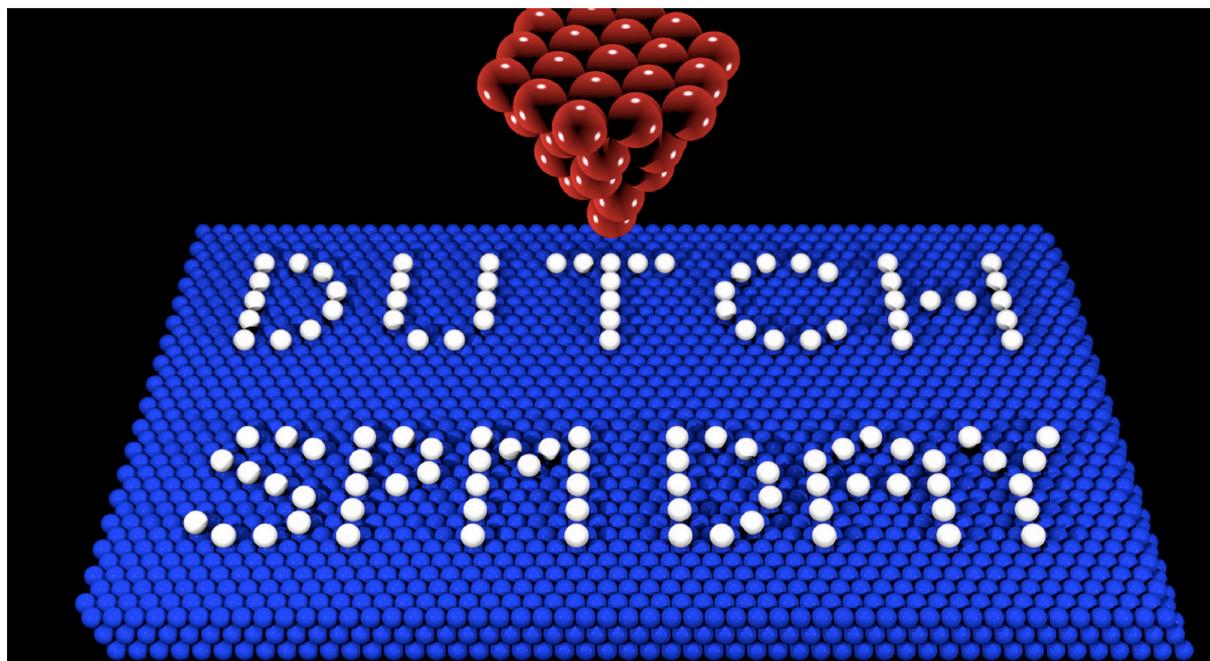




November 8, 2017



2017

**Leiden Institute of
Chemistry
Leiden University**



Leiden Institute of Chemistry



November 8, 2017



Leiden Institute of Chemistry



November 8, 2017

Dutch SPM Day 2017

Organized by:
Leiden University
Irene Groot

Secretary: José Dijkzeul



Leiden Institute of Chemistry



November 8, 2017

Program

9.00 - 9.25: arrival + coffee

9.25 - 9:30: opening

9.30 - 10.20: invited talk **Markus Heyde**, Fritz Haber Institute Berlin, Germany

"Looking into the Structure of Glass by Designing a New 2D Material"

10.20 - 10.40: contributed talk **Samuel Lesko**, Bruker

"High resolution electrical characterization in liquid by conductive nanoelectrode probes"

10.40 - 11.00: contributed talk **Maarten Leeuwenhoek**, Leiden University/TU Delft

"Nanofabricated STM double-tips for probing quantum materials"

11.00 - 11.30: coffee/tea break

11.30 - 11.50: contributed talk **Xin Deng**, Leiden University

"In situ observation of Pt electrode by electrochemical AFM in sulfuric acid under potential cycling conditions"

11.50 - 12.10: contributed talk **Pantelis Bampoulis**, TU Twente

"Pressure induced melting of nanoconfined ice"

12.10 - 12.30: contributed talk **Marlou Slot**, Utrecht University

"(Below) 2D: realizing electronic structures on a Cu(111) surface by atomic manipulation"

12.30 - 14.00: lunch, exhibition, poster session

14.00 - 14.50: invited talk **Volker Rose**, Argonne National Laboratory, USA

"Synchrotron X-ray Scanning Tunneling Microscopy: A Novel Approach for the Nanoscale Characterization of Materials with Chemical and Magnetic Contrast"

14.50 - 15.10: contributed talk **Bay Tran**, RU Groningen

"On-surface formation of cumulene by dehalogenative homocoupling of alkenyl gem-dibromides"

15.10 - 15.30: contributed talk **Leon Jacobse**, Leiden University

"Correlation of surface site formation to nanoisland growth in the electrochemical roughening of Pt(111)"

15.30 - 16.00: coffee/tea break

16.00 - 16.20: contributed talk **Alexander Volodin**, KU Leuven

"Stress dependence of the suspended graphene work function: Vacuum Kelvin probe force microscopy and density functional theory"

16.20 - 16.40: contributed talk **Oleg Kurnosikov**, TU Eindhoven

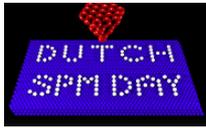
"Functionalization of cleaved probes for STM and AFM"

16.40 - 17.00: contributed talk **Martin de Wit**, Leiden University

"Quantifying spin-induced dissipation using M(R)FM"

17.00: drinks sponsored by Bruker ("Bruker Borrel")





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9:30 Markus Heyde, Fritz Haber Institute Berlin

Looking into the Structure of Glass by Designing a New 2D Material

Markus Heyde, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 14195 Berlin, Germany, Electronic mail: heyde@fhi-berlin.mpg.de

Determining the structure of amorphous materials used to be challenging due to the complexity of this material class. Here I would like to review structural studies of bulk and two-dimensional amorphous silica, focusing on the recent discovery a new 2D amorphous material. For the first time a clear image of an amorphous network structure has been obtained which allowed for the derivation of atomic sites and a detailed analysis from real space coordinates by using scanning tunneling and atomic force microscopy. I will discuss the benefits of these measurements on the newly developed thin silica bilayer film system and consider comparisons between two-dimensional structures and their three-dimensional bulk silica counterparts. Recent experiments which establish 2D amorphous silica as a candidate insulating material for two-dimensional nanoelectronics will also be discussed. Furthermore, I would like to provide you a sneak preview into the latest results pointing towards another amorphous oxide film system.





10.20 Samuel Lesko, Bruker

High resolution electrical characterization in liquid by conductive nanoelectrode probes

*Samuel Lesko, Peter De Wolf, Rakesh Poddar, and Zhuangqun Huang**
Bruker Nano Surfaces, 112 Robin Hill Road, Goleta, California, CA, 93117, USA

**Corresponding Author: Teddy.Huang@Bruker.com*

Battery and catalytic researches are nowadays topics of importance where reactivity and electrical properties heterogeneity of materials on the nanoscale have a significant impact on macroscopic performance. The differences in structural, mechanical, electrical, and/or electrochemical properties locally all together account for this reactivity variation. Therefore, approaches capable of simultaneously capturing correlated multidimensional information on the nanoscale in liquid media are highly desired to perform *in situ*, *in vivo*, non-invasive characterization due to the natures of the samples.

While classic scanning electrochemical microscopy (SECM) using micro-electrodes is known approach for studying local electrochemistry, it suffers challenges on the position control of the tip-sample distance, which is essential for obtaining accurate electrochemical information. In addition, the spatial resolving power is generally low and difficult to achieve sub μm resolution. Likewise, standard AFM based techniques in air for electrical characterization such as capacitance, conductivity, surface potential cannot apply in conductive liquids due to straight current leakage. Furthermore, samples, such as biological or energy materials are often highly soft and fragile which poses another grand challenge for AFM-based imaging using classic contact mode.

Recently, we have developed batch-fabricated, high-quality and robust nanoelectrode probes fully insulated from solution except for the tip apex which exposes Pt-coated conductive tip ~ 200 nm height with diameter of ~ 50 nm. These probes pass > 10 hours continuous electrochemical testing and feature the ease of use. These probes are used in combination with PeakForce Tapping (PFT) mode where probe is sinusoidally modulated at off-resonance, low frequency, with a defined imaging force as low as 20 pN. This enables *in situ*, *in vivo*, non-invasive, stable imaging for challenging fragile and soft samples. PFT is essentially based on fast force curve acquisition and analysis. It naturally allows quantitative mechanical mappings at normal AFM scan rate. Combination of high-quality nanoelectrode probes with PFT, allows us to simultaneously capture multidimensional information in the electrochemical environment at < 100 nm spatial resolution, including topographic, conductivity, mechanical, and electrochemical properties. In addition, the nanoelectrode probe by design also enables all other nanoelectric measurements in liquid as it is fully insulated except for the tip.^{1,2}



This talk showcases a variety of applications examples using nanoelectrodes, including catalysis, surface defects on lamellar, patterned nanostructured electrodes, etc. Associated spectroscopy mapping will be shown to illustrate power of such an approach. Some examples about nanoelectric measurements such as Kelvin probe and PiezoResponse in liquid are also presented. These cases demonstrate the versatility and potential new capabilities through those nanoelectrodes that are well suited for today's highly-multidisciplinary research.

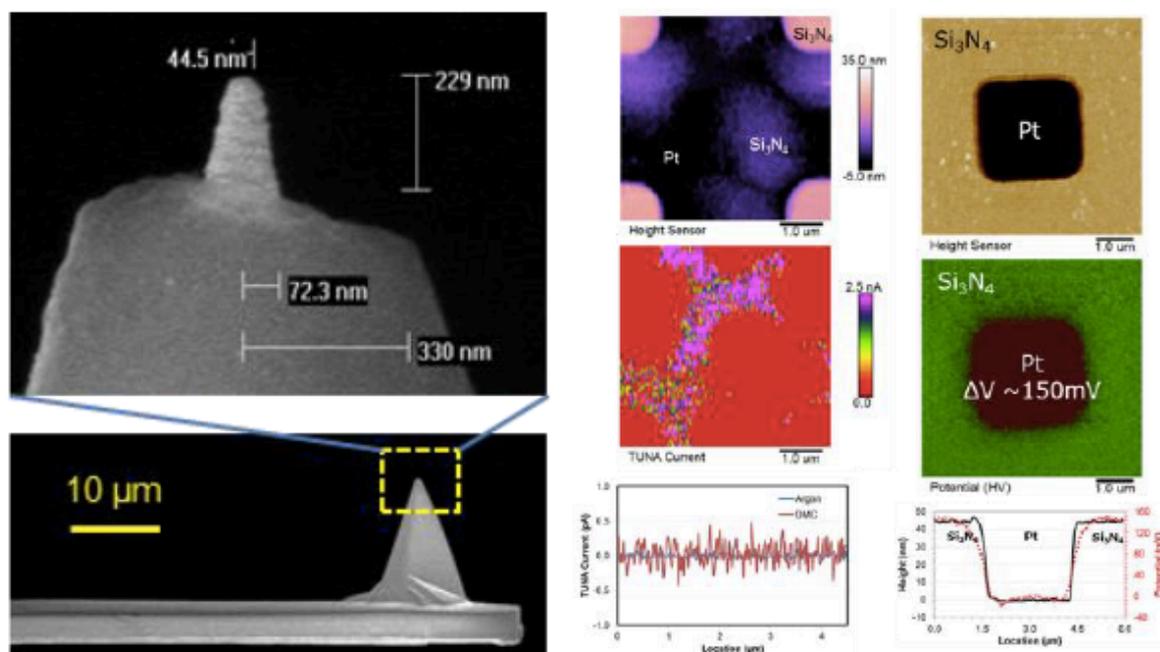


Figure 1. Left: SEM images of the probe. The probe is fully insulated with only the Pt-coated tip apex exposed. Right: Example of electrical characterization in liquid. On center: battery electrode in DMC with contact current mapping in Peak Force Tapping. On left, mesh of Silicon Nitride with opening made of Platinum is measured by Frequency Modulation based Kelvin probe microscopy in milli-Q water.

References:

- 1 Nellist et al., *Nanotechnology*, 2017, **28**, 095711.
- 2 Huang et al., *Microscopy Today*, 2016, **24**, 18.



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10.40 Maarten Leeuwenhoek, Leiden University/TU Delft

Nanofabricated STM double-tips for probing quantum materials

Maarten Leeuwenhoek^{1,2}, Richard Norte¹, Koen M. Bastiaans², Irene Battisti², Yaroslav M. Blanter¹, Jan Zaanen², Milan P. Allan² and Simon Gröblacher¹

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2: Leiden Institute of Physics, Leiden University, Niels Bohrweg 2, 2333 CA Leiden, The Netherlands

Here, we report on the design and fabrication of a new type of smart tip for Scanning Tunneling Microscopy (STM). By fully integrating a metallic tip onto a silicon chip using modern micro-machining and nanofabrication techniques we show the possibility of device based STM tips. Contrary to traditional etched/grinded wire tips, these can be integrated in lithographically defined electrical and photonic circuits, as well as with mechanical systems. We aim at creating two-probe devices with a tip to tip spacing below ~ 50 - 100 nm. Such double tips could not only measure the local density of states at the two individual tips with atomic resolution, but also the current sent through one tip and collected by the other. This would allow us to measure and map the two point propagator of the electrons, providing new information on the transport of correlated electrons at the atomic scale.





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11.30 Xin Deng, Leiden University

In situ observation of Pt electrode by electrochemical AFM in sulfuric acid under potential cycling conditions

Xin Deng, Federica Galli, Marc T. M. Koper

Understanding the electrochemical behavior of Pt at solid/liquid interface is of significant importance to the development of efficient electrochemical devices, such as fuel cells and water electrolyzers. In this work, the evolution of the surface morphology of a polycrystalline platinum under potential cycling condition was investigated by EC-AFM. After 50 cycles between 0.05-1.8 V in 0.1 M H₂SO₄, the Pt surface is coarsened and nanoparticles of several nanometers appear on the surface. The critical upper and lower potential for nanoparticles formation is found to be 1.8 and 0.8 V, respectively. The in situ AFM observation coupled with CV reveals the periodic disappearance and reappearance of the nanoparticles, based on which, the nanoparticles are attributed to be Pt nanoparticles and a model for the nanoparticles formation is proposed. While the formation of a thick oxide layer is the prerequisite, the reduction process is found to have a strong influence on Pt nanoparticles formation as well. This investigation provides the visualization of Pt surface under electrochemical control in a large potential window, which enables a more intuitive understanding of the Pt redox process.





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11.50 Pantelis Bampoulis, TU Twente

Pressure induced melting of nanoconfined ice

Pantelis Bampoulis, TU Twente

The states of aggregation of confined water are highly important and of great fundamental interest in surface chemistry, life and environmental sciences. Due to limited experimental access a coherent understanding of the phase behavior and the occurring phase transitions of interfacial ice is still lacking. Using scanning probe techniques, we show that, at room temperature, ice confined between graphene and muscovite mica undergoes a phase transition to a quasi-liquid phase at a critical applied pressure of 6 GPa. The transition is completely reversible, that is, refreezing occurs when the applied pressure is lifted. Our findings form a paradigm of the classical phenomenon of regelation decoupled from environmental thermal effects. The critical pressure to melt the ice crystals is lower at higher temperature and the latent heat of fusion (0.15 eV/H₂O) is more than two times larger than the corresponding three dimensional value at ambient conditions.





12.10 Marlou Slot, Utrecht University

(Below) 2D: realizing electronic structures on a Cu(111) surface by atomic manipulation

M.R. Slot¹, S.N. Kempkes², T.S. Gardenier¹, P.H. Jacobse¹, G.C.P. van Miert², S.J.M. Zevenhuizen¹, C. Morais Smith², D. Vanmaekelbergh¹ and I. Swart¹

¹ *Debye Institute for Nanomaterials Science, Utrecht University, the Netherlands*

² *Institute for Theoretical Physics, Utrecht University, the Netherlands*

Geometry and dimensionality are decisive factors for the electronic properties of materials. An example is the 2-D honeycomb lattice of graphene, giving rise to Dirac cones in which charge carriers behave as effectively massless particles. A possible route to create novel flat structures in a controlled manner is by confining the surface state electrons of Cu(111) by accurate positioning of adsorbed CO molecules [1]. Employing this strategy, we show the realization of a Lieb lattice [2]: a 2D square-depleted lattice, which is characterized by a band structure featuring Dirac cones intersected by a flat band. Using scanning tunnelling microscopy, spectroscopy and wavefunction mapping, we confirm the predicted characteristic electronic structure of the Lieb lattice. At higher energies, we find states that originate from the artificial p-orbitals of the electronic lattice. Moreover, we move beyond the well-studied integer dimensions. Fractional dimensions could open a new range of possibilities for electronic nanosystems. Here, we realize and characterize a Sierpiński triangle fractal with dimension 1.58. We find that the fractality of this quantum fractal is not only reflected in the geometry and energy levels, but also in the wave functions, which are fractal themselves. The experimental findings were reproduced by muffin-tin and tight-binding calculations.

1. K.K. Gomes et al., *Nature* 483, 306–310 (2012)
2. M.R. Slot et al., *Nature Physics* 13, 672-676 (2017)





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14.00 Volker Rose, Argonne National Laboratory, USA

**Synchrotron X-ray Scanning Tunneling Microscopy:
A Novel Approach for the Nanoscale Characterization of Materials with
Chemical and Magnetic Contrast**

Volker Rose

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The real-space observation of chemistry and magnetic structure using scanning probe microscopy (SPM) methods or synchrotron-based x-ray microscopy (XM) continues to have a tremendous impact on our understanding of functional materials. However, although SPM methods provide high spatial resolution, they typically lack direct chemical contrast and the ability to quantify magnetic moments. On the other hand, XM can provide chemical as well as magnetic sensitivity, but the spatial resolution is inferior. In order to overcome these limitations, various groups have developed a new technique that combines synchrotron radiation with the high spatial resolution of scanning tunneling microscopy (STM). The goal is to combine the spin sensitivity and chemical contrast of synchrotron x-rays with the locality of STM.

Recently, substantial progress was made on Argonne's synchrotron x-ray scanning tunneling microscopy (SX-STM) project. In particular, we demonstrated the power of SX-STM for elemental characterization and topography of individual Ni and Co nano-islands at 2 nm lateral resolution with single atom height sensitivity [1,2], tested new probe tip concepts based on carbon nanotubes [3] and multilayer tips [4], and demonstrated x-ray imaging of nanoscale magnetic domains of an iron thin-film by x-ray magnetic circular dichroism (XMCD) contrast [5]. Further substantial advances are expected using the new low temperature (LT) SX-STM system, which has been developed over the last 3 years and is currently under commissioning.

To fully exploit the special capabilities of the new microscope, XTIP, a dedicated beamline for SX-STM is under construction at the Advanced Photon Source. To meet the scientific objective of the nanoscience and nanomagnetism communities most effectively, we are going to build a soft x-ray beamline with full polarization control operating over the 500-2000 eV energy

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range. The dedicated XTIP beamline will provide researchers access to a one-of-a-kind instrument. Among the potential breakthroughs are “designer” materials created from controlled assemblies of atoms and molecules, and the emergence of entirely new phenomena in chemistry and physics.

This work was funded by the Office of Science Early Career Research Program through the Division of Scientific User Facilities, Office of Basic Energy Sciences of the U.S. Department of Energy through Grant SC70705. This work was performed at the Advanced Photon Source and the Center for Nanoscale Materials, a U.S. Department of Energy Office of Science User Facility under Contract No. DE-AC02-06CH11357.

References:

- [1] N. Shirato et al., Nano Letters 14, 6499 (2014).
- [2] H. Kersell et al., Appl. Phys. Lett., in print (2017).
- [3] H. Yan et al., J. Nanomaterials 2015, 492657 (2015).
- [4] M. Cummings et al., J. Appl. Phys. 121, 015305 (2017).
- [5] A. DiLullo et al., J. Synchrotron Rad. 23, 574 (2016).





14.50 Bay Tran, RU Groningen

On-surface formation of cumulene by dehalogenative homocoupling of alkenyl gem-dibromides

B. Tran,¹ Q. Sun,² L. Cai,² H. Ma,² X. Yu,² C. Yuan,² M. Stöhr,¹ and W. Xu²

¹ Zernike Institute for Advanced Materials, University of Groningen, Groningen, Netherlands

² Interdisciplinary Materials Research Center, Tongji University, Shanghai, China

Recently, on-surface synthesis based on the use of covalent linking has offered an excellent platform to construct novel 1D and 2D carbon-based materials. This new approach has received constantly increasing scientific attention because of its advantages compared to its counterpart solution-based synthesis for building durable, controllable nanostructures for future electronic devices. The most frequently used reaction is Ullmann-type coupling which involves the thermally induced dehalogenation of pre-defined C-X groups (X = halogen atom) for the formation of C-C bonds. So far, all employed halide precursors have only one halogen attached to a carbon atom. Therefore, to extend our fundamental understandings of on-surface C-C coupling reactions it is particular interest to study the effect of introducing more than one halogen atom to a carbon atom with the aim of producing multiple unpaired electrons.

In this work, we successfully fabricated cumulene products on a Au(111) surface via dehalogenative homocoupling reactions by introducing an alkenyl gem-dibromide [1]. The reaction products and pathways were unambiguously characterized by the combination of high-resolution scanning tunneling microscopy (STM) and non-contact atomic force microscopy (nc-AFM) measurements together with state-of-the-art density functional theory (DFT) calculations. This study further supplements the database of on-surface synthesis strategies, and importantly, it provides a facile manner for incorporation of more complicated carbon scaffoldings into surface nanostructures.

[1] Q. Sun, B. Tran, *et al.*, *Angew. Chem. Int. Ed.* **56**, 12165 –12169 (2017).





15.10 Leon Jacobse, Leiden University

Correlation of surface site formation to nanoisland growth in the electrochemical roughening of Pt(111)

Leon Jacobse¹, Yi-Fan Huang¹, Marc T.M. Koper¹, and Marcel J. Rost²

¹ *Leiden Institute of Chemistry, Leiden University, Leiden, The Netherlands*

² *Huygens-Kamerlingh Onnes Laboratory, Leiden University, Leiden, The Netherlands*

Platinum plays a central role in a wide variety of electrochemical devices. Electrode degradation, especially under oxidizing conditions, forms an important barrier for the widespread application of such devices. Although it is known that repeated oxidation and reduction of platinum electrodes results in irreversible surface structure changes, over thirty years of research did not yet yield to a conclusive description of this process on the atomic level; not even for well-defined single crystal surfaces.

Using a special EC-STM, which is capable of measuring the electrochemical signals simultaneously during imaging *in operando*, we directly correlate, for the first time, the evolution of the electrochemical (hydrogen desorption) signal of Pt(111) to the observed roughening of the surface. In the later stages, we find a strong correlation between the evolution of the roughness and the absorption peaks clearly indicating that each created step contributes equally strong to the adsorption signal as well as to the roughness. However, and fully surprising, in the early stages step edges are created that do not contribute to the electrochemical signal.

These results present not only an important step forward in understanding the atomic scale process of the electrochemical roughening of Pt(111), but also provide valuable insight in the degradation of industrially relevant platinum nanoparticles, as a large part of their surface is naturally composed out of {111} planes.



16.00 Alexander Volodin, KU Leuven

Stress dependence of the suspended graphene work function: Vacuum Kelvin probe force microscopy and density functional theory

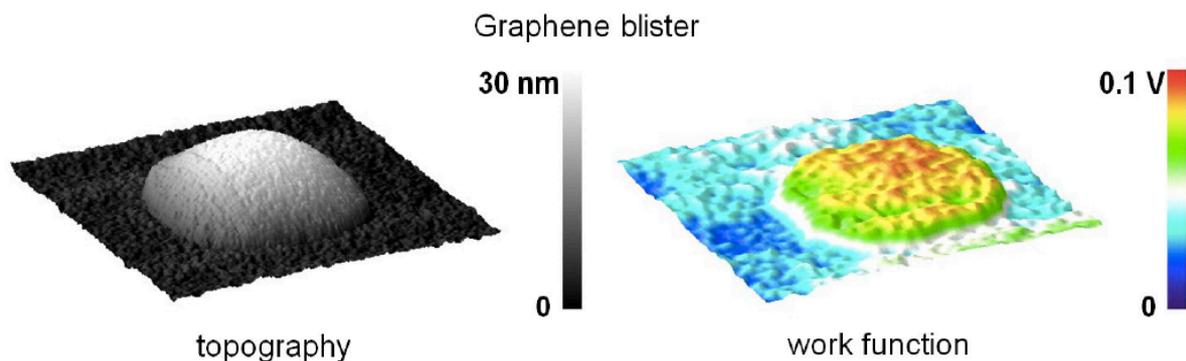
A. Volodin,¹ C. Van Haesendonck,¹ O. Leenaerts,² B. Partoens,² and F. M. Peeters²

¹*KU Leuven, Afdeling Vaste-stoffysica en Magnetisme, Celestijnenlaan 200D, BE-3001 Leuven, Belgium*

²*Universiteit Antwerpen, Departement Fysica, Groenenborgerlaan 171, BE-2020 Antwerpen, Belgium*

We report on work function measurements on graphene, which is exfoliated over a predefined array of wells in silicon oxide, by Kelvin probe force microscopy operating in vacuum. The obtained graphene sealed microchambers can support large pressure differences, providing controllable stretching of the nearly impermeable graphene membranes.

These measurements allow detecting variations of the work function induced by the mechanical stresses in the suspended graphene where the work function varies linearly with the strain and changes by 62 ± 2 meV for 1 percent of strain. Our related ab initio calculations result in work function variation larger by a factor of 1.4 than the experimental value. The limited discrepancy between theory and experiment can be accounted for by a charge transfer from the unstrained to the strained graphene regions.



A. Volodin et al., Appl. Phys. Lett. 110, 193101 (2017)



16.20 Oleg Kurnosikov, TU Eindhoven

Functionalization of cleaved probes for STM and AFM

O. Kurnosikov, H.T. Ciftci, B. Koopmans

An alternative type of probes for STM or tuning fork-based AFM is proposed [1]. Instead of using traditional needle-like tips made from a metallic wire, we use a sharp edge of a cleaved insulating substrate which initially was covered by a thin layer of conducting material. The sharp tip is formed at the intersection of two cleaving lines. Testing the cleaved tips in STM and AFM reveals the resolution down to atomic scale, the same as for convenient STM/AFM tips. However, with our approach, the planar substrates for tip fabrication are very suitable for a very simple and flexible functionalization of the STM and AFM tips, for example by using the cleavable substrates with magnetic, superconducting or other specific layers for tip fabrication. Moreover, profiting from Electron Beam or Focused Ion Beam Lithography (EBL/FIB) we also were able to tailor the metallic layer to obtain two- or multi-terminal structures on the probe. The electric current across a micro- or nanostructure formed near the tip end can modulate the tip-sample interaction and can be used either for a switchable sensing or a sample manipulation on the nanoscale. As an example we present a prototype of magnetically switchable or adaptive MFM probes. The current sent through a 100 nm constriction near the tip end can generate a localized magnetic field of up to 20 mT which is sufficient for magnetic manipulation. In the same way it is also possible to generate short heating pulses for local modification of the sample or nano-printing.

1. T. Siahhan *et al.* *Nanotechnology*, 27, 03LT01 (2016)





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16.40 Martin de Wit, Leiden University

Quantifying spin-induced dissipation using M(R)FM

Martin de Wit, Gesa Welker, Marc de Voogd, Jelmer Wagenaar, Arthur Den Haan, Tjerk Oosterkamp

Magnetic Resonance Force Microscopy is a technique that uses a very soft cantilever to measure the forces between its magnetic tip and the spins in a sample. Typically, Magnetic Resonance protocols are used to manipulate the magnetization of the spin ensemble, thereby changing the tip-sample interaction. However, we have utilized the extreme force sensitivity of our MRFM setup to measure the density and T1 times of paramagnetic spins in the native oxide layer of silicon, without sending resonant pulses. We also determined the density of the free electron spins associated with nitrogen defects in diamond. To expand the number of applications of this technique, we are currently working on a new MRFM setup, nicknamed the Easy-MRFM. It will enable measurements on a larger variety of samples since it is no longer necessary to integrate the sample under investigation with the chip that generates the oscillating B1 field and provides the detection of the motion of the force sensor.





November 8, 2017

Poster Abstracts



Leiden Institute of Chemistry



November 8, 2017

Poster 1

**Imaging Pulsed Laser Deposition oxide growth by in-situ Atomic Force
Microscopy**

W.A. Wessels, T.R.J. Bollmann, D. Post, G. Koster, and G. Rijnders*

*University of Twente, Inorganic Materials Science, MESA+ Institute for Nanotechnology, P.O.
Box 217, NL-7500AE Enschede, The Netherlands*

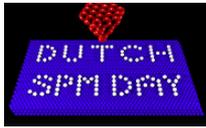
**Corresponding author: t.r.j.bollmann@utwente.nl*

The perovskite oxides are a fascinating class of materials, due to their wealth in available physical properties, such as superconductivity, ferromagnetism, ferro- and dielectricity. Control on the growth of such (ultra)thin film structures is very beneficial as it leads to unanticipated functional properties.

However, to gain deep insight in the growth process, the current in-situ diagnostic tools (RHEED, SXRD, SE) all operate in reciprocal space making interpretation not straightforward and averaging over large surface areas.

In this poster, we present the development of a surface probe microscopy (SPM) technique with which we enable local topography measurements of the thin oxide film growth front in real-space and at the nanometer scale. We present the concept, specifications, design and performance of our atomic force microscope (AFM) in a pulsed laser deposition (PLD) vacuum setup. The demonstrated performance on a typical perovskite oxide surfaces is of great importance not only for the thin film oxide community, but also towards a broader audience as it enables many more future applications for monitoring growth in process conditions.





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Poster 2

Poor electronic screening in lightly doped Mott insulators observed with STM

Irene Battisti et al., Leiden University

The effective Mott gap measured by scanning tunneling microscopy in the lightly doped iridate $(\text{Sr}_{1-x}\text{La}_x)_2\text{IrO}_4$ differs greatly from values reported by photoemission and optical experiments. Here, we show that this is a consequence of the poor electronic screening of the tip-induced electric field, an effect well known for semiconductors and called tip-induced band bending (TIBB). We develop a model able to retrieve the intrinsic Mott gap for doping concentrations $x < 4\%$, leading to a value which is in rough agreement with other experiments. At doping $x \approx 5\%$ we further observe circular features in the conductance layers, indicating higher density of free carriers and presence of a small concentration of donor atoms. We illustrate the importance of considering the presence of TIBB in STM experiments on correlated-electron systems and discuss the similarities and differences between STM measurements on semiconductors and lightly doped Mott insulators.





Poster 3

Local control of atomic vacancies to create artificial materials

*F.E. Kalff*¹, J. Girovsky¹, D. Coffey Blanco¹, J.L. Lado², E. Fahrenfort¹, J.W. van Dam¹, L.J.J.M. Peters¹, P. Schmitteckert⁴, F. Evers⁵, J. Fernández-Rossier^{2,3} and A.F. Otte¹*

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² *QuantaLab, International Iberian Nanotechnology Laboratory (INL), Braga, Portugal.*

³ *Departamento de Física Aplicada, Universidad de Alicante, Alicante, Spain*

⁴ *Theoretische Physik, Universität Würzburg, Germany*

⁵ *Theoretische Physik, Universität Regensburg, Germany*

Large-scale manipulation by means of scanning tunneling microscopy (STM) of individual vacancies in a chlorine monolayer on Cu(100) is used to construct one- and two-dimensional structures of coupled vacancies [1] in various densities and sizes, in order to investigate their electronic and spectroscopic properties.

Local scanning tunneling spectroscopy (STS) measurements reveal the emergence of quasiparticle bands, evidenced by standing Bloch waves, where the number of nodes depends on the energy and the lattice structure. This signifies that changing the lattice properties is a way of tuning the quasiparticle dispersion, which leads to different effective electron masses. The experimental data can be understood in terms of a tight-binding model combined with an additional broadening term that allows an estimation of the coupling to the underlying substrate [2].

1. Drost et al., Nature Physics **13**, 668–671 (2017)
2. Girovsky et al., SciPost Phys. **2**, 020 (2017)





Poster 4

Surface Structure Dependent Physisorption of CO₂

S.V. Auras¹, R.G. Farber², R. van Lent^{1,3}, R. Spierenburg¹, D. Bashlakov⁴, D. Killelea², L.B.F. Juurlink¹

¹ Leiden Institute of Chemistry, Leiden University,

² Loyola University Chicago,

³ Dutch Institute for Fundamental Energy Research,

⁴ ILTPE, National Academy of Sciences of Ukraine

The reactivity of commercial metal catalysts is often significantly influenced by the presence of defects, such as steps, kinks, or adatoms on the catalytic surface.[1,2] Correspondingly, the sticking of CO₂, a crucial step e.g. in methanol synthesis, is expected to be structure dependent. In this process CO₂ is generally predicted to be trapped and react as a molecularly bound species.[3] On bare metal surfaces, the adsorption of CO₂ is considered to be dependent on the work function, which decreases with the introduction of defects.[4]

Our curved crystal approach allows us to model the influence of such defects in a controlled and systematic manner. [5] In this study we employ a curved Pt crystals with (111) at the apex and gradually increasing step densities, ranging from < 1/μm to >1/nm. The two sides of the crystal feature A- and B-type steps respectively.

We examine the sticking of CO₂ by mass spectrometry and scanning tunneling microscopy. Low doses of CO₂ at varying temperatures give us a unique insight into the adsorption and structural organization of CO₂ at wide terraces as well as highly stepped surfaces.

[1] K. Honkala, A. Hellman, I. Remediakis, et al., *Science* **2005**, 307, 555.

[2] L. Vattuone, L. Savio, M. Rocca, *Surf. Sci. Rep.* **2008**, 63, 101.

[3] M. Behrens et al., *Science* **2012**, 336, 893.

[4] U. Burghaus, *Prog. Surf. Sci.* **2014**, 89, 161.

[5] A. J. Walsh et al., *J. Vac. Sci. & Technol. A* **2017**, 35, 03E102;

[5] J. Janlamool et al, *Molecules* **2014**, 19, 10845;

[5] C. Hahn et al., *J. Chem. Phys.* **2012**, 136, 114201.





Poster 5

Mimicking an Atomically-thin 'Vacuum Spacer' to Measure the Hamaker Constant between Graphene Oxide and Silica

Lianyong Chu et al., TU Delft

In nanoscience, control of the separation between surfaces, with sub-nm accuracy, is often important. For instance, when studying Van der Waals (VdW) forces¹ or creating nanogaps for molecule detection and separation²⁻⁴. At nanometre-scale, 1D or 3D spacers, such as nanotubes and nano-particles, are susceptible to deformation⁵. A 2D spacer is expected to yield a more accurately defined separation, owing to the high atom density and strength in planar direction. Herein, atomically thin 2D graphene oxide (GO) was used as nanometre-scale spacer with sub-nm accuracy, to study the VdW interaction. However, using such a physical spacer introduces additional interactions, obscuring the interactions of interest. We demonstrate how these contributions can be eliminated by effectively mimicing the use of a 'vacuum spacer'. In this way, we obtain the effective Hamaker constant between GO and silica.

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Experimental realization and characterization of an electronic Lieb lattice

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Novel two-dimensional materials with promising electronic band structures can be engineered by tailoring their lattice geometries. The honeycomb lattice of graphene, for instance, gives rise to Dirac cones in which charge carriers behave as effectively massless particles. The square-depleted geometry of a Lieb lattice also gives rise to linearly dispersing bands, similar to graphene, with a flat band intersecting the Dirac cones. Whereas optical and photonic Lieb lattices have been studied experimentally [1, 2], an electronic Lieb lattice had not been realized so far.

We report on the realization of an electronic Lieb lattice using a strategy similar to the method employed for creating artificial graphene [3]: carbon monoxide molecules on Cu(111) are positioned into an anti-lattice by lateral manipulation using an STM tip, confining the copper surface state electrons into the desired lattice. The characteristic electronic structure of the Lieb lattice, as simulated using a muffin-tin and tight-binding approach, was confirmed by scanning tunneling spectroscopy and wave-function mapping. Moreover, second-order electronic patterns were observed at higher energies [4].

The Cu(111)/CO system forms a versatile model system, which allows one to study novel lattice geometries and to tune parameters that cannot be accessed in real solid-state materials. The scanning tunnelling microscope facilitates the realization as well as the structural and electronic characterization of the lattice, paving the way for the design of truly novel two-dimensional materials.

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Poster 7

Forensic Application of Atomic Force Microscopy for Age Determination of Bloodstains

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Forensic determination of the age of a bloodstain with little temporal error can provide important information in judicial inquiry. Atomic force microscopy could provide estimations for bloodstain ages based on temporal changes in the elasticity of red blood cells (RBCs). This technology enables RBC nano-indentations with the tip of a cantilever resulting in force measurements and high resolution mapping of nanomechanical cell properties like the Young's modulus of elasticity.

This study applied atomic force microscopy to investigate the elasticity of randomly selected RBCs from the peripheral zone of 4- to 8-day-old bloodstains (controlled laboratory conditions). Special attention was paid to the condition of the silicon probes when continuously used to indent RBCs. The elasticity of 6 RBCs from a 5-day-old bloodstain appeared homogeneous over the cell with a mean Young's modulus of 1.6 ± 0.4 GPa. Data spreading was for only 6.5% caused by the choice of the RBC. Upon ageing of a bloodstain, 4 to 8 days, a significant age effect was observed in RBC elasticity (4 days: 0.8 ± 0.1 GPa; 5 days: 1.7 ± 0.9 GPa; 6 days: 2.3 ± 0.6 GPa; 7 days: 4.5 ± 0.6 GPa; 8 days: 6.0 ± 1.8 GPa; probe spring constants 25.16 - 67.48 N/m). The spreading between the data (4 bloodstains; 3 RBCs/stain) enabled a bloodstain age determination with a 24 h precision only for 6- to 7-day-old stains. In other cases a 48 h temporal accuracy was obtained. The condition of the silicon tip was regularly checked using scanning electron microscopy and an increasing bluntness noticed after 4 to 6 cell indentations. A correction factor for the increase in tip radius was therefore applied for data processing. Particularly for crime scene investigations a more robust tip is required thereby increasing both precision and accuracy of the elasticity data.



Poster 8

NaCl clusters grown on topological insulator Bi_2Te_3 investigated by scanning probe microscopy

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Surface properties and their modifications of topological insulators (TIs) hold great promise for developing high-performance electronics and applications in quantum computing. Here, we choose to deposit nanometer sized atomic clusters of sodium chloride (NaCl) on the bismuth telluride (Bi_2Te_3) TI. The growth and electronic properties of different size NaCl clusters on the Bi_2Te_3 were probed by means of scanning tunneling microscopy and spectroscopy under low temperature and ultra-high vacuum conditions. We observed that the deposition of NaCl on Bi_2Te_3 surfaces at room temperature results in the formation of NaCl clusters, where the cluster size can be controlled via the deposition time. Our findings show that the NaCl clusters alter the electronic properties of the TI surface of Bi_2Te_3 by inducing n-type doping. Combining the active and promising areas of TI materials and clusters, our study may offer a new method for local tuning of the topological properties and may pave the way to the fabrication of nanostructured TI materials.

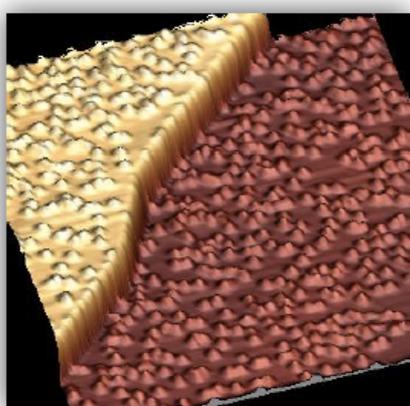


Figure 1: NaCl clusters on the surface of Bi_2Te_3 . $500 \times 500 \text{ nm}^2$ 3D STM topography image illustrating the deposited NaCl clusters that cover the surface of TI Bi_2Te_3 ($V = 1 \text{ V}$, $I = 0.05 \text{ nA}$).



November 8, 2017

Poster 9

Tuning the electronic and mechanical properties of graphene by alcohol intercalation

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In this work, we use conductive AFM and lateral force microscopy to study the electronic and mechanical properties of graphene on a mica substrate. By lowering the relative humidity and consecutively purging with alcohol, we create three distinct intercalant regions: an unaffected double water layer, 2D ice fractals and alcohol intercalated on the 2D ice fractals. The three regions show a clear difference in conductivity and friction. The graphene on the 2D ice fractals shows a high conductivity and friction; the graphene on the double water layer shows a medium conductivity and friction; and the graphene on the alcohol shows a low conductivity and friction. The change in conductivity is caused by doping due to a polarized 2D ice layer, which was validated with STM experiments by Bampoulis et al.[1] So, by changing the intercalant we are able to tune the electronic and mechanical properties of graphene.

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November 8, 2017

Poster 10

Pyridyl-functionalized triarylaminines self-assembled on Au(111)

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The engineering of molecular nanostructures on well-defined inorganic surfaces is considered to be essential for the development of future nanoelectronic devices. In order to build up nanostructures with atomic precision, ultimate control over the underlying interactions is needed.

In our work, we investigated the self-assembly of pyridyl-functionalized triarylaminines on Au(111) by scanning tunneling microscopy (STM), X-ray photoelectron spectroscopy (XPS) and low energy electron diffraction (LEED). For the deposition of submonolayer coverage onto Au(111) held at room temperature, a close-packed phase was observed to co-exist with two structurally different hexagonal nanoporous networks. The close-packed phase is stabilized by hydrogen bonding while the nanoporous networks are held together by metal-ligand interactions with native gold atoms. Thermal annealing at 180°C resulted in the exclusive formation of the Au-coordinated hexagonal nanoporous network with the larger pore size. Based on the STM and LEED data, the structural models could be precisely determined and with the help of the additional XPS data, the intermolecular as well as molecule-substrate interactions were unraveled.





Poster 11

Exploring the potential of graphene nanoribbons as molecular electronic junctions

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Recent developments in the field of on-surface chemistry have facilitated bottom-up fabrication of defect-free graphene nanoribbons (GNRs).¹ State of the art scanning tunnelling microscopy (STM) techniques have highlighted the possibility of forming a molecular junction between a metallic substrate and the STM tip^{2,3}. To counteract the strong electronic coupling to the metal surface, we developed a novel technique where we introduced intercalating layers of NaCl, allowing us to measure the electronic conductance through single ribbons as a function of bias voltage and junction length.⁴ This work is a big step towards the application of GNRs in molecular electronics.

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Poster 12

Universality of pseudogap and emergent order in lightly doped Mott insulators

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High temperature superconductivity as it manifests in the cuprates was for long time suspected to be strongly related to the copper oxide layers, and therefore specific to only this family of materials. Here, we investigate the iridate $(\text{Sr}_{1-x}\text{La}_x)_2\text{IrO}_4$, belonging to a new class of quasi-2D effective Mott insulators [1], which we have shown to exhibit phenomena strikingly similar to the cuprates [2-4]. We use spectroscopic-imaging scanning tunneling microscopy to visualize the electronic structure at different doping concentrations. We measure fully gapped spectra at low doping concentrations ($x < 4\%$) even at the precise location of dopant atoms that are visible in the topograph. This is evidence for an impurity band Mott transition of the extra carriers, and that they are more deeply trapped than in the cuprates. Only at a certain doping threshold, the gap collapses revealing a pseudo gap and complex charge arrangements.

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November 8, 2017

Poster 13

Sensing the non-collinear magnetic structure by combining magnetic exchange and spin-polarized imaging

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Non-collinear magnetic structures, as skyrmions and spin spiral for instance, have been predicted being a candidate for magnetic-based storage and spintronic applications. One key mechanism in these magnetic structures is the Dzyaloshinskii-Moriya interaction (DMI), which emerges in the absence of inversion symmetry and the appearance of strong spin-orbital coupling. The non-collinear magnetic structure also strongly depends on the symmetry of the geometric structure. For investigating non-collinear magnetic structures, spin-polarized scanning tunneling microscopy (SP-STM) has long been used to characterize the magnetism of surfaces with atomic spatial resolution. However, the SP-STM images reveal not only the magnetic information but convoluted with electronic and structural properties. By utilizing SPEX, a new combination of spin-polarized scanning tunneling microscopy (SP-STM) and magnetic exchange force microscopy (MExFM), we obtain more detailed information on both nanoscale skyrmions and a spin spiral system on mono- and bilayers of iron on an iridium surface. We show that we can resolve the skyrmion lattice in the monolayer and reveal the influence of the surface reconstruction on the magnetism in the bilayer.





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Poster 14

Assembled ferromagnetic cluster chains on an ultrathin aluminium oxide layer on Ni₃Al(001)

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Self-assembled chains of Fe clusters have been grown on an ultrathin aluminium oxide layer on Ni₃Al(001) by using atomic beam deposition. The non-wetting property of Fe on an oxidized surface leads to a formation of well-defined clusters arranged in the chains. The diameter of composed clusters can be controlled by the Fe coverage and the substrate temperature. Combining with the in-situ tip manipulation in a scanning tunnelling microscope (STM), Fe clusters on and off the chains can be removed one by one or placed in the existing chain. By this way, an isolated cluster chain is fabricated. The STM-tip manipulation does not only provide a tool to change the geometrical arrangement of the cluster chain but can also be used to modify the clusters in the chain. Fabricated chains of ferromagnetic clusters would demonstrate the effects connecting their ferromagnetic arrangement with current transport in the following in-situ four-probe-STM measurement.





Poster 15

Au(111) is not a suitable substrate to study model CoMoS hydrodesulfurization catalysts

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Using Scanning Tunneling Microscopy and X-ray Photoelectron Spectroscopy, we investigated the direct sulfidation and the oxide route to synthesize Au(111)-supported CoMoS model hydrodesulfurization (HDS) catalysts. The results demonstrate strong cobalt-substrate interactions in the presence of hydrogen sulfide. Direct sulfidation route produces sheets of cobalt sulfide with various stoichiometry that not only corrode the terraces of Au(111), but also displace the MoS₂ crystallites, especially at high cobalt loadings. The resulting surface roughness and clustering of the active phase leads to possible blocking of active sites. The oxide route on the other hand, shows differential sulfidation of the molybdenum and cobalt oxides. The cobalt sulfide phase encapsulates the still sulfiding molybdenum oxide particles and forms a 2D core-shell-like system which prevents the complete sulfidation of MoS₂. The lattice mismatch with Au(111) favors the reduction of the oxide precursors pre-sulfidation. Additionally, the strain induced by the 2D shell formation promotes the growth of molybdenum sulfide over the cobalt sulfide sheets upon extended sulfidation. These effects have not been observed in alumina industrial HDS catalysts reported so far. We recommend that by using an oxide substrate such as rutile TiO₂(110), it may be possible to obtain a model system that better represents the industrial HDS catalysts.





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Poster 16

Defect Dominated Charge Transport and Fermi Level Pinning in MoS₂/Metal Contacts

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Understanding the electronic contact between molybdenum disulfide (MoS₂) and metal electrodes is vital for the realization of future MoS₂-based electronic devices. Natural MoS₂ has the drawback of a high density of both metal and sulfur defects and impurities. We present evidence that subsurface metal-like defects with a density of $\sim 10^{11}$ cm⁻² induce negative ionization of the outermost S atom complex. We investigate with high-spatial-resolution surface characterization techniques the effect of these defects on the local conductance of MoS₂. Using metal nanocontacts (contact area < 6 nm²), we find that subsurface metal-like defects (and not S-vacancies) drastically decrease the metal/MoS₂ Schottky barrier height as compared to that in the pristine regions. The magnitude of this decrease depends on the contact metal. The decrease of the Schottky barrier height is attributed to strong Fermi level pinning at the defects. Indeed, this is demonstrated in the measured pinning factor, which is equal to ~ 0.1 at defect locations and ~ 0.3 at pristine regions. Our findings are in good agreement with the theoretically predicted values. These defects provide low-resistance conduction paths in MoS₂-based nanodevices and will play a prominent role as the device junction contact area decreases in size.





Poster 17

Scanning probe microscopy at magnetic fields up to 34 T

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Up to now, low temperature Scanning Probe Microscopy (SPM) has been limited to a magnetic field strength of 18 T, as the majority of designs have been based on superconductor magnets. For some experimental applications – for example the study of fractal spectra in graphene superlattices, the room temperature quantum Hall effect and some metamagnetic transitions – higher fields are required. Static fields of more than 30 T can be generated in dedicated high-field facilities by water-cooled, resistive Bitter magnets or hybrid resistive-superconducting magnets. However, implementing SPM in a Bitter magnet is a major challenge, due to the high level of vibrational noise produced by the turbulent cooling water, in addition to the strong space constraints resulting from the small magnet bore.

We present a novel cryogenic Scanning Tunnelling Microscope (STM) designed to operate inside a water-cooled Bitter magnet, which can reach a magnetic field of 38 T. The performance of the STM is demonstrated through Landau level tunnelling spectroscopy of graphite, at 4.2 K in magnetic fields up to 34 T.

Additionally we show the design of a highly compact Atomic Force Microscope (AFM) for operation at cryogenic temperatures in an extremely high magnetic field. We present preliminary imaging data on the frustrated spinel CdCr_2O_4 at up to 30 T.





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Poster 18

Low Energy Electron Microscopy at Near Ambient Pressures

Andreas Thissen; SPECS Surface Nano Analysis GmbH, Germany

Low-energy electron microscopy (LEEM) is a spectromicroscopy technique, which allows the study of dynamic processes at surfaces and interfaces, such as thin-film growth, surface reactions, and phase transitions. With the FELEEM P90 from SPECS, which is based on the instrument design by Rudolf Tromp from IBM [1,2], lateral and energy resolution of below 5 nm and 250 meV, respectively, can be achieved. Depending on the excitation source and the settings on the instrument a variety of different imaging modes are possible: mirror electron microscopy, low energy electron diffraction (LEED), phase contrast imaging, reflectivity contrast, dark field imaging and bright field imaging, as well as photoelectron emission microscopy and spectroscopy.

We have enhanced the technical capabilities of the FE-LEEM P90 towards studies under near ambient conditions by developing a special sample geometry. This enables the analysis of materials and devices under near ambient conditions and even in situ during operation.



Poster 19

Local atomic structure and electronic properties of graphene/Cu(111)/Al₂O₃ substrate via scanning tunneling microscopy and spectroscopy

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Graphene is a single layer of carbon atoms arranged in a hexagonal honey comb pattern. This two-dimensional honeycomb structure of carbon has drawn much attention in the past one decade due to its applicability to the most promising nanoelectronics application.¹ Indeed, atomic scale investigations of the electronic properties of graphene are playing a crucial role in understanding and tuning the exotic properties of this material for its potential device applications.² In this regard, scanning tunneling microscopy (STM) and spectroscopy (STS) are unique techniques for atomic scale investigations and have been extensively used in graphene research. In our work, we explore the local atomic and electronic properties of graphene grown on Cu(111) on sapphire substrate. We facilitate to study the atomic and electronic structure behavior of defect free regions of graphene, grain boundaries, effect of intrinsic atomic defects in graphene (carbon vacancy) and point defects in copper. Locally, irregular moiré pattern under negative bias in the absence of grain boundary has been noticed. Position dependent STS at the atomic scale has been used to correlate the atomic structure and electronic property of graphene on Cu(111)/Al₂O₃.

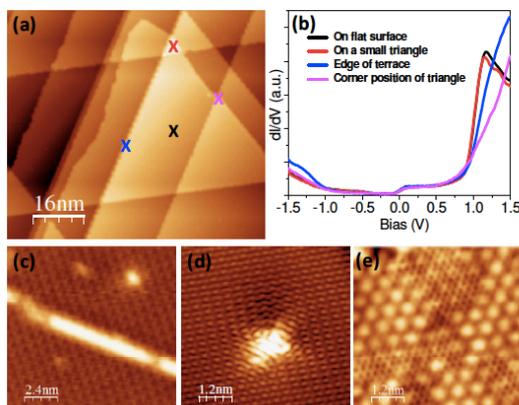


Figure 1. a) An STM image revealing the graphene on atomically flat terraces of Cu(111)/Al₂O₃ substrate. b) dI/dV spectra measured at various locations of graphene suggesting the position dependent electronic structure of graphene. STM images showing a typical grain boundary (c), intrinsic atomic defect (carbon vacancy) (d) and irregular moiré pattern (at negative bias, -1V and 100 pA) in graphene (e).

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November 8, 2017

Poster 20

Magnetic Resonance Force Microscopy: Probing Small Spin Ensembles in 3D

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Magnetic Resonance Force Microscopy (MRFM) is a scanning probe technique for locally probing spin-related material properties with a resolution of 10-100 nm. The use of magnetic resonance allows for detecting spins not only on the sample surface, but also in the bulk. We developed an MRFM with a base temperature of 10mK. As a proof of principle, we demonstrated measuring the nuclear spin-lattice relaxation time of copper at sample temperatures down to 42mK. As expected, we find Korringa-behavior. Furthermore we used our setup to determine the impurity spin density in diamond. These are important steps on the way to investigating more complicated samples with inhomogeneous electron properties, such as iron-doped palladium, oxide interfaces, topological insulators or strongly correlated electron systems.





Poster 21

CO oxidation on Pt₇ on h-BN/Rh(111) : Stability and catalytic activity

*Hamed ACHOUR et al.,
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The complexity of real catalysts in terms of their electronic and geometric structure and their interaction with the support makes fundamental studies much simpler on model catalysts. In this contribution we report on the stability and CO oxidation on mass selected Pt₇ supported on hexagonal boron nitride (h-BN) grown on a Rh(111) surface. In the first case, the idea of our experiments is to reduce the size of catalytic particles to their ultimate limit in order to make car exhaust catalysts that contain less Pt. In the second case, the inert h-BN layer is expected to minimize the effect of the support on the catalytic reaction and will also be used as an ordered array to stabilize the small Pt particles. h-BN forms a single sp² bonded atomic honeycomb layer. The misfit with the underlying Rh(111) surface leads to a strongly corrugated Moiré pattern with 2 nm diameter areas that are strongly bound and surrounded by 1 nm wide areas that are only van-der-Waals bound and 1 Å higher. The low-lying regions of the Moiré are known to be good trapping centers for molecules and clusters. Recent studies show that h-BN presents a great potential as future catalysts support[1]. The stability of the Pt nanoparticles is investigated in-situ by means of Scanning Tunneling Microscopy (STM), operated at 80 K. Catalytic reactions on the catalyst are measured in situ in a custom made UHV reactor. CO oxidation has been studied using alternating ¹³C¹⁶O and ¹⁸O₂ pulses, (at a frequency of 0.2 Hz) on the Pt/h-BN/Rh(111) catalyst as a function of temperature (300-700 K). We find that Pt clusters annealed to 700 K exhibit Smoluchowski ripening, above, intercalation of the Pt under h-BN takes place. The stability is strongly reduced under reaction conditions, a phenomenon which has also been observed for other systems such as Co/h-BN/Rh(111)[2] and Pt/TiO₂(110)[3]. The onset temperature of the CO oxidation is at 500 K and thereby higher than on titania[4]. Intercalated clusters exhibit a by 100 K lower onset temperature of the CO oxidation reaction thus improving the cold start behavior.

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