

Welcome!

As the organizers we take great pleasure in welcoming you to the 5th *International Workshop on Advanced Scanning Probe Microscopy Techniques 2014*. The workshop again will be a forum for stimulating discussions and the exchange of new ideas which will help to explore the nanoworld in more detail. We especially thank our invited speakers and all of you, who contribute to the talks, the posters and the discussions. On the following pages please find the detailed conference program.

The workshop will take place in the lecture hall of the Institute of Nanotechnology (INT) on Campus North of the Karlsruhe Institute of Technology (KIT). A map of the KIT and travel instructions how to reach the Campus North of the KIT are also included in this booklet.

We acknowledge financial support for this workshop from the KIT, the Institute of Microtechnology (IMT), the Institute of Nanotechnology (INT), and the Karlsruhe NanoMicro Facility (KNMF) as well as from the companies Agilent Technologies, Asylum Research, Bruker Nano, JPK Instruments AG, Minasales, NanoAndMore GmbH, Physical Electronics GmbH, Oxford Instruments Omicron NanoScience, Schaefer Technologie GmbH, and SPECS Surface Nano Analysis GmbH. Special thanks go to Oxford Instruments Omicron NanoScience and SPECS Surface Analysis GmbH for sponsoring the Poster Session and the Best Poster Award, respectively.

We hope that you will enjoy the workshop and have a wonderful time in Karlsruhe, and we are looking forward to welcome you at the KIT.

Hendrik Hölscher

Thomas Schimmel

Monday, February 24, 2014

- 10:00 SHUTTLE BUS from the Renaissance Hotel Karlsruhe to the Institute of Nanotechnology (INT) located at the Campus North of the KIT
- 10:30 OPENING COFFEE & PRETZELS
Chair: Hendrik Hölscher & Thomas Schimmel
- 10:50 WELCOME
Hendrik Hölscher & Thomas Schimmel, Karlsruhe Institute of Technology
- 11:00 - To be announced -
Heike Riel, IBM Rüschlikon, Switzerland
- 11:40 Spin spirals and magnetic skyrmions studied with spin-polarized STM
Kirsten von Bergmann, University of Hamburg, Germany
- 12:20 LUNCH
Chair: Regina Hoffmann
- 13:30 Spatially Resolved Surface Photovoltage Spectroscopy
Sascha Sadewasser, International Iberian Nanotechnology Laboratory, Spain
- 14:10 Potential Distributions Inside Perovskite Solar Cells
Rüdiger Berger, Max Planck Institute for Polymer Research, Germany
- 14:30 Understanding STM/AFM Experiments on Single-Atom Junctions from First-principles
Stefan Heinze, CAU Kiel, Germany
- 15:10 Decisive Influence of Substitution Positions in Molecular Self-Assembly
Julia L. Neff, Johannes Gutenberg-University Mainz, Germany.
- 15:30 POSTER SESSION with COFFEE, SNACKS & REFRESHMENTS
Chair: Sascha Sadewasser
- 17:30 Structural Friction Anisotropy on the Nanometer Scale
Gregor Fessler, University of Basel, Switzerland
- 17:45 Preferential sliding directions on graphite
Balakrishna SG, Leibniz Institute for New Materials, Germany

- 18:00 SubSurface- AFM: Looking Below a Surface
Marcel J. Rost, Kamerlingh Onnes Laboratory, Leiden University, The Netherlands
- 18:15 Visualizing the Subsurface of Soft Matter:
Simultaneous Topographical Imaging, Depth Modulation, and Compositional Mapping with Triple Frequency Atomic Force Microscopy
Daniel Ebeling, Justus Liebig University Giessen, Germany
- 18:30 SHUTTLE BUS from the KIT Campus North to Campus South
- 19:00 CONFERENCE DINNER at the Gastdozentenhaus "Heinrich Hertz"
located at the Campus South of KIT
- 22:00 Short guided tour by Thomas Schimmel through the city of Karlsruhe (ends at the Renaissance Hotel Karlsruhe)

Tuesday, February 25, 2014

- 8:45 SHUTTLE BUS from the Renaissance Hotel Karlsruhe to the KIT Campus North
- 9:00 OPENING COFFEE & PRETZELS

Chair: Kirsten von Bergmann

- 9:30 Towards Atomic Force Microscopy with Chemical Contrast
Hideki Kawakatsu, The University of Tokyo, Japan
- 10:10 Engineering carbon-rich networks doped with heteroatoms
Sabine Maier, University of Erlangen-Nürnberg, Germany
- 10:30 Intermodulation AFM with arbitrary Frequency Combs
David Haviland, Nanostructure Physics, KTH, Stockholm,
- 10:50 COFFEE BREAK
- Chair: Stefan Heinze*
- 11:20 Heat dissipation and thermopower in atomic-scale junctions
Fabian Pauly, University of Konstanz, Germany
- 12:00 A hybrid nanoscale magnetometer
Arne Buchter, University of Basel, Switzerland

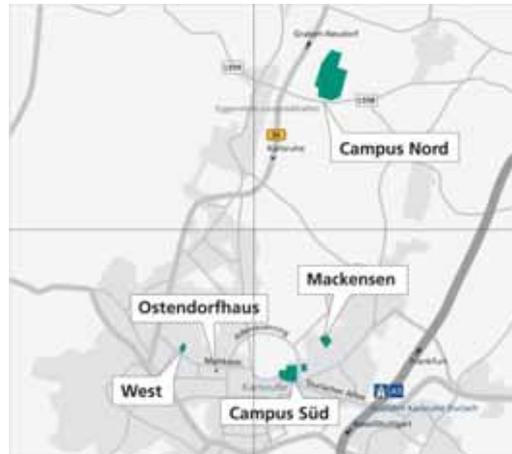
- 12:20 Oxidation Scanning Probe Lithography for the fabrication of silicon nanowire devices
Yu Kyoung Ryu, Instituto de Ciencia de Materiales de Madrid, CSIC, Spain
- 12:40 LUNCH

Chair: Fabian Pauly
- 13:40 Studies of Contact Mechanics Making Use of Quartz Crystal Microbalance
Diethelm Johannsmann, TU Clausthal
- 14:20 Charge-density-wave-induced non-contact friction
Markus Samadashvili, University of Basel, Switzerland
- 14:40 Analyzing Interfacial Friction of Nanoparticles: Contact Ageing vs. Structural Lubricity
Dirk Dietzel, Justus Liebig University Giessen, Germany
- 15:00 Mapping of Elasticity and Damping in an $\alpha + \beta$ Titanium Alloy using Atomic Force Acoustic Microscopy
Walter Arnold, Saarland University, Germany
- 15:20 Imaging of Platelet Activation and Cytoskeleton Reorganization by Scanning Ion Conductance Microscopy
Johannes Rheinländer, Eberhard Karls University Tübingen, Germany
- 15:40 BEST POSTER AWARD & CLOSING REMARKS
- 16:00 SHUTTLE BUS to Karlsruhe Hauptbahnhof (main station)

How to Reach the Karlsruhe Institute of Technology

The Karlsruhe Institute of Technology is distributed over several locations. While the Campus North is situated in the administrative district of Karlsruhe near Eggenstein-Leopoldshafen, the Campus South is about 10 km away in the heart of the city of Karlsruhe.

The ASMT 2014 takes place at the Campus North (Campus Nord) in lecture hall of the Institute of Nanotechnology (INT). You will be asked for your identity card at the front gate (Anmeldung) of Campus North of the KIT.



How to Reach the Campus North by Car

The Campus North is located in Eggenstein-Leopoldshafen (about 12 km north of Karlsruhe). The address for your GPS is: Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen

Coming from North: Frankfurt and Heidelberg (A5)

- Leave the motorway at the exit of Bruchsal and go in the direction of Karlsdorf-Neuthard / Germersheim / B35.
- Stay on this road for some 4 km, and then turn off on the B36.
- Leave the B36 after some 12 kilometers, at the exit of Eggenstein-Leopoldshafen / Forschungszentrum
- After about 1 km, you reach the entrance road to the Campus North on your left.

Coming from Northwest: Landau (A65)

- Leave the motorway at the Knielingen exit and go straight ahead on the road (Rheinbrückenstraße) for another 3 km as far as to the Neureuter Straße junction (after having passed the premises of Siemens AG).
- Then turn left in the direction of Mannheim (B 36). Go straight ahead at all following junctions.
- After some 10 km, turn right at the exit of Bruchsal / Stutensee / KIT Campus Nord.
- Go ahead for another 1 km until you reach the entrance road to the Campus North on your left.

Coming from East: Munich and Stuttgart (A8)

- At the Karlsruhe motorway triangle, turn off in the direction of Karlsruhe / Frankfurt (A5).
- Leave the motorway at the Karlsruhe-Mitte exit and go in the direction of Landau/Pfalz (B10).
- Stay on this road another 9 km and turn off in the direction of Mannheim (B36).
- Drive straight ahead on the B36 at all junctions.
- Leave the B36 after some 11 km at the exit of Bruchsal / Stutensee / Forschungszentrum.
- After about 1 km, you reach the entrance road to the Campus North on your left.

Coming from South: Basel and Freiburg (A5)

- Leave the motorway at the exit No 48 Karlsruhe Süd and go in the direction of Karlsruhe / Landau-Pfalz (B10).
- Stay on this road another 4 km and turn off in the direction of Mannheim (B36).
- Drive straight ahead on the B 36 at all junctions.
- Leave the B 36 after some 11 km at the exit of Bruchsal / Stutensee / KIT Campus Nord.
- After about 1 km, you reach the entrance road to the Campus North on your left.

How to Reach the Campus North by Public Transport

The Campus North is located in Eggenstein-Leopoldshafen. There are various ways to get there by bus or tram from Karlsruhe city or Central Train Station. The exact times of departure are available on the website of the Karlsruher Verkehrsverbund (www.kvv.de). Information about travel data of the German Railway is provided by Deutsche Bahn (www.bahn.de).

Please aware that the tram lines labeled with and without the letter "S" in front are different lines, i.e., the tram line **1** and **S1** go to different directions!

Tram lines S 1 / S 11 from Karlsruhe Hauptbahnhof (Central Station) to the destinations of Leopoldshafen/Hochstetten

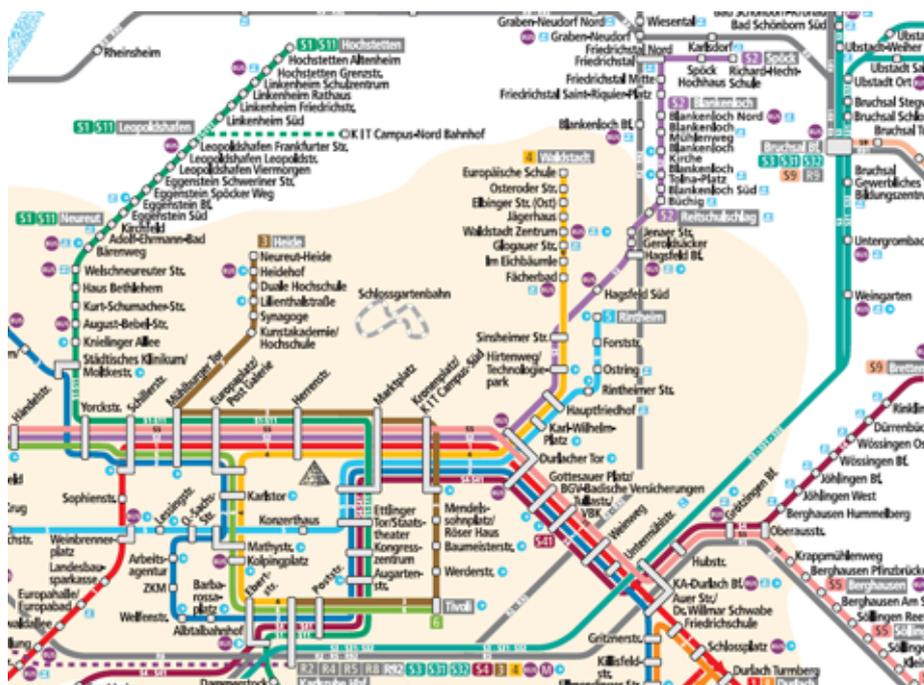
- Trams operate in 20-minute intervals; traveling time is approx. 40 minutes.
- Go by tram to stop "Leopoldstraße" at Leopoldshafen, change to bus No. 195 as far as "KIT Campus Nord Südtor" (southern entrance gate to the Campus North).

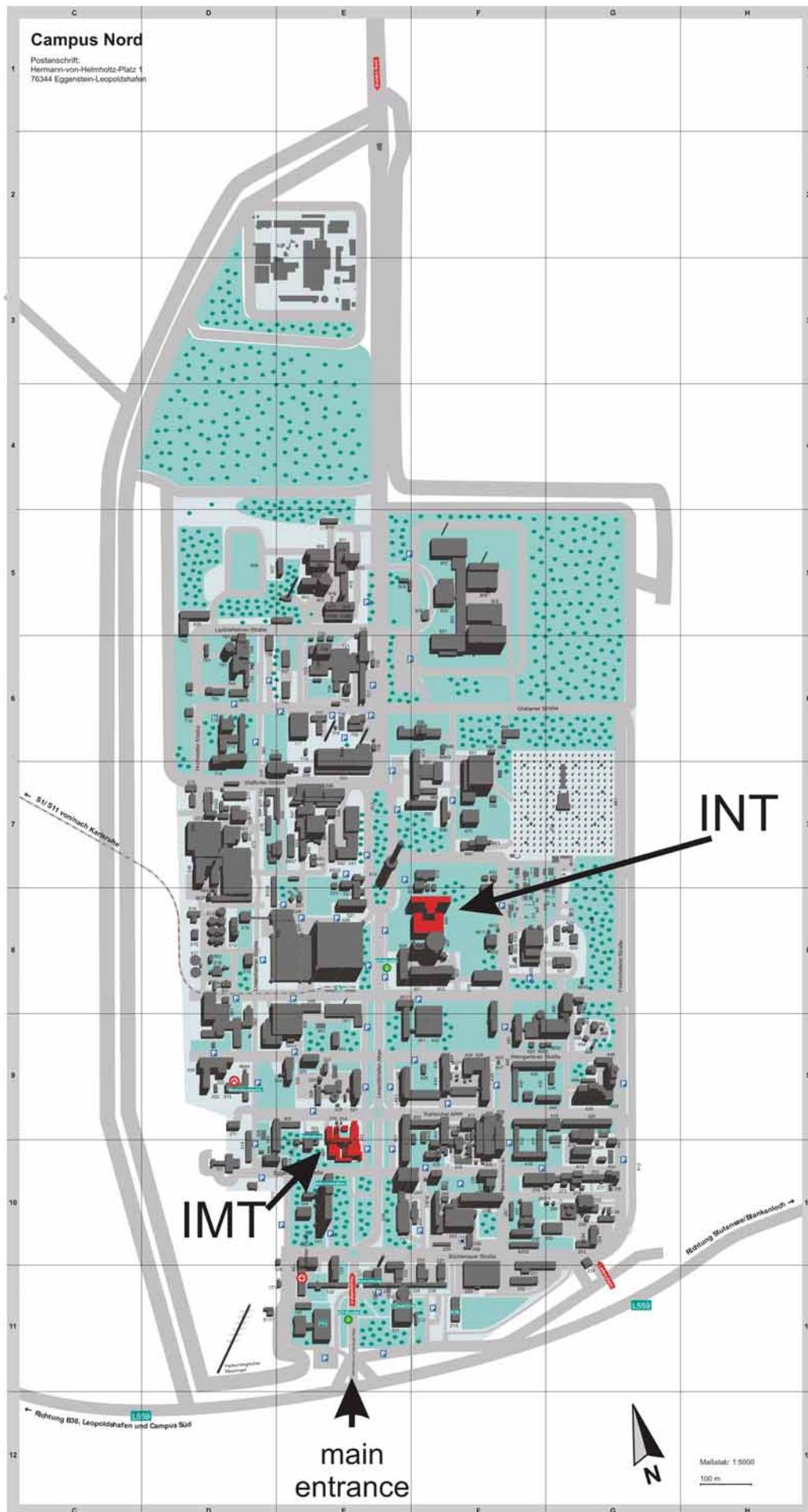
Regional train from Karlsruhe Hauptbahnhof (Central Station) to the destinations of Blankenloch Station/Mannheim.

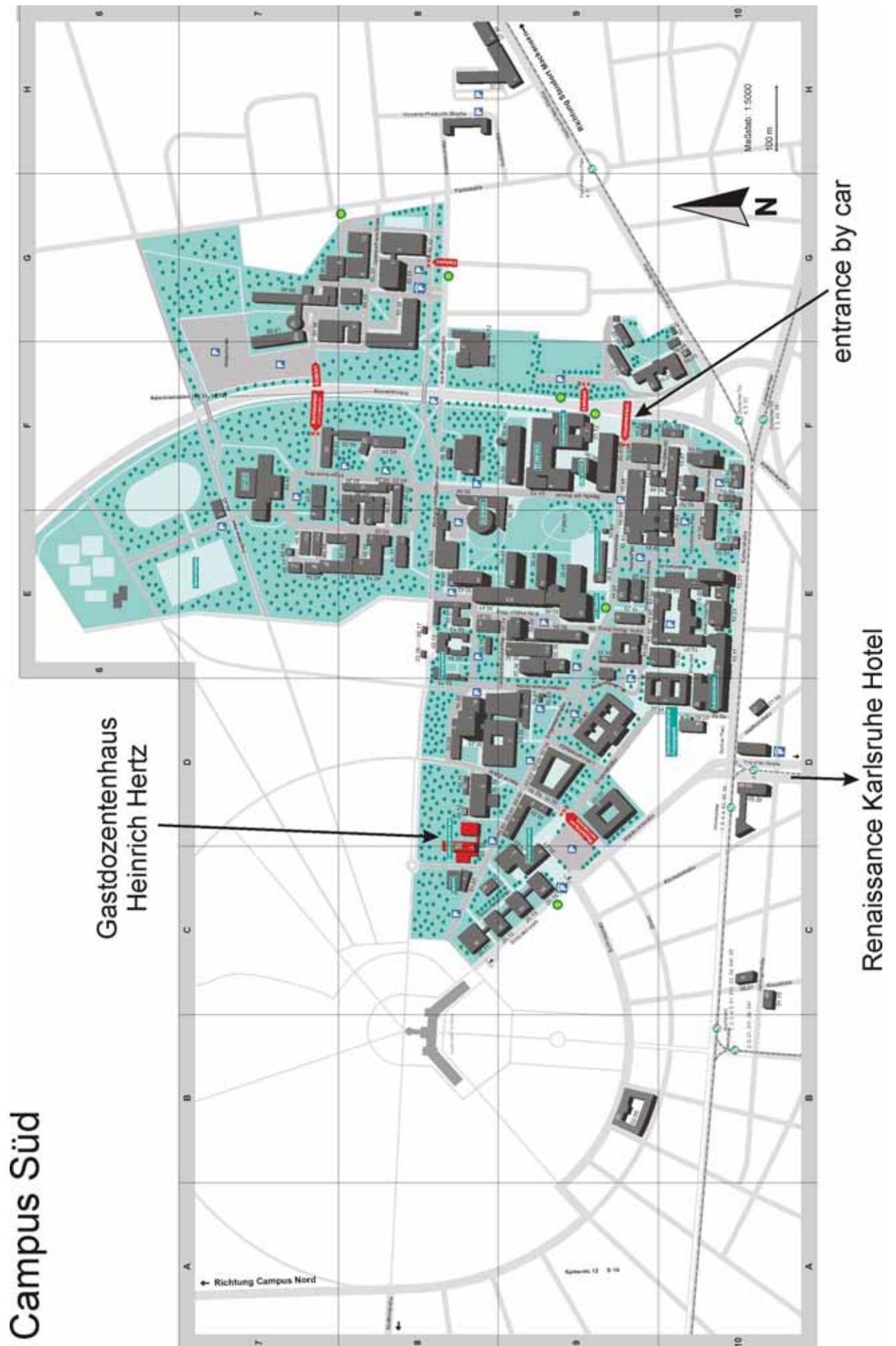
- Trains operate in 60-minute intervals; travelling time is 25 minutes.
- At Blankenloch Station, change to bus No. 195 as far as "KIT Campus Nord"

Tram line S 2 from the Karlsruhe Central Business District (Kaiserstrasse) via Durlacher Tor in the direction of Blankenloch Nord.

- Trams operate in 20-minute intervals; traveling time is 36 minutes.
- At the "Blankenloch Nord" stop, change to bus No. 195 as far as "KIT Campus Nord"







Campus Süd

Spin spirals and magnetic skyrmions studied with spin-polarized STM

Kirsten von Bergmann

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Magnetism in low-dimensions is a versatile topic and broken inversion symmetry due to the presence of a surface can induce the formation of non-collinear magnetic states. In such systems the spin rotates from one atom to the next resulting for instance in spin spirals with nanometer sized magnetic periods [1-3]. In the case of the monolayer Fe on Ir(111) we find a lattice of skyrmions on the atomic scale [4,5]. By covering this system with one atomic layer of Pd we obtain an ultrathin film system in which individual skyrmions can be written and deleted in a controlled fashion with local spin-polarized currents from a scanning tunneling microscope [6]. An external magnetic field is used to tune the energy landscape, and the temperature is adjusted to prevent thermally activated switching between topologically distinct states. Switching rate and direction can then be controlled by the parameters used for current injection. The creation and annihilation of individual magnetic skyrmions demonstrates their potential in future information-storage concepts.

References

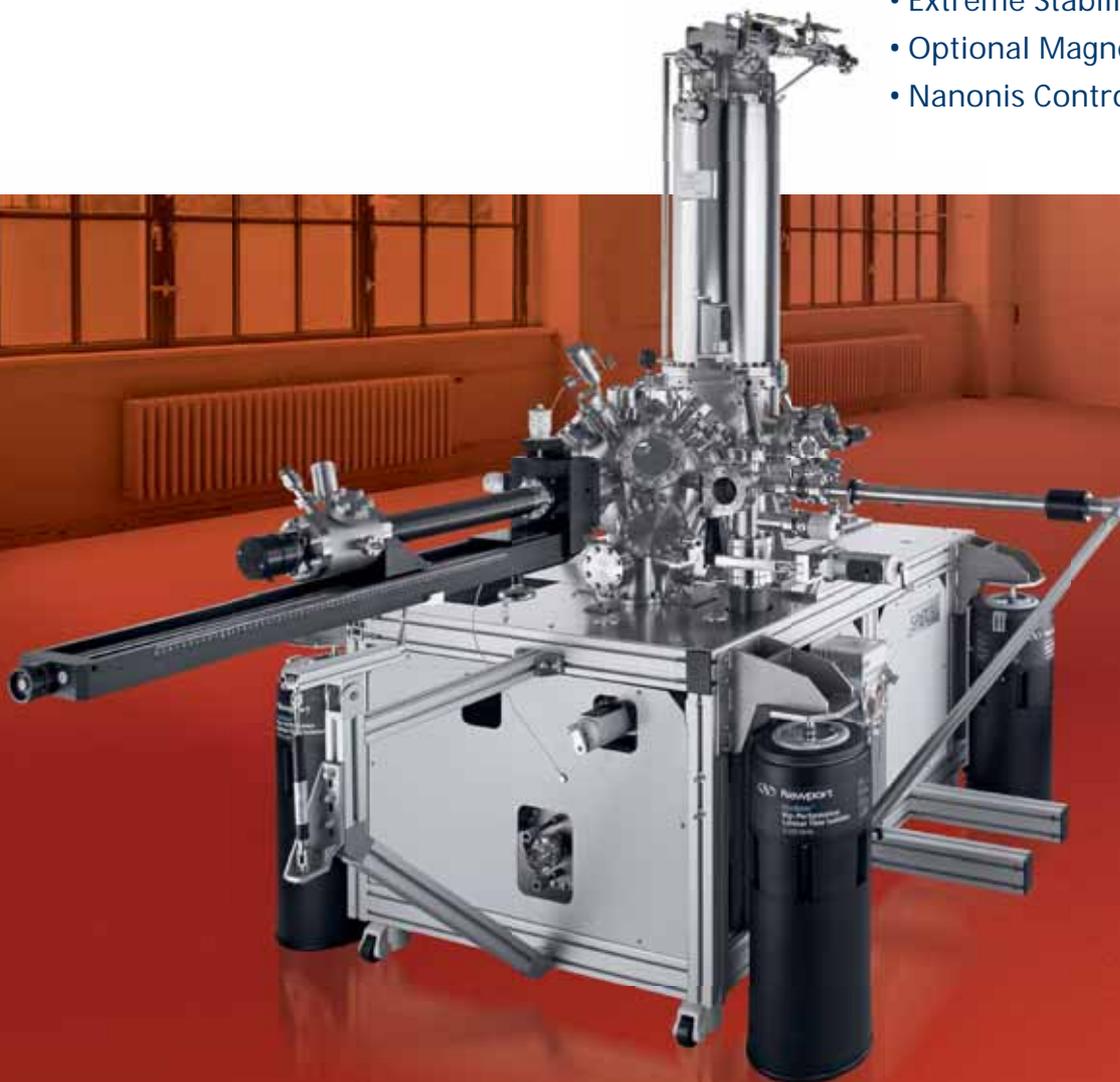
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- [3] M. Menzel et al., *Phys. Rev. Lett.* **108**, 197204 (2012).
- [4] K. von Bergmann et al., *Phys. Rev. Lett.* **96**, 167203 (2006).
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- [6] N. Romming et al., *Science* **341**, 636 (2013).

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Spatially resolved surface photovoltage spectroscopy

Sascha Sadewasser

International Iberian Nanotechnology Laboratory, Av. Mestre José Veiga s/n, 4715-330 Braga, Portugal

Kelvin probe force microscopy (KPFM) is used widely to measure surface potentials and work function of semiconductor surfaces, molecules, electronic devices, etc. By combination of KPFM with sample illumination the surface photovoltage (SPV) can be obtained, which is the change in surface potential between the dark and the illuminated state.

We present here the application of the SPV technique in an ultrahigh vacuum KPFM for the investigation of Cu(In,Ga)Se₂ thin films. Thin (~2 μm thick) Cu(In,Ga)Se₂ layers are used in polycrystalline thin film solar cells and represent the materials class currently reaching the highest power conversion efficiencies, even surpassing multicrystalline silicon solar cells. With typical grain sizes on the order of 1 μm and the resulting high density of grain boundaries, these high efficiency values are surprising. KPFM studies show potential variations at grain boundaries, which for some grain boundaries can be partially reduced upon illumination. We conclude from our experiments to a defect-induced band bending at grain boundaries, which can be reduced by light-induced charge-carrier excitation.

By using monochromatic light with variable wavelength, we investigated the SPV of Cu-Phthalocyanine (CuPc) molecules on a Si(100) surface. We observe a small SPV of a few mV for CuPc islands for photon energies above the HOMO-LUMO distance of CuPc, whereas for lower photon energies no SPV is observed. On the Si surface we do not observe any SPV over the whole wavelength range studied. We demonstrate that this technique is suitable to obtain "band gap" information on a length scale of below 100 nm.

Potential Distributions inside Perovskite Solar Cells

Victor Bergmann¹, F. Javier Ramos³, M. K. Nazeeruddin², Michael Grätzel², Stefan A.L. Weber¹,
Shahzada Ahmad³, Rüdiger Berger¹

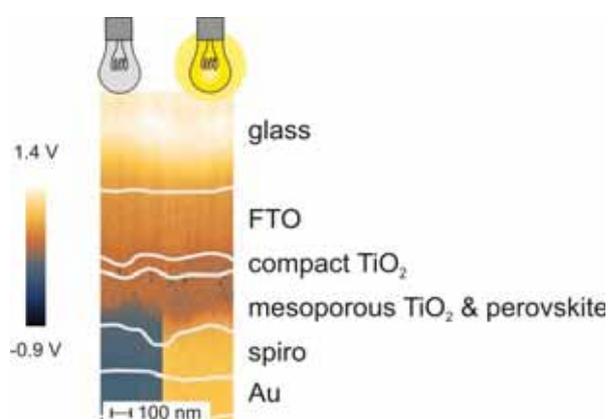
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³ Abengoa Research, C/Energía Solar nº 1, Campus Palmas Altas-41014, Seville, Spain

Recently, solar cells based on perovskite light absorbing materials reached power conversion efficiencies >15%¹. The basic component in such solid state sensitized solar cells is the mesoporous TiO₂ which contains the light absorbing perovskite. Typically the perovskite is deposited onto the mesoporous TiO₂ by spin coating the solution. The loading of perovskite and the exact distribution inside the mesoporous TiO₂ plays a major role for the generation of charges and this influence the efficiency of the solar cell. However there is not much information about the charge generation processes inside solid state sensitized solar cells. Thus the aim of our study is to apply scanning force microscopy (SFM) methods to measure the electrical potentials across the internal interfaces of these solid state solar cells.

After roughly cleaving the device, a focused ion beam (FIB) was used to fine polish the cross section in order to get a smooth topography. This preparation procedure kept the functionality of the cell intact². Directly after the preparation we used frequency modulation Kelvin Probe Force Microscopy



to map the potential distribution in dark (left) and under illumination with white light (right side of Figure). This measurement was performed at open circuit condition where the FTO was grounded and the Au electrode was kept floating. The measurement in dark reflected the difference in work functions of the used materials. Upon illumination the measured surface potential increased inside the mesoporous TiO₂ layer and reached the value of

the open circuit potential at the Au electrode. Interestingly, part of the mesoporous TiO₂ layer had a constant surface potential and did not show a response to illumination. Thus this part of the mesoporous TiO₂ layer appears non-active. This indicates that the perovskite could not penetrate throughout the entire mesoporous TiO₂ layer. Nevertheless this particular solid state solar cell had a power conversion efficiency of 13 %. In conclusion, our measurements showed that SFM can reveal potential distributions inside a working device with nanometer resolution. Thus this will allow us to tailor fabrication processes and improvement in light harvesting abilities in these materials.

References

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Understanding STM/AFM experiments on single-atom junctions from first-principles

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Today, scanning probe microscopy techniques allow creating artificial nanostructures at surfaces atom-by-atom and to locally probe their structural, electronic, magnetic, and transport properties at the atomic level. In order to understand such experiments it is often indispensable to use a first-principles approach based on density functional theory. It is particularly intriguing to explore physical properties at the single atom limit. Here, I will show that it is possible to image the spin direction of single magnetic atoms on surfaces by spin-polarized scanning tunneling microscopy [1] and how the spin-valve effect in single-atom junctions can be explained [2]. We found that in such experiments the interaction between tip and adsorbed atom can become essential and that one can relate the distance-dependent tunneling magnetoresistance to the structural relaxations in the junction [3]. A direct quantitative measurement of the magnetic exchange forces in such junctions is possible using atomic force microscopy with magnetic tips which can be understood based on first-principles calculations [4]. Surprisingly, it is also feasible to detect the spin-quantization axis of single atoms using non-magnetic STM tips [5] due to spin-orbit coupling. This effect – the so-called tunneling anisotropic magnetoresistance – can be implemented into a simple model of STM which allows fast simulation of spin-polarized and non-spin-polarized STM images [6].

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Decisive Influence of Substitution Positions in Molecular Self-Assembly

Julia L. Neff¹, Markus Kittelmann¹, Wojciech Greń², Xavier Bouju², André Gourdon², Ralf Bechstein¹, Angelika Kühnle¹

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Molecular self-assembly provides a versatile tool for creating functional molecular structures on surfaces. A rational design of molecular structure formation requires not only an in-depth understanding of the subtle balance between intermolecular and molecule-surface interactions, but might also involve considering chemical changes of the molecules, such as deprotonation.

Here, we present a systematic investigation of a comparatively simple class of molecules, namely dihydroxybenzoic acids, which, nevertheless, enable the creation of a rich variety of structures when deposited onto calcite(10.4) held at room temperature. Depending on the position of the two hydroxyl groups at the benzene core, six different isomers are possible. Interestingly, out of these six molecules, only two form long-term stable, ordered structures upon sub-monolayer deposition on calcite(10.4). Based on non-contact atomic force microscopy measurements and density-functional theory calculations, our study demonstrates the decisive impact of the position of the hydroxyl groups on the resulting structure formation. The position of the functional groups steers the molecular self-assembly of dihydroxybenzoic acids in three distinct ways, namely by (a) affecting the stability of the molecule itself in terms of deprotonation probability, (b) influencing the intermolecular interaction as already indicated by greatly different bulk structures and (c) altering the molecule-substrate matching. Taking the two long-term stable structures as a reference, we discuss the changes that arise when varying the position of the functional group in the view of these three main aspects. Our results, thus, shed light on the impact of rather small changes in the molecular structure on the structural variety in molecular self-assembly on surfaces.

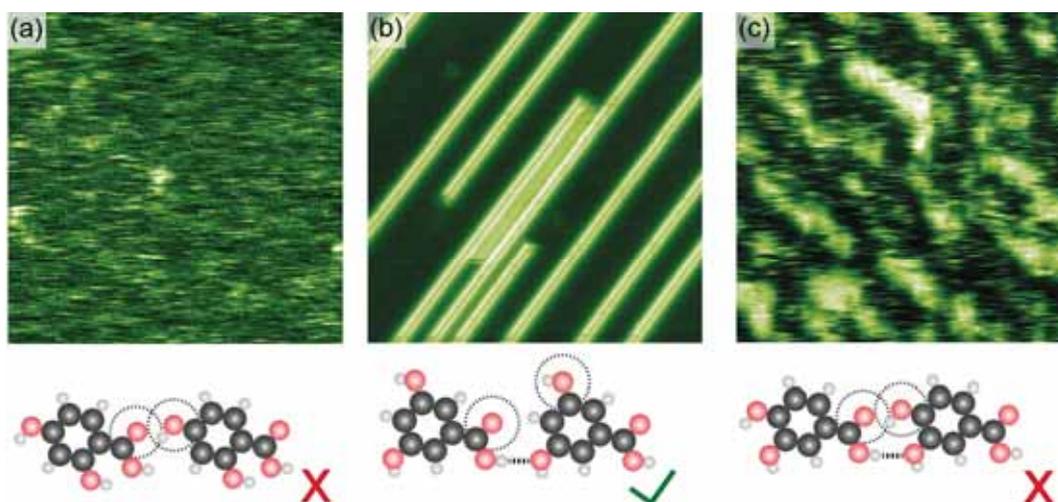


Figure 1: Molecular self-assembly on calcite studied by NC-AFM: (a) 2,4-Dihydroxybenzoic acid; (b) 3,5-Dihydroxybenzoic acid; (c) 3,4-Dihydroxybenzoic acid.

Scanning Probe Microscopy

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Structural Friction Anisotropy on the Nanometer Scale

Gregor Fessler¹, Ali Sadeghi¹, Thilo Glatzel¹, Stefan Goedecker¹, Ernst Meyer¹

¹*University of Basel, Klingelbergstrasse 82, 4056 Basel, Switzerland*

NaCl was investigated by means of friction force microscopy in ultra-high vacuum and at room temperature. A homemade atomic force microscope was used allowing for in situ sample rotation. Therefore, it is possible to measure friction along arbitrary orientations of the NaCl crystal suggesting a decrease of friction towards the [110] orientation. Another benefit of the in situ sample rotation is the ability to precisely tune the scan direction to the desired orientation. With a good alignment of scan direction to the [100] crystal orientation, the tip moves over the same crystallographic positions over the whole scanned line of some nanometers and many unit cells. Different new aspects of friction can be found in this alignment which cannot be reproduced by Prandtl-Tomlinson simulations using the standard potential. Instead a very good agreement is achieved when using an ab initio potential calculated by means of the density functional theory.

ABSTRACT

Preferential sliding directions on graphite

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2 Department of Physics, Stockholm University, SE-106 91 Stockholm, Sweden

Frictional forces are ubiquitous in nature. The invention of Atomic Force Microscopy in 1986 has opened up new possibilities of understanding the microscopic origins of frictional forces. The friction of surfaces moving relative to each other can be related to atomic interaction between the surfaces in contact.

The directional dependence of kinetic friction has been a topic of both research and engineering interest. A directional dependence of friction, also known as friction anisotropy, has been previously observed on larger scales.

Graphene is a novel two-dimensional material, which has attracted scientists and engineers in recent years due to its superior electronic and mechanical properties and extremely high thermal conductivity. It is the building block of graphite, a well-known macroscopic solid lubricant.

In our work we have investigated the nanometer-scale directional dependence of friction on graphite and graphene in a wear-less regime. The Cartesian components of friction forces for different pulling directions at different scales from $(100\text{nm})^2$ down to $(5\text{nm})^2$ were measured and the corresponding friction hodographs plotted. For a surface with isotropic friction response, the hodograph is a circle of equidistant points. We have observed strong friction anisotropy on graphitic surfaces, where the points on the friction hodograph are grouped into six clusters forming a hexagonal pattern evidencing six preferred sliding directions. Our results are interpreted by simulations based on Prandtl-Tomlinson model.

SubSurface-AFM: looking below a surface

Marcel J. Rost

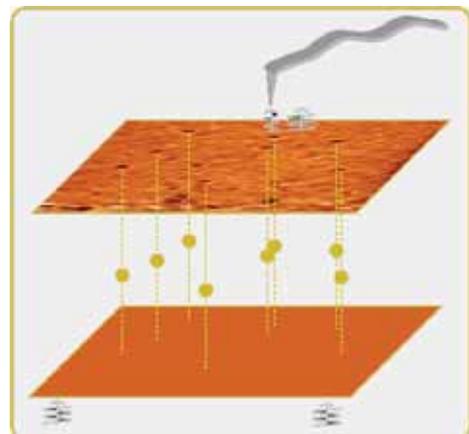
*Kamerlingh Onnes Laboratory, Leiden University
P.O. Box 9504, 2300 RA Leiden, The Netherlands*

Imaging subsurface structures with nanometer resolution has been a long-standing desire in science and industry in particular and microscopy in general. To obtain subsurface information one usually applies ultrasound, like e.g. in echocardiography. The implementation of ultrasound in an Atomic Force Microscope (AFM) gives access to additional information, which enables, under certain conditions, the imaging of subsurface structures with nanometer resolution. The most promising candidate for imaging *deeply* buried objects or structures with *nanometer resolution* is based on a special excitation scheme, which makes use of two ultrasound excitations (one through the sample and one through the cantilever). This technique is called Heterodyne Force Microscopy (HFM).

Despite some reported subsurface observations that clearly demonstrate the power of this technique, a decent quantitative understanding of the physical contrast mechanism was (until now) still missing.

This talk focuses on the poorly understood elements in Heterodyne Force Microscopy. We studied the ultrasound propagation in the sample [1], the dynamics of an ultrasonically excited cantilever near a sample that is also vibrating at a slightly different frequency [2, 3], and the generation of the heterodyne signal [4]. This recent insight in the basic working principles of HFM [1-4] enabled us to perform a quantitative analysis of our measurements on a well-characterized sample, which contained 20nm large gold nanoparticles buried more than 100nm deep underneath a soft polymer matrix. We demonstrate not only the subsurface imaging capability, but we also determine the physical contrast mechanism. Totally unexpected, the contrast is neither related to ultrasonic Rayleigh scattering nor elasticity variations in the sample, but to the *rattling* motion (and the involved friction) of *shaking nanoparticles* [5].

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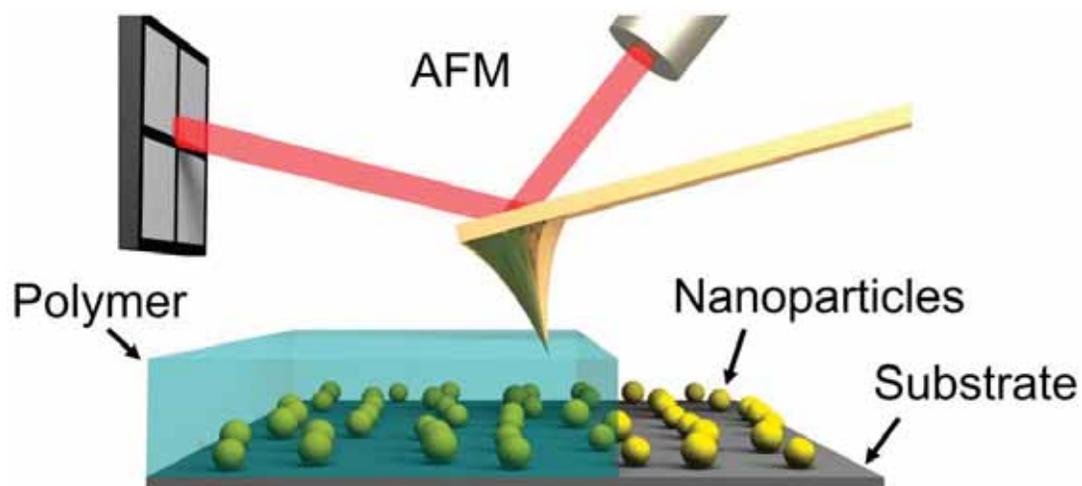
Visualizing the Subsurface of Soft Matter: Simultaneous Topographical Imaging, Depth Modulation, and Compositional Mapping with Triple Frequency Atomic Force Microscopy

Daniel Ebeling^{1,2}, Babak Eslami², and Santiago D. Solares²

¹ Institute of Applied Physics, Justus Liebig University Giessen, Giessen, Germany

² Department of Mechanical Engineering, University of Maryland, College Park, Maryland, United States

Characterization of subsurface morphology and mechanical properties with nanoscale resolution and depth control is of significant interest in soft matter fields like biology or polymer science, where buried structural and compositional features can be important. However, controllably “feeling” the subsurface is a challenging task for which the available imaging tools are relatively limited. In this presentation we describe a trimodal atomic force microscopy (AFM) imaging scheme, whereby three eigenmodes of the microcantilever probe are used as separate control “knobs” to simultaneously measure the topography, modulate sample indentation by the tip during tip-sample impact, and map compositional contrast, respectively [1]. We illustrate this multifrequency imaging approach through computational simulation and experiments conducted on ultrathin polymer films with embedded glass nanoparticles in ambient air. By actively increasing the tip-sample indentation using a higher eigenmode of the cantilever, we are able to gradually and controllably reveal glass nanoparticles which are buried tens of nanometers deep under the surface, while still being able to refocus on the surface.

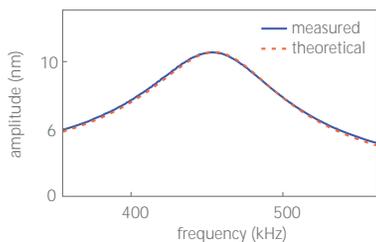


[1] D. Ebeling, B. Eslami, S.D. Solares, *ACS Nano* 7, 10387 (2013)

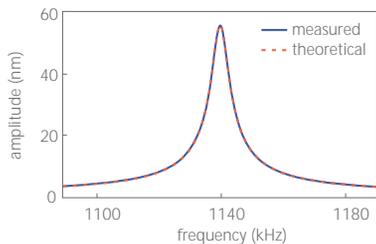
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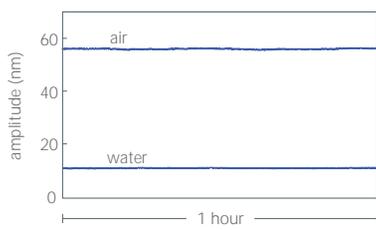
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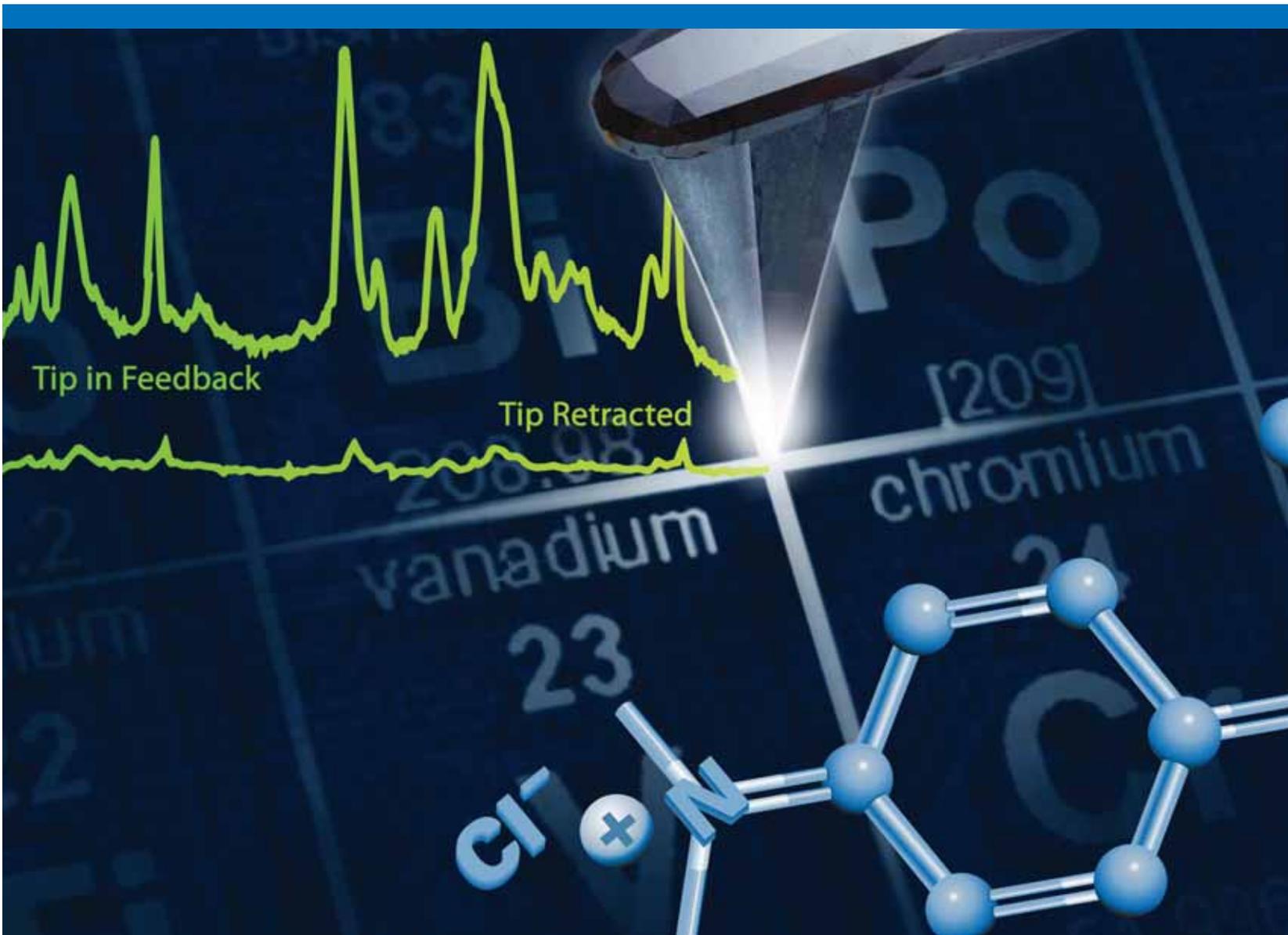
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Towards atomic force microscopy with chemical contrast

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It is of great importance to implement microscopy with chemical contrast at the atomic level. Scanning probe methods have the possibility of offering a versatile, non-destructive imaging method with such capability since the short range interaction used for tip to sample distance regulation includes important information on the chemical species of the atom under the tip. We have introduced a new control scheme in atomic force microscopy where tip to sample distance is regulated to give the minimum self-excitation frequency of the AFM cantilever during image acquisition. Since the local minima in the frequency shift curve reflects the minimal force or potential, mapping of the minimum frequency shift, with different colours assigned to different frequency shift, is expected to give a "colour image" with chemical contrast. Imaging of Si and solder were performed successfully with the proposed method. The images showed angstrom sized features with discreet population of different minimum frequency of oscillation or "colours". The method also worked with the torsional mode of the cantilever. The torsional frequency shift curve, though not confirmed on all samples, showed a w like profile, and by maintaining the working point at the local minima further away from the sample surface, atomic features could be resolved.

The chemical contrast could be improved by selecting operating conditions such as the amplitude of drive of the cantilever, dither amplitude and frequency. We will also report on simulation results and latest results on imaging of quasi-crystals and metastable surfaces.

Engineering carbon-rich networks doped with heteroatoms

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From recent studies it became apparent that organic molecules on metal surfaces are ideal precursors for forming new carbon allotropes in a bottom-up approach using on-surface synthesis, e.g. for constructing graphene nanoribbons [1] or π -conjugated networks [2,3]. Here, we demonstrate a low temperature scanning probe microscopy study, showing the formation of novel carbon-rich networks doped with heteroatoms using functionalized triarylamine molecules as precursors on metal and insulating surfaces.

On Au(111) we achieved to form extended hydrogen-bonded networks using triarylamine derivatives with carboxylic and diaminotriazine as end-groups. We found that the hydrogen-bonding triarylamine moieties also wet a KBr(001) surface by forming well-ordered films. Halogen atoms as end-groups lead to covalently bound networks and macrocycles on metals. In contrary on KBr, halogen end-groups support the growth of π - π stacked linear structures with the molecules standing nearly upright on the surface. In conclusion, we can direct the growth of triarylamine building blocks from linear structures to flat-lying networks by carefully balancing the molecule-surface interaction and molecule-molecule interaction owing to different end-groups.

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Intermodulation AFM with arbitrary Frequency Combs

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Dynamic AFM methods are typically sorted in to one of two basic categories: Amplitude Modulated (AM-AFM) or Frequency Modulated (FM-AFM). A third basic category called Intermodulation AFM (ImAFM) encompasses aspects of both AM-AFM and FM-AFM. ImAFM uses a multi-frequency drive to *actively* modulate the cantilever motion, and force is reconstructed from a record of how the tip-surface interaction changes this active modulation. We describe ImAFM with coherently synthesized frequency combs, or drive waveforms that periodically modulate either the amplitude or the frequency, or both.

As single frequency methods, both AM-AFM and FM-AFM are limited to acquiring two pieces of information: the amplitude and phase of the response at the drive frequency, in a given measurement bandwidth, B . Much more information can be gained by collecting response at multiple frequencies, separated from one another by B . That is to say, we probe the response with a frequency comb, or waveform with period $T=1/B$. Even if the system under test is nonlinear, periodicity of the response at the long time scale T ensures that there exists a mapping from any input frequency comb to the output frequency comb. In multi-frequency AFM we reconstruct this mapping (the nonlinear tip-surface force) for a given input comb from the measured output comb. The challenge is to preform this reconstruction of force from a measurement of cantilever motion, where the later is limited to a frequency band near a high quality resonance.

We have devised an intermodulation lockin analyzer capable of driving the AFM cantilever with an arbitrary frequency comb of up to 24 discrete tones and recording both amplitude and phase of the response at the same 24 tones. We describe the use of this instrument to create different of drive modulations, and we discuss how these modulations effect the reconstruction of tip-surface forces. We find that a simple drive comb consisting of only two tones centered at resonance appears to be optimal for reconstruction of surface typical surface forces encountered in AFM.

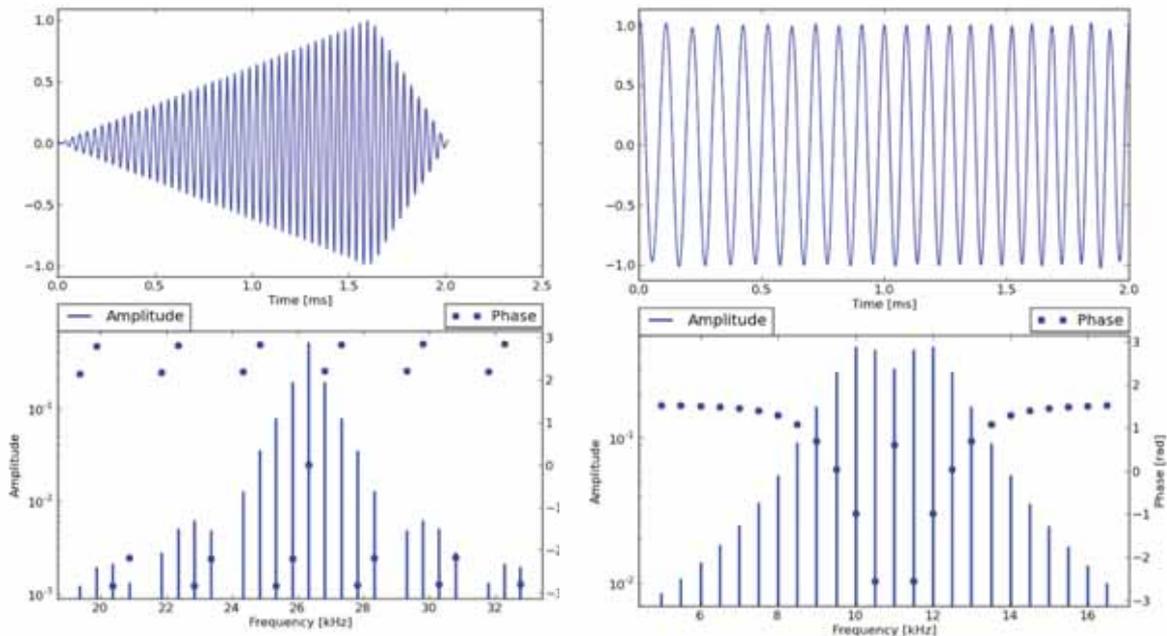


Figure 1: Time and frequency domain representations of drive signals used to probe nonlinear response of an AFM cantilever interacting with a surface. Left: Skewed linear ramp of amplitude with constant frequency. Right: Chirp, or linear ramp in frequency (quadratic phase ramp) with constant amplitude.

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Heat dissipation and thermopower in atomic-scale junctions

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Single-atom and single-molecule junctions represent the ultimate limit to the miniaturization of electrical circuits. They are also ideal platforms for testing quantum transport theories that are required to describe charge and energy transfer in novel functional nanometer-scale devices. Recent work has successfully probed electric and thermoelectric phenomena in atomic-scale junctions. However, heat dissipation and transport in atomic-scale devices remain poorly characterized owing to experimental challenges. In this talk, I will present our recent combined experimental and theoretical efforts to understand the heat dissipation in atomic-scale junctions [1,2]. Using custom-fabricated scanning probes with integrated nanoscale thermocouples, we find that if the junctions have transmission characteristics that are strongly energy dependent, this heat dissipation is asymmetric – that is, unequal between the electrodes – and also dependent on both the bias polarity and the identity of the majority charge carriers (electrons versus holes). In contrast, junctions consisting of only a few gold atoms (‘atomic junctions’) whose transmission characteristics show weak energy dependence do not exhibit appreciable asymmetry. Our results unambiguously relate the electronic transmission characteristics of atomic-scale junctions to their heat dissipation properties, establishing a framework for understanding heat dissipation in a range of mesoscopic systems where transport is elastic – that is, without exchange of energy in the contact region. The close relation between heat dissipation and thermopower provides general strategies for exploring fundamental phenomena such as the Peltier effect or the impact of quantum interference on the Joule heating of molecular junctions [2].

If time permits, I may show further recent results obtained on inelastic electron tunneling spectra [3], current-induced atomic rearrangements that allow to realize single-atom memories [4], or conductance enhancement by light [5].

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A hybrid nanoscale magnetometer

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We realize a setup, where an ultrasoft Si-cantilever, tipped with a nanomagnet, is hanging in the pendulum geometry above a nanoSQUID [1]. The setup is placed in a ³He cryostat equipped with a 6T-magnet and allows for the measurement of individual nanomagnets simultaneously by cantilever and SQUID magnetometry.

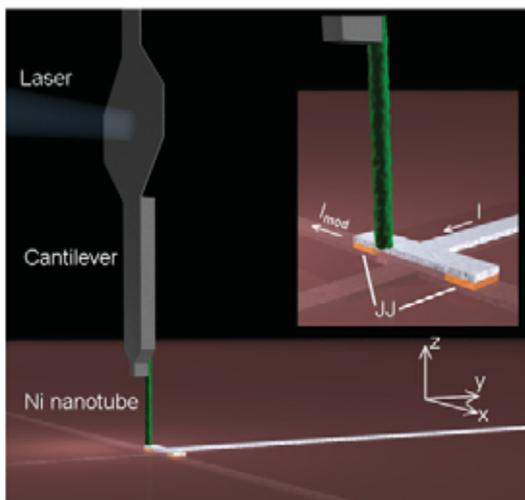


Figure 1: Shown is the setup of the hybrid nanoscale magnetometer. It consists of an ultrasoft Si cantilever with a Ni nanotube sample glued to its tip and a Nb/HfTi/Nb nanoSQUID.

By scanning the three dimensional-halfspace above the nanoSQUID with an individual Ni-nanotube glued to the tip of the cantilever we determine the spatial coupling map of the nanoSQUID, i.e. the amount of flux coupled to the SQUID loop by the magnetic nanotube. The results fully agree with numerical simulations [2].

With the Ni-nanotube at the position of optimal coupling we measure hysteresis in the magnetization of the nanotube both by cantilever and SQUID magnetometry. Whereas the nanoSQUID is sensitive to the nanotube's stray field and the cantilever torque magnetometry measures its integrated magnetization [3].

By using the magnet-tipped cantilever's functionality as a magnetic force scanning probe, we are able to detect Abrikosov vortices entering the superconducting structure. Therefore we are able to discriminate spurious signals emerging from vortices coupling to the SQUID from signals originating from magnetic reversal in the nanomagnet [2].

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Oxidation Scanning Probe Lithography for the fabrication of silicon nanowire devices

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Keywords: oSPL, Silicon nanowire, FETs, nanopillars

Oxidation Scanning Probe Lithography (oSPL) has been proved to be a high-resolution, top-down technique that enables the fabrication of both very small transistors with arbitrary shape and accurate location [1] and very sensitive biosensors [2] based on silicon nanowires (SiNWs). The electrical characterization of the SiNW field effect transistors (FETs) fabricated by oSPL reveals that the values of the on/off current ratio, electron mobility and subthreshold swing, of about 10^5 , $200 \text{ cm}^2/\text{V s}$ and 500 mV/dec , respectively, are similar to those reported from other techniques, both top-down and bottom-up [3]. The potential of oSPL to make high aspect ratio arrays of Si nanopillars is also being explored.

We acknowledge support from the European Union under Grant Agreement No. 318804 (SNM).

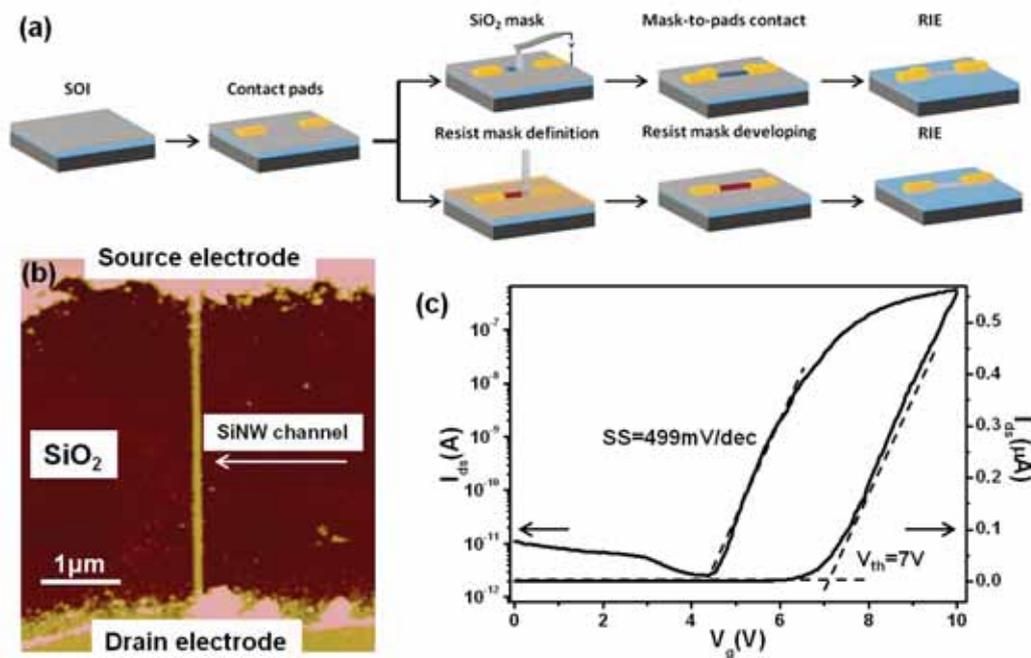


Figure 1. (a) Fabrication steps of SiNW FETs by oSPL. (b) AFM image of a SiNW FET. (c) Transfer curve of a typical device.

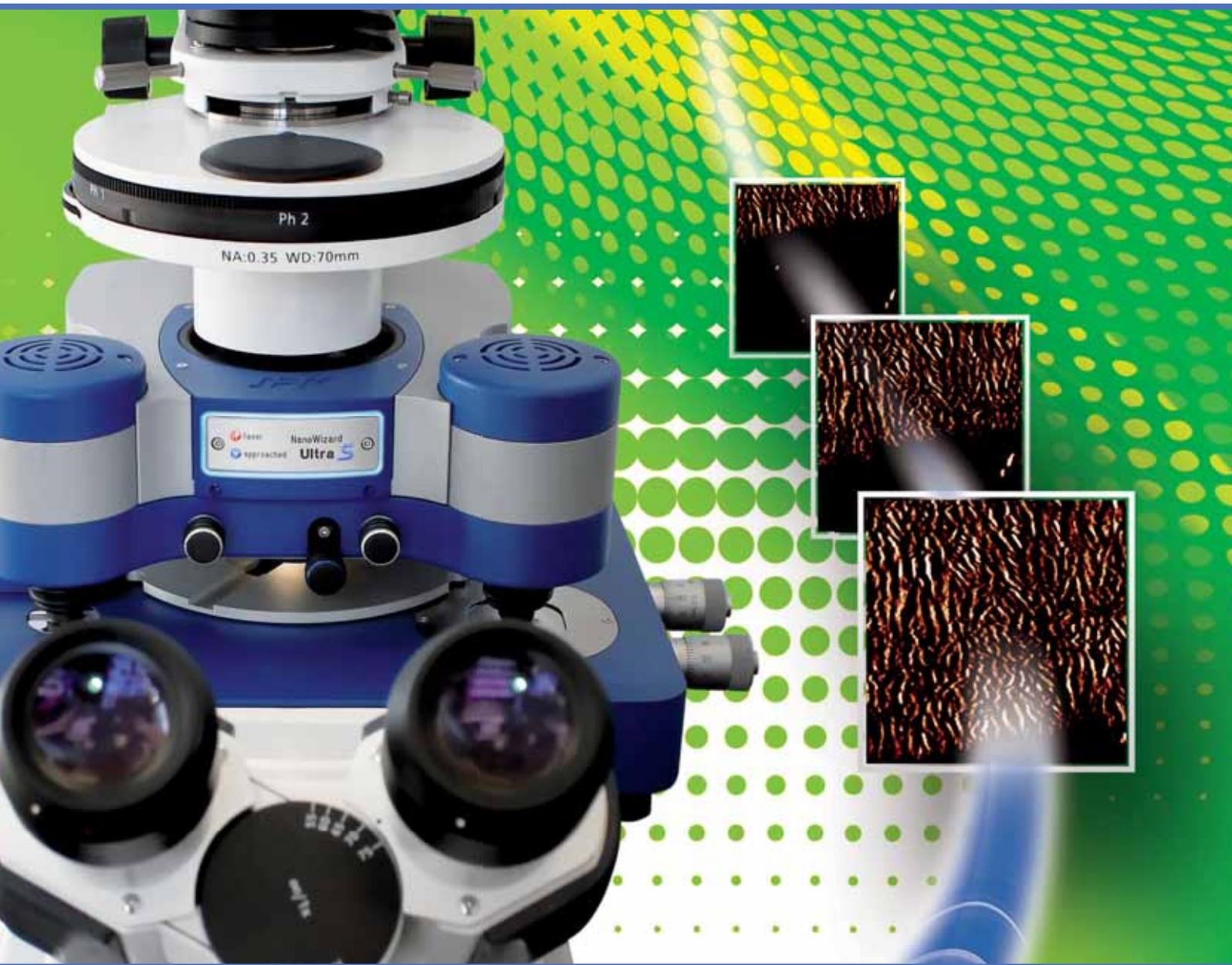
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Studies of Contact Mechanics Making Use of Quartz Crystal Microbalance

Diethelm Johannsmann, TU Clausthal

The quartz crystal microbalance (QCM) is an established instrument for the determination of film thickness and for microgravimetry. Less widely known are its capabilities in contact mechanics. When touching the QCM surface with an external object, the stiffness of the contact can be inferred from the frequency shift, Δf . The talk gives an overview of the QCM in contact mechanics and then focuses on the weakening of contacts at high amplitudes of oscillation, which give rise to partial slip. It turns out that nonlinear behavior (with partial slip being an example) affects the Δf in the same way, as nonlinear force-distance relations affect the resonance frequency of a cantilever in dynamic AFM. Differing from dynamic AFM, the nonlinear regime is approached from the tightly adhered, viscoelastic state. This entails the possibility of hysteresis. The formalism predicts the dependence of frequency and bandwidth on the amplitude of motion. The amplitude of motion thereby turns into a novel sensing dimension, which opens up an entire new field of research to the QCM.

Charge-density-wave-induced non-contact friction

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Understanding nanoscale friction and dissipation is central to nanotechnology. The recent detection of the electronic friction drop caused by the onset of superconductivity in Nb [1] by means of an ultrasensitive non-contact pendulum atomic force microscope (AFM) raised hopes that a wider variety of mechanical-dissipation mechanisms become accessible. Here, we report a multiplet of AFM dissipation peaks arising a few nanometers above the surface of NbSe₂, a layered compound exhibiting an incommensurate charge density wave (CDW) [2,3]. Each peak appears at a well-defined tip–surface interaction force of the order of a nanonewton, and persists up to 70 K, where the short-range order of CDWs is known to disappear. Comparison of the measurements with a theoretical model suggests that the peaks are associated with local, tip-induced 2π phase slips of the CDW, and that dissipation maxima arise from hysteretic behavior of the CDW phase as the tip oscillates at specific distances where sharp local slips occur.

Published in Nature Materials [DOI: 10.1038/NMAT3836]

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Analyzing Interfacial Friction of Nanoparticles: Contact Ageing vs. Structural Lubricity

Dirk Dietzel, Michael Feldmann, and André Schirmeisen

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The investigation of friction by AFM assisted nanoparticle manipulation has presented itself as a very useful approach to gain insight into tribological processes of extended nano- and mesocontacts. Currently, this approach is utilized to analyze the significance of dynamic processes at the interface formed between particles and substrates under UHV conditions. Our studies reveal that some experiments, like the sublinear contact area dependence of friction, can be explained by purely structural considerations of rigid interfaces [1]. Other experiments however, additionally highlight the influence of dynamic interface processes [2]. More specifically, a strongly non-monotonic relation between friction and temperature/velocity was observed during combined velocity and temperature dependent measurements of particle friction. This result cannot be explained either in terms of a purely structural model for interfacial friction or by a simple thermally activated Prandtl-Thomlinson model. We therefore introduced a new model that is based on the Prandtl-Thomlinson concept, but also includes a characteristic time evolution of the contact energy barrier. The corresponding simulations are in very good agreement with the experimental findings when using an energy barrier for contact breaking that increases logarithmically with time, at a rate governed by thermal activation. This effectively transfers the macroscopically well-known concept of contact ageing down to the nanoscale.

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Mapping of elasticity and damping in an $\alpha + \beta$ titanium alloy using atomic force acoustic microscopy

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Abstract

Rapid development in microscopy techniques is providing paths to measure elastic properties at various dimensions. For the past two decades, atomic force acoustic microscopy (AFAM), an advanced scanning probe microscopy (SPM) technique, has played a promising role in materials characterization with a good lateral resolution at micro/nano dimensions. The principal difference between AFAM and other forms of SPMs is the addition of a transducer at the bottom of the sample which induces longitudinal out-of-plane vibrations in the specimen. The vibrations are sensed by a probe during contact. The amplitude of this probe's vibrations at excitation at various frequencies are detected from the AFM photodiode signal by use of a lock-in-amplifier. The probe's response as a function of frequency can be acquired by sweeping a wide range of frequencies. The contact resonance spectra obtained are used to calculate the contact stiffness (k^*) and the local contact damping using suitable models for the tip-sample contact enabling one to image and measure local elasticity and damping of sample surfaces with a spatial resolution of a few tens of nanometers.

Ti-6Al-4V, the most widely used $\alpha+\beta$ titanium alloy, is considered in the applications where a combination of high strength at low to moderate temperatures, light weight and excellent corrosion resistance are required. Some of the many applications where this alloy has been used include aircraft turbine engine components, aircraft structural components, aerospace fasteners, high-performance automotive parts, marine applications, medical devices, and sports equipment. Ti-6Al-4V samples of dimensions $25 \times 25 \times 4$ mm³, heat treated at different temperatures in the range of 923 K - 1123 K for an hour followed by water quench were investigated in this study. Specimens were finely polished using 0.05 microns colloidal silica to obtain scratch-free and strain-free surfaces. Specific software were developed in NOVA Script and LabVIEW for acquiring and processing the contact resonance spectra, respectively, to calculate the indentation modulus (M) and the local damping (E''/E') based on the vibrational behavior of the cantilever as a distributed mass system and not as a point-mass oscillator.

For the first time ever, simultaneous mapping of elasticity and damping due to local internal friction is reported in a metallic structural material with a lateral resolution of 50 nm. The study indicated that the metastable β phase has the minimum modulus and the maximum damping followed by α' and α phases in Ti-6Al-4V alloy. The elasticity and damping measured using AFAM are correlated with the bulk elastic properties and attenuation measured by ultrasonic technique. The moduli estimated by the two techniques are found to be within 10%.

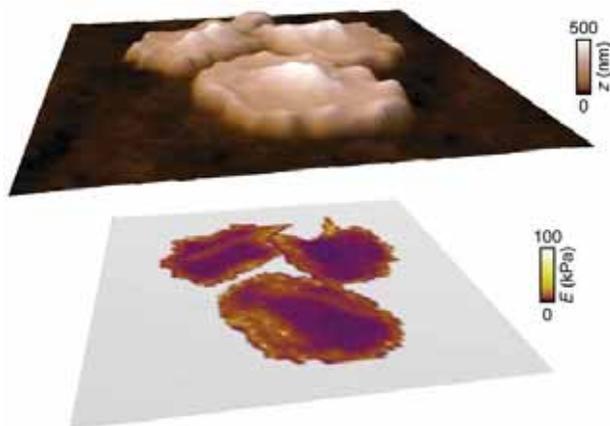
Key words: Atomic Force Acoustic Microscopy, Elasticity, Damping, Internal Friction, $\alpha+\beta$ titanium alloy

Imaging of Platelet Activation and Cytoskeleton Reorganization by Scanning Ion Conductance Microscopy

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Platelets are cell fragments circulating in the blood stream of mammals to maintain hemostasis after injuries of blood vessels. When a damage of the blood vessel endothelium is detected, platelets adhere to the site of the injury to form a plug as a temporal sealing and thereby play a major role in primary hemostasis and wound healing. A fundamental issue of platelet function is their activation, which involves numerous processes in the platelet including a reorganization of the platelets' cytoskeleton. We used the scanning ion conductance microscope (SICM) to investigate the changes of shape and mechanical stiffness of live human platelets during two fundamental processes: the process of spreading on a substrate surface and the process of thrombin-induced activation. We show that the activation resulted in a significant reduction of the platelets' average Young's modulus, which can be explained by a disassembly of the platelets' actin cytoskeleton. By analyzing the spatial distribution of the softening we found that it was mainly located in the lamellipodium of the platelet. In comparison, cytochalasin D-induced depolymerization of the cytoskeleton resulted in similar softening, which was more uniform over the cells. The results support current hypotheses for the morphological and cytoskeletal transformation process of platelets during activation and show that the SICM is a valuable measurement technique for the investigation of blood cells.



SICM image of three live platelets adhered to a polycarbonate substrate. Height (top) and Young's modulus (bottom). Scan range $20 \times 20 \mu\text{m}^2$.



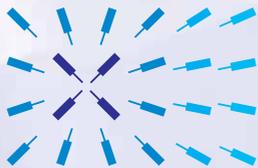
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Measurement of local internal friction in metallic glasses

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Abstract

Rapid development in microscopy techniques is providing paths to measure elastic properties at various dimensions. For the past two decades, atomic force acoustic microscopy (AFAM), an advanced scanning probe microscopy (SPM) technique, has played a promising role in materials characterization with a good lateral resolution at micro/nano dimensions. AFAM is based on inducing out-of-plane vibrations in the specimen. The vibrations are sensed by the AFM cantilever when its tip is in contact with the material under test. The amplitude of the cantilever's vibrations at various frequencies are detected from the AFM photodiode signal by use of a lock-in-amplifier. From the contact-resonance spectra obtained, one determines the real and imaginary part of contact stiffness k^* and from these two quantities the local damping factor Q_{loc}^{-1} which is proportional to the imaginary part γ of the contact stiffness. The evaluation of the data is based on the cantilevers' mass distribution with damped flexural modes and not on an effective point-mass approximation for the cantilevers motion. The given equation is simple to use and has been employed to study the local Q_{loc}^{-1} of amorphous PdCuSi metallic glass and its crystalline counterpart as a function of position of the AFM tip on the surface. The center value of the distribution curve for Q_{loc}^{-1} coincides very well with published data, based on global ultrasonic or internal friction measurements. The width of the distribution changes dramatically from the amorphous to the crystalline case as expected from the consequences of the potential-energy landscape picture. This is compared to Q_{loc}^{-1} measured in crystalline SrTiO₃, which exhibits a narrow distribution, as expected.

Scanning Microwave Microscopy used for detection of ionic movement

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Besides other chalcogenides Ag_2S , due to its remarkable silver ion conductivity, is a very interesting material for cation-based switching devices^[1]. By reversible incorporation / supply of silver cations it can build up and break down conducting silver connections between two metal electrodes using external voltages^[2].

By now this phenomenon is mainly investigated focused on the switching itself. Although there are several publications and patents on this topic, the processes taking place before the switching moment are not yet fully explained.

To observe the phenomena correlated to the switching effect, Ag_2S thin films on a silver background were investigated as well as bulk Ag_2S pressed on silver pellets.

The formation of metal structures on the samples was investigated by means of Atomic Force Microscopy. Using the AFM-tip as electrode, silver depositions with height differences up to the micrometer scale were induced. Using Scanning Microwave Microscopy, these processes were monitored and charge accumulation under the tip was analyzed.

Results are presented in terms of different sample thickness and electrode setup with a focus on cation conduction.

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Noise in Dynamic Scanning Force Microscopy: calibration and imaging

(Oral contribution)

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In general, noise is a nuisance that limits the quality of experimental measurements. From a fundamental point of view, generally either statistical or quantum noise limit the ultimate resolution. On the other hand, according to the dissipation-fluctuation theorem, (some) noise is related to the dissipation of a physical system. In the present work, we will discuss noise in the tip-sample interaction of a Scanning Force Microscope (SFM), and investigate what information can be extracted from this noise. We will show, on the one hand, that thermal noise allows to precisely calibrate the oscillation amplitude in Dynamic SFM [1,2]; and on the other hand that it is possible to acquire “noise” images showing a well defined pattern, different from topography or other acquisition channels.

A typical electronics used to process the dynamic motion of the cantilever can be adjusted to transfer the thermal noise of the cantilever motion from its resonance frequency to a much lower frequency within the typical bandwidth of the corresponding electronics. If the force constant of the cantilever is known then the oscillation amplitude can be precisely calibrated from the thermal power measured in the output signals of a lock-in type dynamic SFM electronics.

For typical applications thermal noise is considered the most important noise source in a SFM setup. We show, however, that additional noise sources may be present in Dynamic SFM measurements, which may be significantly larger than the well-known thermal noise. Using different SFM techniques (Force Spectroscopy, Kelvin Probe Microscopy, 3D modes, etc), we analyze this noise for amphiphilic molecules (SDS) adsorbed on a graphite substrate. Our experiments show that the different chemical nature of these materials induce a different magnitude of the measured noise, leading to an image with “chemical” contrast (see figure 1).

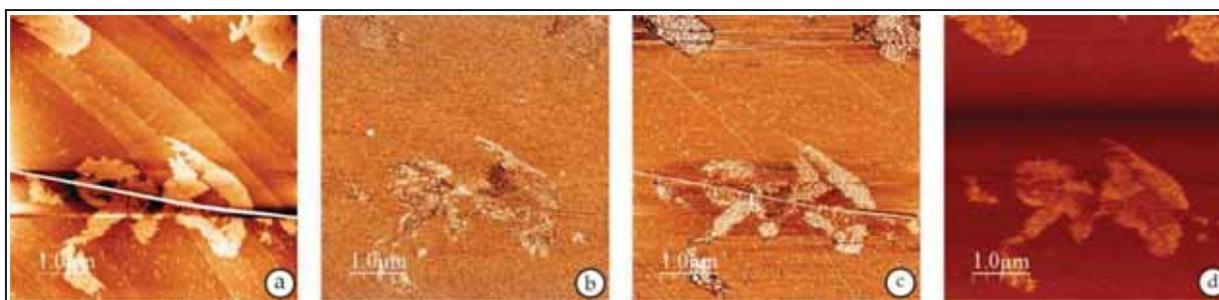


Figure 1: Topography (a), frequency shift (b), contact potential (c) and noise images (d) acquired simultaneously in the so called Amplitude Modulation mode (topography feedback performed keeping the oscillation amplitude constant).

[1] J. Colchero, M. Cuenca, J.F. González Martínez, J. Abad, B. Pérez García, E. Palacios-Lidón and J. Abellán. “Thermal frequency noise in dynamic scanning force microscopy”; JAP 109, 024310 (2011). doi:10.1063/1.3533769 95

[2] J.F. González Martínez, I. Nieto Carvajal and J. Colchero. “Calibration of oscillation amplitude in Dynamic Scanning Force Microscopy”; Nanotechnology 24 (18) 185701 (2013). <http://dx.doi.org/10.1088/0957-4484/24/18/185701>

We demonstrate the ability to measure Stern potential and Debye length in dilute ionic solution with atomic force microscopy. We develop an analytic expression for the second harmonic force component of the capacitive force in an ionic solution from the linearized Poisson–Boltzmann equation. This allows us to calibrate the AFM tip potential and, further, obtain the Stern potential of sample surfaces. In addition, the measured capacitive force is independent of van der Waals and double layer forces, thus providing a more accurate measure of Debye length.

See: *Nanotechnology* 24 435701 (2013). doi:10.1088/0957-4484/24/43/435701

Crystallographic and electronic structure of graphene on the pseudomorphic Cu/Ir(111) substrate

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Understanding the nature of the interaction at the graphene/metal interfaces plays a critical role for the correct description of the graphene-based electron- and spin-transport devices. Here, several factors, such as doping level or/and hybridization of the electronic states of graphene and the metal around the Fermi level defines the properties of such interfaces. Starting from *p*-doped nearly free-standing graphene on Ir(111), we tailor its properties via intercalation of one monolayer of Cu. The crystallographic and electronic structures of the resulting *n*-doped nearly free-standing graphene layer on the lattice mismatched pseudomorphic Cu/Ir(111) substrate were studied by means of scanning probe microscopy (STM and 3D NC-AFM) and photoelectron spectroscopy in combination with state-of-the-art density functional theory calculations. These results allow to understand mechanisms that are responsible for the modification of the electronic structure of graphene at the Dirac point (doping and the band-gap opening). These findings for the graphene/metal system will help in the engineering of the future graphene-based devices.

Temperature determination of AFM cantilevers from thermal noise spectrum analysis

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Abstract:

For high temperature surface imaging using AFM, the sample is usually heated with an integrated heating element. The temperature of the cantilever also increases due to radiation and possibly heat conduction through the tip-sample contact. It is important to determine the exact temperature of the cantilever so as to calculate the limit of the force sensitivity. We calculate the temperature of the cantilever from the thermal noise spectrum.

A sample is heated in an UHV-AFM to a temperature within the range of 300-600 K while the cantilever is approximately 20 nm away from the sample surface. The optical beam-deflection system is used to characterize the dynamic behavior of the cantilever. For several different temperatures the cantilever was calibrated by using force distance curves, the surface was imaged in the non-contact-mode and the thermal noise spectra of the cantilever deflection were recorded, which are dominated by peaks at the mechanical resonances of the cantilever.

We compare two methods to determine the temperature of the cantilever: The first method is based on the integral of the power spectrum density at the first bending resonance which is proportional to the temperature. The second method uses a shift in the resonance frequency with increasing temperature, which is caused by the temperature dependence of the Young's modulus.

Conducting AFM study of colloidal nanoparticle assemblies

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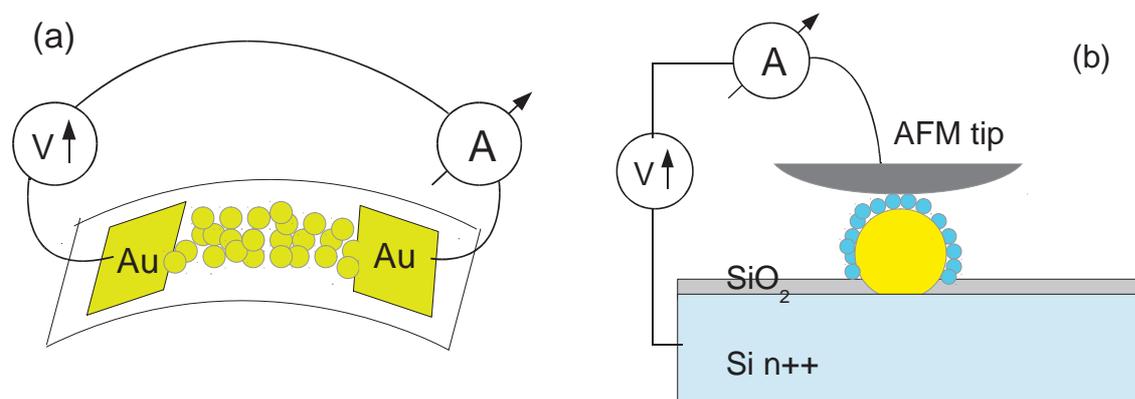


Figure 1: (a) Electromechanical characterization of colloidal nanoparticle assembly on flexible substrate under strain and (b) C-AFM and force spectroscopy measurement on an individual nanoparticle coated by a ligand.

Electronic transport through nanoparticle arrays, fabricated by convective self-assembly from colloidal nanoparticles, has been studied macroscopically, on flexible substrate, as function of strain and temperature. For most ligands, the particles are estimated to be weakly coupled since the interparticle conductance between neighboring particles is below the quantum conductance. A priori, a Coulomb tunneling model is favored for the so-formed regular arrays. However, some controversy remains on the role of the Coulomb charging energy between temperature and strain dependent measurements¹, and the role of the ligand in determining the barrier height.

In this work, AFM studies are performed both on colloidal nanoparticle assemblies and on individual nanoparticles forming an ohmic contact with the substrate and covered by molecular layers. The AFM is used simultaneously as conductive probe in contact mode and for force spectroscopy measurements. Characterizing monolayers or even single particle junctions allows dissociating the Coulomb charging energy and mechanical multilayer effects from the tunneling decay constant and hence focusing on the effect of the ligand on the barrier height and a possible implication of its molecular levels.

1) Electron transport in gold colloidal nanoparticle-based strain gauges

H. Moreira, J. Grisolia, N.M. Sangeetha, N.M., N. Decorde, C. Farcau, B. Viallet, K. Chen, G. Viau, and L. Ressier, NANOTECHNOLOGY 24(9), 095701 (2013)

Noise behavior of frequency modulation Kelvin force microscopy (FM-KFM)

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Noise behavior of a phase-locked loop (PLL) based frequency modulation Kelvin force microscope (FM-KFM) is studied. Starting from probe deflection noise with its two components (thermal excitation and deflection sensor noise), the noise propagation is modeled throughout the setup¹. We adopt the “noise gain” formalism commonly used in operational amplifier (OpAmp) design. It is shown that noise performance can be considered widely independent from stability and ideal loop response. Thermal and detector noise are sufficient as noise sources for modeling the output noise in agreement with measurement, demonstrating that the PLL components neither modify nor contribute noise. A design rule suggests setting the AC modulation frequency at about 4 times the desired output bandwidth which shall be made equal for distance and Kelvin loops, in order to obtain the best sharing of the available PLL bandwidth. A criterion allows to determine as a function of bandwidth, temperature and probe parameters whether thermal or detector noise is the preponderant noise source. Probe merit factors are then established for either case, revealing the influence of probe parameters on noise. merit factors of known probes are compared. This comprehensive study is a step toward a more integral performance assessment. Finally, we address the question of the ultimate performance limit and how far existing setups are still away.

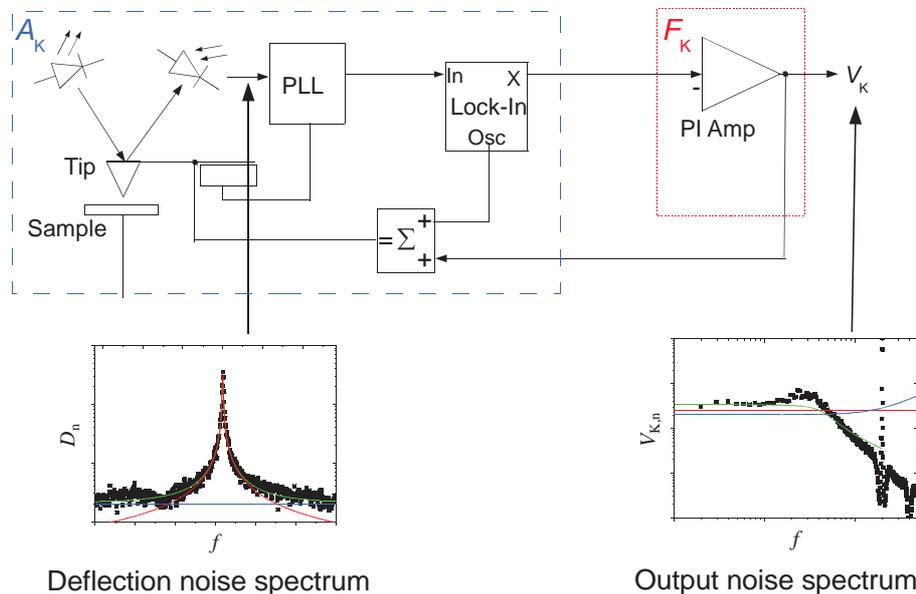


Fig.1 : PLL based FM-KFM setup and noise power spectral densities of the probe deflection noise and of the Kelvin voltage output noise.

1) Noise performance of frequency modulation Kelvin force microscopy, H. Diesinger, D. Deresmes, and T. Mélin, Beilstein J. Nanotechnol. 2014, 5, 1-18

Quantitative measurement of the nanomechanical properties of bio- and polymeric materials

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The nanomechanical properties of single human immunoglobulin G and M (IgG, IgM) antibodies were measured with sub-10-nm spatial resolution in a liquid environment using a fast force-volume technique (Peak-Force tapping mode). Ultrastructural details of these macromolecules were resolved in topographical images (Fig. 1). Simultaneously, physiological important physical quantities, such as elasticity, adhesion and deformation, were measured. Considering their dimensions and adsorption onto the substrate, the IgM antibodies were observed to be highly flexible, with a low elastic stiffness (34 ± 10) MPa and high deformability (2.8 ± 0.7) nm.

In a second approach, we studied the nanomechanical properties of elastomeric polypropylene (ePP), a semi-crystalline polymer, applying the same technique (Fig. 2). Wet-chemical ablation allowed us to obtain the elasticity, adhesion, deformation, and dissipation on amorphous and crystalline regions in multiple depth layers of the sample. The elastic moduli of these regions were found to be significantly lower than those of pure atactic and isotactic polypropylene samples. The sample surface, however, revealed similar values of the mechanical properties of both regions due to the existence of a thin amorphous top layer (≈ 22 nm). Liquid-liquid phase separation of short and long isotactic polymer chains during sample preparation led to the formation of crystalline defect regions within the whole volume of the sample, which we found to be faster removed than the surrounding area by the etching.

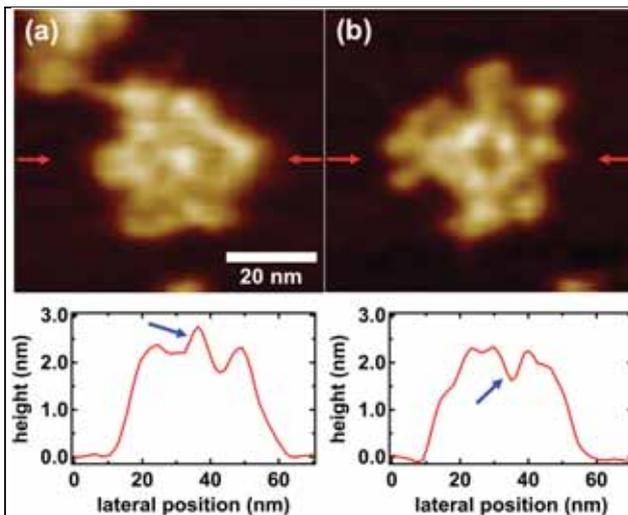


FIG 1. High-resolution topographical images (top) and cross-sectional profiles (bottom) of single human IgM antibodies in two different configurations (a, b) measured in a liquid environment.

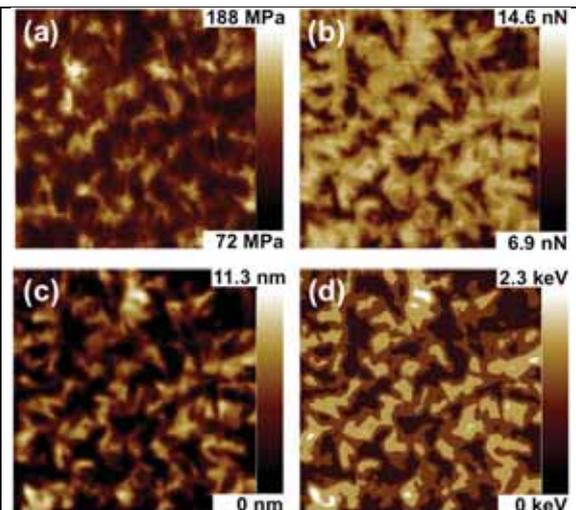


FIG 2. Surface properties of an unetched ePP sample measured by quantitative nanomechanical mapping. (a) Elastic modulus, (b) adhesion, (c) deformation, and (d) dissipation.

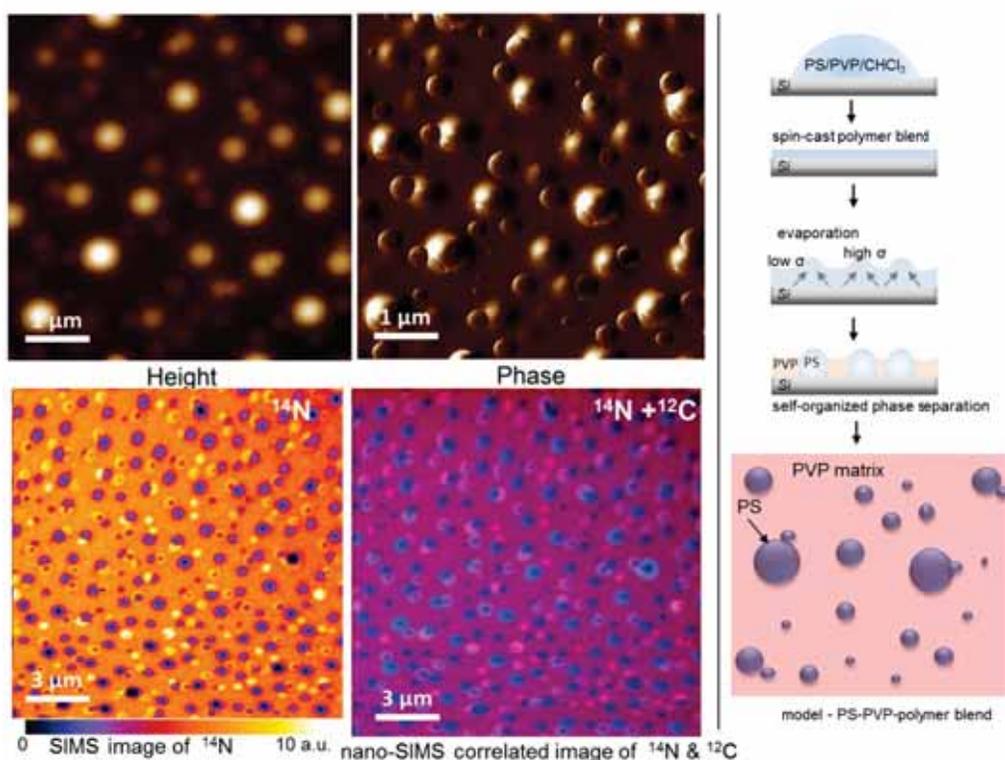
Mapping nanophase of polymer blends polystyrene/poly(vinylpyrrolidone)

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In thin films of polymer blend, in addition to composition and molecular weight of components, the structure is dependent on the surface energies of the blocks and on geometrical constraints induced by confinement in a thin film. Due to the large surface-to-volume ration, the structure of separating phases is different from that in the bulk. Since block polymers assembly is highly dependent on surface energies these boundaries can effectively direct the orientation of nanoscopic polymer domains. Therefore, the thin films of blends have found potential in different applications (e.g. biomedical, surface coating). Moreover, the surface behavior of a two polymer mixture depends on the chemical structure of the polymer components and the interaction between the two polymers. Hence, the nanoscopic morphology and the surface composition using chemical “fingerprint element” need to be known in order to fully understand and utilize the thin film properties. A polystyrene/poly(vinylpyrrolidone) (PS/PVP) polymer blend has been used to create nanosize-domains structures on silicon oxide substrate via simple solvent evaporation of spin-coating films. The phase information and the surface topography are investigated using tapping mode atomic force microscopy (TM-AFM) technique. A dynamic second ion mass spectroscopy technique (nano-SIMS) provides the chemical mapping of the surface using CN as “fingerprint” element for PVP areas. The PS/PVP polymer blend was thermally annealed and an “unexpected” structure in topography was observed. Nano-SIMS images show the formation of a “stable” nanostructure on the annealed surface but AFM studies show these nanostructures as “shifted” in topography due to the contribution of surface tension and temperature dependence of the both component polymers.



Keywords: surface pattern, polymer blend, phase separation, AFM, nano-SIMS

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Electrostatically-formed Nanowires as Gas Sensing Devices

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A novel gas sensing technology is proposed that is based on electrostatically-formed nanowires (EFN). Recently, Shalev *et al.* have demonstrated biosensing with an EFN device in an aqueous environment [1]. The EFN is composed of a doped silicon region surrounded by four gates, a back gate (V_{BG}), two lateral junction gates (V_{JG1} and V_{JG2}) and an active top layer that acts as a molecular sensing gate. Appropriate biasing of the various gates form depletion regions at the gate-silicon interfaces and junctions around the n-type silicon body, forming a small accumulated conductive silicon region that has a diameter of a few nanometers.

The actual EFN dimensions are obtained by correlating the 3D electrostatic simulations with I-V measurements and Kelvin probe force microscopy (KPFM). KPFM as an advanced atomic force microscopy technique allows the quantitative characterization of the surface potential with nanometer precision. Gas sensing was demonstrated for the first time for volatile organic compounds (VOC) with ppm sensitivity.

It has to be emphasized that such an EFN device was realized with conventional low cost high volume manufacturing (CMOS processing) as no nanometer features are defined during the process. Instead, the nanometer scaling of the device is performed electrostatically post fabrication.

1. G. Shalev, G. Landman, I. Amit, Y. Rosenwaks, I. Levy, *NPG Asia Materials*, 2013, 5, e41.

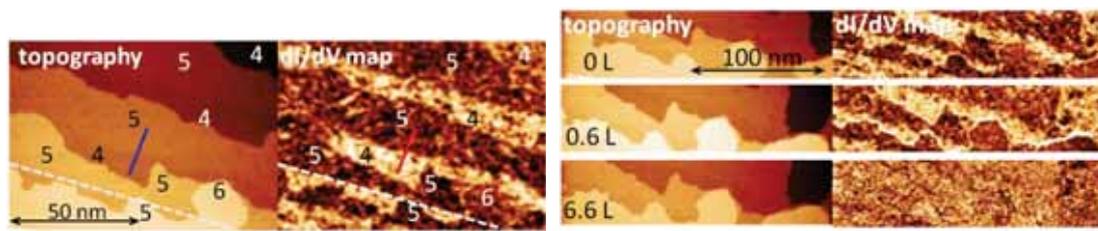
STM study of oxygen adsorbate-induced dI/dV contrast tuning in fct-Mn films

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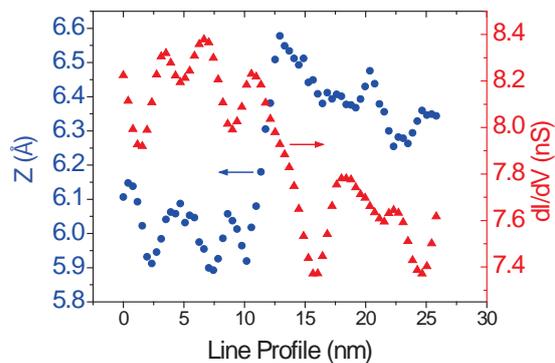
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Oxygen adsorption on metal surfaces is an interesting means to modify system properties in surface science. We have applied scanning tunneling microscopy (STM) in connection with the lock-in technique to simultaneously record differential conductance maps and constant-current mode topography images of 4.2 atomic monolayers (ML) fct-Mn on 5.0 ML Co/Cu(001). Before dosing oxygen into the ultra-high vacuum chamber, a clear bright and dark contrast is observed in the dI/dV maps at a bias voltage of +0.2 V for regions of 4 and 5 ML Mn thickness, respectively, as seen in (1-a). The dashed white line indicates a buried Co step edge underneath a Mn terrace and corresponds to the contrast reversal boundary in the dI/dV map. On the same Mn terrace, dark regions in the dI/dV map are 36 pm higher in topography than bright regions. This is shown in (1-b), which represents line scans of the topography (blue) and the dI/dV signal (red) along the same line indicated in (1-a). This height difference is close to the difference in perpendicular lattice constants of Mn and Co, revealing the position of overgrown steps in the Co layer. Subsequently, 0.6 Langmuir (L) and 6 L oxygen is dosed. Differential conductance maps, recorded also at +0.2 V, are shown in (2-a). Comparing histograms of dI/dV maps on clean Mn, 0.6 L oxygen/Mn, and 6.6 L oxygen/Mn (2-b) reveals that the contrast is more and more blurred with increasing oxygen dosage. After dosing 6.6 L oxygen, the Mn surface should be completely covered with oxygen, since the sharp 1×1 pattern in low-energy electron diffraction has disappeared. However, the contrast in differential conductance maps recorded at other sample bias voltages is still present on 6.6 L oxygen/Mn. Comparing dI/dV-V curves for the three thicknesses, similar peaks close to the Fermi level are present on both the clean and the oxygen-covered Mn surface, while their intensity in the latter case shrinks towards the Fermi level. The bias dependence of the dI/dV contrast and the dependence on the amount of oxygen have to be attributed to the oxygen-induced modification of the local density of states of Mn.

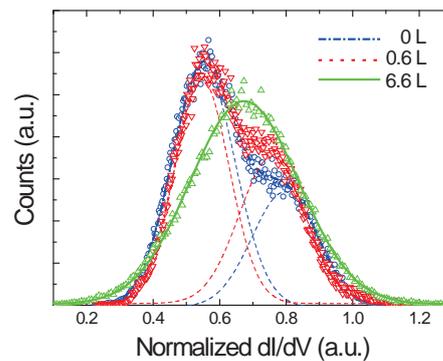


(1-a)

(2-a)



(1-b)



(2-b)

Quantifying the atomic-level mechanics of single long physisorbed molecular chains

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In-situ polymerized fluorene wires of 10-100nm length linked by single C-C bonds are vertically pulled from a Au(111) substrate by the probing tip of a low-temperature atomic force microscope (AFM).[1] The observed force gradient shows strong, nearly periodic oscillations which correspond to the sequential detachment of fluorene units. In contrast to previous tunnelling microscopy experiments,[2] these oscillations persist until complete pull-off. Calculations within an extended Frenkel-Kantorova model reproduce the periodicity and magnitude of those oscillations and reveal their connection to the detachment force and energy of individual units. The remaining part easily slides along the surface, only leading to small oscillations, owing to the high stiffness of the units and to their near incommensurability with respect to the substrate atomic structure.

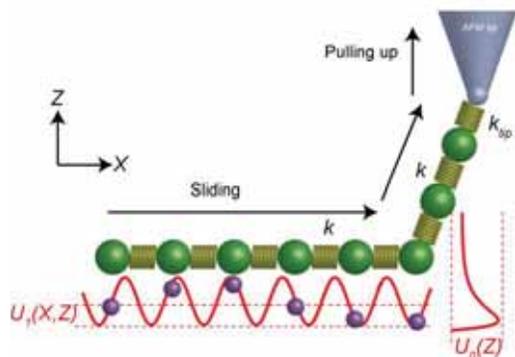


Fig.1 Schematic drawing of the tip-sample interaction while pulling up the molecular wire.

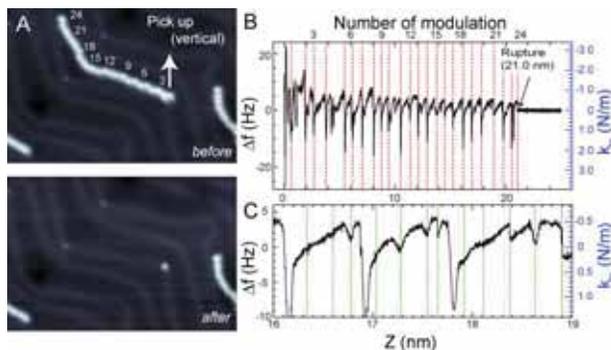


Fig.2 (A) STM images of the same surface area before and after pulling up a molecular wire. (B) Δf curve measured while retracting the tip (and pulling the wire), and (C) magnification around $z=17.5$ nm.

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Effect of tip functionalization on Kelvin probe force measurements

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Atomic force microscopy (AFM) measurements provide new insights into the functioning of battery materials on the micro- and nanoscale. Kelvin Probe Force Microscopy (KPFM) [1] is an AFM mode, where in addition to the sample topography the surface potential of the material can be mapped. On hard substrates this imaging mode can normally be executed quite easily, but sticky samples such as soft polymers can provide an obstacle. This can be avoided by using specially functionalized cantilevers which do not stick to the surface. A model experiment setup was used to gather information on the effect of this functionalization on the measured surface potential, and to determine if KPFM can be a valuable additional method for research on polymer electrolyte coated electrodes in lithium polymer batteries [2].

Cantilevers with Pt/Ir or Au coated tips (PPP-NCSTPt or PPP-NCSTAu by Nanosensors) were cleaned thoroughly by dipping in conc. H_2O_2 for 30 s and then immersing in a mixture of 0.1 M HCl and 0.1 M H_2O_2 for 16 h. The tips were then coated with self-assembled decanethiol or dodecanethiol monolayers using a 5 mMol 1-decanethiol or 1-dodecanethiol solution in trichloromethane by immersing overnight.

After coating, the tips were first controlled for impurities using light microscopy. Test measurements to guarantee tip quality were executed using an x-y silicone test grid. Afterwards, the tips were applied for measurements on a gold/glass substrate using amplitude modulated KPFM mode. Comparisons of the KPFM signal intensity were made. In addition, the “de-sticking” effect of the samples was tested on a polysiloxane film, which was applied on a glass slide. The topography image quality was compared to commercially available CF_3 -functionalized tips.

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On-Surface Synthesis by Thermal and Photochemical Activation

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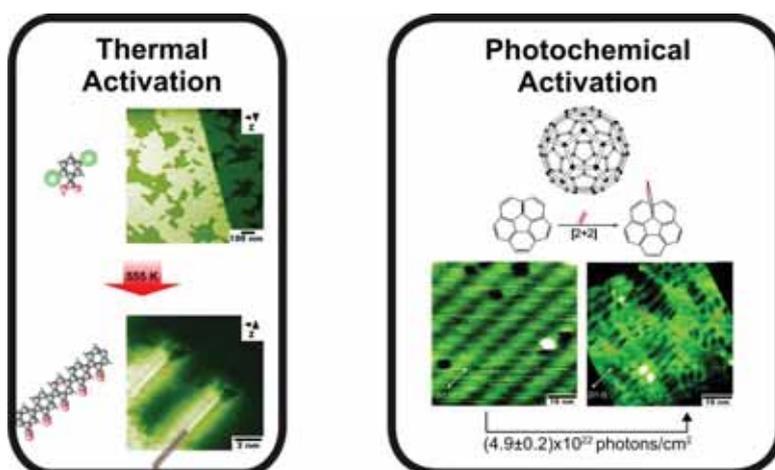
now at: Department of Physics & Astronomy, University of Utah, Salt Lake City, USA

The bottom-up construction of functional devices from molecular building blocks offers great potential in tailoring materials properties and functionality with utmost control. An important step towards exploiting bottom-up construction for real-life applications is the creation of covalently bonded structures that provide sufficient stability as well as superior charge transport properties over reversibly linked self-assembled structures. On-surface synthesis has emerged as a promising strategy for fabricating stable, covalently bound molecular structure on surfaces [1].

However, so far, the experimental studies have mainly been limited to metallic substrates [2-3]. Nevertheless, for many applications such as molecular electronics or molecular optoelectronic, it would be exceedingly attractive to transfer this technique to bulk insulator substrates to provide electronic decoupling.

Here, we present the first successful examples of polymerization reactions on the surface of a bulk insulator by using thermal and photochemical activation. First, we focus on the covalent linking of halide substituted benzoic acid molecules by thermal activation [4-5]. For the covalent linking step, we exploit dehalogenation and linkage of the resulting radicals.

Second, C₆₀ fullerenes are polymerized by non-thermal [2+2] cycloaddition coupling upon irradiation [6]. The substrate-guided photochemical polymerization of C₆₀ results in well-oriented polymeric domains. Thus, our work constitutes a promising route towards fabrication of covalent networks on bulk insulators.



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Surface Studies of Wurtzite and Zincblende Heterostructure InAs and GaAs Nanowires Using Scanning Tunneling Microscopy and Spectroscopy

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With the high surface to volume ratio of nanowires (NWs), the surface characteristics and morphology of the NW side-facets will be strongly coupled to the electronic properties of the NW. Hence, in order for NWs to be realized as future photonics and electronics devices surface studies are essential. In addition, III-V NWs provide the opportunity to study the wurtzite (Wz) crystal structure, giving access to surfaces not found in other III-V geometries. Regardless, very few atomically resolved surface studies have been published^{1,2}

We have previously presented the first scanning tunneling microscopy/spectroscopy (STM/S) study on III-V NW surfaces¹. With our current ability to engineer III-V NWs with axially alternating Wz and zincblende (Zb) crystal phases^{3,4} our experimental toolbox is now further improved by the multitude of facets and interfaces provided by such NWs.

Here we present STM/S studies on all common low index facets in addition to interfaces and edges of well-defined Au-seeded heterostructure InAs and GaAs NWs. The NWs were grown using metal organic vapor phase epitaxy and tuning of the crystal structure was realized by varying the precursor gas flow such that NWs with axial stacking of Zb {110}, and {111}A/B, as well as Wz {10-10}, and {11-20}, type facets could be grown in a controlled manner. We have performed detailed imaging of the surface structure and morphology during different phases of wire overgrowth when a transition occurs from the initial saw-tooth {111} type facet morphology of Zb twin superlattice to smooth {110} type facets.

In addition, investigations of the non-polar NW facets revealed unreconstructed surfaces implying that STS data will correspond to the bulk electronic properties of the material without being perturbed by surface states related to reconstructions. The polar facets, {111}A/B, were however found to be reconstructed, which was not seen on analogous surfaces of wafers, suggesting that NW surfaces have very different surface energies.

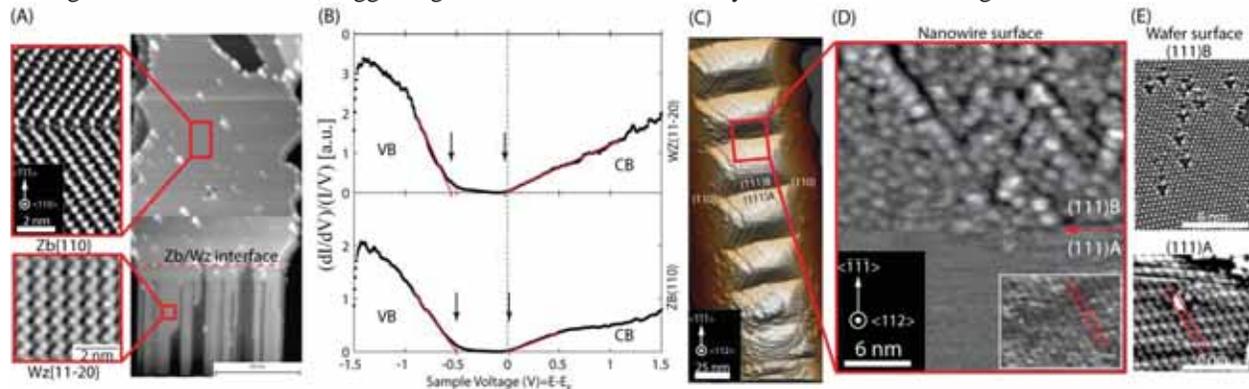


Figure 1. STM results on InAs NWs. All images were obtained at negative sample bias. (A) STM image of interface between Zb {110} (top) and Wz {11-20} (bottom). Inset shows atomic structure of each facet. (B) STS data obtained on two neighbouring Wz and Zb facets in a single NW, depicting the local density of states. (C) Overview STM image of Zb twinned superlattice. (D) High resolution image of {111}A/B facet of NW TSL showing a highly reconstructed {111}B. Inset shows {111}A. (E) {111}A/B surfaces of wafers.

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Modelling dissipation in Dynamic Scanning Force Microscopy as a function of tip-sample distance

(Poster contribution)

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The quality factor is a key parameter for Dynamic Force Microscopy, since it determines a series of important properties of the system, such as the energy stored in the system, the width of the resonance curve, the resonance enhancement, but also the response to non-linear interactions, and the response of a Phase-Locked Loop in the so called Frequency Modulation mode. For operation in non-vacuum environment –that is in air or liquids, the quality factor is essentially determined by the surrounding viscous medium. In the present work the behaviour of a Scanning Force Microscopy (SFM) cantilever in viscous media is studied, with particular emphasis on how the viscosity of these media reduces the quality factor. A simple theoretical model is proposed based on the assumption of a composed probe model:

SFM probe = microscopic cantilever + mesoscopic tip cone + nanometric tip apex

This model correctly describes the dissipation for a variety of cantilevers in different media (fig. 1). It is shown that the physical dimensions of the cantilever as well as the viscosity of the medium where it is immersed determine the dependence of the quality factor with the cantilever-sample distance. For the range of distances explored in this work, it is shown that the Reynolds number is a key parameter to select a cantilever in a specific medium, in order to achieve the highest quality factor at small cantilever-sample range.

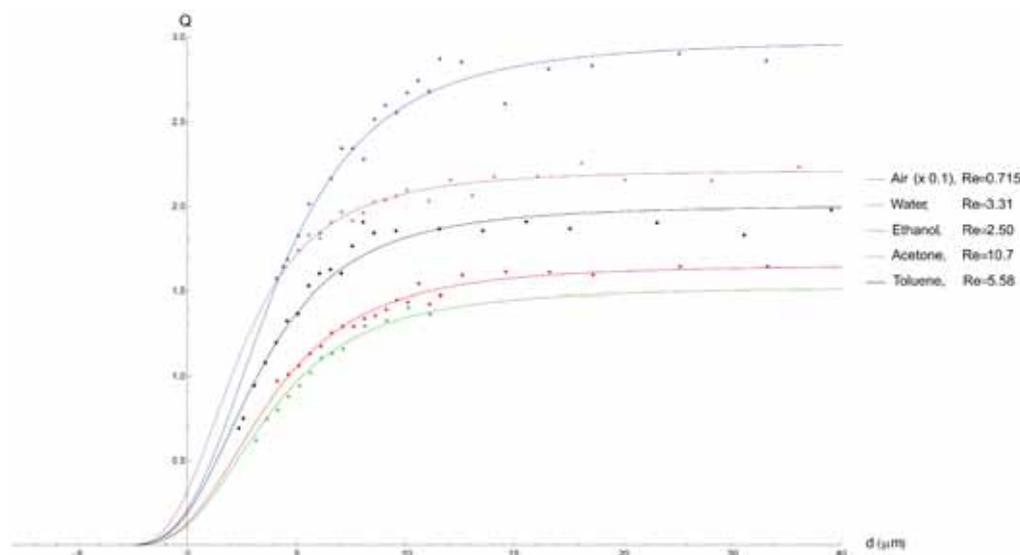


Figure 1: This graph shows the dependence of the quality factor with distance for different media (air, water, ethanol, toluene and acetone). Note that for better visibility the curve in air has been divided by 10. The Reynolds number (Re) for each case has also been added. In this particular case, a silicon nitride cantilever (length, 100 microns; width, 20 microns; elastic constant, 0.046 N/m) has been used.

Length-Dependent Thermal Transport along Molecular Chains

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Molecular layers are used today mostly as passive layers like adhesion promoters or photoresists with the goal to use them as active materials, e.g. as electrical conductors or insulators or as thermal interface materials. Thermal transport across molecular layers is of high relevance for current and future nanoelectronic devices. In those layers, the one dimensional molecular chains are peculiar systems as they do not behave like thermodynamic ensembles and do not equilibrate which causes unexpected effects in their thermal transport properties like signatures of ballistic phonon propagation, effects of phonon localization and interference.

Here, we present thermal transport measurements conducted with a vacuum-operated scanning thermal microscope (SThM) to study the thermal conductance of monolayers of nine different alkane thiols self-assembled on gold Au(111) surfaces and four alkane silanes coupled to silicon oxide surfaces as a function of the length and bonding conditions of the alkane chains. The key element of our microscope is a microfabricated resistive silicon probe tip which allows probing local thermal conductance at a lateral resolution down to 10 nm with a relative sensitivity well below 10^{-12} W/K/molecule. We found the thermal conductance of molecular layers highly dominated by the thermal interface and developed a new method to quantify the thermal conductance by tuning the contact area of the thermal interface between the probe and sample. We achieved this by repetitive making and breaking contact between tip and sample and varying the tip's load rather than scanning the tip. This approach also allows monitoring the mechanical properties of the sample and an analytical description of the contact mechanics between the tip and the sample. Combining both, mechanical and thermal response of the sample, we are able to extract the thermal conductance of the samples' molecules. Finally, we discuss the results in terms of the apparent thermal conductance of the molecules and their boundaries to the thermal contacts at different coupling strengths. Moreover, the dependence of the thermal conductance on the chain length is compared to theoretical predictions.

FM-SFM and KPFM studies on vicinal Si(111) with atomic resolution

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We have studied the structure of vicinal Si(111) with frequency modulation scanning force microscopy (FM-SFM) and Kelvin probe microscopy (KPFM) at room temperature. Vicinal Si(111) surfaces inclined towards the $[-1 -1 2]$ direction contain steps with the height of one and three interplanar distances at temperatures below 870°C [1]. With increasing miscut angle the amount of triple steps also increases [1]. For an angle of $\sim 10^\circ$, it has been found that only triple steps regularly spaced (111) terraces with a width equal to that of a unit cell of the Si(111)-7x7 surface structure appear [2]. This stepped well-ordered surface can be used as a nanotemplate for the fabrication of one-dimensional nanostructures.

Although the measurements of step edges are challenging for SFM and KPFM, we have partially resolved the triple step structure between the Si(111) 7x7 terraces using sputtered silicon cantilevers, some of them coated with PtIr. Based on STM images, Kirakosian et al. found that the surface has the $(5 5 7)$ orientation, being the period of the staircase of triple steps 5.73 nm [2]. However, in the literature the structure of the triple step is still subject of debate [1,3-4]. After analysing our atomic resolved FM-SFM images of such steps, we conclude that the structure corresponds to the one proposed by Teys et al. [1], which is ascribed to a $(7 7 10)$ orientation of the surface rather than the initially proposed $(5 5 7)$ [2-3]. Moreover, KPFM images with atomic resolution of the surface reveal strong surface potential variations on the terraces and the steps. We found that the local contact potential difference is high at the dimer positions, whereas it drops at the adatoms, extra rows, and defects at step edges.

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Nanoscale mechanical, thermal and infrared spectroscopy with the atomic force microscope

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Abstract

We have employed AFM-based infrared spectroscopy (AFM-IR), Lorentz Contact Resonance (LCR) and nanoscale thermal analysis (nanoTA) to map nanoscale chemical, thermal, and mechanical properties in polymers, and biological materials as well as on hard surfaces like minerals, metals and semiconductors. The AFM-IR technique irradiates the sample with light from an infrared laser and measures the absorption of this light on a sub-wavelength scale using the tip of an AFM. Measurement of the absorbed light over a broad wavelength range generates nanoscale infrared absorption spectra that can be used to chemically analyze samples at the nanoscale. Simultaneous measurements of the cantilever's contact resonance frequency as excited by the IR absorption provides a complimentary measurement of mechanical properties. We have used these techniques to chemically identify individual chemical components in polymer nanocomposites and multilayer films and to perform subcellular spectroscopy and chemical imaging on biological cells. Recent advances with top side illumination have enabled measurements on a broader range of real world samples on almost arbitrary substrates including metals and semiconductors. Resonant and electric field enhancement techniques have also enabled nanoscale infrared absorption spectra to be obtained on extremely thin samples.

Additionally, using self-heating AFM probes we have employed LCR and nanoTA to measure and map thermal and mechanical properties of polymeric materials, including spatial mapping of polymer glass transitions and melting temperatures. These self-heating AFM probes have also been used to measure temperature dependent mechanical and chemical properties of materials as well as to enable local mass spectroscopy measurements.

Towards ultra-low dopant concentration carrier profiling and quantification by complementary Scanning Probe Microscopy (SPM) techniques

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In power electronics, superjunction compensation devices offer the possibility to tune the ability of the device to withstand high breakdown voltages while maintaining a low on-resistance [1]. Also in this field novel wide band gap semiconductor materials with superior properties [2] compared to silicon, have illustrated great opportunities, but require controlling dopant profiles down to an unprecedented low concentration regime.

Scanning Probe Microscopy, e.g. Kelvin Probe Force Microscopy provides direct access to dopant profiles as has been recognized early on. So far SPM techniques have been used to provide information which is complementary to what can be revealed by techniques like Scanning Resistance Probe (SRP), Secondary Ion Mass Spectroscopy (SIMS) or Scanning Electron Microscopy (SEM). These well-established techniques, however, proved unsuitable for precise monitoring of charge compensation during several epitaxial steps in demanding fabrication processes [3] and are challenged by low dopant concentrations. In SPM derived methods, on the other hand, quantitative dopant profile assessments are comparably challenging [4].

In this contribution we demonstrate that SPM techniques, such as Kelvin Probe Force Microscopy (KPFM), Scanning Capacitance Force Microscopy (SCFM) and Scanning Spreading Resistance Microscopy (SSRM) can be used to assess dopant profiles down to the concentration regime needed for wide band gap semiconductors such as SiC or GaN. In our detailed study the three techniques are performed in the same measurement environment to map and quantitatively determine the electronically active dopant concentrations present at the surface region of the specimen. We show carrier distributions inside silicon trench structures mapped at high sensitivity and at concentrations as low as $7 \cdot 10^{14} \text{ cm}^{-3}$ [5].

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Towards the formation of extended polycyclic aromatic hydrocarbons doped with nitrogen

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The formation of extended polycyclic aromatic hydrocarbons (PAHs) by on-surface synthesis on metal surfaces has recently attracted increased interest owing to the current developments in the field of building new carbon allotropes in a bottom-up approach. We used planarized triarylamine molecules functionalized with diphenylmethylidene side groups and end groups of different types (e.g. alkylchains and trialkylsilyl moieties) as molecular building blocks. We investigated the potential of the functionalized triarylamine molecules to undergo a cyclodehydrogenation reaction on the Au(111) and Cu(111) surface to create novel extended PAHs doped with nitrogen heteroatoms. The thermal-stability of the different functional side and end groups as well as the adsorption configuration of the individual molecules will be discussed in detail by means of low temperature scanning tunneling microscopy under ultra-high vacuum condition.

Micromechanical properties of multilayered tissue investigated by AFM force mapping

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The mechanical properties of tissue samples are often investigated by large-scale tensile tests. These tests give large-scale information about the whole sample, but they do not resolve multilayered structures. In two studies, we applied AFM force mapping to cryosections of multilayered porcine corneas [1] and heart valves [2], to gain insight on the layer-specific mechanics on the micrometer scale.

The cornea is the transparent curved outer part of the eye covering the pupil. It mainly consists of a thick, physiologically weak middle layer (stroma) covered by an epithelial and an endothelial cell layer. A disorder of the cornea (Keratokonius) leads to a progressing deformation of the natural corneal curvature of the affected eye. In a standard method of treatment, riboflavin and UVA light are used to stiffen the outer region of the collagen-containing stroma by creating collagen cross-links. This helps to increase the strength of the cornea to resist further deformations. We investigated the mechanics of the several hundred micrometer deep stroma of cross-linked and non-cross-linked control corneas with a high depth resolution (Fig. 1A). For the cross-linked corneas, we found an exponentially declining stiffening effect starting from the corneal surface (Fig. 1B) and we identified a maximum depth of effective stiffening within the stroma. There was no significant stiffening in the control corneas.

In the second study, we investigated porcine heart valves, which mainly consist of three layers: two stabilizing outer layers and one physiologically weak inner layer. By performing force mapping on vertical cryosections of single heart valve leaflets, we determined the layer-specific Young's moduli and showed that the two outer layers were about 2 times stiffer than the inner layer. These parameters are useful in cardiac tissue engineering for research on artificial heart valve replacements.

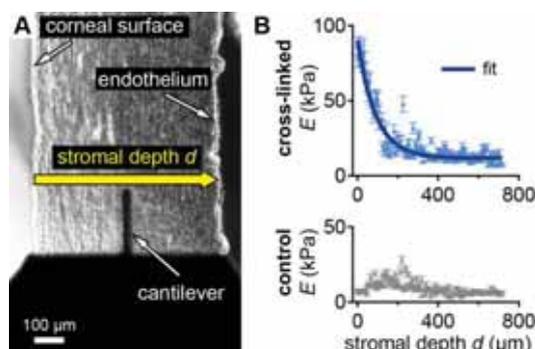


Figure 1: A) Optical image of a cryosection of a cross-linked cornea in the AFM setup. Force mapping was performed along the whole stromal depth starting from the corneal surface towards the endothelium. B) Profile of the Young's modulus along the whole stromal depth of a cross-linked and non-cross-linked control cornea.

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Simultaneous nc-AFM/STM characterization of subsurface defects on B:Si(111)- $\sqrt{3}\times\sqrt{3}$ surface

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B:Si(111)- $\sqrt{3}\times\sqrt{3}$ surface has gained a lot of interest in surface science, due to its prominent electronic and structural properties. Compared to bare silicon surface, this system has reduced chemical reactivity, which makes it a suitable candidate for deposition of molecular complexes without a risk of their decomposition. Here we will report combined nc-AFM/STM measurements using qPlus sensor with atomic resolution of the B:Si(111)- $\sqrt{3}\times\sqrt{3}$ surface supported by total energy DFT calculations. STM reveals slight modulation of the tunneling current over surface area, which we attributed to presence of subsurface boron defects. We combine KPFM and lock-in STS measurements with large scale DFT calculations to characterize position of boron subsurface defects and their influence on surface electronic structure.

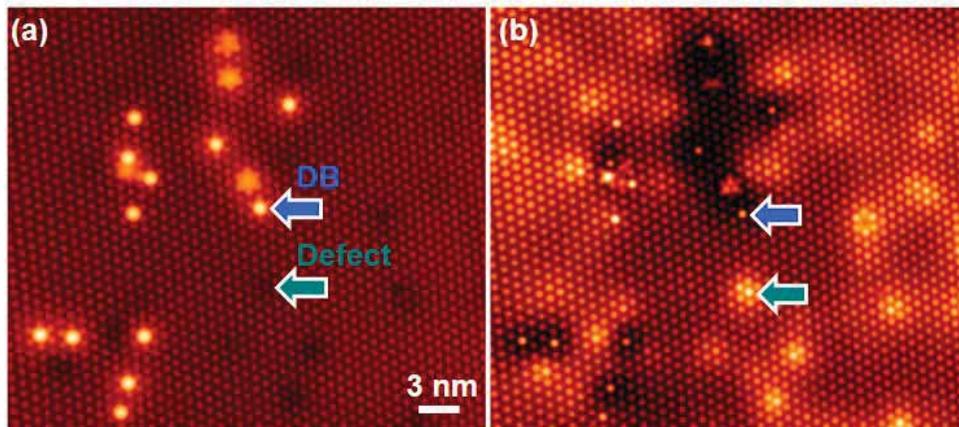


Figure 1. STM image of the empty (a) and filled (b) states of the Si:B $\sqrt{3}\times\sqrt{3}$ surface. $I_s = 0.5$ nA, $V_{\text{empty}} = +1.4$ V, $V_{\text{filled}} = -0.5$ V.

Progressive nitrogen-doping of graphene on SiC(0001)

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Doping of epitaxial graphene on SiC substrates was achieved by direct nitrogen ion implantation and stabilization at temperatures above 1300 °K. Scanning tunneling microscopy reveals very well-defined single substitutional defects on single and bilayer graphene. Repeated nitrogen implantation and stabilization leads to formation of double defects, which comprise of two nitrogen defects in a second-nearest-neighbour (meta) configuration. DFT calculations and scanning tunneling microscopy simulations are used to evaluate the electronic properties and to explain varying contrast of these defects in the atomically-resolved images, depending on the probe type. A mechanism of defect formation is proposed.

Quantum size effects on the work function of Pb islands on Si(111)

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When the thickness of a metal film approaches the *de Broglie* wavelength of the confined electrons quantum size effects become pronounced. This leads to a variation and even an oscillatory behavior of many physical properties as a function on the thickness of the metallic film, such as the electric resistivity, the superconducting critical temperature, the surface free energy, and the work function. Among other systems, Pb on Si(111) has attracted enormous interest since it exhibits a bilayer oscillatory behaviour and flat Pb islands are formed under suitable deposition parameters.

We have studied the influence of quantum size effects on the work function of Pb islands grown on Si(111) with frequency modulation scanning force microscopy (FM-SFM) and Kelvin probe microscopy (KPFM). We have evaporated Pb on the Si(111)- 7×7 surface in ultra-high vacuum at room temperature. To prevent dewetting during the measurements, the sample was cooled down to ~ 110 K. The local work function has been correlated with the height which ranged from four to nine monolayers. We found that the local work-function difference between the islands and the wetting layer shows an even-odd oscillatory dependence on the island's height. The amplitude of the oscillation decreases with increasing film thickness. This is in good agreement with calculations [1] and previous investigations with other techniques [2].

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Hydrogen-bonded networks of triarylamine molecules on noble metal surfaces

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Hydrogen bonding is a highly directional interaction that has successfully been applied towards the construction of complex supramolecular architectures both in bulk and on surfaces. Here, we present a low temperature scanning tunneling microscopy study of hydrogen-bonded networks on noble metal surfaces using triarylamine derivatives functionalized with diaminotriazine and carboxylic moieties as molecular building blocks. While on Au(111) extended hexagonal hydrogen-bonded networks with different pore-sizes are formed, the strong surface-molecule interaction leads to different hydrogen-bonded assemblies on Cu(111). In addition, we performed comparative STM measurements of the same triarylamine networks formed from solution on HOPG in ambient conditions and discuss their structure and stability. Such self-assembled organic nanostructures formed at room temperature and atmospheric pressure are highly appealing with respect to applications.

Cold Tip SPM

A new generation of variable temperature SPM for spectroscopy

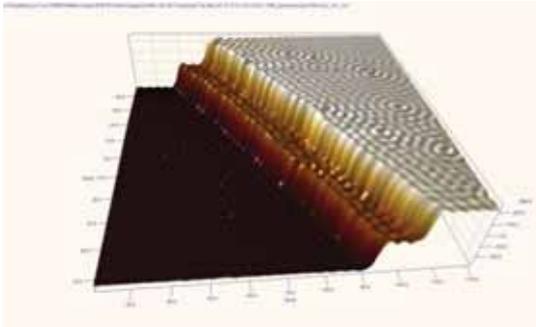
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¹Oxford Instruments Omicron NanoScience, 65232 Taunusstein, Germany

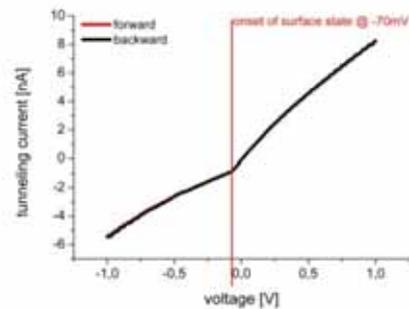
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Abstract: We present design and first results of a new generation of variable temperature scanning probe microscope (SPM) that has been developed to enhance the performance in tunneling spectroscopy at lower and variable temperatures. The new microscope for ultra high vacuum is based on a new stage design using a flow cryostat compatible for cooling with liquid nitrogen or helium. In contrast to earlier established designs of variable temperature SPM's where only the sample is cooled, this new SPM also cools the scanner with tip. This is realized by a new developed compact and microscope stage with thermal shields and a dedicated cooling management system. With the new design we achieve lower temperatures and improve drift by more than an order of magnitude compared to previous variable temperature stages. Sample temperatures down to 10 K (with helium) and 95 K (with nitrogen) have been achieved. The temperature stability is better than 5mK / min and the measured thermal drift is below 1 pm/s. During cooling the mechanical z stability is better than 3 pm. These conditions enhance spectroscopy measurement capability. "Loop off" times of up to 10 s per single spectroscopy curve have been measured. The new flow cryostat also allows for changing between nitrogen cooling and helium cooling in less than 90 min during a running experiment. Pre-cooling with nitrogen during the starting phase of an experiment also reduces running costs for liquid helium. This new SPM is configured for imaging in STM as well as AFM with a non-optical sensor in a temperature range between 10-400 K. Switching between the 2 modes can be accomplished without breaking vacuum.

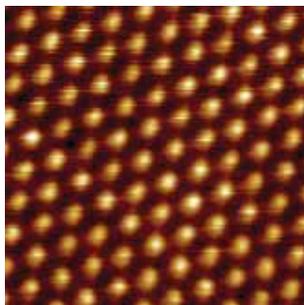
Figures:



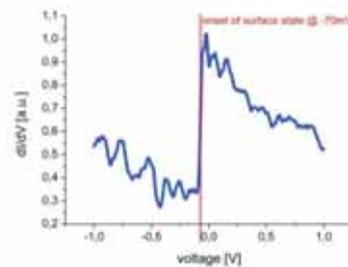
STM on Ag(111) at 10K showing electron standing waves



I(V) on Ag(111) at 10K, loop off for 10s during voltage ramp from -1V to 1V and back to -1V



STM on Au(111) at 95K using nitrogen
z stability < 3pm



Simultaneous measured dI/dV on Ag(111) at 10K , loop off for 10s

Exploring the retention properties of CaF₂ nanoparticles as possible additive in dental care application with tapping-mode atomic force microscope in liquid

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The addition of fluoride in dental care products has a beneficial influence on the reduction of caries progression [1].

CaF₂ nanoparticles have been shown to have the ability for reducing caries by acting as a potential source of fluoride preventing teeth demineralization [2,3]. The anti-caries activity of calcium fluoride nanoparticles is mainly determined by two factors, the solubility of the nano-composite at a certain pH and their adhesion strength to the teeth enamel upon application. In this work we explored the retention properties of as-synthesized calcium fluoride nanoparticles adsorbed on mica and on polished teeth enamel with amplitude-modulation atomic force microscopy (AM-AFM) in liquid. By recording the phase lag of the excitation signal with respect to the vibrating tip at the point the particles were manipulated, we were able to calculate the power needed to dislocate the particles [4]. This enabled us to examine the substrate dependent adhesion properties of CaF₂ nanoparticles which vary in size, shape and surface morphology. We show that the same CaF₂ nanoparticles show up to ten times higher retention on teeth enamel compared when adsorbed on mica [5].

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