, preliminary observations therefore suggest point defects may apply a screening effect to the impact of a-domains, yielding anomalous behaviour that is locally constrained around a-domains in the non-bombarded sample, while being statistically similar on a larger scale to ion-bombarded datasets.

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Piezoresponse force microscopy on ZnO platelets, thin films, and sintered material

Dr. Markus Kratzer
Montanuniversitaet Leoben, Austria

Session Thursday, 24 September: Advanced research on ferroelectric materials I Join the session

As a nano/micro–scale method, piezoresponse force microscopy (PFM) is on the rise for the characterization of ferroelectric/piezoelectric properties of microsystems. Frequently PFM is utilized to study ferroelectric domain patterns, which can be challenging already. However, usually, the materials are smooth, strongly responsive, and strictly insulating allowing the extraction of quantitative information. [1] By using vector piezoresponse force microscopy [2], we could reconstruct the domain distribution function on lead zirconate titanate. [3] In contrast to such model samples, we also measured the piezoelectric material zinc oxide (ZnO) in form of crystalline ZnO platelets [4], CVD deposited thin films [5], or sintered polycrystalline varistor material. In these cases, the measurements are experimentally more challenging and the interpretation/quantification is difficult. Significant surface roughness, finite conductivity, and poor mechanic stability are parameters that should be considered.

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Integrated Simultaneous Chemical, Surface Potential, Mechanical, and Topographic Imaging at <10 nm Spatial Resolution

Ass. Prof. Xiaoji Xu
Lehigh University, USA

Session Friday 25 September: Correlative microscopy techniques for comprehensive material characterization | *Join the session*

Multimodal measurements of chemical composition, electrical properties, mechanical properties, and topography by scanning probe microscopy (SPM) deliver correlations across properties at the nanoscale, and provide clues to the structure-function relationship of materials. In the past, measurements with these modalities are operated separately with different operational modes of SPM. Not only the sequential measurements require additional operation time, are subject to scanner/sample drift, but also, different modalities of SPM have different spatial resolutions, which undermine correlative analysis. For example, the popular frequency-modulated Kelvin Probe Force Microscopy measures the surface potential with 30~50 nm spatial resolution under the ambient conditions, whereas the SPM measurements of chemical composition, mechanical properties, and topography can routinely achieve < 10 nm spatial resolution.

At the conference, we will present our invention of an integrated SPM mode that can simultaneously provide chemical, surface potential, mechanical, and topographic imaging at < 10 nm spatial resolution under the ambient conditions. We name it peak force infrared-Kelvin probe force microscopy (PFIR-KPFM), as we achieved it through an integration of peak force infrared microscopy and pulsed force Kelvin probe force microscopy. In a single scan, the integrated PFIR-KPFM delivers simultaneous multimodal measurement at a comparable and high spatial resolution of < 10 nm. As a demonstration, we measured a naturally-degraded $CH_3NH_3PbBr_3$ perovskite single crystal. AFM topography, mechanical modulus, contact potential difference (CPD), and nano-IR imaging at infrared absorption of perovskite are simultaneously acquired and shown in Figure 1 (a-d) respectively. Correlation analysis among infrared absorption, mechanical modulus, and CPD are performed and displayed in Figure 1e-f.

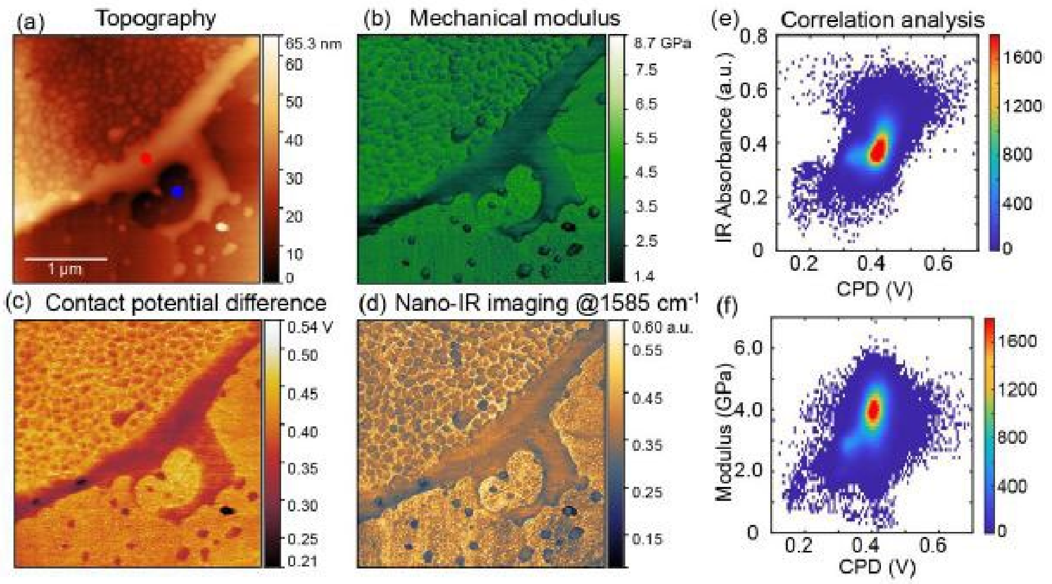

Figure 1. PFIR-KPFM measurement of a naturally degrade $CH_3NH_3PbBr_3$ perovskite single crystal. (a) topography. (b) Modulus. (c) CPD. (d) The nano-IR image at the infrared frequency of 1585 cm-1 (e) Correlation between CPD and IR signal. (f) Correlation between CPD and modulus.

Accelerating Your Research Using Nature Research Group's AI Powered Nanotechnology Platform

Pranoti Kshirsagar
Database Group, Nanoscience & Technology, Springer Nature, Heidelberg, Germany

Session Friday 25 September: Correlative microscopy techniques for comprehensive material characterization | *Join the session*

Growing public and private investment into nanotechnology has led to a significant rise in nanotechnology data generation, with increasing research output and patents over the last decade. Nanotechnology has made a significant impact on a wide range of industries from medicine to aerospace – in developing new products and applications. However, utilizing this rapidly growing data remains a critical challenge. The increasing number of research articles and patents leads to a gap between data generation and data access. Nano (nano.nature.com) is an artificial intelligence powered research solution under the Nature Research portfolio that aims to provide highly indexed and structured information related to nanoscience derived from peer-reviewed journals across all major publishers. Figure 1 shows the three categories on Nano and the content.
Scanning probe techniques are crucial in understanding the correlations between surface structure and property of nano- and bio-materials. As seen in figure 2, 22,000+ nanomaterials characterized by atomic force microscope, 120,000+ Articles and 66,000+ Patents are part of the Nature Nano database.
This talk will illustrate how Nano can aid nanotechnology research communities to obtain fast and precise insight into the wealth of nanotechnology based scholarly knowledge.

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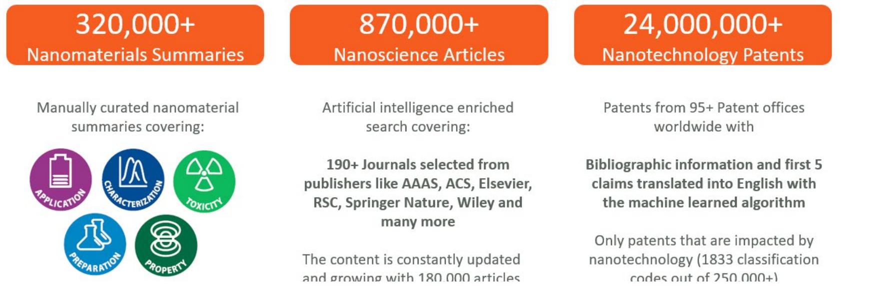
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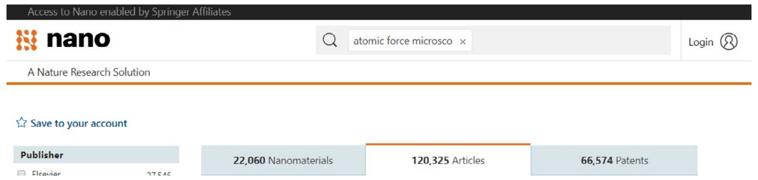
Figure 1: Nano (nano.nature.com)'s content as of Aug 2020 (left)

Figure 2: Nano's content screenshot with atomic force microscopy as the keyword as of Aug 2020. (below)



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66,574 Patents

pySurf - a Python library for analysis of surface data

Dr. Vincenzo Cotroneo
The National Institute for Astrophysics, Italy

Session Friday 25 September: Overcoming barriers in AFM | *Join the session*

OEM software provided with AFMs (or other instruments) usually offer a large amount of functionalities and a quick way to analyze data and generate reports. However, this simplicity of use, often comes with a limitation in flexibility. This is especially felt in a research context, where custom analysis can be required, or when AFM data need to be integrated with other instruments, each one with its own software, formats and operational parameters.
pySurf is an open-source flexible library for the realization of complex surface analysis, written in Python, probably the most widespread programming language in scientific communities.
The features of Python allow an easy integration with the many highly specific analysis and visualization tools available, and the possibility of integration in several flavours of interfaces, from GUI, to notebooks, to command line or scripting. The self documentation capabilities of python also enhance maintainability and allow a soft learning curve.
We will give an overview of this package and present some usage example taken from real-life research, mainly on metrology of astronomical X-ray mirrors.

Instrument Development for New AFM Capabilities

Fangzhou Xia
Massachusetts Institute of Technology, USA

Session Friday 25 September: Overcoming barriers in AFM | Join the session

Microscopy instruments are important in nano-technology research for imaging of nanoscale phenomena. Among such tools is the atomic force microscope (AFM) for nanoscale imaging and surface characterization. An AFM scans a micro-cantilever over the sample surface to measure various quantities from the probe-sample interaction. With high-speed imaging, dynamic processes can be visualized to improve fundamental understanding of microscopic interactions. Scientists can use videos, in addition to images, to observe and compare experimental data with theoretical predictions, and verify models without speculating about intermediate dynamics. However, conventional AFMs have limited throughput that allows for static imaging only and require transparent working environments.

In our group, we develop AFM subsystems to remove such AFM restrictions and enable advanced visualization capabilities. Example applications include visualizing chemical reactions and biological responses in their native environments. We design new generation nano-positioners to address the low imaging throughput limitation and enabled high-speed video-rate AFM imaging. To resolve the transparency limitation, active cantilever probes with embedded piezoresistive sensing and thermomechanical actuation are developed with nano-fabrication techniques. We apply coating to protect the functional structures to enable AFM imaging in chemically harsh opaque liquid enabled. This allows observation of samples in their native environment such as non-transparent acid, crude oil, cells in blood, etc.

In this talk, the development of high-bandwidth nano-positioning systems, active cantilever probes and other enabling technologies will be presented. We will show AFM images and videos to demonstrate the new capabilities. The high-speed imaging capability is used to capture videos for calcite etching process and electrochemical deposition of copper on gold substrate. Images taken in opaque acidic liquid and crude oil are used to verify the functionality of the coated active cantilever probes. We conclude by discussing related applications and broader impacts of the development on both the AFM user and instrument designer communities.

Improving image contrast and estimating material properties by Biharmonic AFM

Prof. Babak Eslami
Widener University, USA

Session Friday 25 September: Overcoming barriers in AFM | Join the session

Since invention of atomic force microscopy (AFM) in 1980s, researchers have focused on developing new characterization methods to measure different materials properties. These efforts have been mostly toward characterizing soft matters such as polymers or biological samples with the goal of minimizing surface damage during imaging process. One way to increase accuracy and sensitivity in AFM is exciting the cantilever's higher modes (i.e., Higher Modes AFM) or exciting multiple modes simultaneously (Multifrequency AFM). Bimodal AFM captures both topography and material composition of a material especially for soft heterogeneous material in a single-pass measurement. Based on the rectangular beam theory, the cantilever's second to first eigenmode frequency is 6.27. Due to the fact that they are not multiple integers, there are irregular taps over the surface and high signal-to-noise ratio for the higher modes excitations. This can cause nonlinear vibrations in the cantilever in addition to the fact that the probe does not interact with each pixel similarly. Therefore, exciting the cantilever with higher harmonics instead of the eigenmodes in multifrequency AFM mechanisms and its advantages are discussed.

In this talk, a new imaging technique known as biharmonic AFM is presented. Similar to bimodal AFM, in biharmonic AFM, the cantilever is excited with two frequencies simultaneously. The first excitation frequency is at or near the first eigenmode frequency. The second excitation frequency is the closest harmonic frequency to the second eigenmode frequency. Based on this theoretical discussion, this study provides the guideline for selecting the correct harmonic according to cantilever's type and geometry. It is found that the ratio of second to first eigenmode frequency heavily depends on the geometry of the cantilever and applicable environment. Additionally, it is found that cantilevers with lower eigenmode frequency ratio, excited with the first eigenmode frequency and higher harmonic, can provide higher phase contrasts in comparison to bimodal AFM. Numerical and experimental studies prove that for enhancing phase contrast and accurately measuring material properties the ratio of 2nd to 1st frequency should be minimized. Additionally, the second excitation frequency should be the closest n-th harmonic to this ratio.

Pulsed Force Kelvin Probe Force Microscopy for < 10 nm Contact Potential Mapping in Ambient Conditions

Devon Jakob
Lehigh University, USA

Session Friday 25 September: Overcoming barriers in AFM | Join the session

Measurement of the contact potential difference (CPD) and work functions of materials are crucial in the development and study of new electronically active materials and devices. Kelvin probe force microscopy (KPFM), an imaging technique based on atomic force microscopy, is a robust and popular tool for CPD and work function mapping at the nanoscale. However, the conventional KPFM variants are typically limited in their spatial resolution to 30 – 100 nm under ambient conditions. The continuingly decreasing size and increasing complexity of photoactive materials, semiconductor devices, and related materials present challenges in uncovering their important electrical properties through KPFM. In this talk, a new paradigm of contact potential measurement in KPFM is presented. The new imaging technique, pulsed force Kelvin probe force microscopy (PF-KPFM), is developed and can reliably obtain CPD and work function images with ~ 10 nm spatial resolution, on a wide range of samples.

The operating principle of PF-KPFM deviates from the fundamental paradigm of all current KPFM techniques, as there is no requirement of external oscillating voltage to produce the KPFM signal. As a result, PF-KPFM can avoid many of the intrinsic limitations associated with other KPFMs which limit their achievable spatial resolution; most notably the requirement for lift mode and the stray capacitance effect. Here, we explain the operating principle of PF-KPFM, how it compares to conventional frequency modulated KPFM (see Figure 1a-d), and present results on several materials to exemplify the robustness of our technique. In particular, we show that PF-KPFM is suitable for probing the interfaces between metals and semiconductors, for probing individual ferroelectric domains and boundaries on ferroelectric materials (see Figure 1e-f), and for imaging nanoscale perovskite domains. From these measurements, new insights into the nanoscale electrical properties are established.

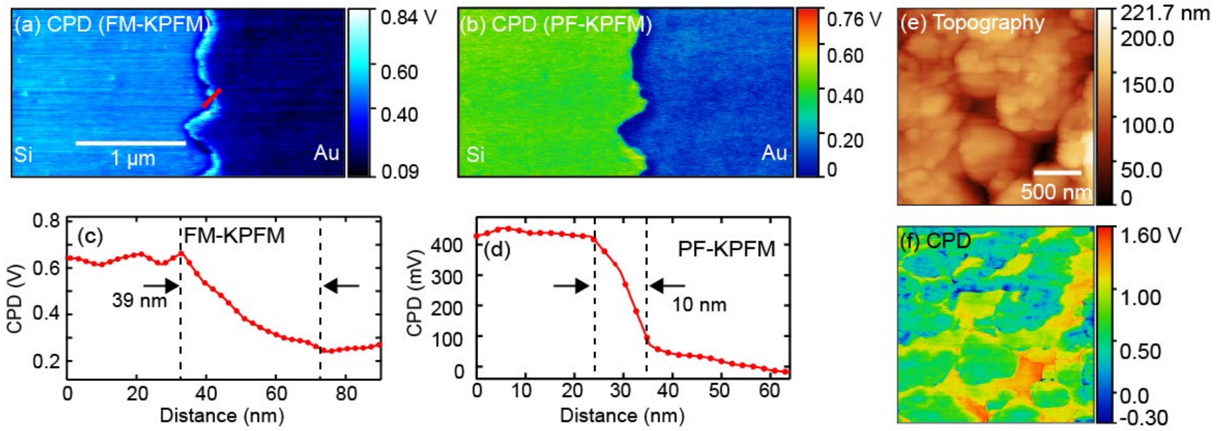

Figure 1. Comparing the spatial resolution of FM-KPFM and PF-KPFM on a standard KPFM sample composed of silicon (on the left) and gold (on the right) (a) FM-KPFM on the sample. (b) PF-KPFM over the same area. (c) The spatial resolution for FM-KPFM is estimated at 39 nm, found from the signal cross section marked by the red line in (a). (d) The spatial resolution of PF-KPFM over the same area is estimated to be 10 nm. (e) Topography of a BaTiO3 ferroelectric sample. (f) PF-KPFM measurement on the region, showing the heterogeneity of the contact potential over nanoscale domains.

Using scanning electrochemical cell microscopy (SECCM) architecture for high-temporal resolution stochastic electrochemistry

Marc Brunet Cabre
Massachusetts Institute of Technology, USA

Session Wednesday 23 September: Resolving Electrochemical Properties on Functional, Sustainable Materials I Join session

Stochastic collision electrochemistry[1] requires high-resolution and high-bandwidth current amplification due to the low magnitude and short duration of the current signals. However, increasing the current amplifier bandwidth leads to increased current noise levels, which in turn obscures the current signal generated from stochastic collision electrochemistry experiments[2,3] Noise levels are very sensible to the input capacitance of the current amplifier when operating at high bandwidth.[4]
In this presentation a new strategy will be introduced to minimise the parasitic effect contribution over the input capacitance to a current amplifier for stochastic collision electrochemistry. This is achieved by using a movable microscale electrochemical cell, formed at the end of a micropipette using a scanning electrochemical cell microscopy approach,[5] to conduct electrochemical experiments in close proximity (~300 µm) to a custom design transimpedance amplifier. We demonstrated this via electro-oxidation of single Ag nanoparticles detected at 1 MHz bandwidth.

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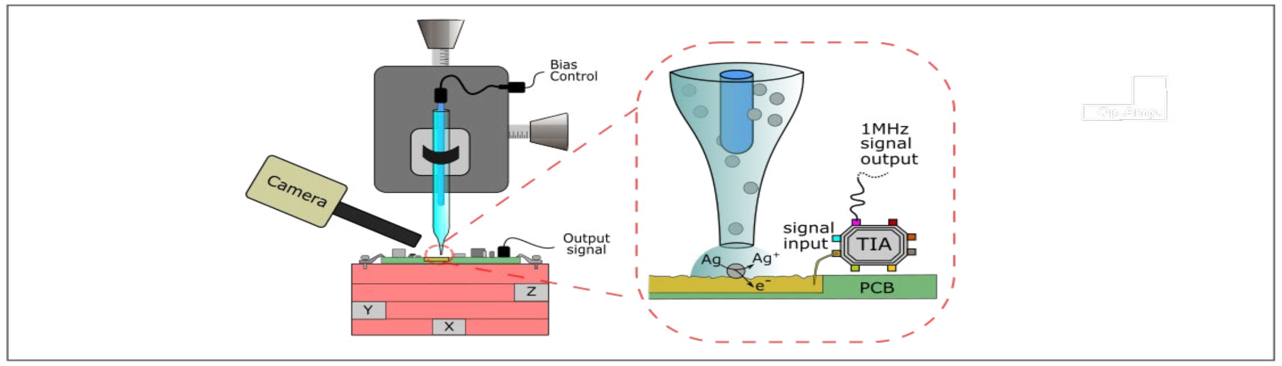

Figure 1: On the left, scheme of SECCM set up. On the right, magnification over the end of the nanopipette, where a microscale electrochemical cell is formed in close proximity of the transimpedance amplifier (TIA). It is also detailed the nano-impact oxidation of silver nanoparticles.

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Session / Topic	Author	Abstracts
Session Topic: Resolving Electrochemical Properties on Functional, Sustainable Materials		link
Scrutinizing the bonding of 2-mercaptobenzimidazole on Cu(111) by STM and DFT	Marion Antonia van Midden	
Challenges in Dopamine Sensing at Carbon-Based Electrodes: Studies of Dopamine Adsorption Properties and Electrode Fouling at Physiological pH	Filip Grajkowski	
Atomic Force Microscopy analysis on the modified separator of the Lithium-Sulfur batteries	Giovanni Saenz-Arce	
Study of vanadium oxide state of vanadium films thermally oxidized	Juan Fernández	
Session Topic: 2D materials and nanoelectronics		link
Structuring and Integration of Molecular Nanomagnets on Sensors and Quantum Devices by Deposition with Atomic Force Microscopy	Maria Carmen Pallarés	
2D talc nanosheets obtained via liquid phase exfoliation	Mariana Prado	
Crystal-Oriented Wrinkles with Origami-Type Junctions in Few-Layer h-BN	Camilla Oliveira	
Anomalous Response of Supported Few-Layer Hexagonal Boron Nitride to DC Eletric Fields: A Confined Water Effect?	Camilla Oliveira	
Exfoliated lead sufide characterization by Scanning Probe Microscopy	Felipe Beato	
Scanning Probe Microscopy in the Control of the Electromechanical Properties	Maurício Vasconcelos Bessa	
AFM investigation of electropolished copper foils for CVD graphene growth	Eduardo Antônio Silva	
Atomic scale study of strain effects on 2D materials and their electronic properties	Jack Maughan	
Investigating Triboelectric Charging by Kelvin Probe Force Microscopy	Qiwei Hu	
Scanning Tunneling Microscopy and Spectroscopy of Pt-based Transition Metal Dichalcogenides	Kuanysh Zhussupbekov	
A Study of Topography and Magnetic Domain of Galfenol Thin Films	Yu Tang	
Session Topic: Polymer- and Biotechnology		link
Self-assembly of tetracycline molecule by Scanning Probe Microscopy	Alexia Dias	
Nanomechanical properties of nanocelulose by Scanning Probe Microscopy	Renata Maria de Paula	
Study of the interaction of melanin with different acid bleaches by Probe Scanning Microscopy	Thamires Almeida Sanches	
Development and characterization of type I bovine collagen and nanostructured talc nanobiocomposites for bone repair	Ana C.F. de Brito	
Heating effect in the calcination process of nano selenite	Thiago R.G. Silva	
Biomechanics of primary hippocampal neurons and SH-SY5Y cells in oxygen and glucose deprivation (OGD) model	Tomasz Zielinski	
AFM as a tool in studies of stroke – changes in elasticity of organotypic hippocampal cultures after OGD	Natalia Bryniarska	
Atmospheric pressure He plasma jet for surface treatment: correlation between plasma optical / electrical parameters and surface properties via AFM and SWCA	Andrei Nastuta	
SPM monitoring of laser surface designing of polyimide films for efficient light management in a solar cell device	Iuliana Stoica	
Session Topic: Overcoming barriers in AFM		link
Gypsum: an environment-friendly, inexpensive and robust height calibration standard at nanometer-scale for atomic force microscopy	Ana Paula Barboza	

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2020 NanoScientific Forum Europe
Scanning Probe Microscopy (SPM)
23-25 September I VIRTUAL EVENT

Friday, September 25 I 11-00-12.00 CEST — Join Session: Link

Session / Topic	Author	Abstracts
Session Topic: Polymer- and Biotechnology		link
Investigation of humidity-induced self-assembly Phe-Phe in solid-state organic film	Semen Vasilev	
Properties of the nanoparticles of different nature studies by atomic force microscopy	Irina Chelnokova	
Ultrasound assisted nanocomposite Chitosan ZnO copper oxide and their application for the improvement of the cooling system	Insaf Ould Brahim	
Nanomechanical properties of blood cell surface after exposure to extremely low frequency electromagnetic field	Maria Starodubtseva	
Liposome-An effective nano carrier	Snehal Kawade	
Enhancement of antimicrobial action of cationic fullerene derivatives in the presence of 4-hexylresorcinol	Hike Nikiyan	
Atomic Force Microscopy of aquatic spun silk	Janusz Strzelecki	
Nanomechanical changes of endothelial cells as a marker of toxicity of silver nanoparticles	Agnieszka Kolodziejczyk	
Cell adhesion heterogeneity in bacterial culture studied by AFM	Dariusz Laskowski	
Effect of post-annealing process on the mechanical and piezoelectric properties of Poly (L-lactic acid) electrospun nanofibers	Cuong Nguyen Thai	
The influence of chemotherapeutics on viscoelastic properties of cancerous and non-malignant bladder cancer cells	Joanna Zemła	
Significance of finite thickness correction for AFM measurements of prostate cancer cells lamellipodia	Andrzej Jan Kubiak	
Mechanical Characterization of Bladder Cancer Cells by means of Atomic Force Microscopy	Kajangi Gnanachandran	
AFM-based local nanomechanical characterization of pulsed UV laser-nanoinduced patterns on azo-polyimide films	Iuliana Stoica	
Session Topic: Advanced research on ferroelectric materials		link
Formation and characterization of self-organized ferroelectric ß-glycine films	Daria Vasileva	
At the origin of surface charge effect in piezoresponse force microscopy experiments	Huan Tan	
Session Topic: Overcoming barriers in AFM		link
Multiscale: A Processing Tool for Scanning Probe Microscopy and Other n-Dimensional Datasets	Ralph Bulanadi	
Single-pass magnetic force microscopy technique for rapid survey scans	Antoniu Moldovan	
Hurst exponent calculations as a new method to analyze AFM images	Andrea Ehrmann	
Fabrication of a Berkovich tip on an AFM cantilever using a focused ion beam for the nanoelectromechanical characterization of nanomaterials	Zhi Li	
Numerical Model for Adhesion Force Prediction on Contaminant Particles on EUV Reticle	Benny Ku	
Data-driven, dual actuation controller design for a high-speed atomic force microscope	Navid Asmari Saadabad	
A new Approach for Nanowire Diameter Measurement using Atomic Force Microscope	Nikita Chhetri	
What you cut is what you get	Rebecca Savage	
Session Topic: Correlative microscopy techniques for comprehensive material characterization		link
Insights from application of photo-induced force microscopy (PiFM) for investigation of multicore magnetic nanoparticles (MCNPs)	Daniela Täuber	

Read the abstracts:

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