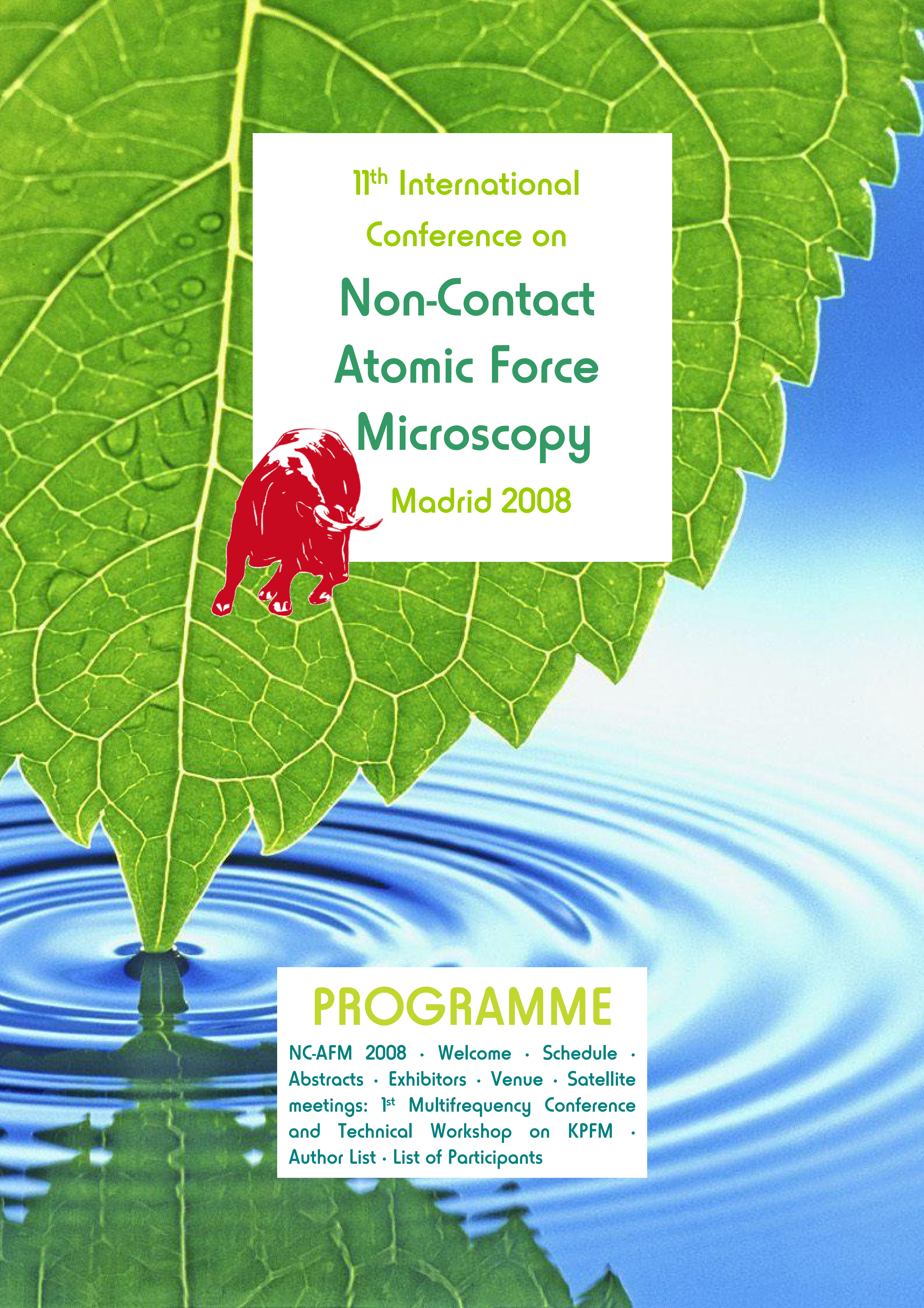

11th International Conference on

# Non-Contact Atomic Force Microscopy

Madrid 2008

## PROGRAMME

NC-AFM 2008 · Welcome · Schedule · Abstracts · Exhibitors · Venue · Satellite meetings: 1st Multifrequency Conference and Technical Workshop on KPFM · Author List · List of Participants

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CSIC
CONSEJO SUPERIOR DE INVESTIGACIONES CIENTIFICAS

GOBIERNO DE ESPAÑA
MINISTERIO DE CIENCIA E INNOVACIÓN

Comunidad de Madrid

¡MADRID!
MADRID CONVENTION BUREAU

## Past Conferences

Osaka, Japan (1998)
Pontresina, Switzerland (1999)
Hamburg, Germany (2000)
Kyoto, Japan (2001)
Montreal, Canada (2002)
Dingle, Ireland (2003)
Seattle, USA (2004)
Osnabrück, Germany (2005)
Kobe, Japan (2006)
Antalya, Turkey (2007)

## Forthcoming Conference

Yale, USA (2009)

# 1 Welcome

Welcome to the 11th International Conference on Non-Contact Atomic Force Microscopy in Madrid. The 2008 meeting has attracted a large number of participants (almost 170 registrations at the time of writing these lines) that will enjoy a strong programme with 128 scientific contributions (including 44 oral presentations) and 5 exhibition booths. These figures reflect the maturity of the field, where an increasing number of groups are developing a strong activity that involves both new challenging methodologies and applications, and that covers areas well beyond the UHV-imaging niche where NC-AFM was born.

This year's programme confirms the tendencies already seen in the 2007 Conference in Antalya. (1) The development of sophisticated force spectroscopy methodologies that are able to map the complete 3D tip-sample force field on different surfaces. (2) Great achievements in the resolution obtained with KPFM (reaching, in some cases, the atomic scale) are accompanied by a thorough, quantitative understanding of the contrast observed. (3) Atomic resolution imaging on insulating substrates is now a mature technique capable of making significant contributions to the microscopic understanding of the properties and chemical activity of these surfaces. (4) New instrumental and methodological developments pave the way to the characterization of the magnetic and electronic properties of nanostructures. (5) Last but not least, the different dynamic modes for high-resolution operation in liquids are already making a significant impact in the highly-competitive field of biological imaging under physiological conditions.

The scientific sessions of NCAFM-2008 (running from Tuesday 16th to Friday 19th, September, 2008) are complemented by two satellite meetings that will take place prior to the conference during Sunday 14th and Monday 15th: The 1st Multi-frequency AFM Conference and The Technical Workshop on Kelvin Probe Force Microscopy. We have been happily surprised by the large demand (74 and 85 participants respectively) for these focused sessions that, we hope, will further contribute to the high-quality programme of NCAFM-2008. We would like to thank Thilo Glatzel and Sascha Sadewaser for their initial suggestion and the invaluable contribution to put together the KPFM programme.

The conference in Madrid has been made possible by the enthusiastic contribution of the Local Organizing Committee. Special thanks go to Pablo Pou for his work as Scientific Secretary, and to Giulio Biddau for maintaining the Conference Web site. As organizers, we very much appreciate the financial support of the Universidad Autónoma de Madrid (UAM) and the local sponsors: Comunidad de Madrid (Madrid Regional Government) and Ayuntamiento de Madrid (Madrid City Council). We are extremely fortunate to have the collaboration of Udo Schwarz and the help of the publishing team at the Institute of Physics (IOP) in order to disseminate the results of the conference through a Topical Issue of the journal Nanotechnology and the on-line publication of the Proceedings as part of the Journal of Physics Conference Series.

We hope that you will enjoy the Conference and have a wonderful time in Madrid.

Rubén Pérez and Ricardo García

# Contents

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# Committees

## International Steering Committee

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Franz J. Giessibl Regensburg Universität (Germany)

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Ricardo García
Instituto de Microelectrónica de Madrid (Spain)

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Jose R. Lozano
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Elena Tomas
Inst. de Microelectrónica de Madrid (Spain)

# General Information

## Registration:

The registration desk at the conference venue is open 8:00-11:00 (14th September), 17:30-20:00 (15th September) and 8:30-9:00 (16th - 19th September). Onsite registration is also possible.

## Welcome party:

All participants are invited to a welcome party at the conference venue from 19:00 to 20:00 (15th September)

## Conference Hall:

Eating and smoking are not allowed in the conference hall.

## Oral Presentations:

The time allocated for the contributed papers is 20 minutes including 5-minute discussion. An LCD projector is available in the conference room. Please note that overhead projectors, 35-mm slide projectors or any other equipment are NOT available.

## Poster Presentations:

Two Poster sessions will be held during the conference. The poster boards are 153 cm height and 90 cm wide. Pins will be available for mounting a poster on the board. Authors are required to stand at their posters during the sessions.

## Banquet:

Conference banquet will be held on 18th September (20:00) at the Restaurant Pedro Larumbe, C/ Serrano 61 4th floor, 28006 Madrid, Spain, tel. 915 751 112 (www.larumbe.com)

## Internet access:

Wireless internet access is available in the conference venue. Username and password will be provided at the registration desk

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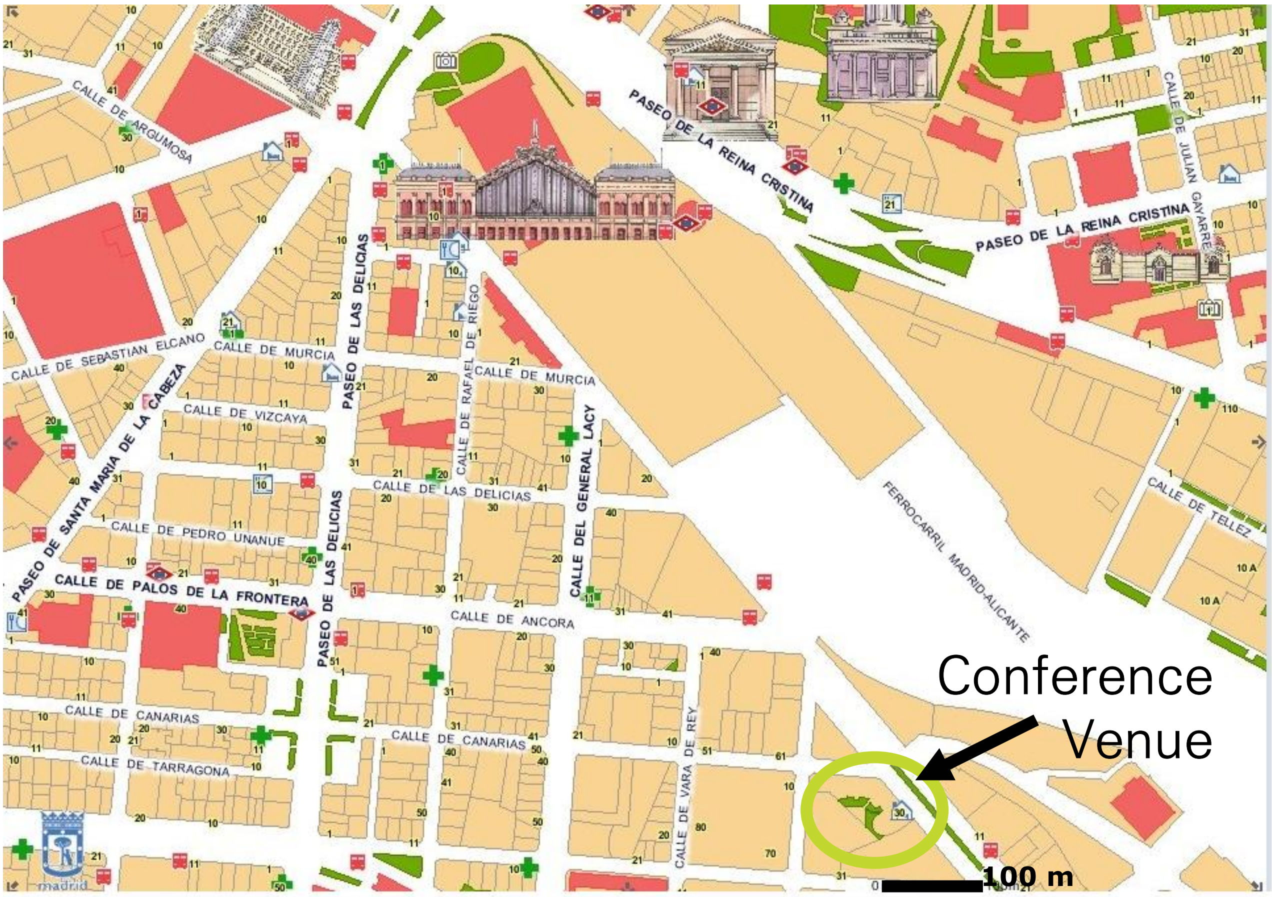
# Local Maps

## CONFERENCE VENUE

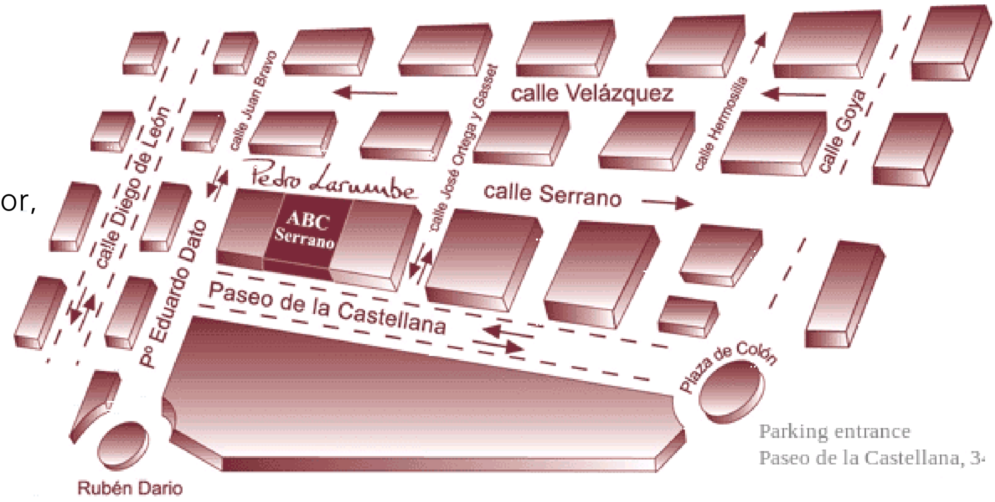
Hotel Rafael Atocha



## BANQUET

Pedro Larumbe
Restaurante

C/ Serrano 61, 4th floor,
28006 Madrid, Spain
tel 915 751 112



# 6 Exhibitors

NANONIS
The SPM Application Experts

NANOTEC
ELECTRONICA

Omicron
NanoTechnology

RHK Technology
Imaging the Future of Nanoscience

Veeco

- Novel Instrumentation and techniques in dynamic AFM.
- Small amplitude and lateral force measurements
- Atomic resolution imaging on surfaces and molecular systems.
- Highest resolution imaging of clusters, biomolecules and biological systems.
- Atomic- and molecular-scale manipulation.
- Theoretical analysis of contrast mechanisms. Forces and tunnelling phenomena.
- Simulation of images and virtual SFM systems.
- Measuring tip-sample interaction potentials and mapping force fields.
- Mechanisms for damping and energy dissipation.
- Amplitude modulation versus Frequency modulation imaging.
- Imaging and spectroscopy in liquid environments.
- Measuring nanoscale charges, work function and magnetic properties.
- Characterisation and modification of force microscopy tips at the atomic scale.

# Topics

# Conference Programme

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# NC-AFM 2008 Sessions:

- Force Spectroscopy I:
  Tuesday 16, 9:20 – 10:40
- KPFM I:
  Tuesday 16, 11:20 – 12:40
- Manipulation:
  Tuesday 16, 14:30 – 15:50
- Oxides:
  Wednesday 17, 9:00 – 10:40
- KPFM II:
  Wednesday 17, 11:20 – 12:40
- Liquids and biomolecules I:
  Wednesday 17, 14:30 – 15:50
- Magnetic and electronic properties:
  Thursday 18, 9:00 – 12:00
- Liquids and biomolecules II:
  Thursday 18, 12:00 – 13:00
- Insulators and molecules:
  Friday 19, 9:00 – 10:40
- Force Spectroscopy II:
  Friday 19, 11:20 – 12:40

| | Sunday 14, Sept. | Monday 15, Sept. | Tuesday 16, September | Wednesday 17, September | Thursday 18, September | Friday 19, September |
|---|---|---|---|---|---|---|
| *8:30* | ***1st Multi-Frequency AFM Conference*** | ***1st Multi-Frequency AFM Conference*** | *Registration* | *Registration* | *Registration* | *Registration* |
| **9:00** | | | Opening Remarks | M. Reichling | A. Schwarz | T. Filleter |
| 9:20 | | | M. Ashino | J. V. Lauritsen | H. Holscher | S. Gauthier |
| 9:40 | | | S. A. Ghasemi | M. Heyde | G.Rubio-Bolllinger | P. Rahe |
| 10:00 | | | U. D. Schwarz | H. P. Pinto | Y. Miyahara | S. Burke |
| 10:20 | | | Y. Sugimoto | R. Bechstein | S. Huant | S. Torbrugge |
| *10:40*<br>*40 min* | | | *Coffee Break* | *Coffee Break* | *Coffee Break* | *Coffee Break* |
| **11:20** | | | S. Sadewasser | Ch. Loppacher | P. Jelinek | R. Hoffmann |
| 11:40 | | | Th. Glatzel | E. Tsunemi | B. Uder | O. Custance |
| 12:00 | | | S. Watanabe | P. Milde | H. Yamada | M. Abe |
| 12:20 | | | A. Baratoff | R. Bennewitz | W. Hofbauer | F. J. Giessibl |
| ***12:40*** | | | ***Lunch***<br>*110 min*<br>*(12:40-14:30)* | ***Lunch***<br>*110 min*<br>*(12:40-14:30)* | E. Sahagun | Closing Remarks |
| *13:00* | | *14:00-19:00*<br>***KPFM Workshop*** | | | ***Lunch***<br>*(13:00- )* | |
| **14:30** | | | Y. Sugawara | T. Fukuma | | |
| 14:50 | ***1st Multi-Frequency AFM Conference*** | | P. Pou | N. Oyabu | | |
| 15:10 | | | A. Foster | N. F. Martinez | | |
| 15:30 | | | T. Trevethan | M. Tsukada | | |
| **15:50**<br>**\|**<br>**18:00** | | | Poster Session<br>I<br>*Coffee Service*<br>*(15:50-18:00)* | Poster Session<br>II<br>*Coffee Service*<br>*(15:50-18:00)* | | |
| | | ***19:00 - 20:00***<br>*Registration*<br>*Welcome*<br>*Party* | | | ***20:00 - 23:30***<br>*Banquet* | |

# Sunday, 14 September

## 1st MULTIFREQUENCY AFM CONFERENCE

September 14-15, 2008
Madrid (Spain)

| Time | Speaker | Abstract Title |
|---|---|---|
| 8.30-8:40 | **Ricardo García** | Welcome |
| 8:40-9.15 | **Roger Proksch (I. E.)** | |
| 9.15-9.50 | **Stephen Jesse (.I E.)** | Sinc or Sine: Band Excitation Method in SPM |
| 9.50-10.10 | **Richard Arinero** | Recent advances in Amplitude-Controlled AFM under vacuum |
| 10.10-10.30 | **Raoul Enning** | Controlling bistability in AFM through multifrequency excitation |
| **10.30-11.00** | Coffee Break | |
| 11.00-11.20 | **Nicolás F. Martinez** | Bimodal AFM imaging with sub-pico Newton force sensitivity |
| 11.20-11.40 | **Elena T. Herruzo** | Bimodal atomic force microscopy of proteins in water |
| 11.40-12.00 | **José R. Lozano** | Theory of Multifrequency Atomic Force Microscopy |
| 12.00-12-20 | **Alex M. Gigler** | Time-traces and Fourier analysis of bimodal atomic force microscopy |
| **12.20-12-30** | Break | |
| 12.30-13.05 | **Arvind Raman (I.E.)** | Higher eigenmodes in the AFM: Laser vibrometry and momentary excitation in liquids |
| 13.05-13.25 | **Johannes Preiner** | Higher Harmonics Imaging in Liquids |
| **13.25-15.00** | lunch | |
| 15.00-15.20 | **Robert Stark** | Numerical Simulations of Bimodal AFM |
| 15.20-15.40 | **Alvaro San Paulo** | Multifrequency atomic force microscopy for dynamic characterization of nanoelectromechanical systems |
| 15.40-16.00 | **David Haviland** | Intermodulation AFM |
| 16.00-16.20 | **Carsten Hutter** | Theoretical Framework for Intermodulation Spectroscopy |
| **16.20-16.50** | Coffee Break | |
| 16.50-17.10 | **Huiling Li** | Harmonic concentrated-mass cantilevers for tapping-mode atomic force microscopy |
| 17.10-17.30 | **Seref Burak Selvi** | Accuracy of elastic modulus prediction from higher harmonic data of a tapping mode harmonic cantilever |
| 17.30-17.50 | **Ali Fatih Sarioglu** | Time-resolved force measurements and material mapping using differential interferometric force sensors |

# Monday, 15 September

| Time | Speaker | Abstract Title |
|---|---|---|
| 8.20-8.55 | **Hendrik Hölscher (I. E.)** | Quantitative measurement of tip-sample interactions in amplitude- and phase modulation AFM |
| 8.55-9.15 | **Allard J. Katan** | Quantitative force versus distance measurements in Amplitude Modulation AFM: a novel force inversion technique |
| 9.15-9.50 | **Ozgur Sahin (I. E.)** | Theory and applications of time-varying tip simple force measurementes in tapping-mode AFM |
| 9.50-10.10 | **Samuel Lesko** | New scanning probe microscopy technique for high resolution mapping of material properties |
| 10.10-10.30 | **Philippe Leclere** | Quantitative mechanical properties of pressure sensitive adhesives determined by Torsional Harmonic Cantilevers operated in tapping Mode |
| 10.30-11.00 | Coffee Break | |
| 11.00-11.35 | **Tilman Schaefer (I. E.)** | Optimizing Optical Detection in Multimodal AFM |
| 11.35-11.55 | **Aykutlu Dana** | Parametrically Coupled Multiharmonic Force Imaging |
| 11.55-12.15 | **Dominik Ziegler** | Multifrequency Kelvin Probe Method to Compensate Electrostatic Forces in AFM |
| 12.15-12.25 | Break | |
| 12.25-12.45 | **M. Teresa Cuberes** | Detection of ultrasonic excitation at lower frequency using atomic force microscopy cantilevers |
| 12.45 -13.05 | **Franz J. Giessibl** | |
| 13.05-13.10 | **Ricardo García** | Coming Events |

# Technical Workshop on KPFM

*Chair: Th. Glatzel*

14:00 Kelvin Probe Microscopy: A comparison of differert methods and their resolution in experiement and simulation
Christian Loppacher, *Universites Paul Cezanne, 13397 Marseille, FRANCE*

14:45 Quantitative KPFM: Semiconductors and self-assembled monolayers
Yossi Rosenwaks, T*el-Aviv University, Ramat-Aviv, 69978, ISRAEL*

15:30 Atomistic simulations of AFM junctions using SciFi code with possible applications for KPFM
Lev Kantorovich*, King's College London, London WC2R 2LS, UNITED KINGDOM*

16:15 *Coffee break*

*Chair: S. Sadewasser*

16:45 Some aspects of high –resolution imaging in KPFM
Laurent Nony, *Universites Paul Cezanne, 13397 Marseille, FRANCE*

17:30 Charge Ttansfer induced by adatoms and admolecules
Hiroshi Onishi, *Kobe University, Kobe 657-8501, JAPAN*

18:15 Discussion
*Chairs:*
Adam Foster, Helsinki University of Technology, FIN-02015 HUT, FINLAND
Roland Bennewitz, Leibniz Institut fur Neue Materialien, Saarbrucken, GERMANY

Venue: Hotel Rafael Atocha

# Tuesday, 16 September

| | | |
|---|---|---|
| 09:00 | *Opening Remarks*<br>R. Perez and R. Garcia | |

## Force Spectroscopy I

### Chair: E. Meyer

| | | |
|---|---|---|
| 09:20 | *Atomic-Resolution 3D Force and Damping Maps on Nanotube Peapods*<br>M. Ashino, D. Obergfell, S. Roth, S. Berber, D. Tomanek, R. Wiesendanger | 25 |
| 09:40 | *Ubiquitous Mechanisms of Energy Dissipation in Non-Contact Atomic Force Microscopy*<br>S. A. Ghasemi, A. Baratoff, S. Goedecker, T. Lenosky, E. Meyer, H. J. Hug | 26 |
| 10:00 | *Three-Dimensional Force Imaging and Quantification with Atomic Resolution*<br>U. D. Schwarz, B. J. Albers, T. C. Schwendemann, M. Z. Baykara, N. Pilet, E. I. Altman | 27 |
| 10:20 | *Vertical and lateral force mapping on the Si(111)-(7x7) surface by dynamic force microscopy*<br>Y. Sugimoto, T. Namikawa, K. Miki, M. Abe, S. Morita | 28 |
| 10:40 | Coffee | |

## KPFM I

### Chair: H. Onishi

| | | |
|---|---|---|
| 11:20 | *Distinct short-range electrostatic interaction on Si and substitutional Pb atoms at the Si(111)-(7x7) surface*<br>S. Sadewasser, O. Custance, Y. Sugimoto, M. Abe, S. Morita | 29 |
| 11:40 | *Atomic scale Kelvin Probe Force Microscopy Studies of the Surface Potential Variations on the TiO2(110) surface*<br>T. Glatzel, G.H. Enevoldsen, M.C. Christensen, J.V. Lauritsen, E. Meyer, F. Besenbacher | 30 |
| 12:00 | *Quantum Mechanical Simulation of Kelvin Probe Force Microscopy for a Simple Metal Surface*<br>S. Watanabe, S. Shibata, T. Tada | 31 |
| 12:20 | *Theory of atomic scale contact contrast in amplitude modulation Kelvin probe force microscopy - application to Si(111)-7x7*<br>A. Baratoff, S. Kawai, T. Glatzel, E. Meyer | 32 |
| 12:40 | Lunch | |

## Manipulation

### Chair: F. J. Giessibl

| | | |
|---|---|---|
| 14:30 | *Atom Manipulation on Cu(110)-O Surface with Low Temperature Noncontact AFM*<br>Y. Sugawara, S. H. Lee, Y. Naitoh, Y. J. Li, M. Kageshima | 33 |
| 14:50 | *Unveiling the atomic processes during the manipulation of single atoms at semiconductor surfaces using the FM-AFM in the repulsive regime*<br>P. Pou, P. Jelinek, Y. Sugimoto, M. Abe, O. Custance, S. Morita, R. Perez | 34 |
| 15:10 | *Water chemistry and manipulation on alkaline earth halide surfaces*<br>A. S. Foster, S. Hirth, F. Ostendorf, M. Reichling | 35 |
| 15:30 | *Controlling the motion of large organic molecules on insulating surfaces through molecular structure and localised forces*<br>T. Trevethan, A. Shluger | 36 |

## Poster Session I

### Chair: A. Oral

| | | |
|---|---|---|
| 15:50 | *Poster session I & Coffee Service (15:50 – 18:00)* | PI |

# Wednesday, 16 September

## Oxides

*Chair: A. Shluger*

| | | |
|---|---|---|
| 09:00 | *Characterizing reactivity of water molecules on CeO2(111) using atomic force microscopy*<br>M. Reichling, S. Torbruegge, O. Custance, S. Morita | 39 |
| 09:20 | *Atomic-scale ordering of the a-Al2O3(0001) surface studied with atom-resolved nc-AFM*<br>J. V. Lauritsen, M. C. Christensen, K. Venkaramani, B. Hinnemann, S. Helveg, F. Besenbacher | 40 |
| 09:40 | *Beyond Imaging: Dynamic Force Spectroscopy on Thin MgO Films*<br>M. Heyde, T. Koenig, G. H. Simon, H.-J. Freund | 41 |
| 10:00 | *Simultaneous Atom-Resolved AFM and STM Studies of the Hydroxylated TiO2 (110) Surface*<br>H. P. Pinto, G. H. Enevoldsen, M. C. Christensen, W. Hofer, J. V. Lauritsen, A. S. Foster, F. Besenbacher | 42 |
| 10:20 | *"All-inclusive" imaging of TiO2(110)*<br>R. Bechstein, C. Gonzalez, P. Jelinek, R. Perez, A. Kuehnle | 43 |
| 10:40 | Coffee | |

## KPFM II

*Chair: O. Custance*

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| 11:20 | *Interface Dipole Formation of Different ZnPcCl8 Phases on Ag(111) Observed by Kelvin Probe Force Microscopy*<br>C. Loppacher, P. Milde, U. Zerweck, L. M. Eng, M. Abel, L. Giovanelli, L. Nony, M. Mossoyan, L. Porte | 44 |
| 11:40 | *Development of Two-probe Frequency Modulation AFM/KFM for Nanometer-scale Electrical Measurement*<br>E. Tsunemi, N. Satoh, K. Kobayashi, K. Matsushige, H. Yamada | 45 |
| 12:00 | *Interface and single molecule analysis of organic molecules in a Ferroelectric-OFET*<br>P. Milde, K. Haubner, U. Zerweck, D. Koehler, E. Jaehne, L. M. Eng | 46 |
| 12:20 | *Kelvin probe force microscopy measurements of epitaxial graphene*<br>R. Bennewitz, T. Filleter, J. McChesney, A. Bostwick, E. Rotenberg, T. Seyller | 47 |
| 12:40 | Lunch | |

## Liquids and Biomolecules I

*Chair: J. Colchero*

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|---|---|---|
| 14:30 | *High-speed Frequency Modulation Atomic Force Microscopy in Liquids*<br>T. Fukuma | 48 |
| 14:50 | *High-resolution Imaging of Biomolecules in Liquids by FM-AFM*<br>N. Oyabu, S. Ido, K. Kimura, K. Kobayashi, Y. Hirata, H. Yamada | 49 |
| 15:10 | *Bimodal atomic force microscopy imaging of isolated antibodies*<br>N. F. Martinez, J. R. Lozano, E. T. Herruzo, F. Garcia, R. Garcia | 50 |
| 15:30 | *Theoretical simulation of dynamic mode AFM in water*<br>M. Tsukada, K. Tagami, N. Watanabe | 51 |

## Poster Session II

*Chair: A. Schwarz*

| | | |
|---|---|---|
| 15:50 | *Poster session II & Coffee Service (15:50 – 18:00)* | PII |

# Thursday, 18 September

## Magnetic and Electronic Properties

*Chair: U. D. Schwarz*

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11:40 *QPlus AFM on single crystal insulators with small oscillation amplitudes at 5 K* 61
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## Liquids and Biomolecules II

*Chair: T. Fukuma*

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12:20 *Small Amplitude Dynamic Force Microscopy of Liquids Near a Solid Surface* 63
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12:40 *Adhesion Hysteresis and Capillary Interactions in Dynamic Atomic Force Microscopy* 64
E. Sahagun, J. J. Saenz

13:00 Lunch

# Friday, 19 September

## Insulators and Molecules

*Chair: M. Reichling*

| | | |
|---|---|---|
| 09:00 | *Heteroepitaxy of ultrathin films studied by high-resolution nc-AFM*<br>T. Filleter, R. Bennewitz, S. Maier, W. Paul, T. Glatzel, O. Pfeiffer, E. Meyer | 67 |
| 09:20 | *Evidence for tip structural reversible transformations during NC-AFM imaging on KBr(001)*<br>S. Gauthier, M. A. Venegas de la Cerda, J. Abad, D. Martrou | 68 |
| 09:40 | *Repulsive interaction and contrast inversion in non-contact atomic force microscopy imaging of adsorbates*<br>P. Rahe, R. Bechstein, J. Schuette, F. Ostendorf, F. Loske, A. Kuehnle | 69 |
| 10:00 | *Molecule–Insulator interactions: Growth, epitaxy and electronic properties of PTCDA on NaCl*<br>S. Burke, W. Ji, J. LeDue, J. Topple, S. Fostner, J. Mativetsky, H.-J. Gao, H. Guo, P. Grutter | 70 |
| 10:20 | *Resolving atomic scale features of a polar metal oxide surface*<br>S. Torbrugge, F. Ostendorf, M. Reichling | 71 |
| 10:40 | Coffee | |

## Force Spectroscopy II

*Chair: S. Morita*

| | | |
|---|---|---|
| 11:20 | *Atomic Scale Force Vector Fields on NaCl-type (001) surfaces*<br>R. Hoffmann, K. Ruschmeier, D. Weiner, A. Schirmeisen | 72 |
| 11:40 | *Force spectroscopy using cantilever higher flexural modes*<br>O. Custance, Y. Sugimoto, M. Abe, S. Morita | 73 |
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| 12:20 | *Qplus, quo vadis?*<br>F. J. Giessibl | 75 |

| | |
|---|---|
| 12:40 | *Closing Remarks*<br>R. Perez and R. Garcia |

# Poster Session I

Tuesday
16 September

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## Force Spectroscopy

## KPFM

## Manipulation

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# Oral Presentations

## Tuesday, 16 September

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# Tuesday,
## 16 September

# Atomic-Resolution 3D Force and Damping Maps on Nanotube Peapods

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Single-walled carbon nanotubes (SWNTs) consist of single graphene sheets rolled up into cylinders with nanometer-scale diameters. Nanotube Peapods are created by encapsulating fullerene molecules inside the hollow cores of SWNTs. Using three-dimensional force field spectroscopy (3D-FFS), we have found that the short-range inter-atomic van der Waals forces are dominant for atomic-scale contrasts over the SWNTs [1]. Here, we report that atomic-resolution 3D mapping of short-range forces $F_{\mathrm{sht}}$ and damping $U_{\mathrm{dmp}}$ reveal sub-surface structures of the nanotube Peapods in the atomic scale [2]. Furthermore, we discuss correlations between $F_{\mathrm{sht}}$ and $U_{\mathrm{dmp}}$ to understand their detailed mechanisms.

The surface topographies obtained over the nanotube Peapods using dynamic AFM in ultrahigh vacuum (< $10^{-10}$ mbar) and low temperature (< 13 K) represent undulating features with atomically-superimposed corrugations. 3D mapping of $F_{\mathrm{sht}}(x, y, z)$ and $U_{\mathrm{dmp}}(x, y, z)$ over the area (1.2 nm x 1.2 nm) including a topographic protrusion demonstrates the specific features originating from the inner fullerene molecules, as shown in Figs. 1 and 2: Figure 1 corresponds to one section, i.e. $F_{\mathrm{sht}}(x, \mathrm{z})$ (upper) and $U_{\mathrm{dmp}}(x, z)$ (bottom). The distance z dependences (i.e. lines in Fig. 1) are plotted in Fig. 2.

1) M. Ashino, A. Schwarz, T. Behnke, R. Wiesendanger, *Phys. Rev. Lett.* **93**, 136101 (2004).
2) M. Ashino *et al.*, Nature Nanotech. (in press).

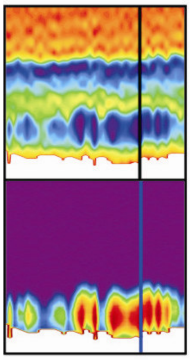

Fig. 1.

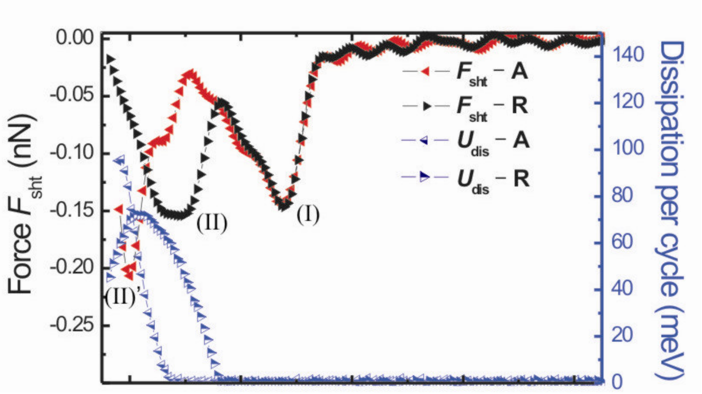

Fig. 2.

# Ubiquitous Mechanisms of Energy Dissipation in Non-Contact Atomic Force Microscopy

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Atomistic simulations considering larger tip structures than hitherto assumed reveal novel dissipation channels in near-contact Atomic Force Microscopy [1]. The potential energy surfaces of realistic silicon tips exhibit many energetically close local minima that correspond to different configurations. Many of them easily deform, thus causing dissipation arising from hysteresis in force vs. distance characteristics below a critical approach distance [2]. Above that distance thermally activated jumps can occur [3]; moreover, saddle points which connect local minima can suddenly switch to connect different minima. Configurations driven into metastability by the tip motion can then relax into lower energy structures. Thermally activated jumps between temporarily equilibrated groups of tip configurations cause an intermittent redistribution of their populations and sudden energy losses, albeit not in every oscillation cycle. Somewhat noisy, but stable images and dissipation can nevertheless be recorded in a finite distance range without irreversible damage. Indeed, sudden losses are averaged over the relatively long response time of the amplitude controller or the even longer recording time at each pixel. Sudden contrast changes on otherwise defect-free areas, often attributed to “tip changes” are caused by activated jumps over barriers higher than those likely to be surmounted during the measuring time.

The phenomena in question occur if the short-range tip-sample interaction is sufficiently strong to modify the potential energy landscape of the whole system. This is the case if the tip and sample are made of the same material, e.g. silicon in our simulations. However, more general validity is expected because the softer material is likely to be transferred to the other side in some of the initial oscillation cycles in the near-contact range.

[1] S.A. Ghasemi et al., Phys. Rev.Lett., in press
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[3] L. N. Kantorovich and T. Trevethan, Phys. Rev. Lett. 93, 236102 (2004)

# Three-Dimensional Force Imaging and Quantification with Atomic Resolution

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Atomic resolution images in noncontact atomic force microscopy (NC-AFM) reflect planes of constant frequency shift. To draw conclusions on the chemical activity at specific surface sites, however, the force acting between tip and sample should be known locally rather than the frequency shift. This is not an easy translation due to the non-linear nature of the relationship between the two.

To overcome this problem, several groups have developed an extension to NC-AFM, *dynamic force spectroscopy*, which allows the precise, distance-dependent measurement of tip-sample forces. The forces are determined from frequency shift versus distance curves by mathematical analysis. Even though this approach had some success, prior attempts resulted either only in two-dimensional atomic resolution force *xz*-maps or in data sets of relatively low resolution, as long-term drift stability has been a problem.

Using our recently completed home-built low temperature, ultrahigh vacuum NC-AFM [1], we were able to map the complete three-dimensional (3D) force field over a surface virtually drift free. Simultaneously, the tip-sample interaction potential and the energy dissipation of the oscillation process were obtained. As a test material, we used highly oriented pyrolytic graphite due to its importance as a carbon-based, $sp_2$-bonded model material. Individual data points have been acquired over a surface area comprising several unit cells in a 3D grid with about 2 pm grid size in vertical and <7 pm in lateral direction. From this data set, representations of planes in any direction can be produced. While constant height force maps show atomic resolution with pN force resolution, vertical force maps visualize how the attractive force fields of the atoms extend into the vacuum space. An in-depth discussion of the obtained data will highlight differences between the contrast mechanisms in the dissipation and the force data channels as a function of the tip-sample distance, which give rise to complementary information. We expect that the technique will find applications in fields of science where a local knowledge of interaction forces is beneficial, such as catalysis, chemical imaging, thin film growth, device fabrication, and tribology.

[1] B. J. Albers, M. Liebmann, T. C. Schwendemann, M. Z. Baykara, M. Heyde, M. Salmeron, E. I. Altman, and U. D. Schwarz. Rev. Sci. Instrum. **79,** 033704 (2008).

# Vertical and lateral force mapping on the Si(111)-(7×7) surface by dynamic force microscopy

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The topographic image of dynamic force microscopy (DFM) keeping the frequency shift ($\Delta f$) constant at tiny cantilever oscillation amplitude ($A$) corresponds to the constant-vertical force ($F_z$) gradient surface, while the interpretation becomes complicated at larger $A$. We discuss how $F_z$ and the potential energy ($U$) act on the tip during DFM topographic scan at various $A$ by measuring the $\Delta f$ map on the Si(111)-(7×7) surface at room temperature as shown in Fig.1. The $\Delta f$ map with high spatial resolution [(1024×1024) points] is numerically converted into $F_z$ and $U$ maps [1]. And DFM topographic curves with keeping the $\Delta f$ constant at various $A$ are numerically derived using the experimentally obtained $F_z$ map. One of the important findings is that the corrugation of the topographic profile is drastically enhanced with the decrease of $A$ [2]. The derived topographic profiles in Fig.2 shows that the atomic corrugation at $A$=0.1 Å (small $A$ limit) is almost twice larger than that at $A$=100 Å (large $A$ limit). In addition, we investigate how the lateral force ($F_x$), which plays an important role in the friction and lateral atom manipulation, acts on the tip at various tip-surface distances on various surface sites by the $F_x$ map converted from the $\Delta f$ map. Furthermore, it is demonstrated that lateral force microscopy (LFM) performed at $A$=1 Å enables direct measurement of the $F_x$ gradient by numerically deriving the $\Delta f$ caused by $F_x$. Thus, the force mapping measurements provide a great deal of information about not only the tip-surface interaction but also the imaging mechanism of DFM/LFM.

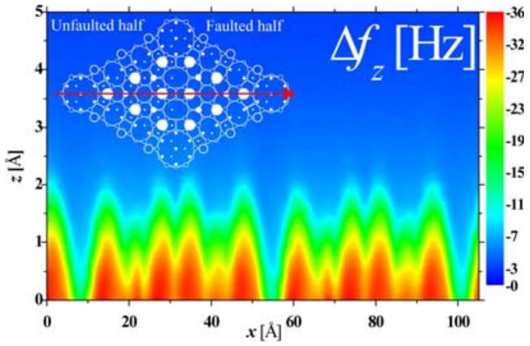

Fig. 1

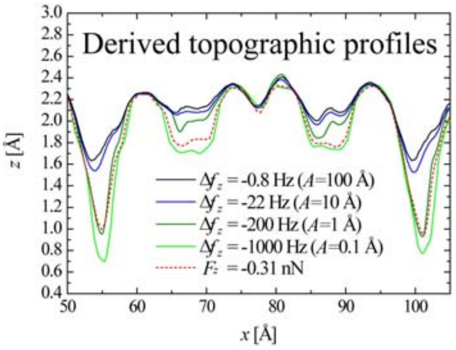

Fig.2

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2) Y. Sugimoto, et al. Phys. Rev. B 77 (2008) 195424.

# Distinct short-range electrostatic interaction on Si and substitutional Pb atoms at the Si(111)-(7x7) surface

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Recently, several Kelvin probe force microscopy (KPFM) studies have shown atomic contrast in contact potential difference (CPD) images [1-4]. Different proposals have been made [5, 6] to explain the observed contrast as due to artifacts of the measurement method.

Using low temperature non-contact atomic force microscopy (NC-AFM) and KPFM, in this contribution we unambiguously show the presence of CPD changes on atomic scale. In our experiments, the CPD is extracted from the minimum of a parabolic fit to the recorded frequency shift ($\Delta f$) as a function of sample bias voltage, $\Delta f(V_s)$. We performed such measurements using 3D-mode imaging [7], recording $\Delta f(V_s)$ as a function of the lateral tip position in constant height tip displacements and as a function of the tip-surface distance above different sites of the Si(111)-(7×7) unit cell containing substitutional Pb adatoms. Clear variations of the CPD between Si adatom sites and substitutional Pb atoms are observed. When using KPFM with an active feedback on the sample bias to adjust the local CPD value, almost identical dependencies of the CPD on the tip-surface distance are obtained.

We discuss the experimental results in view of several mechanisms to understand the local CPD variation on the atomic scale, as local dipoles in the Si(111)-(7×7) surface, induced dipoles in the tip, and tip induced charge transfer between different dangling bond states.

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4) T. Eguchi, et al. *Phys. Rev. Lett.* **93**, 266102 (2004).
5) T. Arai, M. Tomitori, *Phys. Rev. Lett.* **93**, 256101 (2004).
6) K. Okamoto, Y. Sugawara, S. Morita, *Jpn. J. Appl. Phys.* **42**, 7163 (2003).
7) C. Gomerz-Navarro, et al., *Nanotechnology* **13**, 314 (2002).

# Atomic scale Kelvin Probe Force Microscopy Studies of the Surface Potential Variations on the $TiO_2$(110) surface

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From an interplay of simultaneous Kelvin Probe Force Microscopy (AM-KPFM) and non-contact Atomic Force Microscopy (nc-AFM) we study the surface potential variation on the atomic scale on the rutile $TiO_2$(110) model oxide surface [1]. We show that both of the simultaneously recorded nc-AFM topographic and KPFM images reveal the atomic scale structure of the $TiO_2$ surface shown in Fig. 1. Out of a detailed correlation of the topographic trace of the tip and the corresponding simultaneously recorded KPFM signal we conclude that the KPFM signal indeed resolves genuine atomic scale variations of the surface potential. The observed AM-KPFM contrast is interpreted in terms of the so-called Local Contact Potential Difference (LCPD), which lends its name to the macroscopic CPD, but it is a potential which has a superimposed spatial modulation set up by the surface potential from the fixed positively or negatively charged ions in the $TiO_2$ surface lattice. Subsequently, we show how a simple model for the charge distribution at the tip apex by may successfully be applied to explain the fine structure effects in the recorded KPFM images and their relation to the topographic AFM signal.

[1] G.H. Enevoldsen et al., accepted for publication in Phys. Rev. Lett. (2008).

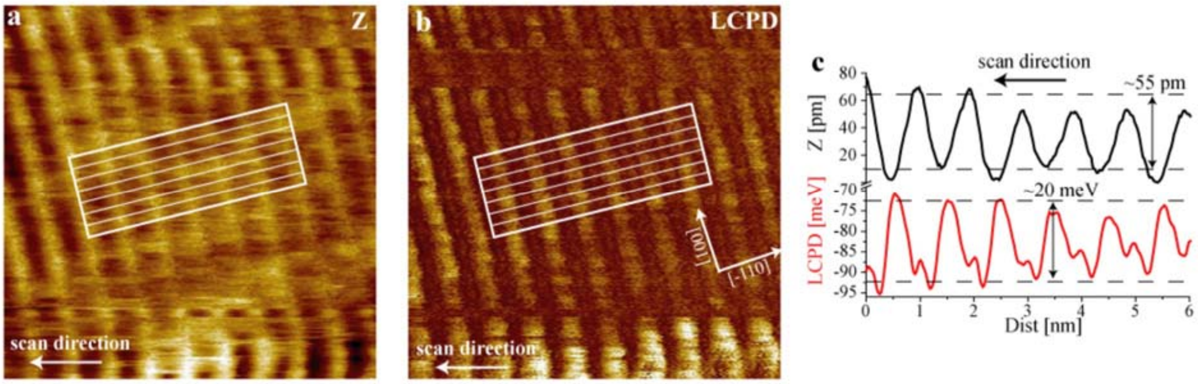

**Fig. 1.:** (a) nc-AFM topography image (z). Size: 10×8 $nm^2$, $\Delta f = -121.2$ Hz, $A_{p\text{-}p} = \sim 28$ nm, $f_0 = 70691$ Hz, $f_{mod} = 451978$ Hz, $U_{AC} = 300$mV, scanning speed = 5 nm/s. (b) Simultaneously recorded LCPD. (c) 99-line average twin cross-sections of z and LCPD, as indicated by the white boxes in (a) and (b).

Tu-1200

# Quantum Mechanical Simulation of Kelvin Probe Force Microscopy for a Simple Metal Surface

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Kelvin probe force microscopy (KFM) has enabled us to observe the local variation of contact potential difference (CPD) with high resolution. However, the interpretation of nanoscale variation of CPD is not necessarily easy. Besides the effects of tip shape, quantum effects such as the deviation of charge distribution from classically estimated one might be considerable. However, quantum mechanical KFM simulations have not been performed yet as far as we know. Bearing this situation in mind, we have performed quantum mechanical simulations of KFM.

In the simulations, the sample is modeled by Al (100) layers on a semi-infinite jellium electrode, and two models were adopted for the tip: bare semi-infinite jellium with an Al atom (Tip-1), and the model same as the sample except an additional Al atom (Tip-2). The electronic states and forces were calculated using the boundary-matching scattering-state density functional method developed by our group [1]. For the tip-sample distance of 16 a.u. (=0.85 nm), the calculated force vs. bias voltage curves have reasonable parabolic shapes (Fig. 1). The values of local CPD estimated from the curves are −0.33 and +0.78 eV for the cases of Tip-1 and Tip-2, respectively. The work functions calculated using the same method are 4.40, 3.95 and 4.35 eV for the sample, Tip-1 and Tip-2, respectively, and thus the difference of work function between the tip and sample is comparable to the local CPD for Tip-1, but not for Tip-2. The origin of the large local CPD for Tip-2 is under investigation, and will be discussed in the presentation.

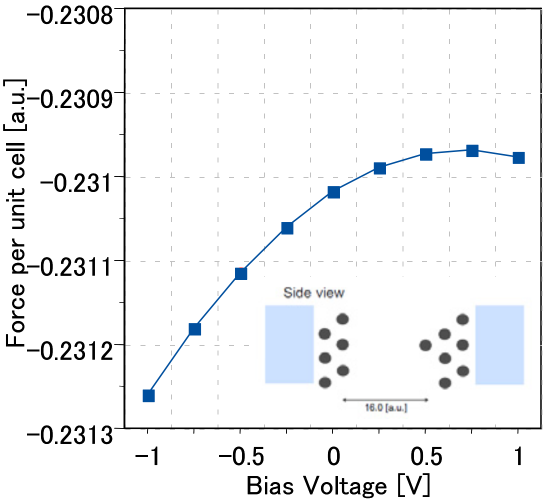

Fig. 1. Bias voltage dependence of the calculated force between the sample and Tip-2. The Model used in the calculation is shown in the inset.

Reference

1) S. Furuya, Y. Gohda, N. Sasaki and S. Watanabe, Jpn. J. Appl. Phys. **41**, L989 (2002).

# Theory of atomic scale contact contrast in amplitude modulation Kelvin probe force microscopy – application to Si(111)-7×7

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KPFM measurements are conventionally interpreted in terms of a long-range electrostatic interaction $\frac{1}{2} C(V - V_c)^2$ , where the tip-sample capacitance C and the contact potential difference (CPD) $V_c$ depend on the relative position of the tip versus surface features of interest, In the case of atomic-scale CPD variations, recently detected by several groups, ours in particular [1], the ultimate hope is to extract information about the surface charge distribution. This distribution, which determines the electrostatic potential outside the surface and the average CP, is overall neutral, but the applied voltage and the tip (via its different CP) induce opposite charges which are in part concentrated around surface and tip apex atoms.

Recent observations of two local CPD components in constant height KPFM images on the Si(111) 7 x 7 surface [2] suggest that additional voltage-dependent electrostatic forces with distinct distance dependences become significant at closest approach distances comparable to the adatom spacing. We identify these components with interactions between the deviation of the surface charge distribution from its smooth background with 1) the long-range electric field and 2) the dipole at the tip apex. Only the last component is responsible for atomic-scale CPD variations. A similar model (with some important differences) reasonably describes sublattice-resolved KPFM images on KBr(001) [1]. Computations based on an analytic approximation to the long-range field [3] and on effective charges and polarizabilities of Si atoms with unsaturated dangling bonds [4-6] are in progress.

Results will be compared with KPFM images obtained via amplitude modulation at the second cantilever resonance simultaneously with the frequency shift of the fundamental resonance [2]. Because the two resonance frequencies are incommensurate, it is sufficient to average the relevant force components over the fundamental oscillation, as long as the oscillation induced by the voltage modulation is comparatively small. Most averages can be performed analytically.

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[6] R. Losio, K. N. Altamnn, F. J. Himpsel, Phys. Rev. B61, 10845 (2000)

# Atom Manipulation on Cu(110)-O Surface with Low Temperature Noncontact AFM

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Manipulation of single atoms and molecules is an innovative experimental technique of nanoscience. So far, a scanning tunneling microscopy (STM) has been widely used to fabricate artificial structures by laterally pushing, pulling or sliding single atoms and molecules. However, the driving forces involved in manipulation have not been measured. Recently, an atomic force microscopy (AFM) has been also used to manipulate single atoms and molecules. Atom manipulation with an AFM is particularly promising, because it allows the direct measurement of the required forces.

In the present study, we investigated the forces in atom manipulation by using AFM. We found, for the first time, that the forces, that are needed to move an atom laterally on the surface, strongly depend on the chemical nature of tip-sample interaction.

The experiment was performed by using homebuilt noncontact AFM using frequency modulation technique operating in ultrahigh vacuum at a temperature of about 77 K. As a sample surface, Cu(110)-O surface was used, which was prepared by exposing Cu(110) surface to the oxygen gas of 1000 L at room temperature. The AFM tip apex was coated with Cu or O atoms in situ by slightly making a tip-sample mechanical contact on the Cu(110)-O surface prior to the imaging. AFM lateral manipulation was carried out for a top single Cu atom (supper Cu atom) on the surface by moving the tip parallel to the surface and by precisely controlling the frequency shift of the oscillating cantilever. The nature of atom movements and the type of tip-atom interaction during lateral manipulation was determined from the AFM feedback signals.

In the case of Cu-adsorbed AFM tip, the super Cu atom on the surface was pulled at a lateral tip position on the adjacent binding site. In the case of O-adsorbed AFM tip, in contrast, the super Cu atom was pushed over the top of the super Cu atom. Thus, we found that the forces (attractive or repulsive forces) to move an atom laterally on the surface strongly depend on the atom species of the AFM tip apex and the surface. These experimental results strongly suggest that the chemical nature of tip-sample interaction plays an important role in lateral atom manipulation. Furthermore, we found that the tip-sample energy landscape estimated reflects this nature.

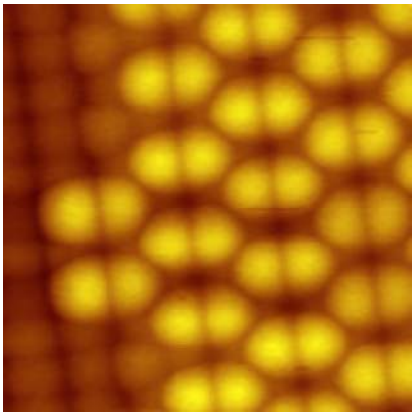

Fig. 1 Manipulated atoms on Cu(110)-O surface at 77K.

# Unveiling the atomic processes during the manipulation of single atoms at semiconductor surfaces using the FM-AFM in the repulsive regime.

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The artificial assembling of complex atomic patterns by the reproducible vertical interchange of atoms between the tip apex of an atomic force microscope and a semiconductor surface has been achieved using the FM-AFM at room temperature and in a very favorable time scale [1]. At variance with previous methods [2], these novel manipulations are produced by the gentle exploration of the repulsive part of the short-range chemical interaction between the closest tip-surface atoms. While imaging and manipulation in the attractive regime involve mainly the bonding interaction between the closest tip-surface atoms [3], in the so far unexplored repulsive regime a larger contact including several atoms is expected, leading to a very complex energy landscape. The structure of the contact at closest approach determines the most likely output among the different competing processes -atom interchange, atom transfer to the tip, deposition of tip atoms, or recovery of the original structure- when the tip is pulled out from the surface. In this work, using first-principles calculations, we cast some light on the complex mechanisms driving these vertical atomic interchanges; we characterize the key atomic processes involved and the magnitude of the energy barriers between the relevant atomic configurations leading to vertical manipulation. Furthermore, the energy landscape associated with these vertical atomic interchange processes provides an explanation for both the dissipation signal and the intriguing features exhibited by the experimental short-range force curves at close tip-surfaces distances.

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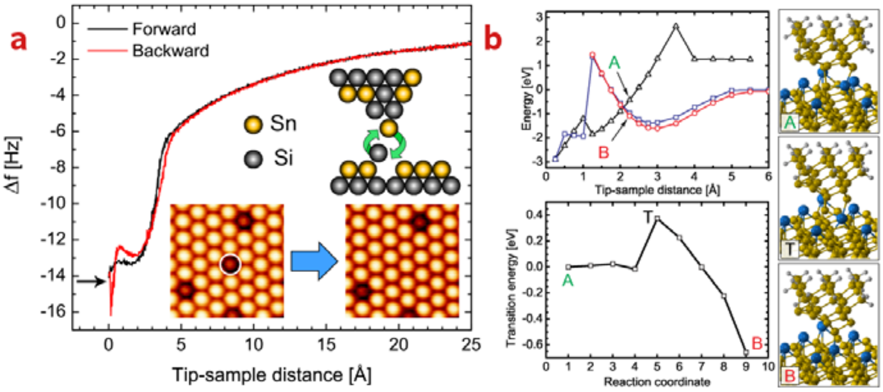

# Water chemistry and manipulation on alkaline earth halide surfaces

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While scanning tunneling microscopy (STM) offers routine manipulation on conducting samples, only non-contact atomic force microscopy (NC-AFM) can provide atomic resolution and molecular manipulation on insulating surfaces. As yet, mechanical manipulation of molecules with NC-AFM on insulating surfaces remains in its infancy, with very few published studies [1-3]. Our previous studies on $CaF_2$ (111) [2] demonstrated the potential of manipulating defects on the surface, and in recent water dosage experiments, we observed the development of two characteristic defects - one which can be manipulated by the tip and one which cannot.

In order to understand this, and interpret the experimental images, we have performed systematic first principles calculations of water chemistry and mobility on the surface using the VASP code. We calculated the binding energies for molecular water and hydroxide groups on the perfect surface, as well as at neutral and charged fluoride vacancies that might act as pinning or dissociation sites for these molecules. Water was found to bind more strongly to vacancies than to the perfect surface, while the binding strength for hydroxides is weaker. Calculations of the reaction and diffusion barriers suggest that molecular water adsorbed at a charged vacancy is accessible to AFM manipulation, while dissociated water at neutral vacancies is more strongly pinned. In order to provide a comprehensive comparison to experiment, we take the lowest energy configurations of water predicted by first principles, and use atomistic methods to simulate NC-AFM imaging and tip-induced manipulation. We also use simulations to suggest explanations for the observed differences in manipulation behaviour seen on $BaF_2$ and $SrF_2$ (111) surfaces.

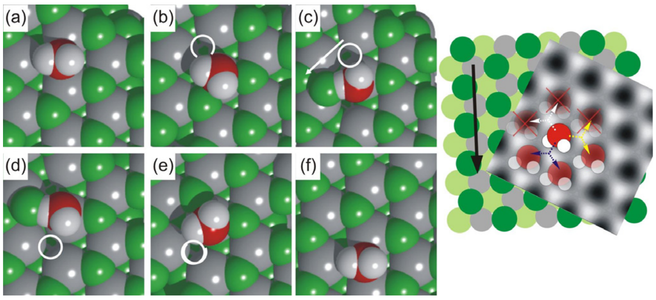

*Figure 1: Mechanism for manipulation of water at a neutral vacancy in the $CaF_2$ (111) surface.*

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# Controlling the motion of large organic molecules on insulating surfaces through molecular structure and localised forces

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Control over the position and mobility of large organic molecules adsorbed on insulating surfaces is important to many areas of nanotechnology and materials science, but particularly in the formation of self-assembled layers and in the development of single molecule devices. We have performed calculations to model the adsorption, mobility and manipulation of a set of large organic molecules with polar binding groups on the $TiO_2$ (110) surface to explore some of the general features common to this type of complex system, using multi-scale simulation methods.

These calculations involved determining the accessible states of the complex potential energy surface of the molecule-surface system and the barriers that separate them using extensive atomistic simulations and then using the kinetic Monte Carlo method to determine the mechanisms of diffusion and effective diffusion rates over laboratory time-scales. We find that the overall structure and flexibility of the molecule has a profound effect on the potential energy surface of the system and hence the mechanism of diffusion and the effective mobility of the molecule.

We investigated how localised forces from an atomic force microscope tip changethe potential energy surface of the molecule/surface system and how this can facilitate controlled manipulation of the molecule. We show how modifying only a small part of the potential energy surface with the tip, the thermally driven motion of the molecule can be induced in a single direction. These calculations suggest ways in which the position and mobility of large molecules adsorbed on surfaces can be controlled.

# Oral Presentations

## Wednesday, 17 September

# Wednesday,
## 17 September

# Characterizing reactivity of water molecules on $CeO_2(111)$ using atomic force microscopy

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NC-AFM is a technique with tremendous potential for the characterization of atomic scale processes taking place on insulating surfaces. One of the most promising insulators in energy-related industrial applications is cerium dioxide (ceria, $CeO_2$). This material —with a band gap of ~6 eV— plays a fundamental role as noble metal supporting substrate for the enhancement of the water-gas shift reaction (WGS, $CO+H_2O \rightarrow H_2+CO_2$) and the massive production of hydrogen [1]. A detailed understanding of the chemical reaction paths of the WGS-reaction on ceria-based catalysts is still missing [2], being a crucial intermediate state the dissociation of molecular water.

Trying to clarify the dissociation process of molecular water on ceria, we have conducted extensive NC-AFM experiments involving single-molecule manipulation, different levels of water dosage, and characterization over a temperature range spanning from 10 to 300 K (Fig. 1). In this contribution we present experimental evidence of high energy barriers that inhibit the diffusion of single molecules on the surface even at room temperature, whereas the characteristic triangular shape of individual molecules is ascribed to an internal rotational motion apparent even at the lowest temperatures this system has ever been explored (see Fig. 1). We have also applied single-molecule manipulation techniques to explore adsorption sites and the interaction of water molecules with surface oxygen vacancies [3]; experiments that provide new insights into the reactivity of molecular water on the $CeO_2(111)$ surface at the atomic scale.

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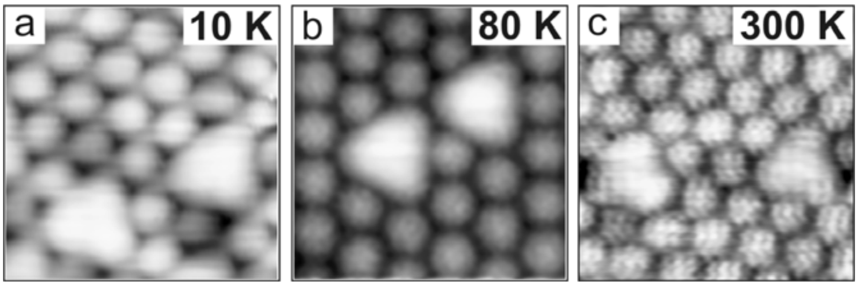

Fig. 1: Topographic NC-AFM images at various temperatures of individual water molecules adsorbed on the $CeO_2(111)$ surface.

# Atomic-scale ordering of the α-$Al_2O_3$(0001) surface studied with atom-resolved nc-AFM

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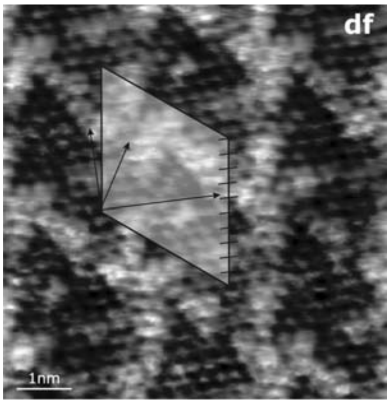
Aluminum oxide ($Al_2O_3$) is a versatile ceramic used in a very broad range of technological applications. Common to most of these applications is the fundamental importance of atomic interactions at the oxide interface. However, relatively little is known about the detailed surface structure of alumina due to its insulating nature, and in this context the AFM is often highlighted as a tool that can provide new insight. Using an interplay between atom-resolved nc-AFM studies and density functional theory (DFT) calculations, we perform here a new detailed study of the surface structure of the intriguing, and surprisingly stable, $\sqrt{31}\times\sqrt{31}R°9$ reconstructed state of $Al_2O_3$(0001). Our atom-resolved AFM images reveal an almost perfectly ordered surface, characterized by a large rhombic grid reflecting the $\sqrt{31}\times\sqrt{31}$ unit cell, inside which atomic subdomains with a very characteristic triangular or hexagonal appearance are visible (Fig. 1). The distinct pattern observed by AFM can be used to determine the exact nature of the whole interface region. The subdomains arise due to variations in the stacking between a single atomic Al layer on top of an Al-rich $Al_2O_3$(0001) substrate (so-called Al-Al-O-$Al_2O_3$), leading to a sub-Å modulation of the perpendicular Al-distance relative to the substrate. DFT calculations are used to calculate surface relaxations and the energetics confirm that the structure proposed is the most stable under the conditions of our experiment. The new proposed atomic model is consistent with previous structural studies based on x-ray diffraction or nc-AFM [1, 2], but differs in a significant way as the surface layer is concluded to be well ordered. The new insight may be used to explore the use of the alumina surface as a nanotemplate and understand the adhesion of adsorbates and metals on alumina surface in general. We have performed preliminary AFM studies to elucidate the morphology, growth and thermal stability of Cu and Ni nanocluster, and despite the similarities of the metals they show a distinctly different morphology on the surface [3].



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# Beyond Imaging: Dynamic Force Spectroscopy on Thin MgO Films

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Here we present atomically resolved measurements obtained with our dual mode dynamic force and scanning tunneling microscope (DFM/STM) on thin MgO films grown on Ag(001). Experiments have been performed in ultrahigh vacuum at a temperature of 5 K using a custom-build quartz tuning fork sensor (Fig. A) operated at small oscillation amplitudes [1]. With high resolution we have been probing inequivalent surface sites on thin MgO films to provide atomic-scale information on surface chemical reactivity (Fig. B,C). Recent STM measurements have already shown evidence for the presence of color centers on the MgO surface. Singly and doubly occupied color centers were identified by their specific appearance in STM images as well as by spectroscopic data revealing the electronic structure of the respective defects [2]. Here we focus especially on combined site selective DFM/STM spectroscopic measurements taken on those color centers. Furthermore, the possibility to derive work function measurements with this setup on MgO films of different thicknesses will be presented in detail. Since the work function and the thickness are crucial parameters for the charge state and hence catalytic properties of adsorbed species. The DFM spectroscopy (Fig. D) and field emission resonances will be discussed (Fig. E) and compared to recently published density functional theory calculations by Giordano et al. [3].

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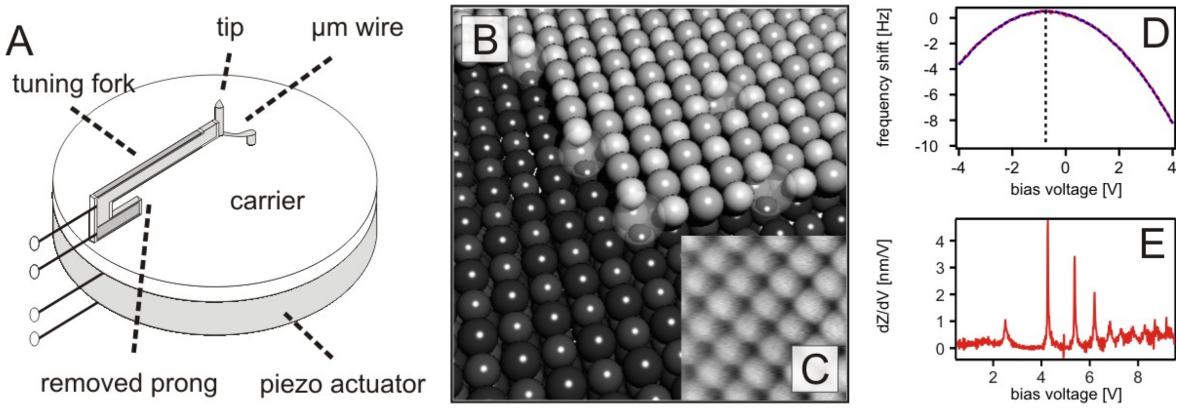

Fig. A) Schematic setup of the quartz tuning fork sensor for dual mode DFM/STM. B) Electron trapping sites on MgO. C) DFM image of MgO on Ag(001), $1.5\times1.5$ nm$^2$, $\Delta f = -8.5$ Hz. D) Contact potential measurement. E) Field emission resonance measurement.

# Simultaneous Atom-Resolved AFM and STM Studies of the Hydroxylated $TiO_2$ (110) Surface

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From an interplay between simultaneously recorded non-contact Atomic Force Microscopy (nc-AFM) and Scanning Tunneling Microscopy we present here a detailed account of the atomic scale contrast obtained in both imaging channels on the prototypical metal oxide surface, $TiO_2$(110). From the nc-AFM topography and tunneling current images obtained on the rutile $TiO_2$(110) surface [1, 2], we clearly identify the different species and point defects present on the surface along with the surface ionic sublattice. The interplay between nc-AFM and $I_t$ images offers complementary information about the surface under inspection, which greatly aids in the interpretation of the images, and provides much more information than would be possible with a single approach.

In order to understand and interpret the experimental results, we use first principles calculations to calculate the tunneling current as a function of the AFM trajectory, and investigate its dependence on the structure and material of the tip, and tip-induced deformation of the surface. We can clearly match the contrast seen in AFM and STM images with specific tip models, and identify several characteristic tips. This knowledge provides a background to understand the tip-induced motion of hydrogen observed in AFM images, but barely visible in STM images. Our calculations show that a probable explanation is the displacement of hydrogen from an oxygen-bridging site to a subsurface site. The calculated barriers for diffusion are within the range accessible to the influence of the tip, and subsurface hydrogen has a similar signature to surface OH in simulated STM images. In contrast to images of the ideal surface, we show that STM imaging of hydrogen requires the inclusion of on-site Coulomb interactions corrections (GGA+U) and finite temperature effects.

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# "All-inclusive" imaging of $TiO_2(110)$

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Understanding the (110) surface of rutile titanium dioxide from direct visualization by scanning probe techniques has been the focus of numerous publications in the last decade [1, 2]. Usually, STM shows the titanium atoms bright while the bridging oxygen atoms are invisible [3]. An NC-AFM study combined with numerical calculations revealed that at least two contrast modes exist, imaging titanium atoms bright and oxygen dark or vice versa [4].

Here, we present an NC-AFM study demonstrating that NC-AFM is capable of imaging both, the bridging oxygen atoms as well as the titanium rows *simultaneously* (Fig. 1). Furthermore, oxygen vacancies, single and double hydroxyls, as well as further defects ascribed to subsurface impurities [5] are also imaged in one single frame. We employ density-functional theory calculations to understand the relation between the atomic tip structure and the observed contrast modes.

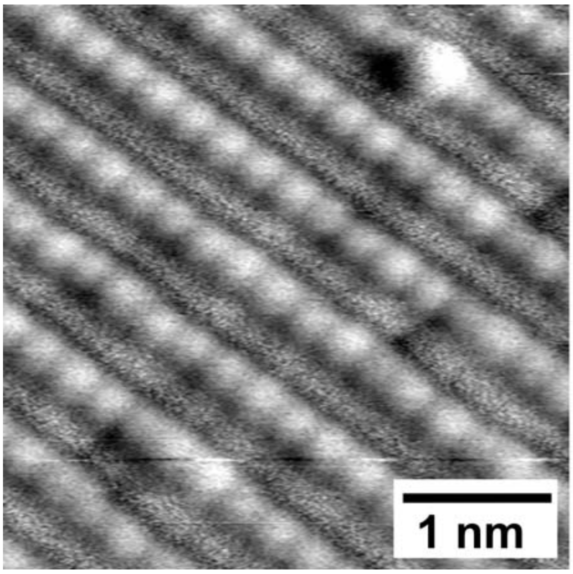

Fig. 1. Constant height NC-AFM detuning image. Bridging oxygen atoms are bright and titanium atoms are faint bright.

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# Interface Dipole Formation of Different $ZnPcCl_8$ Phases on Ag(111) Observed by Kelvin Probe Force Microscopy

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Recently, we investigated the adsorption of octachloro zinc phthalocyanine ($ZnPcCl_8$) on Ag(111) by scanning tunneling microscopy. Compared to the standard phthalocyanine, halogenated phthalocyanine molecules show a much more complex binding behavior, which results in the formation of three different structural phases. These phases follow from the ordering process with formation of 8, 4, and 0 intermolecular hydrogen-halogene bonds [1].

In the present work [2] we investigate these phases by Kelvin probe force microscopy in order to quantitatively deduce the electric interface barrier of the first monolayer. Our measurements reveal that the binding behavior does not only affect the structural ordering but also the interface dipole formation, which leads to different work functions. The fact that we observe interface barriers of opposite signs between ordered and disordered molecular layers underlines the importance of exactly knowing the molecular arrangement at the interface when assembling organic molecule devices.

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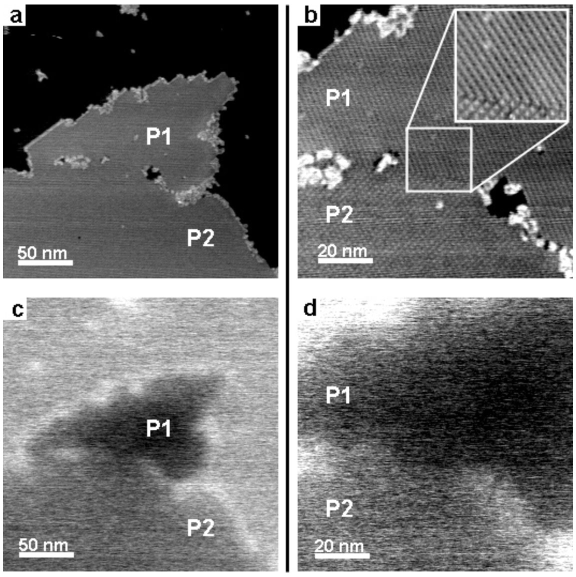

(a/b) Topography and (c/d) KPFM of 0.25 ML $ZnPcCl_8$ vacuum deposited onto Ag(111). Molecular islands measure 2.1 Å in height. Kelvin pictures reveal different contrast for the different structural phases, with the Kelvin greyscale measuring 180 mV and 150 mV in (b) and (d) respectively. Note the different KPFM contrast for phase P1 and P2. The frame size of the inset in (b) measures 25 nm.

# Development of Two-probe Frequency Modulation AFM/KFM for Nanometer-scale Electrical Measurement

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Atomic Force Microscopy (AFM) has been widely used as a nanometer-scale characterization method for investigating various surface properties. However, its simultaneous, multiple uses of various applications are often limited because only a single probe is used in AFM. Thus, the implementation of two or more probe tips can tremendously expand the capability of AFM. It allows us to make a multi-probe electrical measurement or to study the local response which is caused by a mechanical/electrical local stimulus to various materials including biological samples using two probes.

We have developed a two-probe AFM system working with the frequency modulation (FM) detection method for the investigations of nanometer-scale structures [1, 2]. The electrical properties of organic thin films (α-sexithiophene: α-6T) deposited on an insulating substrate (300 nm-$SiO_2$/p-Si) were preliminarily investigated by the two-probe AFM using FM Kelvin probe force microscopy (FM-KFM) mode. While probe-1 having a bias voltage was in contact with an α-6T thin film island, the surface potential was measured by probe-2 working as a KFM tip, as shown in figure 1. Figure 2 shows the obtained temporal change of the surface potential after the charge injection into the film.

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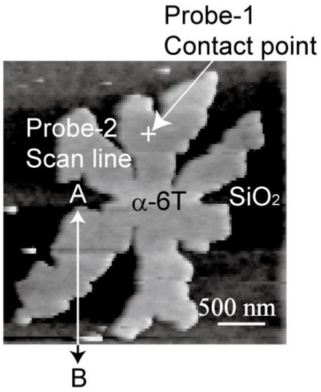

Fig. 1: FM-AFM image of an α-6T thin film island.

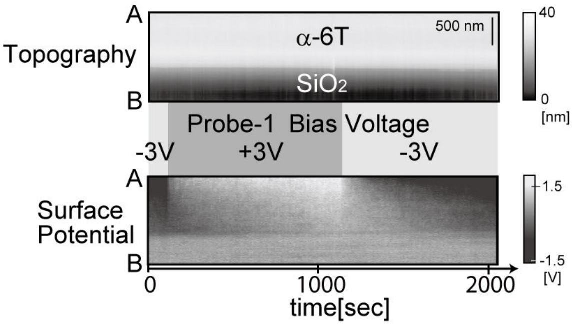

Fig. 2: The temporal change of the surface potential (*lower image*) along the line A-B indicated in Fig. 1. Upper image is the topographic contrast of the corresponding positions.

# Interface and single molecule analysis of organic molecules in a Ferroelectric-OFET

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Organic field effect transistors (OFETs) and their application gain considerable interest these days [1] due to significant advances in molecular design and integration. For an OFET-design where the substrate gate is made of a ferroelectric (FE), we may expect even more functionality, due to the strong and remanent electric field arising from bound surface charges at the FE/molecule interface [2]. Furthermore, for the aim of miniaturization, a high degree of intermolecular ordering is mandatory in order to achieve excellent electric properties [3].

We report here on the high resolution and potentiostatic inspection of the organic-electrode (see Fig. 1) and the organic-ferroelectric interfaces, as well as the self assembly properties of the active oligothiophene layer as being the most critical part in such a Ferro-OFET structure. Using non-contact AFM in combination with Kelvin-Probe Force Microscopy [4] we deduce structural and electric characteristics down to the molecular scale. Measurements reveal a very high degree of self assembled order within the organic layer and at the organic-electrode interface. This considerably enhances the electric transport properties of the proposed Ferro-OFET device.

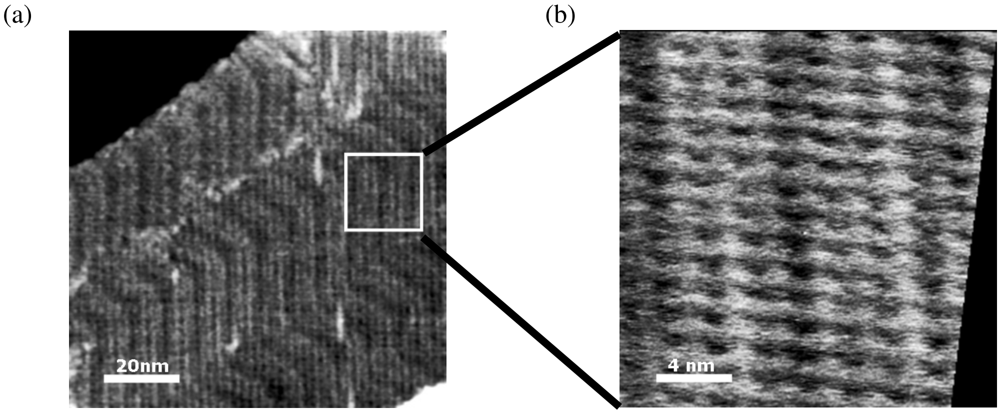

*Fig.1:*
*(a) 1ML dibutylsexithiophene on Au(111), and (b) molecular structure measured in the marked region in (a).*

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# Kelvin probe force microscopy measurements of epitaxial graphene

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Graphene has emerged as a strong candidate for future electronic applications due to its exciting and exotic electrical properties. The epitaxial growth of graphene films on SiC provides a potential method for the technological realization of graphene devices as well as facilitating fundamental studies of pristine single and bilayer films.

Epitaxial graphene films grown on SiC(0001) have been characterized using Kelvin probe force microscopy [1]. Experiments have been performed at room temperature under UHV conditions to avoid surface contamination typically found in air environments. The Kelvin probe method allows for the direct identification of the layer thickness due to a contrast in the local work function of subsequent layers. The local work function is found to increase with film thickness for 0th (interface layer), 1st, and 2nd layer graphene films. The difference between both 0th - 1st and 1st - 2nd layers is found to be 0.14 eV. The films are also found to exhibit a regular superstructure originating from the underlying SiC surface reconstruction. The periodicity of the superstructure is found to be 6 x 6 and its corrugation decreases with layer thickness as previously observed in STM studies [2]. The method also provides true topographic measurements at transitions between single and bilayer films allowing for the identification of the underlying SiC substrate step configuration.

The capability to directly identify single and bilayer films by a scanning probe method could facilitate further understanding of the unique properties of single and bilayer graphene films. For example we have used this technique in conjunction with friction force microscopy to investigate the frictional properties of graphene films.

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We-1430

# High-speed Frequency Modulation Atomic Force Microscopy in Liquids

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Frequency modulation atomic force microscopy (FM-AFM) was proven to have the capability of true atomic resolution imaging even in liquids [1]. The technique has been successfully used for the molecular-scale research on the biological systems such as lipid membranes [2] and amyloid fibrils. However, there still remain technical issues that have greatly limited the application range of FM-AFM in biology. Amongst the most evident is the insufficient feedback bandwidth of the tip-sample distance regulation, which has hindered imaging dynamic events, fragile macromolecular assemblies, and heterogeneous complexes of various biomolecules.

The present work aims at improving the imaging speed of FM-AFM for biological applications. This requires enhancing the bandwidth of all the components in the main feedback loop as well as improving the signal-to-noise ratio in the force detection. One of the major improvements that we made is shown below. Figure 1 shows the frequency response of the developed scanner in the z-axis. The profiles illustrate the clean resonance peak at 540 kHz. This is significantly higher than the typical resonance frequency of a tube scanner in the z-axis (< 10 kHz). Figure 2 shows the FM-AFM image of mica taken in liquid with the developed scanner, showing the individual atoms constituting the honeycomb-like lattice of the cleaved mica surface. The result demonstrates the applicability of the scanner to the atomic-resolution imaging.

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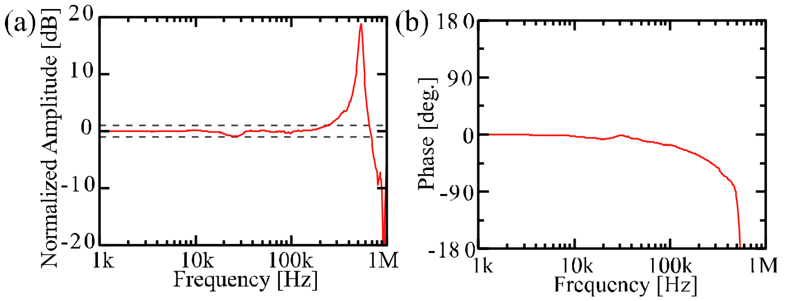

Fig. 1 Frequency response of the developed scanner in the z-axis. The resonance frequency is about 540 kHz.

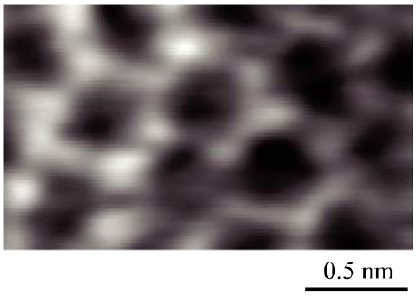

Fig. 2 FM-AFM image of mica taken in liquid

# High-resolution Imaging of Biomolecules in Liquids by FM-AFM

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Recent progress in frequency modulation atomic force microscopy (FM-AFM) in liquids has opened a new way to direct visualization of "*in vivo*" molecular-scale biological processes. The phase noise in the self-oscillation loop is a dominant noise source in FM-AFM working in low Q-factor environments. We successfully reduced the phase noise by decreasing the coherence of the laser light used in the cantilever deflection sensor, which was experimentally performed by the laser power modulation with a high-frequency signal.

In this presentation subnanometer-resolution imaging of biomolecules such as membrane proteins and DNA molecules in liquids using the improved FM-AFM is described. Figure 1 (a) shows a high-resolution FM-AFM image of a plasmid DNA molecule (pUC18) on a mica substrate. The image was taken in pure water while the sample was prepared in a buffer solution for DNA adsorption to the substrate. The major grooves (2.2 nm wide) indicated by gray arrows and the minor grooves (1.2 nm wide) indicated by white arrows along the DNA chains are clearly resolved. In addition, by force mapping method in FM-AFM we recently succeeded in visualizing the hydration structures, which play essential roles in the stabilization of higher-order protein structures as well as in various bio-function processes.

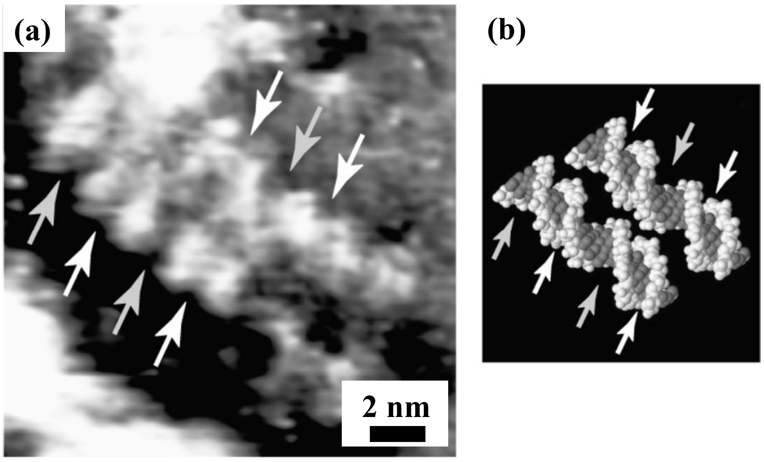

Fig. 1: FM-AFM image of a DNA molecule in pure water (a) and the DNA structural model corresponding to the obtained image (b).

# Bimodal atomic force microscopy imaging of isolated antibodies

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We develop a dynamic atomic force microscopy (AFM) method based on the simultaneous excitation of the first two flexural modes of the cantilever. The instrument, called bimodal AFM, allows us to resolve the structural components of antibodies in both monomer and pentameric forms. The instrument operates in both high and low quality factor environments, i.e., air an liquids. We show that under the same experimental conditions, bimodal AFM is more sensitive to compositional changes than amplitude modulation AFM. By using theoretical and numerical methods, we study the material contrast sensitivity as well as the forces applied on the sample during bimodal AFM operation.

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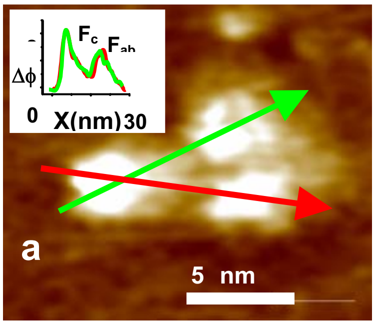

Fig. 1.

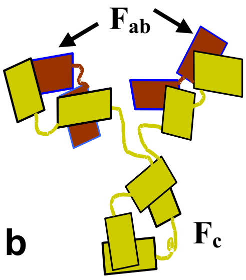

Fig.2.

# Theoretical simulation of dynamic mode AFM in water

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## • Introduction

For the AFM of soft-materials as polymers, proteins, DNA, measurements in water are important, since properties of such systems in vacuum are different from those in water. We are developing basic simulation methods for describing the cantilever nonlinear oscillation dynamics including liquid motion, as well as the detailed features of the tip sample interactions mediated by water molecules[1]. Toward our final goal to establish a useful theoretical simulator for dynamic AFM in liquids supporting experiments, some very recent remarkable results will be reported.

## ・ Cantilever oscillation analyses

As for the cantilever dynamics, the elastic body oscillation of cantilever in water very close to the sample is analyzed, by solving the fluid dynamics of water simultaneously by a specially designed finite element method. The calculated resonance curve showed strong nonlinear features, as well as the reduction of resonant frequency and lose of the sharpness of the resonance. This method is useful for designing cantilever shapes, and oscillation modes. Then, the effect of the tapping process by the sample is included by a visco-elastic model of samples. Sticking and detaching of the tip to the sample, which causes large disturbance of the cantilever motion, are also analyzed. With including these processes altogether, the frequency shift, dissipating energy, phase delay of the oscillation are obtained and used to calculate tapping mode images of proteins.

## • Tip-sample interaction meduated by water

We developed two methods, i.e., molecular dynamics method(MD) and the RISM method to calculate tip-sample interaction force mediated by water with the atomistic model. Some remarkable features so far obtained are introduced, including the oscillatory solvation structure of water molecules near the surfaces of the tip and the sample, and their compression and collapse which give characteristic structures in the force-distance curve. The three-dimensional force map is presented for some typical substrate surfaces and compared with experiments.

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# Oral Presentations

## Thursday,
## 18 September

# Thursday,
## 18 September

# Magnetic Exchange Force Microscopy on Fe/W(001)

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Magnetic exchange force microscopy (MExFM) is a novel technique [1] that senses the short-ranged magnetic exchange interaction between magnetic moments of an atomically sharp tip and a surface at sub-nanometer separations. Thus, it can image the arrangement of magnetic moments at the surface with atomic resolution.

In this presentation we report MExFM experiments and density functional calculations performed with an iron tip on a iron monolayer pseudomorphically grown on W(001) resulting in a p(1x1) chemical surface unit cell. Hybridization with the tungsten substrate leads to an c(2x2) antiferromagnetic structure of the iron magnetic moments with out-of-plane anisotropy [2]. While AFM images every iron atom as protrusion, MExFM data clearly resolve the antiferromagnetic structure, i.e., only every second atom appears as a protrusion. Comparing our experimental data with ab-intio calculations and simulations we can elucidate the complex interplay between chemical and magnetic exchange interactions, which are both short-ranged and electron-mediated. Moreover, since for the first time a multi-atom tip was employed in density functional calculations for a magnetic tip-sample system, we can study the role of relaxations in tip and sample. They alter magnitude and distance dependence of the tip-sample forces. Particularly, the onset of significant exchange forces is shifted towards larger tip-sample separations.

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# Ab-initio Simulation of Magnetic Exchange Force Microscopy

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Magnetic exchange force microscopy (MExFM) is a promising new technique to perform magnetic imaging with atomic resolution by measuring the magnetic exchange force between a magnetically coated atomic force microscopy tip and a magnetic sample [1]. However, many questions remain open concerning e.g. the strength of the observed forces and how they depend on the specific tip and sample. Here, we apply density functional theory using the full-potential linearized augmented plane wave method to investigate the exchange forces between a magnetic tip and an Fe monolayer on W(001), which is a model system of a two-dimensional antiferromagnet [2]. First, we consider Cr, Mn, and Fe single atom tips and show that the long-range exchange forces are independent of the chemical species while the short-range forces depend in sign and magnitude on the atom type. As a more realistic tip model, we use a Fe cluster and include relaxations the cluster and the surface. Surprisingly, relaxation effects of tip and sample depend sensitively on the local magnetic configuration. Therefore, relaxations play a crucial role for the magnetic signal. In particular, the onset of the exchange forces is shifted to larger distances which facilitate their experimental observation. Based on the calculated force-distance curves we simulate MExFM images which display a competition of chemical and magnetic forces. The obtained images are in excellent agreement with recent experiments [3].

[1] U.Kaiser, A.Schwarz, R.Wiesendanger, *Nature* **446**, 522 (2007).

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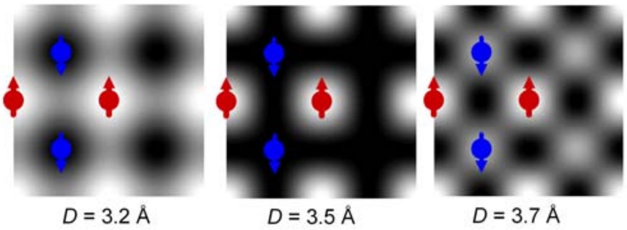

**Fig. 1**. Simulated MExFM images for different tip-sample distances. The contrast for *D* = 3.5 Å agrees with the experimentally observed image [3]

# Manipulation of local charges in graphene nanoribbons

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Graphene [1], a single layer of graphite, is a 2D material whose charge density can be manipulated by an external electrostatic gate at a nanometer scale.

We deposit graphene by mechanical exfoliation of HOPG graphite on an oxidized silicon substrate [2]. The silicon substrate top oxide layer is 285 nm thick, allowing for a fast identification of graphene flakes by phase contrast optical microscopy. We have found that, in some cases, 10 nm thin ribbons (which can be microns long) are deposited during the fabrication process. Topography images of graphene flakes and nanoribbons are obtained by FM-AFM microcopy using a piezoelectric quartz tuning fork force sensor [3]. A metallic tip is attached to the free end of the tuning fork. Both trapped charges in the silicon oxide layer and the background gate voltage applied to the silicon substrate control the charge density in the graphene 2D layer [4]. Electrostatic force images are obtained to find the charge density landscape in graphene flakes and nanoribbons.

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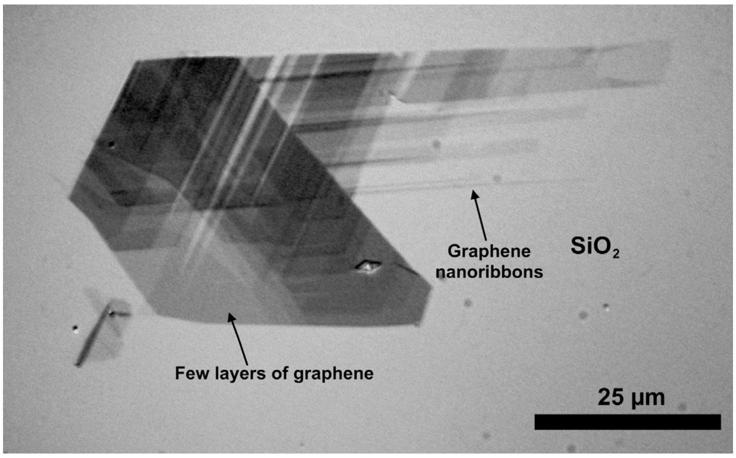

Phase contrast optical microscopy image of graphene flakes and protruding nanoribbons.

# Observation of Coulomb oscillation rings in InAs quantum dots by dissipative electrostatic force detection

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The Coulomb oscillation rings were observed around individual quantum dots (QD) in the dissipation images taken by frequency modulation mode atomic force microscopy (FM-AFM). The dissipation images show the concentric rings around the QDs which correspond to the increased cantilever dissipation.

An electron tunneling back and forth between the QD and the 2DEG across the InP tunneling barrier accounts for this increase in the cantilever dissipation. The condition for the tunneling is determined by the voltage across the barrier which has to overcome the Coulomb charging energy of the QD for each single electron tunneling (Coulomb blockade effect).

The experiments were performed on self-assembled InAs QDs grown on an InP substrate by chemical beam epitaxy. A two dimensional electron gas (2DEG) formed in the InGaAs quantum well was used as the back electrode. A conductive AFM tip is scanned over the QD sample with a dc-bias voltage applied between the tip and the 2DEG at a constant height while the resonant frequency and dissipation of the oscillating cantilever are acquired.

Similar ring structures have already been observed in the QD formed in carbon nanotubes, semiconductor nanowires and lithographically defined semiconductor QDs. In most of these experiments, the dc transport measurement are performed in the QD wired to two external electrodes where the AFM tip was used simply as a movable gate electrode. The present method requires no electrode to be wired to the QDs and makes use of the AFM tip as a high-sensitive charge detector as well as a movable gate. It is therefore applicable to such small QDs as epitaxially grown self-assembled QDs and nanocrystal QDs. The dissipation arises from the delayed response of the tunneling electron to the oscillating electric field caused by the oscillating tip and could give us the information on the dynamics of the tunneling electrons.

We will elucidate the origin of the observed dissipation and the mechanism of the dissipation imaging. The difference between the dissipation images and the frequency shift images will also be discussed.

# Scanning-Gate Microscopy images the electronic LDOS inside nanostructures

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In the last decades, techniques to probe low-dimensional electron systems have flourished, triggered by the invention of the STM. However, STM is useless when the electron system is buried under an insulating layer. To get local information in such cases, *an electric variant of the non-contact AFM* has emerged [1], namely the "Scanning Gate Microscopy" (SGM). In SGM, the charged tip of an AFM induces a local electrostatic perturbation within the electronic system. The perturbation alters the transmission of electrons through the device, and this can be imaged by recording the device conductance as the tip scans over it without contact.

Here, we investigate on the link between the electron local density-of-states (LDOS) and SGM images measured on quantum rings (QRs) fabricated from InGaAs based heterojunctions. We performed SGM measurements at low temperature (4.2 K) so that transport is in the ballistic and coherent regime. Conductance modulations are observed in SGM images when the tip scans over the QR area and in its vicinity. In the latter case, the modulations originate from the Aharonov-Bohm effect and correspond to iso-phase lines for electrons [2].

To elucidate the origin of conductance modulations observed when the *tip scans directly over the QR area*, we first perform simulations of the LDOS and of the conductance of mesoscopic QRs. Then, we introduce a scanning perturbing potential in the simulation to mimic the effect of the SGM tip. We finally compare the simulations of QR conductance modulations induced by this perturbation to the LDOS maps and analyze the correspondence between both [3]. This comparison permits us to establish a direct link between simulated SGM images and LDOS for sufficiently narrow perturbation amplitude. This link still holds in the presence of random impurities. A major conclusion of our work is that SGM can be viewed as the analog of STM for imaging the electronic LDOS in buried open mesoscopic systems.

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Th-1120

# A nontrivial behavior of the resonant tunneling current along the tip-sample distance on semiconductor surfaces

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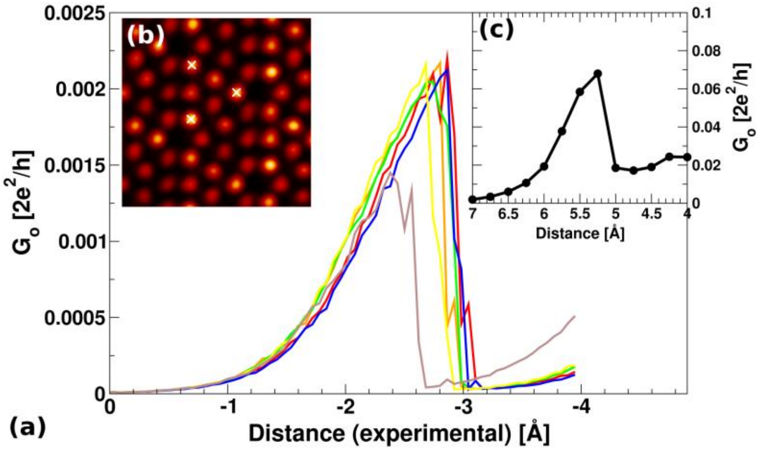
While standard as Scanning Probe Microscope (SPM) image procedures operate mostly at so called the tunneling regime, more and more applications take place in the near-to-the contact regime (see e.g. [1]). Extensive experimental and theoretical studies of the transition regime for different metallic surfaces [2] have been performed in the last years. In general, all these works confirmed the intuitive picture of the increase of the tunneling current with the reduction of the tip-sample distance showing the exponential shape of current curves.



In this work, we combine STM measurements, first principle DFT total energy methods and calculations of the electron currents based on Green's function techniques [3] to clarify the mechanism of the resonant tunneling through 'dangling bond' states of Si adatoms on the Si(111)-(7x7) surface. We show that the commonly accepted assumptions about the above discussed behaviour of the tunneling current is violated in the case of a resonant tunneling processes on semiconductor surfaces [4]. In the near-to-contact regime, a substantial decrease of the tunneling current, almost to zero, is both experimentally (see Fig. 1) and theoretically (see inset Fig.1) observed. As we demonstrate here, this effect is driven by the substantial modification of surface 'dangling bond' states due to the short range chemical interaction between the tip apex atom and the surface adatom [5].

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# QPlus AFM on single crystal insulators with small oscillation amplitudes at 5 K

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The creation and investigation of nano-structures, molecules or atomic structures on insulating surfaces is a key approach for electronic decoupling from the substrate. It pushes AFM as an complementary imaging and spectroscopy technique to STM. Ideally, the used AFM probe should simultaneously or alternatively work in STM/STS modes without performance compromises on the latter. Based on a proven low temperature (5K) LT STM platform, we have integrated a QPlus [1] sensor, which employs a quartz tuning fork for force detection in non-contact AFM. For combined STM operation, this sensor has key advantages over conventional cantilevers: (i) a solid metal tip for optimal STM/STS and (ii) high stiffness and high stability, i.e. low vibrational noise due to small self-resonance amplitudes.

For quantitative force spectroscopy on insulating thin films or semiconductors, decoupling of tunneling current and piezo-electrically induced AFM signal is important. By measurements on Si(111) and Au(111) we prove that only a dedicated pre-amplification technique can solve this problem. In addition, extremely low signals require the first amplification stage to be very close to the sensor, i.e. to be compatible with low temperatures. STS measurements using a Niobium tunneling tip reveal the superconducting gap with a FWHM of approx. 2.5 meV and prove a probe temperature of approx. 5K.

The high stiffness (1800 N/m) of the sensor allows for operation with extremely small amplitudes to (i) more precisely keep the sensor with a certain force interaction regime, (ii) increase sensitivity especially for short range forces and (iii) allow for force measurements during atom manipulation experiments without disturbing the manipulation event as such [2]. As benchmark measurement, we present atomic resolution imaging on single crystal NaCl with oscillation amplitudes down 100pm (peak-to-peak) in constant df imaging feedback. Optimal S/N ratio is achieved with a frequency noise down to 30mHz (peak-to-peak). We also present atomic resolution measurements on MgO(100), C60 molecules on Ag(111), and first evaluation measurements of the QPlus sensor in Kelvin Probe (KPM) mode operation.

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# Molecular-scale Hydration Structures Investigated by Frequency Modulation Atomic Force Microscopy

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Molecular-scale investigations of hydration structures at a solid-liquid interface are essentially important for understanding the mechanisms of various biochemical processes. However, theoretical calculations such as molecular dynamics simulations have been so far the only ways to study hydration structures at a molecular level. We recently found that oscillations with the period close to the liquid molecular size appeared in the force-distance curves measured by FM-AFM and that the oscillatory structures reflected local hydration structures.

In this study molecular-scale hydration structures at an interface between a muscovite mica surface and an aqueous solution containing salt were investigated both experimentally and theoretically using an FM-AFM force mapping method and a three-dimensional reference interaction site model (3D-RISM) calculation. Figure 1 shows a two-dimensional (*x*- and *z*-directions) frequency shift mapping (reflecting interaction forces), which was taken above a mica surface in a 10 M KCl aqueous solution. A bright, horizontal corrugation with a periodicity of 0.52 nm at the bottom of the image corresponds to the honeycomb core structure of the mica surface. We can see three-layer structure on the surface. The frequency shift contrast in the first layer is strongly modulated depending on the surface crystal structure. Note that a large positive frequency shift (strong repulsive force) is actually induced in the first layer at each surface depression, which corresponds to the hexagonal center of the mica surface, because the measured frequency shift includes gradual, negative background frequency shift caused by the van der Waals interaction. This site-dependent contrast is probably due to the water-molecules regularly adsorbed onto the mica surface, which is also supported by the 3D-RISM calculation.

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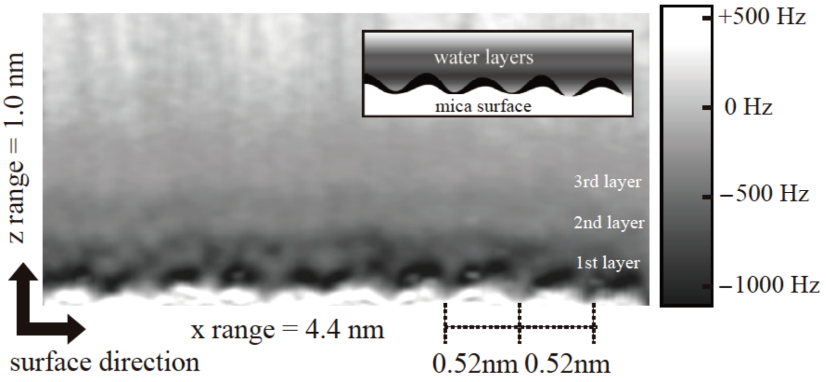

Fig. 1: Two-dimensional frequency shift ($\Delta f$) mapping in a KCl aqueous solution on a mica crystal surface. Inset shows a schematic of the hydration structure.

Th-1220

# Small Amplitude Dynamic Force Microscopy of Liquids Near a Solid Surface

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Confinement of liquids between two surfaces (such as a solid substrate and an AFM tip) can lead to the observation of ordered layers of liquid molecules. In some systems, such ordering may also be induced by the substrate alone. Such solvation layers have been extensively studied using force spectroscopic methods.

Recent advances in the sensitivity of liquid AFM (i.e. FM-AFM) have made it possible to not only detect such layers by their solvation force signatures, but also to obtain high-resolution topography images of the different layers. We will demonstrate that solvation layers with 3D crystalline ordering can be imaged up to several nanometers away from a solid HOPG surface, supporting molecular dynamic models of "surface-enhanced freezing".

The transition from the bulk liquid to a solid-like interfacial phase affects the fluid dynamic properties of the liquid (such as viscosity). Hence, we will expand the force spectroscopic characterization of such interfacial layers by measuring not only their stiffness, but also variations in damping (dissipation) encountered by an AFM tip vibrating at sub-nanometer amplitudes in liquids at different temperatures. This is important not only for understanding the state of the confined material, but also for dynamic mode imaging in liquid environments in general.

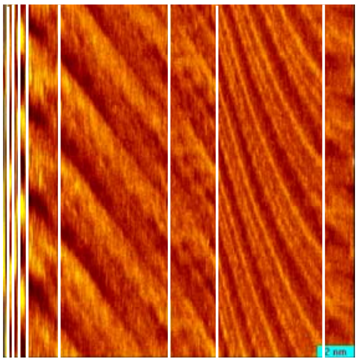

Fig.1: Flattened topography of C12OH on HOPG, showing successive "jumps" between layers.

Th-1240

# Adhesion Hysteresis and Capillary Interactions in Dynamic Atomic Force Microscopy

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Dissipation spectroscopy in amplitude-modulation AFM (AM-AFM), based on the analysis of the dissipated power as a function of the cantilever oscillation amplitude, has been shown to be a powerful tool to identify specific energy dissipation processes at the nanoscale [1]. As a general approach, power dissipation in AM-AFM is naturally considered synonymous of energy dissipated per cycle. Assuming that the dissipation takes place in each oscillation cycle, it would be proportional to the oscillation frequency, $\nu_0$, i.e. $P_{dis}=\Delta E \nu_0$, being ΔE the energy dissipated in the contact process. In striking contrast to this apparently natural argument, we show that the time-averaged dissipated power is not always proportional to ΔE due to a beating phenomenon where the interaction is occasionally dissipative [2,3].

In air ambient condition, when the tip approaches the sample, water condensation from the humidity can induce the formation of a nanometer-sized water bridge and dissipation can be strongly influenced by capillary forces [2]. Even in vacuum, intrinsic mechanical instabilities may lead to the formation of an atomic-scale connective neck [4]. With the help of numerical simulations, dissipation contrast in AM-AFM is shown to be a result of a non-trivial interplay between the energy dissipated in each rupture process and the bi-stable motion of the cantilever. In the repulsive regime (see Fig. 1), the dissipated power is approximately constant and independent of the amplitude as expected. Working in this regime, and for large relative humidity, energy dissipation images in air can be regarded as surface hydrophobicity maps [2]. In contrast, in the attractive regime, after the contact process, the cantilever, which has lost energy, will not reach the same amplitude as before the contact, and the tip may not hit the sample surface during the next swings. The power dissipation is then lower than expected.

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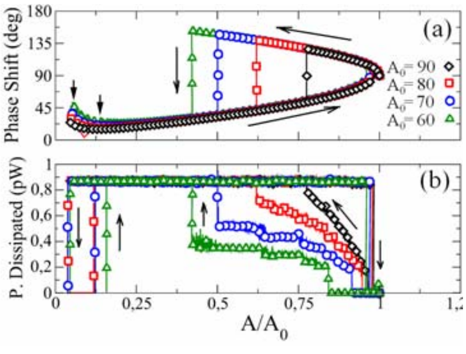

Figure 1: Simulated phase (**a**) and power dissipated (**b**) vs normalized amplitude (After Ref. [2]).

# Oral Presentations

## Friday,
## 19 September

# Friday,
## 19 September

# Heteroepitaxy of ultrathin films studied by high-resolution nc-AFM

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The structure of ultrathin films is the result of a competition between the the structure of the substrate surface, molecular interactions within the film, and surface effects towards the vacuum. In the case of ultrathin films of alkali halide salts the ionic bonds between its constitutents are so dominating that the cubic structure with a (100) surface is usually observed. We will report on high-resolution non-contact AFM studies of two systems: KBr on Cu(100) surfaces and the reciprocal heteroepitaxy of KBr and NaCl.

For KBr/Cu(100) we observe a Moire structure which reflects the different lattice constants of substrate and film [1]. The periodicity of the Moire pattern allows for a precise determination of strain in the film. Furthermore, atomic resolution imaging reveals the expected enhanced interaction at low-coordinated surface sites. We will show how these nc-AFM results help in the intepretation of friction force microscopy on the same film system.

For the reciprocal system KBr/NaCl(100) and NaCl/KBr(100) we have observed an intriguing difference for the interface [2]. Ultrathin films of KBr grow on NaCl(100) with the KBr lattice constant starting from the first layer. Consequently, a Moire pattern is observed on the films. On the other hand, NaCl films adapt the larger lattice constant of the KBr substrate for the first few layers. We assume that the asymmetry of the molecular bond is the reason for this difference.

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Fr-0920

# Evidence for tip structural reversible transformations during NC-AFM imaging on KBr(001)

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We present atomic resolution images of step edges on the KBr(001) surface imaged by NC-AFM at room temperature in UHV. In certain experimental conditions, we observe a systematic and reversible change in the atomic contrast when the tip crosses a step (figure 1) and [1]. This change is the consequence of the reversal of the polarity of the ionic tip under the influence of the tip-substrate interaction in the immediate vicinity of the step edge. This polarity reversal is attributed to a change in the atomic structure of the tip and is described as a transition between the two potential wells of a two-level system localized near the tip apex. The case of two monoatomic steps imaged in succession is also investigated in detail.

This tip bistable behavior is also observed on the Δf(z) curves obtained with the same tip (figure 2). More generally, a few examples of force curves obtained on KBr(001) at RT which show anomalous behaviors similar to that shown in figure 2 will be discussed. They will be tentatively interpreted in terms of reversible structural transformations of the tip apex under the influence of the tip-substrate interaction.

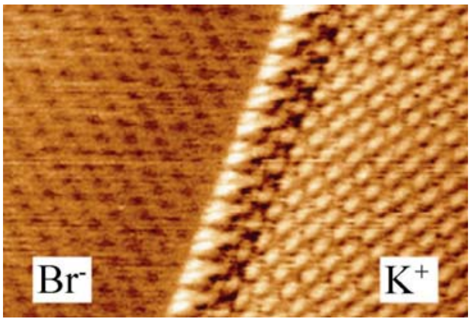

Fig. 1.

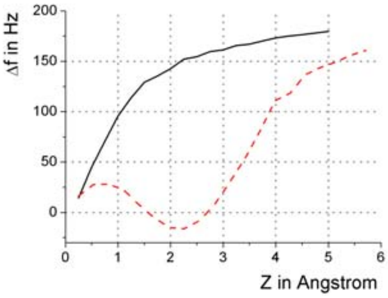

Fig.2.

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# Repulsive interaction and contrast inversion in non-contact atomic force microscopy imaging of adsorbates

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To understand contrast formation in non-contact atomic force microscopy (NC-AFM), we investigate whether or not repulsive tip-sample interaction contributes to contrast formation. Especially above protruding adsorbates contrast inversion is frequently observed, turning data interpretation into a rather delicate task [1]. Contrast inversion has also been observed on bare surfaces such as Au(111) [2] and Si(111)-(7x7) [3].

Here, we present experimental evidence for contrast inversion above protruding adsorbates on mica as well as on $C_{60}$ islands deposited on rutile $TiO_2(110)$. Based on simple potentials, we explain the mechanisms leading to the observed contrast inversion [4]. The influence of the repulsive regime on the detected frequency shift is discussed in dependence on the oscillation amplitude, providing means for differentiation between the attractive and repulsive regime in NC-AFM imaging.

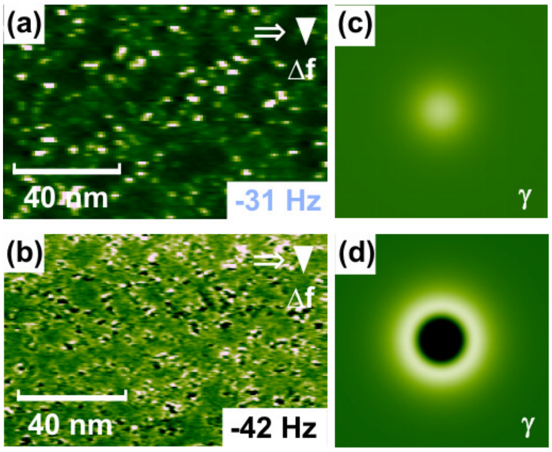

Fig.1. Contrast inversion of adsorbates on mica. (c) and (d) are simulated images for a single adsorbate.

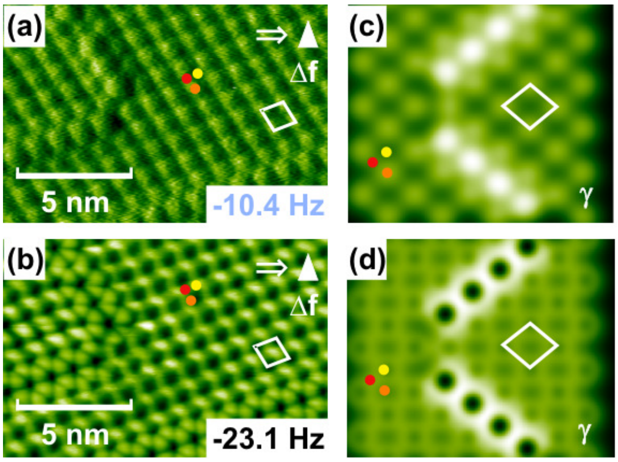

Fig.2. Contrast inversion on a $C_{60}$ island grown on $TiO_2$. (c) and (d) present simulated images for a $C_{60}$ island with a bridging row.

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# Molecule–Insulator interactions: Growth, epitaxy and electronic properties of PTCDA on NaCl

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The study of molecules on insulating surfaces has increased dramatically in the past 5 years, in part due to the development of tools which better allow study of these systems such as nc-AFM, but also with an eye toward using insulating surfaces to decouple molecules from their environment for use in single molecule and organic thin-film electronics. We have explored the assumption that insulating substrates, often ionic crystals, have little influence on molecular orbitals, as well as studied growth considerations and the nature of the molecule-surface interaction by investigating PTCDA on NaCl through both nc-AFM experiments and modeling.

Nc-AFM imaging of various coverages of PTCDA on NaCl(001) revealed an interesting bimodal growth with diamond shaped monolayer (ML) islands, and multilayer islands similar to those observed on other surfaces. A dewetting transition from predominantly ML islands to crystallites occurs at a critical coverage of ~0.85ML. Through high-resolution imaging, the epitaxy of the ML islands was found to be a square p3x3 arrangement. As this is not compatible with the herringbone structure observed for multilayer crystallites, we propose that the interface structure is adapted to accommodate additional layers during growth. To more thoroughly investigate this, both the p3x3 epitaxy as well as a proposed p2x3 interface structure were modeled by molecular mechanics. While the p2x3 structure was found to be more favourable, the p3x3 structure maximizes interactions with the surface through electrostatic interactions between the negative carbonyl groups and the $Na^+$ ions of the surface. The modeling also revealed that while an ordered, bulk-like second layer was easily formed on the p2x3 interface, no ordered second layer was found on a p3x3 interfacial layer supporting our hypothesis. Also, based on the epitaxial structures determined from the AFM data, DFT calculations were carried out showing that there is minimal alteration of molecular orbitals for PTCDA on NaCl, indicating that, at least for this combination, an insulating substrate *can* be used to maintain the expected properties of a molecular device.

Additionally, the growth of PTCDA on a pit-templated NaCl surface was examined and found to result in a third coexisting structure: a confined p2x3 monolayer. Electrostatic characterization under laser illumination of all three of these structures shows differing photoinduced responses of the contact potential difference for each, indicating different opto-electronic properties as a result of epitaxial structure. The combination of high resolution characterization by nc-AFM with hybrid optical-electrostatic characterization allows a critical connection to be made between molecular scale structure and functional properties.

1) S.A. Burke *et al.* PRL **100**, 186104 (2008)

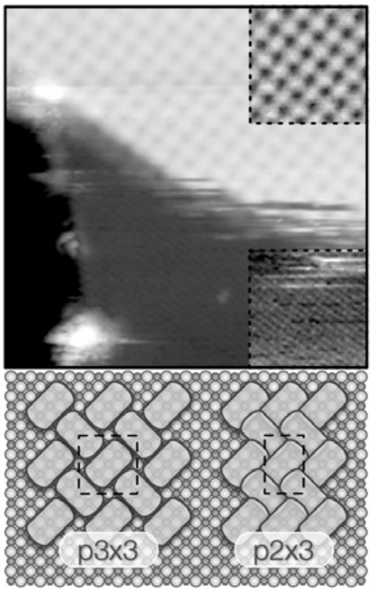

Fig. 1: PTCDA on NaCl high resolution, below: schematic of p3x3 ML epitaxy and

# Resolving atomic scale features of a polar metal oxide surface

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By atomically resolved scanning force microscopy operated in the non-contact mode (NC-AFM) under UHV-conditions, for the first time we directly reveal the details and atomic arrangements of the Zn-terminated ZnO(0001) surface that excels by its well known triangle reconstruction [1]. Upon high temperature preparation an additional faceting by formation of step arrays with stunningly high regularity and symmetry spanning over several 100 nm is observed [Fig. 1(a)] [2].

Besides the known nanoscopic triangular and faceting morphology we furthermore resolve a novel coexisting (1x3) reconstruction on the same surface on areas where no triangular islands and pits are found [Fig. 1(b)]. The (1x3) reconstruction is ascribed to missing rows of Zn-atoms [Fig. 1(c)]. Based on highly resolved images we have derived a model, where hydrogen adsorbs on the remaining Zn-rows, yielding zinc-hydride species, stabilizing areas where the (1x3) reconstruction is not perfect. Our results strongly suggest that there is a common stabilization mechanism for both polar ZnO surfaces, namely the formation of ordered Zn- (O) vacancy arrays, leading to a (1x3) surface reconstruction of both the Zn-ZnO(0001), and the O-ZnO(000-1) [3] surface.

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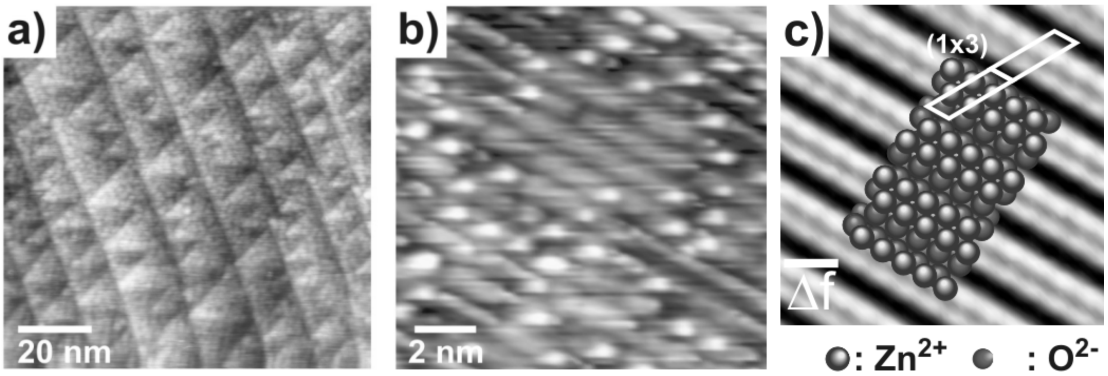

Figure 1.

# Atomic Scale Force Vector Fields on NaCl-type (001) surfaces

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The magnitude and direction of forces acting between individual atoms as a function of their relative position can be described by atomic scale force vector fields. We present non-contact atomic force microscopy measurements of the force fields between an atomically sharp tip and the (001) surface of a KBr crystal in conjunction with atomistic simulations. The direct overlap of experiment and simulation allows identification of tip and surface ions. Superposition of vertical and lateral forces reveal the spatial orientation of the interatomic force vectors on the KBr sub-lattice. In addition the case of NaCl will be discussed.

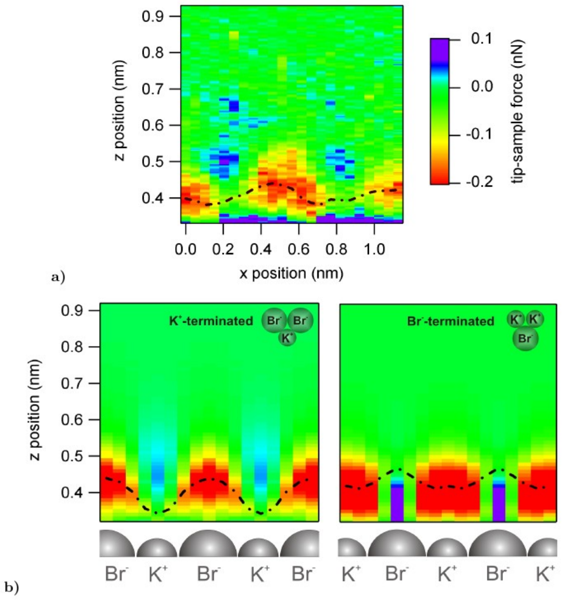

Fig. 1. a) The 2-dimensional map of the site-specific vertical tip-sample forces. The x axis covers a distance of 1.13 nm whereas the z axis extends over a distance of 0.6 nm. b) Simulated map of the site-specific tip-sample forces for a K+ terminated KBr-cluster (left), and for a Br− terminated KBr-cluster (right). The dotted black lines indicate the minimum of the force curves with respect to the z-axis at each x-position.

# Force spectroscopy using cantilever higher flexural modes

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There is a clear trend in NC-AFM towards increasing the sensitivity to the short-range interaction —responsible of atomic contrast— by reducing the cantilever oscillation amplitude to the sub-ångstrom regime, well below the characteristic decay length of these forces [1, 2]. Several approaches have been proposed to beat this technical challenge [3, 4]. Among them, a straight forward strategy for a cantilever-based AFM is to use the higher harmonics of the cantilever oscillation [5-7].

Force spectroscopy [8] using frequency modulation detection [9] has become a fundamental tool to clarify complex processes taking place between tip and surface in NC-AFM [10-12]. However, the formalism in which force quantification is based, in principle, cannot be directly applied for data acquisition using cantilever higher flexural modes, and a parameterization of the relation between frequency shift and tip-surface interaction force is required [13].

In this contribution, we use the first and second flexural modes of the cantilever oscillation to perform force spectroscopic measurements, and experimentally corroborate the validity of this parameterization [14]. Comparison of spectroscopic characteristics acquired under the same experimental conditions with these two flexural modes yields to an estimation of the cantilever effective stiffness for the second eigenmode larger than the value predicted from theory. This large effective stiffness enables us to perform measurements with cantilever oscillation amplitudes as small as 3.6 Å, and to provide valuable information regarding force measurements with higher harmonics and enhancement of the signal-to-noise ratio, that will be presented here.

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# Imaging and Mapping for discriminating atom species using Non-contact Atomic Force Microscopy

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Discrimination of atom species on surface is one of the important techniques for studying chemical reaction and/or atom manipulation. We have proposed that site-specific force spectroscopy using atomic force microscopy (AFM) can be applied to this purpose [1]. Difference of the maximum attractive force in the short-range interaction force brings us information of the atom species. We have shown that the ratio of the maximum attractive force is constant within experimental error.

In this contribution, we propose two methods in which one can obtain images and two-dimensional force maps featuring on atom difference. Figures 1 show simultaneous images of (a) AFM topographic and (b) atom discriminative images of the surface of Si, Sn, and Pb alloy on the Si(111) substrate. In the AFM topographic image, three species cannot be distinguished. In order to discriminate the species, we modulated tip-sample distance at amplitude nearly equivalent to attractive interaction force region and at frequency of larger than the band width of the *Z*-feedback circuit. In this scheme, in a certain condition, there is a response in the frequency shift at the same modulation frequency depending on the values of the maximum attractive force as in Fig.1 (b). Although it is difficult to mention the condition for obtaining images with quantitative color contrast at present, one can perform a quick look of the atom species of the surface with this method.

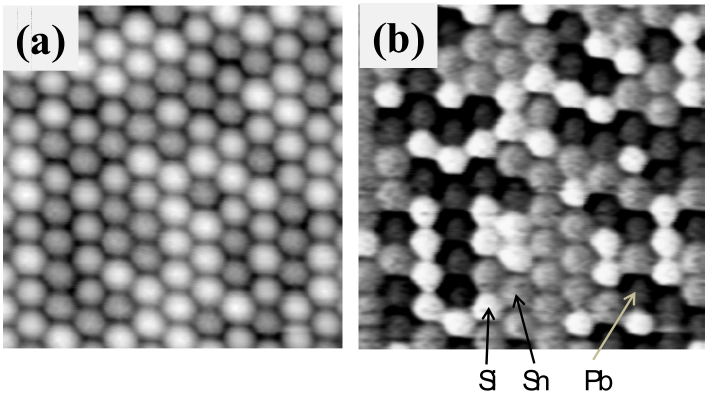

Fig.1 (a) AFM Topographic image of a surface alloy composed by Si, Sn and Pb atoms on a Si(111) substrate and (b) atom discriminative image. Both images were obtained simultaneously.

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# ¿Qplus, quo vadis?

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The first scientific publication about the qPlus sensor has appeared exactly a decade ago. Today, other groups[1,2,3] and commercial AFM manufacturers[4] have started to utilize this alternative to the traditional silicon cantilever. In the first ten years of its existence, several new possibilities of AFM were developed at the University of Augsburg: subatomic resolution,[5] lateral force microscopy with atomic resolution,[6] simultaneous STM and AFM with atomic resolution,[7] subatomic resolution with sub-Å lateral resolution by higher harmonic force microscopy,[8] simultaneous STM and AFM spectroscopy,[9] simultaneous force microscopy with work-function measurements[10] and in a collaboration with the IBM Research Lab in Almaden, the measurement of forces acting during atomic manipulation.[3] Many new users of the qPlus sensor like the very simple electric deflection measurement that reduces the experimental difficulties in measuring the cantilever deflection. But the small-amplitude operation feasible by qPlus has other benefits. The focus of our work in Regensburg is twofold: 1. decrease the deflection noise density of the sensor and 2. find new ways to prepare tips that are well characterized on the atomic level. The talk will discuss recent progress in these domains.

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4) See company webpages of Createc-Fischer GmbH (Erligheim, Germany), RHK Technology (Troy, MI, USA) and Omicron Nanotechnology (Taunusstein, Germany).
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10

# Poster Presentations

Tuesday,
16 September

# Session I

## Tuesday,

## 16 September

# Measurement of lateral tip-sample forces in the attractive regime with picometer resolution in three dimensions using NC-AFM

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Two decades ago, the first atomic-scale contact mode lateral force microscopy measurements [1] impressively demonstrated the validity of the Prandtl-Tomlinson model of stick-slip motion [2,3] for atomic friction. As a consequence of this discontinuous type of motion, it is impossible to measure the lateral forces acting on a sharp probe tip for many surface locations by contact mode measurements. Alternatively, three-dimensional dynamic force spectroscopy, i.e., the acquisition of frequency shift vs. distance curves in a dense raster over a surface, can be employed to map the *continuous* tip-sample interaction potential with high local resolution within the attractive regime. From these data, lateral tip-sample forces can be obtained by calculating the derivative in the appropriate directions. This ability makes this new technique interesting for high-resolution nanotribology.

Using our recently completed home-built low temperature, ultrahigh vacuum NC-AFM [4], we were able to probe the tip-sample interaction potential over highly oriented pyrolytic graphite in three dimensions with atomic resolution. Graphite was chosen due to its superior qualities as a prototype solid lubricant. From the recorded data set, lateral forces have been recovered quantitatively with pN resolution in a grid with only a few picometer spacing. We will discuss the measurement principle, the site-specific distance-dependence of the static lateral forces, their local distributions with regard to the underlying lattice at constant heights, as well as influences of the tip shape. Specifically, we find that significant lateral forces occur mainly within a relatively small area around the locations that show the largest attractive vertical force. To conclude, we will speculate on how these results might help explain graphite's extraordinary low frictional resistance experienced under most circumstances, and connect our data to Amontons' law of friction.

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# 3D-Force-Spectroscopy and -Dissipation Data of an Organic-Inorganic Interface: PTCDA on Cu(111) and Ag(111)

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Organic semiconductors have attracted intensive research over the last years. Especially the adsorption of π-conjugated organic molecules on metal substrates in view of potential applications in organic and molecular electronics gained a lot of interest. One of the most studied molecules is 3,4,9,10-perylenetetracarboxylic-dianhydride (PTCDA) and it has been investigated on a wide range of substrates.

Using non-contact atomic force (ncAFM) microscopy molecular resolution in the topography as well as in the dissipation has been achieved for PTCDA crystallites on KBr(001) [1]. Furthermore, experiments with PTCDA adsorbed on Cu(111) revealed, that the intramolecular contrast of the PTCDA-molecules depends strongly on the local adsorption environment [2]. Here we present first experimental ncAFM results of three-dimensional force-spectroscopy and dissipation experiments of PTCDA adsorbed on Cu(111) (Fig. 1) and on Ag(111) with submolecular resolution. The dissipation is understood as a hysteresis of forces between approach and retraction of the tip and is caused by bistabilities in the potential energy surface of the tip-sample system. Therefore the dissipation signal can reveal additional information of the tip-sample system.

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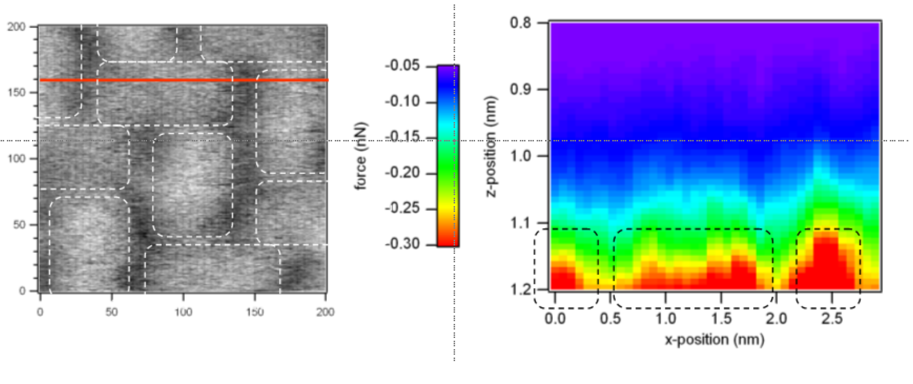

**Fig.1: PTCDA on Cu(111), 3x3 nm² scan, Δf = -50 Hz and 2D slice of vertical force along the solid line**

# Simulation on Non-contact Atomic Force Microscopy for Dihydride Si(001) Surface

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A simulation on the non-contact atomic force microscopy (ncAFM) is given for a dihydride Si(001) surface by a continuum model to the long range van der Waals force[1] and the density-functional based tight-binding (DFTB) method to the short range chemical force[2].

As for the dihydride Si(001) surface, an exotic ncAFM image has been reported in experiment[3]. The ncAFM image shows pattern changing that depends on a distance between the tip and surface. That is, arrangement of the ncAFM bright spots changes from 1x1 via 2x1 to 1x1 periodic structures as the tip-surface distance decreases.

On the assumption that a surface distortion affects the pattern changing of the ncAFM image, we performed the ncAFM simulations using a small tip cluster model: $Si_4H_9$[4]. As a result, we obtained the pattern changing of the ncAFM image when the tip-surface distance decreases. In the simulation result, the pattern changing is caused by not the surface distortion but a superposition of the force field generated by each adsorbed H atom. Though the simulation pattern did not agree with the experiment one, we found that the dihydride Si(001) surface is more stiff than we expected, so that we concluded that the surface distortion does not affect the pattern changing.

We expect that another cause of the pattern changing is a tip distortion. In the present study, we prepare a slightly large cluster for the tip model, e.g. a pyramidal shape consisting of several layers with Si atoms. The tip model is optimized by some conditions: gentle relaxation of the isolated system, collision with another tip or surface model, and so on, which will become meta-crystalline, amorphous, etc. Using those optimized tip models, we perform the ncAFM simulations on the dihydride Si(001) surface and discuss results.

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# NC-AFM observation of Ge(001) surface by improved cantilever with sharp tungsten tip

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Noncontact atomic force microscopy has an ability to image various surfaces and adsorbate structures at atomic scale. However, Si cantilever used predominantly in AFM has some drawbacks: anomalous atomic scale structures are imaged from a semiconductor surface due to complex interaction between the tip apex dimer and the surface dimer, and its complicated electronic state makes difficult to analyze spectroscopic data. To prevent the difficulties well defined metal tip with sharp apex should be desirable. In this study, we tried to produce a cantilever with sharp tip coated by tungsten thin film and investigated the availability of it utilizing Ge(001) surface.

At first, the commercial Si cantilever was sputtered by $Ar^+$ bombardment to remove contaminants and oxide layers on the tip. The cantilever tip was then faced to the tungsten plate and tungsten layer was formed on the tip by $Ar^+$ sputtering of the plate. This process tends not to change its resonant frequency (～170kHz). Electron micrograph of the tip apex of a tungsten coated cantilever is displayed in Fig. 1. The tip was found to be covered by 3-5 nm thick tungsten layer with 3nm radius of curvature. Figures 2(a) and (b) show topographic FM-AFM images of Ge(001) surface acquired by tungsten coated and Si tips at room temperature, respectively. For the tungsten tip imaging, the surface atomic configuration of the dimer structure, the c(4x2) phase, is always detected as shown in Fig. 2(a). On the other hand, the atomic resolved image obtained by the Si tip in Fig. 2(b) does not show the same c(4x2) configuration. We will show the detailed imaging mechanism and the tungsten coated cantilever tip has higher performance than Si ones.

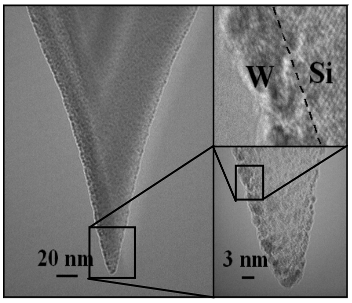

Fig. 1. TEM images of tungsten coated tip apex of a cantilever.

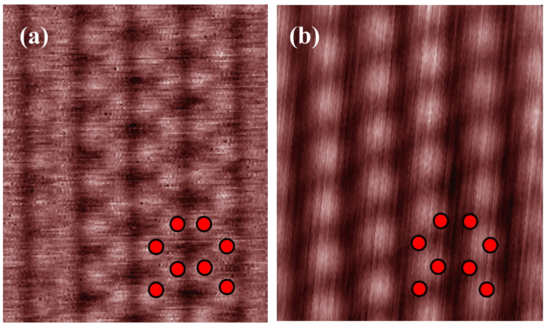

Fig.2. Topographic images of Ge(001) surface obtained with (a) tungsten coated tip (Δf=-56Hz) and (b) Si tip (Δf=-18Hz). Both scan areas are 4 x 5 $nm^2$.

# Simulation and theoretical study of small-amplitude lateral force microscopy – Effect of amplitude on AFM images of Si(111) 7×7 surface

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Recently attempts to detect the lateral force gradient between the single atoms at non-contact region have been carried out [1,2]. However the large peak-to-peak dithering amplitudes of 6 Å or 2 nm, and intently or accidentally given tilt angles from the parallel to the surface confuse us the interpretation of the observed images. Therefore small-amplitude lateral dynamic force microscopy with high atomic resolution on the order of sub-Å has been developed [2,3]. Nevertheless mechanism of atomic resolution has not been clarified yet.

We have developed theory and simulation method of two-dimensional map of Si(111)7×7 surface of small-amplitude lateral AFM. In this talk, effect of dithering amplitude and scanning direction on the two-dimensional map is studied and compared with experiment to study the mechanism of atomic resolution of lateral AFM.

Tip-surface interaction is modeled by the Morse potential [4] fitted to the first-principles simulated results [5] of the Si tip-Si(111)7×7 surface interaction force (Fig.1). Using this potential, two-dimensional lateral AFM images are simulated. Peak to peak amplitude of the tip oscillation is set as 0 Å < App < 10 Å, and the tip-apex height is set as 4 Å < z < 7 Å.

For App = 0 Å, the sharp bright spots appear on the adatom. However, when the App increases to the length equivalent to the distance between neighboring Ce-F and Ce-UF atoms, two bright regions due to the Ce adatoms are broadened along scan direction, and remarkable change of the AFM pattern occurs as shown in Fig.1. Furthermore, if the tip is retracted from the surface for z > 0.5nm, clear bright spots between neighboring adatoms come to become unclear and connected. Here simulated image patterns can reproduce experimental ones well as shown in Fig.2.

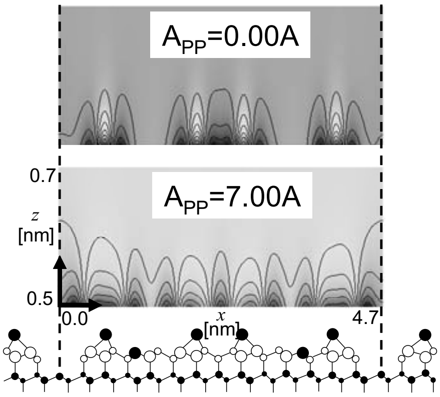

Fig.1 Δν along diagonal line of Si(111) 7*7 surface for each tip height

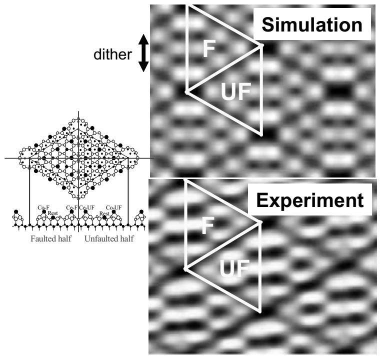

Fig.2 L-AFM images obtained by simulation and experiment for Si(111)7*7 surface

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# Automatic procedure for tip apex simulation

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In the last decade computer simulation played a vital role in understanding the NC AFM apparatus, including its unique atomic-scale and chemical resolution and nanomanipulation capabilities. Unlike substrates, which are usually reasonably well known, the shape and morphology of the NC AFM tip apex are typically only barely known, which severely limits the predictive power of any realistic simulation.

We propose an automatic procedure for generation of tip apex realized in the experiment under investigation. As an input the procedure uses the experimental force-distance curve(s) (FDC). The method is based on a Monte-Carlo (MC) fit of the FDC in an inverse MC manner. The procedure uses a weighted sum of squares of the forces acting on all mobile atoms in the model and sum of squared differences between forces acting on fixed atoms in the model tip and experimental tip-sample forces in each point of the FDC simulated as a penalty function. The first contribution drives the procedure towards minimum-energy structures, whereas the other drives the structural/morphological tip apex modifications. Initially the ability of the method to recreate the correct experimental tip apex structure was successfully tested on a number of systems/apexes which could be modeled by emprical interatomic potentials. Further development is under way where results obtained with empirical potentials are further refined by tight-binding/DFT modeling.

Our procedure provides an important missing link to a complete and reliable simulation of the NC AFM apparatus.

# Direct quantitative measurement of lateral forces at atomic scale on Si(100)(2x1) surface using non-contact Atomic Force Microscopy (nc-AFM)

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Atomic Force Microscopy has shown its capability to manipulate single atom and molecules laterally, however quantitative measurement of lateral forces that govern the activated or induced motion of atomic or molecular scale objects along the surface has still remained a challenge. To investigate this important issue, we designed and constructed a nc-AFM, capable of measuring lateral stiffness simultaneously with the tunneling current. In our technique, a sensitive fiber interferometer is aligned at the side of a home–made tungsten cantilever with typical stiffness of about 150 N/m. An RF circuit is also designed to inject RF current into the laser diode to improve the noise performance. A noise level of ~$9\times10^{-5}$ Å/$\sqrt{Hz}$ is routinely obtained. The cantilever is dithered in lateral direction with respect to the sample with sub-Ångstrom oscillation amplitudes ($A_0$ ~ 0.25 Å) at a frequency well below the resonance frequency and the changes in lateral oscillation amplitudes due to the tip-surface interaction are measured with a lock-in amplifier. We will present lateral stiffness and STM topography images on Si(100)(2×1) surface obtained with this ncAFM.

# FM-Kelvin Probe Study of Nanoindentations on KBr(100)

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## Body of abstract

Indentations on KBr(100) have been produced using the contact force microscopy and examined with non-contact force microscopy in ultrahigh vacuum (Fig. 1). Cleaved samples of KBr(100) were imaged before and after indentation, where the sharp tip of the microscope's probe was used both for imaging and indenting. Discontinuities in the force-distance curve recorded during indentation are a result of atomic scale plasticity and nucleation of single or multiple dislocations.

In the initial study of these indentations, it was observed that the surface charge varied significantly between areas of undisturbed KBr and in the vicinity of the indent [1]. The use of Frequency Modulated Kelvin Probe Microscopy (FM-KPM) has allowed for the compensation of the changes in the surface states of the material, yielding a pseudo-contact potential difference map over the surface of the insulator (Fig. 2). FM-KPM enables for the observation of the change in surface charge over the indentation site itself, as well as on sites of atomic dislocations, and kink sites at step edges. We will discuss the problems and the perspectives of the slow charge relaxation on bulk insulating crystals in FM-KPM.

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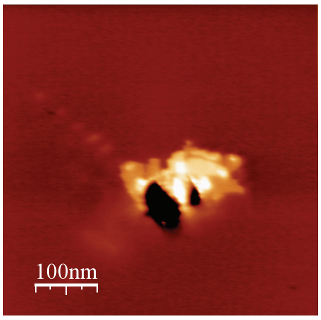
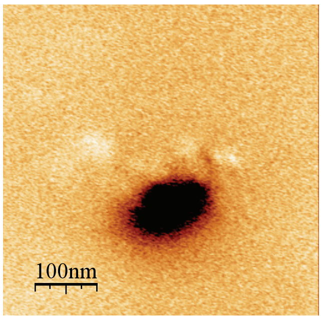
Fig. 1.



Topographic Image

Fig.2.



Kelvin Probe Image

# Tip-induced reversible potential change of ferrocenylthiol molecules using atomic force microscopy

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Alkanethiol self-assembled monolayers (SAMs) have been studied for more than 20 years due to their precisely controlled thickness and well-ordered structures. They also allow us to easily design and control the surface chemical functionalities of substrates. Recently, several research groups reported on studies of ferrocenylthiol SAMs as electrochemically-active ultrathin films, which are expected to be applied to memory devices and molecular electronics with control of their electrochemical states [1, 2].

In this study, we demonstrated local redox manipulation on 11-ferrocenyl-1-undecanethiol (11-FUT) SAMs using atomic force microscopy (AFM). A DC bias voltage was applied to the 11-FUT SAM while the tip was connected to the ground in order to manipulate local electrochemical states of the SAM under the conditions shown in fig. 1(a). The electrochemical states of the SAMs were detected as surface potential (SP) changes using Kelvin probe force microscopy (KFM). All the AFM/KFM experiments were performed in an atmosphere at room temperature. The SAMs were made on Au(111) surfaces by immersing the substrates into its ethanol solution (1 mM, 3 hours). Figure 1(b) and (c) show an AM-AFM (topographic) image of the SAM and the corresponding KFM (SP) image, respectively. The SP of an area where a negative bias voltage was applied was approximately 10 mV higher than that of an area where no bias voltage was applied. In addition, the SP was reversely changed when we applied a positive DC bias voltage. We also carried out the same experiment on the 1-decanethiol (DT) SAMs, which have no electrochemically-active functional groups, and no SP change was detected.

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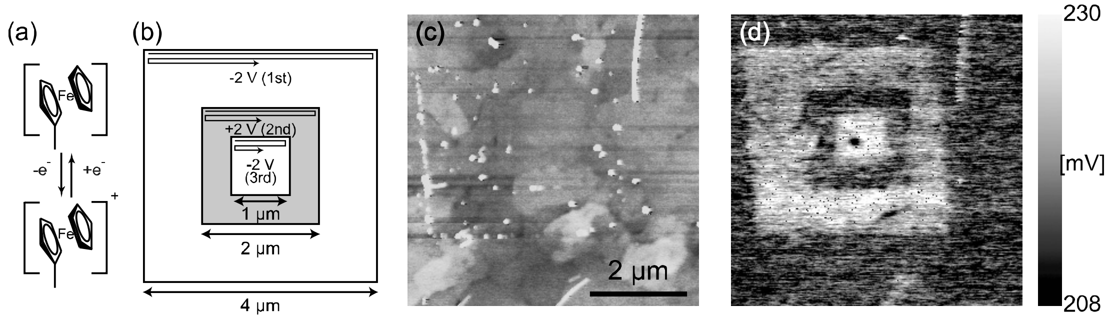

Fig.1 (a) the redox of a ferrocenyl group, (b) the schematic for explaining the condition of redox manipulation, (c) an AM-AFM image of the 11-FUT SAM, (d) the corresponding KFM image.

# Atomic scale contact potential variations on the Si(111)-7×7 surface detected by amplitude modulation Kelvin probe force microscopy

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We present atomically resolved Kelvin probe force microscopy (AM-KPFM) measurements of the Si(111)-7×7 reconstructed surface with the amplitude sensitive technique at the second resonance of the cantilever [1]. Enhanced sensitivity due to the high mechanical quality factor in ultra-high vacuum enables local contact potential difference (LCPD) measurements of individual adatoms as shown in Fig. 1. The measured LCPD variations between adatom and corner hole changed by the tip-sample distance, and values were increased up to 117 mV. The distance dependence was investigated by a quasi-constant height mode. The CPD of the entire surface also shifted, and this phenomenon indicates that the short-range electrostatic force, arising from the individual surface atoms, plays a major role in KPFM, especially at small tip-sample distances. Moreover, the condition of the tip apex strongly affects the measured LCPD as shown in Fig. 2.

Our theoretical calculations indicate that amplitude sensitive KPFM at the second resonance has a higher sensitivity than frequency modulation KPFM in practical cases. Moreover, further enhancement of the sensitivity can be expected by using smaller amplitudes of the fundamental mode for topographic imaging.

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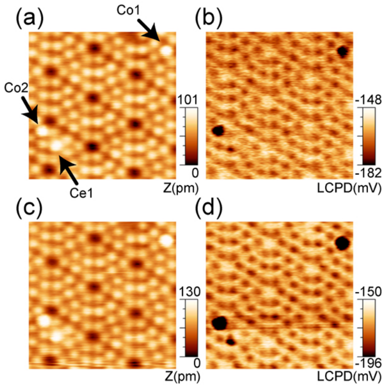

Fig. 1. (a) and (c) DFM topography. (b) and (d) simultaneously detected CPD images. A = 12 nm. (a) and (b) df = -5.45 Hz. (c) and (d) df = -6.05 Hz.

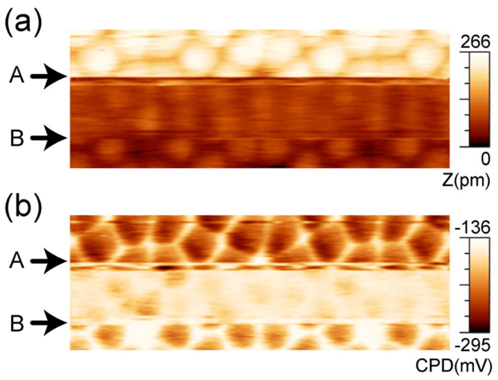

Fig. 2. (a) DFM topography and (b) simultaneously detected CPD image. df = -9.05 Hz. The tip was accidentally crushed on the surface at A and B points.

# Lateral Resolution and Potential Sensitivity in Kelvin Probe Force Microscopy: Towards Understanding of the Sub-nanometer Resolution

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We report on high resolution potential imaging of heterogeneous surfaces (clean surface of compound semiconductor InSb(001) and the same surface with some submonolayer coverages of KBr and Au) by means of Kelvin probe force microscopy, working in frequency modulation mode (FM-KPFM), performed in ultrahigh vacuum. It has been found that long- and short-range bias-dependent interactions, acting between the tip and the surface, can be detected and that both interactions contribute to the measured Contact Potential Difference (CPD) signal.

On the one hand, when only the long-range electrostatic interactions between the tip and the surface are active, the CPD map provides the distribution of the local surface potential on the imaged sample with the lateral resolution and the correctness of the measured values depending on the measurement conditions. For this case, the experimental findings are compared with the predictions of theoretical calculations based on a realistic model for the cantilever-sample geometry.

On the other hand, when the short-range and bias-dependent interactions are detected, FM-KPFM provides even the sub-nanometer contrast in the CPD signal. In this situation, however, the measured CPD signal is not related to the sample surface potential but reflects the properties of the front tip atom-surface atom interactions.

# Coupled Kelvin Force Microscopy and Transport in carbon nanotube field effect transistors

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Carbon nanotubes (CNTs) can be used as charge detectors with single-electron sensitivity, as demonstrated recently by the observation from transport measurements of elementary charging events in metallic particles coupled to CNT field-effect transistors (CNT-FETs) [1].

The aim of this presentation is to investigate the mechanisms of the response of CNT-FETs to local charge gating using combined transport and local electrostatic mapping by Kelvin and electrostatic force microscopy. To do so, we generate out-of-equilibrium local charges in the vicinity of CNT-FETs by charge injection from an atomic force microscopy tip in the surface of the oxide layer supporting the CNT device (typically +100e or -100e at a ~200nm distance from the CNT).

Electronic transport measurements (source-drain current $I_{ds}$ as a function of gate bias $V_g$) show that the local positive (resp. negative) injected charge induces an effective shift of the $I_{ds}(V_g)$ CNT-FET characteristics towards negative (resp. positive) $V_g$, i.e. of opposite sign compared to the backgate.

This is explained by KFM measurements which show that the contacted nanotube responds to the charge injection by emitting a charge pattern delocalized along the CNT-FET channel (i.e. up to a few µm in length). This effect is attributed to the charging of the CNT-FET channel and its environment [2] from its leads, in agreement with the effective $V_g$ shifts of the $I_{ds}$ ($V_g$) characteristics. The delocalized response of the CNT-FET upon local gating will be discussed. Preliminary KFM data obtained in ultra-high vacuum will also be presented.

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# Analytical and numerical approaches to the local contact potential difference on (001) ionic surfaces: implications for Kelvin Probe Force Microscopy

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Last year, the issue of atomically resolved contact potential difference (CPD) images on KBr(001) had been addressed with our nc-AFM simulator. The images had been acquired in the Amplitude-Modulation Kelvin Probe Force Microscopy (AM-KPFM) mode. Although it was possible to reproduce a numerically-estimated contrast of the CPD with an atomic periodicity compatible with the one of the crystal, its strength did not match the one of the Madelung surface potential at an equivalent height from the surface. This work however, relied on a somewhat raw estimation of the electrostatic force between the biased tip and the (001) surface of the ionic crystal.

In the present work, a more refined analytical model of the electrostatic force is reported [1]. The expression of the force can be split into two major contributions: the first stands for the coupling between the microscopic structure of the tip apex and the capacitor formed between the tip, the ionic crystal and the counter-electrode; the second term depicts the influence of the Madelung surface potential on the mesoscopic part of the tip, independently from its microscopic structure. The former has a short-range character with the lateral periodicity of the Madelung surface potential whereas the latter rather has a medium-range character and acts as a static component which shifts the total force. These electrostatic forces are in the range of ten pico-Newtons. Beyond the dielectric properties of the crystal, a major effect is the ionic polarization of the sample due to the influence of the tip/counter-electrode capacitor. When explicitly considering the crystal polarization, an analytical expression of the bias voltage to be applied on the tip to compensate for the local CPD, *i.e.* to cancel the electrostatic force, is derived.

Indeed, the analytical expression of the compensated CPD exhibits the lateral periodicity of the Madelung surface potential. However, the strong dependence of the force upon the atomic structure of the tip and upon the tip-sample distance makes questionable the possibility to quantitatively interpret KPFM measurements in the short-range regime (local CPD). Nevertheless, the analytical approach remains helpful since it allows us to address in details the influence on the CPD of each contribution to the total force.

With this goal in mind, new simulations which mimic the usual KPFM bias spectroscopy curves have been performed with the nc-AFM simulator. When weighting differently the two contributions (short *vs.* medium-range), it is possible to derive a panel of trends, some of them having been experimentally observed. We have particularly focused on: i-the frequency shift of the first eigenmode of the cantilever, which is crucial while FM-KPFM experiments and ii-the evolution of the modulated amplitude of the second eigenmode of the cantilever, which is rather used while AM-KPFM experiments.

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# Investigations of Pt Clusters on $TiO_2(110)$ by Kelvin Probe Force Microscopy in a $N_2$ Gas Environment with an Atmospheric Pressure

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Frequency modulation atomic force microscopy (FM-AFM) is a powerful method not only for imaging surfaces on an atomic scale but also for investigating surface properties. However, high-resolution FM-AFM imaging has been limited in vacuum environments where the Q-factor of the cantilever resonance usually exceeds 10,000. It is heavily reduced in an environment with an atmospheric pressure and hence the effective force sensitivity is decreased. We have developed a high-resolution FM-AFM working in both air and liquids based on a commercially available AFM (Shimadzu: SPM-9600) and have obtained high-resolution FM-AFM images of a polydiacetylene single crystal surface and lead-phthalocyanine thin films on a $MoS_2$ surface in air[1]. In this study, we have developed a high-resolution Kelvin probe force microscopy (KFM) utilizing FM-AFM for mapping local surface potentials (SPs).

Platinum is a highly efficient cocatalyst for photocatalytic reactions on a titanium dioxide ($TiO_2$) substrate. Improved catalytic performance is attributed to electron trapping by nanometer-sized Pt clusters as well as to their catalytic activity. Figures 1 show high-resolution KFM images of Pt clusters on $TiO_2(110)$ substrate in $N_2$ gas with atmospheric pressure. The bright particles marked by white circles in the topographic image (a) are the Pt clusters. The surface potentials of the Pt clusters marked with the circles are higher in the SP image (b) than those on the surrounding part of the clusters. In contrast, the surface potentials on the bright particles indicated by arrows in (a) are lower, as shown in (b). These results demonstrate that our KFM is capable of mapping surface potentials with a few nanometer resolution even in an atmospheric pressure environment.

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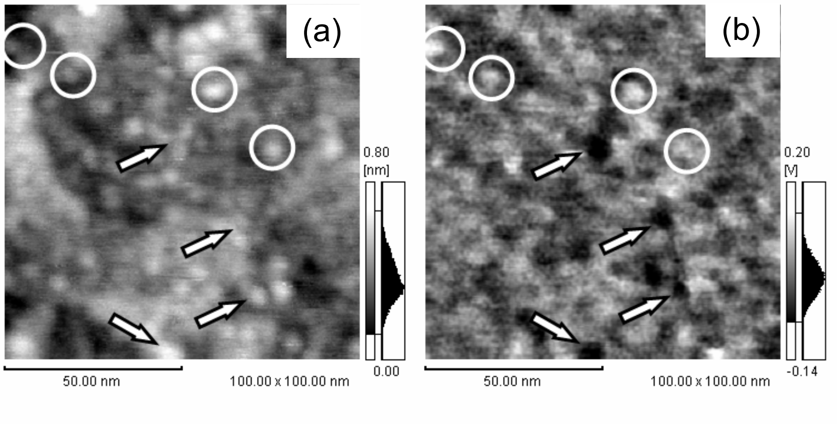

Fig. 1. KFM images of Pt clusters on a $TiO_2$ substrate in an atmospheric pressure $N_2$ gas environment. (100nm x 100nm, $\Delta f$=-80Hz, A=5.2 nm, Q=436) (a) Topographic image and (b) surface potential image.

# High-Resolution Surface Potential Mapping on Operating Organic Field Effect Transistor by FM-KFM

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Investigation of the local structures and the electrical properties of organic thin films is becoming more and more important for development of the high performance organic field-effect transistor (FET) devices. The properties in OFETs are often limited by the morphology, molecular structure and electronic state at the interface between the molecules and the metal electrodes. Frequency modulation AFM with Kelvin-probe force microscopy (FM-KFM) is an attracting tool to investigate those morphological and electrical properties at a nanometer-scale resolution. In this study, we fabricated OFET by bridging the crystalline semiconductor film between metallic electrodes on silicon oxide and performed high-resolution mapping of local surface potential (SP) on operating OFET.

We fabricated a crystalline island of oligothiophene (sexithiophene, α-6T) molecules connected across Pt electrodes patterned by electron beam (EB) lithography. Figure 1 shows a topographic image of α-6T island by FM-KFM operated in vacuum. The island between the electrode exhibited crystal habits indicating its high crystallinity. Figure 2 shows a series of SP images obtained during the bias voltage was applied to the upper electrode. The SP on α-6T domain was continuously changed depending on the applied bias voltage.

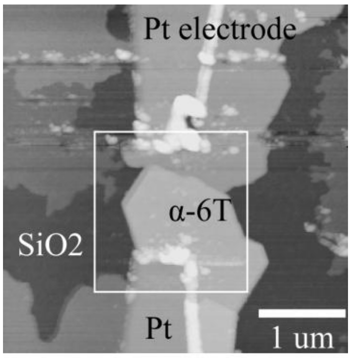

Fig.1 Topographic image of α-6T film deposited around the Pt gap electrodes.

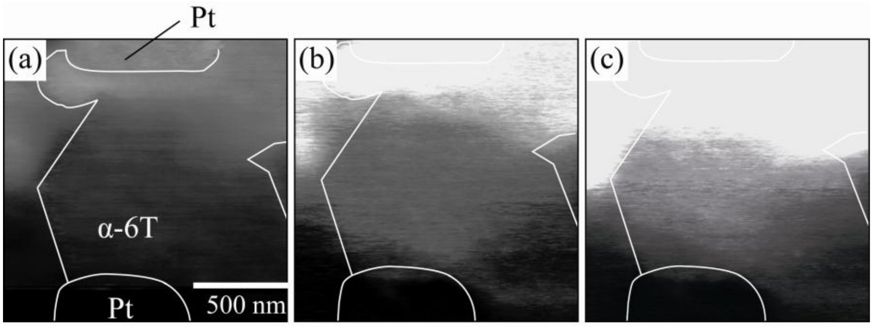

Fig.2 Surface potential images of 6T film (enlarged images in white square in Fig.1) during bias voltage was applied to the upper electrode. (a) 1.0 V, (b) 2.0 V, and (c) 3.0 V.

# KPM on Pd nanoclusters: work function changes due to gas interaction

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Work function (WF) measurements of supported metal nanoclusters are of vital importance for a better understanding of the catalysis involved in different processes. These measurements give direct information about the electronic structure of the clusters which, in turn, determines their catalytic behavior [1,2]. In this work we use the Kelvin probe microscopy (KPM) in UHV [3, 4] to study the WF variations of Pd nanoclusters deposited on graphite under different preparation conditions.

It has been found that the WF of as-prepared nanoclusters strongly depends on the substrate temperature and on the residual gas pressure during the Pd deposition. By changing the latter growth conditions, the cluster WF can vary within a range of 600 meV and more. Moreover, we observed that, irrespectively of the initial cluster WF state, the cluster WF always decreases to a constant value, what happens even at very low pressure conditions in the UHV chamber ($10^{-10}$-$10^{-9}$ mbar). The WF change is always faster for smaller clusters than for larger ones (Fig. 1). These measurements show that the WF change of the Pd clusters is very sensitive to chemical reactions between the clusters and the residual gas of the UHV. In order to discriminate between different possible chemical reactions, post-treatments of the clusters in oxygen, hydrogen and in $CO_2$ at different temperatures and partial pressures will be discussed in the contribution.

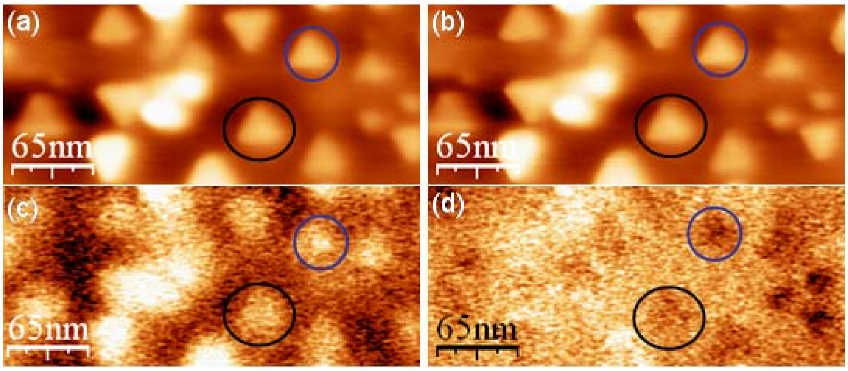

**Fig. 1.** Topography of the Pd nanoclusters: (a) as-prepared sample (b) 24h later. (c) and (d) are corresponding KPM images where the cluster WF change with respect to graphite as reference is clearly evidenced.

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# Nanoscale Strain and Composition Mapping Inside Quantum Dots Using UHV Kelvin Probe force microscopy

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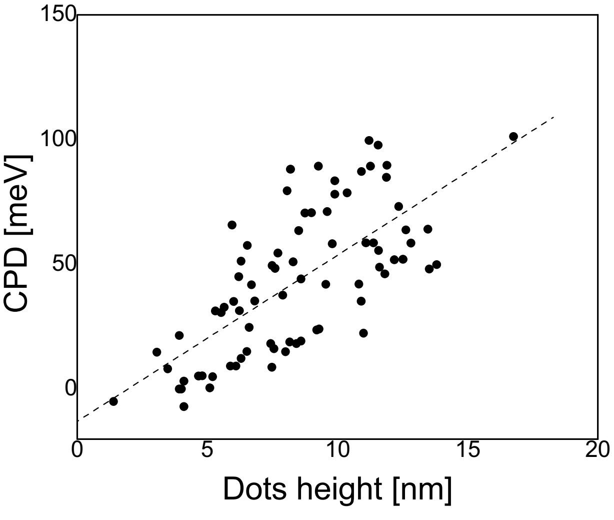
One of the key factors in improving quantum dots (QDs) electrical properties and QDs based devices is the ability to control the crucial parameters of composition, uniform doping, size, and strain distribution within the QDs, as well as the crystalline structure at the QDs-substrate interface and surface chemistry.

We present ultra high vacuum Kelvin probe force microscopy (KPFM) measurements of the work function inside and around InSb and InAs QDs. The QDs were grown on highly Te-doped GaAs, InSb, and GaSb (100) oriented substrates by the droplet heteroepitaxy (DHE) method, producing a wide variety of different strain and composition conditions. The KPFM measurements were conducted in a modified UHV AFM (VT AFM, Omicron Inc.) operated at pressure $\leq 10^{-10}$ mbar, where the topography is measured in the non-contact frequency modulation mode at the first cantilever resonance, while the AC voltage applied to the tip for the KPFM measurements is tuned to the second resonance frequency.

It was found that the measured contact potential difference (CPD) is very sensitive to size of dots as shown in the figure below; small and thus higher strained dots show bigger change in their work function (relative to the substrate) than big relaxed dots. The work function changes with strain due to changes in the conduction band minimum energy, $E_C$. The results were analyzed using a two-dimensional model which assumes a linear elastic behavior, with smooth cosine-like variations of the composition and strain profiles for both the substrate and the dots. A good correlation between the model and the measured work function was achieved, showing that the nanometer resolution work function measurement is a powerful non-destructive method for mapping strain and composition in QDs and QD arrays.



# Kelvin probe force microscopy on locally modified self-assembled monolayers on Si substrates

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Self-assembled monolayers (SAMs) have been intensively studied due to their highly ordered structures and uniform thickness. Since 1-hexadecene (HD) SAM on Si(111) substrates, where the HD molecules are covalently bonding to the Si substrates, has high durability to chemical etching, the SAM is preferable as ultrathin resist films for nano-micro processing of Si. Studies on local properties, particularly chemical properties, as well as on structures of the modified regions will help us to understand the patterning mechanism of the HD-SAM and to develop reliable nano-processing techniques. Kelvin probe force microscopy (KFM) is well known as a tool for investigating surface potential (SP) distributions at nanometer scales. Since SP of an organic monolayer on an inorganic substrate reflects its molecular dipole moment, chemical changes induced in the monolayer can be monitored with a higher sensitivity compared to structural analysis [1].

In this study, the HD-SAM was locally modified or degraded using some technique and was further investigated using KFM. For example, Figures 1 (a) and (b) show an AM-AFM image of a HD-SAM and the corresponding KFM image, respectively. The SAM was modified by applying DC bias voltages using an AFM tip under the conditions shown in fig. 1(c). Both the topographic and the SP changes were detected where a DC bias voltage (>= 6 V) was applied to the sample, which must be due to the anodization of the Si and the degradation of the HD molecules.

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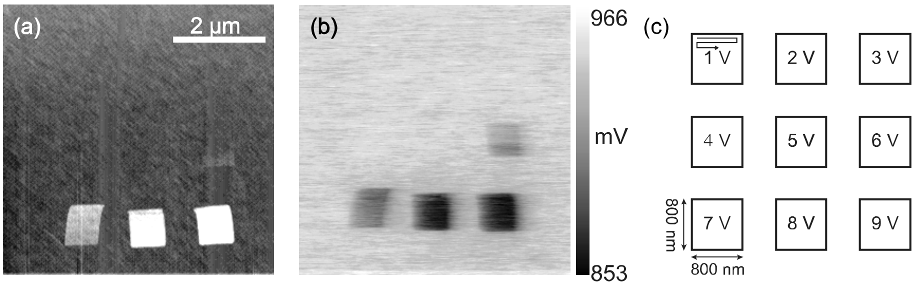

Fig.1 (a) AM-AFM image of a locally modified HD-SAM, (b) Corresponding KFM image, (c) Schematic illustration explaining the condition of AFM-anodization.

This work was supported by KAKENHI (Grant-in-Aid for Scientific Research) No. 19049010 on Priority Area "Strong Photons-Molecules Coupling Fields (470)" from the Ministry of Education, Culture, Sports, Science and Technology of Japan.

# Local Surface Potential Measurements of Co-ferritin nanodot by Kelvin Probe Force Microscopy

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We fabricated Co (cobalt metal) cores structure by using a low energy $Ar^+$ ion beam. A monolayer of ferritin molecule ($Co_3O_4$ : cobalt oxide) was adsorbed on the thermal silicon oxide layer. The bombardment energy was optimized using Ar gas by changing the input power after the protein of the monolayer was eliminated with $UV/O_3$. Though it was resulted in a poor reduction when the time of ion beam was less than 30 sec, $Ar^+$ ion beam enabled completely reduction when the time of ion beam was in 60 sec. We reduced the core particles to conductive Co nanodots. X-ray photoelectron spectroscopy (XPS) measurements confirmed the reduction of the cores. As a result, the diameter of the Co ferritin nano-structure was 7 nm, which was not identical to that of the iron core in the ferritin after ion beam. Additionally, the Kelvin Probe Force microscopy (KPFM) profile was not almost identical between $Co_3O_4$ and Co cores. It is very difficult for conventional $Ar^+$ beam processes to fabricate such fine structure of Co cores, because the high energy ions enhanced the bombardment damage of the cobalt core in the conventional reduction processes. Furthurmore, the diameter of the Co ferritin core decreased from 7 nm to 5 nm after the ion beam process. This report, that the electron confinment in the quantum nanodots are produced usng ferritin, is the convincing evidence that the biomolecule can be introduced in to the fabrication process of electronic devices. The results obtained here will open up a new field combining semicoductor technologyand biotechnology.

# Growth and manipulation of $C_{60}$ islands on $TiO_2(110)$

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Non-contact atomic force microscopy was employed to study the system of $C_{60}$ molecules on the rutile $TiO_2$ (110) surface *in situ* at room temperature. At submonolayer coverages, the molecules adsorb preferentially at substrate step edges. Upon increasing coverage, islands grow from the decorated step edges on the lower terraces. Simultaneous imaging of the substrate's bridging oxygen rows and the $C_{60}$ island structure reveals that the $C_{60}$ molecules arrange in a rhombic superstructure, with the molecules lying centered in the troughs between the bridging oxygen rows of the substrate (Fig. 1). Although the $TiO_2$ (110) surface exhibits a high density of intrinsic defects, the observed $C_{60}$ islands were of striking perfection. This indicates that the $C_{60}$ intermolecular interaction is sufficient for growing rather perfect molecular islands on a defective surface. Domain boundaries were determined to run parallel to the superstructure's basis vectors and are characterized by a single strand of protruding $C_{60}$ molecules along the junction (Fig. 2). When scanning on the $C_{60}$ islands, holes were successfully created into these islands by scanning at increased detuning setpoints. Besides hole formation, also the movement of a domain boundary was observed.

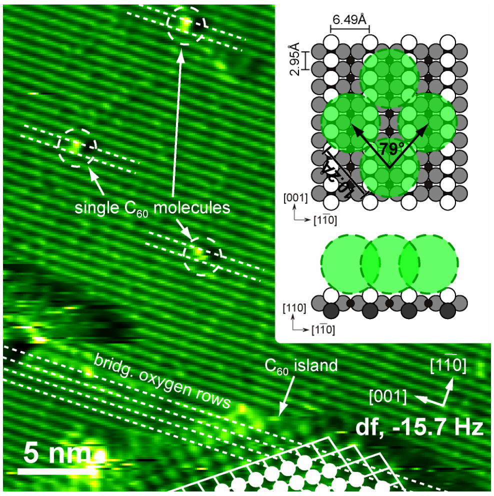

Fig 1.: NC-AFM image showing the substrate with single $C_{60}$ molecules on the upper terrace and a $C_{60}$ island on the lower terrace anchored at the step edge. Inset shows the rhombic $C_{60}$ adsorbtion structure.

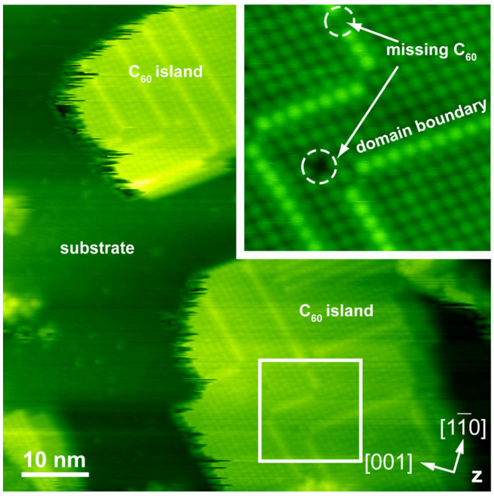

Fig 2.: NC-AFM image of $C_{60}$ islands adsorbed on the $TiO_2$ (110) surface. Inset shows protruding domain boundaries and defects on the plain $C_{60}$ islands.

# Theoretical modelling of the effect of an AFM tip on intra-molecular rotation

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Atomic force Microscopy (AFM) is an invaluable tool for the imaging of surfaces and adsorbates. In particular, a strong dissipation signal in AFM may be a sign of tip-induced processes taking place, such as adsorbate diffusion or intermolecular rearrangements.

We investigate theoretically the origin of a strong dissipation signal in the NC-AFM image of dimethyl-PTCDI on a Ag(111) substrate, observed in the experiments done at the University of Duisburg [1]. We suggest that the tip-induced rotation of the $CH_3$ group may be responsible for this dissipation signal. Therefore, we perform density-functional theory calculations to find the energy barrier of the $CH_3$ group rotation and to investigate the effect of the surface and the oscillating AFM tip on this barrier. We show that while the barrier of the $CH_3$ rotation is very low for the molecule in the gas phase, the tip hinders this rotation. Thus, the increase in the dissipation signal in AFM images may be due to the tip interfering with and stopping the rotation of the $CH_3$ groups of dimethyl-PTCDI.

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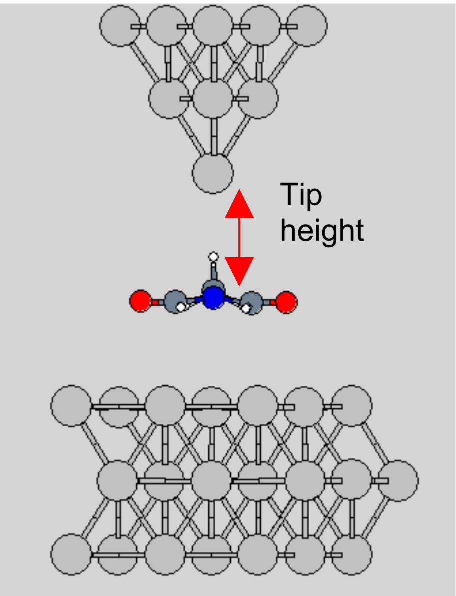

Fig. 1. The substrate-molecule-tip system used in our calculations.

# Tip effects in dissipation imaging: Challenges for interpretation

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It is a common feature of scanning probe techniques that the probe structure and properties influence the measurement. Indeed, great effort is undertaken to understand these effects on imaging resolution and contrast, particularly in nc-AFM. We present experimental results obtained on PTCDA on KBr at both room temperature and ~100K which demonstrate the influence of the imaging tip on dissipation contrast, both at the molecular scale and across molecular island edges. Between differing macroscopic tips (different cantilevers) and changes in microscopic tip shape (tip changes of the same cantilever), significant variation in contrast states was observed at both temperatures including inversion of contrast, and no observable contrast. The most striking examples occur for tip changes during imaging where dissipation contrast suddenly appears and then disappears with very little change in the topographic image (see fig. 1 below).

The variability of the results obtained due to different macroscopic and microscopic tip shape and/or composition lead us to urge caution in interpreting the outcome of individual dissipation experiments. Without a detailed tip characterization, even qualitative statements might be problematic. Additionally, there is a significant variation in imaging conditions used for dissipation measurements, which points to the open challenge of finding the optimal imaging parameters and tip configurations for obtaining reproducible dissipation results.

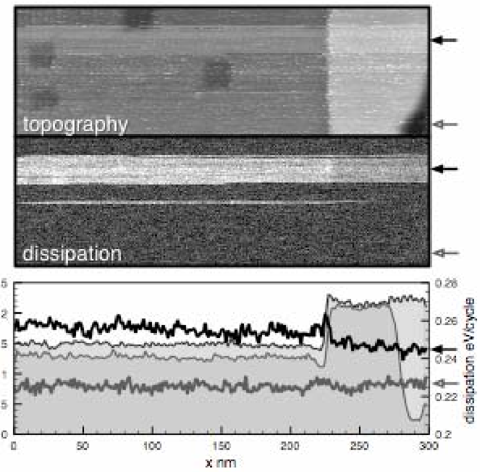

Fig. 1. Measurement of topography (top) and dissipation (middle) exhibiting a tip change with minimal effect in topographic imaging, but with a dramatic effect in the dissipation channel.

# Structure of water films on ionic crystals: The role of surface defects and lattice mismatch.

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A non contact Atomic Force Microscopy mode called Scanning Polarization Force Microscopy (SPFM)[1] and non-contact dynamic AFM was used to study water structure on $BaF_2(111)$ and $CaF_2(111)$ surfaces. $BaF_2(111)$ is known to expose hexagonal arrays of barium and fluoride ions whose lattice constant almost matches that of the basal face of Ih ice. This structural coincidence is believed to induce an ice-like water films adsorbed on the surface even at room temperature. $CaF_2(111)$ has a similar structure but the lattice mismatch with Ih ice is so important that no induced ice-like structure is expected on this surface. Contrary of what was expected a high density of defects (steps) on $BaF_2(111)$ surface was found to stabilize macroscopic ice grown on the surface.[2] Here we present a study of the role of defects and lattice mismatch at nanometer scale on the structure of adsorbed water films.

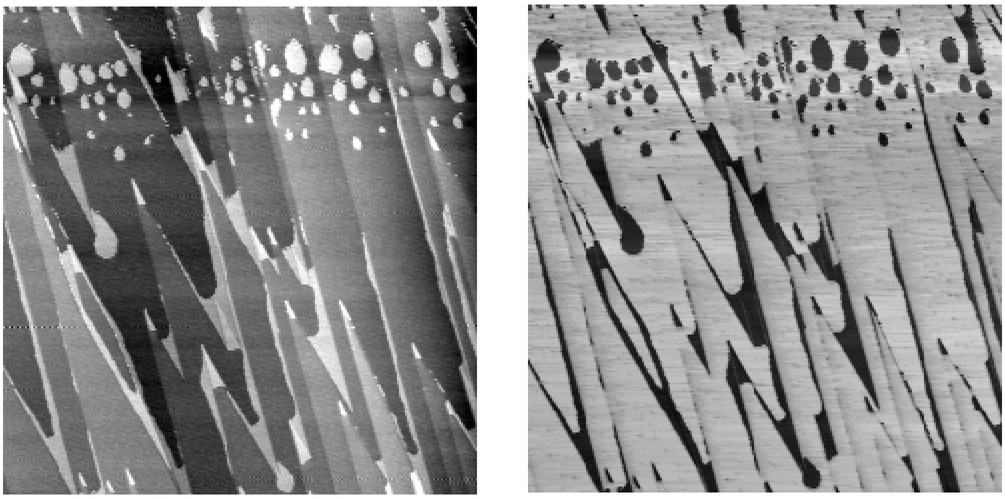

Fig 1: Topographic (left) and phase (right) images of a stepped BaF2(111) surface at room conditions. Water films can be observed preferentially at the triangular steps.

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# Characterization of the NaCl Suzuki structure by scanning force microscopy

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Due to their insulating character, the (001) surfaces of bulk alkali halide crystals are emerging as important substrates in many nanosystems. Particular focuses are supported metal nanoclusters for the study of catalytic properties and the adsorption of molecules to the surface for applications in nanoelectronics. For simultaneous characterization of both the substrate and nano-object at the atomic scale, Scanning Force Microscopy (SFM) remains the tool of choice, but on the highly symmetric alkali halide surfaces, unambiguous interpretation of contrast is difficult. Possible solutions, such as using force spectroscopy, are extremely challenging both experimentally and theoretically, and do not offer a routine approach to the problem. It is clear that a much more desirable setup would provide interpretation directly from images alone.

Recently, it was proposed to dope NaCl crystals with divalent impurity cations as part of a combined SFM and Kelvin probe microscopy study [1]. Above a certain divalent impurity content, the doped NaCl system creates precipitates in their well-known Suzuki phase [2] on the surface. The precipitates are embedded in the NaCl(001) matrix, so that two different types of surfaces regions, which are well separated, can be found. The atomic unit cell of the Suzuki phase is twice as large as the one of NaCl(001) and is composed of three different ions including Na vacancies. In this work we combine SFM experiments with *ab initio* and atomistic simulations to show quantitatively that all ions in the Suzuki structure ($NaCl:Mg^{2+}$ or $NaCl:Cd^{2+}$) can be unambiguously identified in images with atomic resolution, independently of the tip's polarity and the chemical nature of the divalent impurity ions. We explore in detail the dependence of contrast on the tip-surface distance dependence and the role of atomic displacements in observed features.

Our results demonstrate that doping NaCl crystals with divalent impurity cations nanostructures the (001) surfaces with Suzuki precipitates, offering templates for adsorption and growth. The resulting ease of interpretation of atomically resolved SFM images, means that the contrast formation is also expected to be much more accessible for supported nano-objects.

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# Bimodal Imaging on Charged Surfaces

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Bimodal atomic force microscopy on surfaces like chlorite allows for a clear distinction between the chemically different layers of the mineral. While contrast is already pronounced in common intermittent contact mode imaging, it is enhanced with the detection of the second eigenmode oscillation. Our results are summarized in figure 1. Fig. 1 (a) shows the topography of the cleaved chlorite surface, while (b) and (c) show the lateral contrast by the first eigenmode amplitude (d) and phase (e) signals, respectively. Using the mechanically driven second eigenmode concurrently with the first one, amplitude and phase contrasts are strongly enhanced. It is strikingly obvious that layers of the same material have the same second eigenmode amplitude and phase results. Thus, brucite-like and T-O-T layers can be clearly discerned. From conventional Kelvin probe force microscopy it is known, that the two different layers are charged differently. Thus, the strong contrast between the different materials may be mainly attributed to the charge differences.

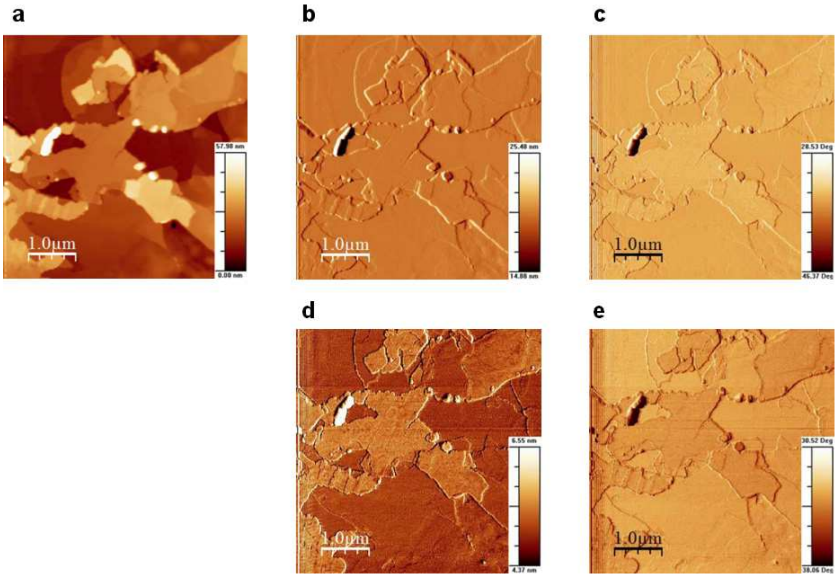

Fig. 1: Multimodal AFM imaging of chlorite. (a) topography, amplitude (error signal) and phase signal of the first eigenmode (b) and (c), and amplitude (error signal) and phase signal of the second eigenmode (d) and (e).

# Can the NC-SFM sort out the unresolved questions of the natural materials? The Antigorite, example of a complex mineral

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During the last years the UHV-AFM non contact has become an important technique in the surface characterization of insulating materials at an atomic scale [1]. However, most of the studies have been performed on synthetic crystals [2] and only few, on minerals [3]. In this work, we show that with an appropriated sample preparation, the UHV NC-SFM can be used to visualize the surfaces of minerals giving new invaluable information about the unknown structural aspects of these complex materials. The antigorite is a phyllosilicate that belongs to the serpentines family [4]. The sample used in this study is an antigorite pseudocrystal that has been detached from the original rock and cleaved in UHV ($2x10^{-10}$ mbar) before the SFM imaging. After the cleavage the (001) surfaces are exposed. Two different kinds of surfaces, have been clearly distinguished: cleavage surfaces and growth surfaces. In both cases the visualization of the geometric modulation has been possible for the first time. The topography of the perfect cleavage and growth surfaces has been distinguished by analyzing the modulation amplitudes and comparing them with the classical models of the atomic structure of this mineral. Finally the structure of the cleavage surface has been confirmed with the atomic resolution images.

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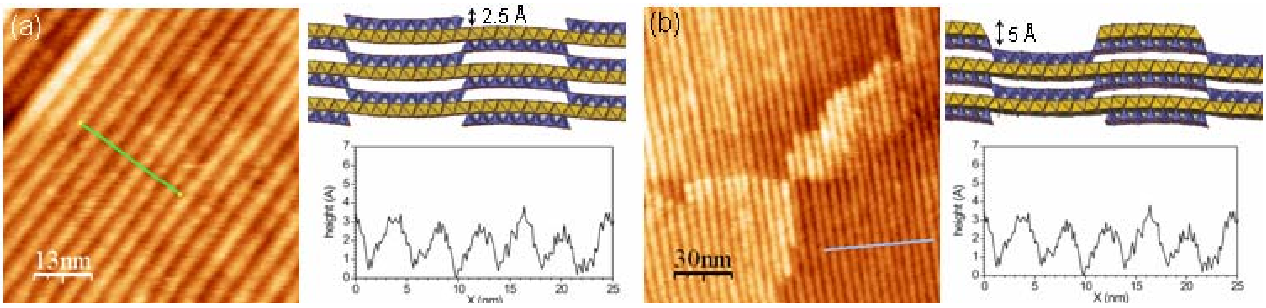

Fig 1: Topography images of (a) growth surface and (b) cleaved surface of antigorite (001) planes. In each surface type the amplitude modulation measured experimentally (profiles) are compared with the corresponding theoretical model.

# Ice on BaF2(111) at room temperature: a non-contact scanning force microscopy study.

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A non contact Atomic Force Microscopy mode called Scanning Polarization Force Microscopy (SPFM)[1] was used to study water adsorption on a $BaF_2(111)$ surface at room temperature. This surface exposes hexagonal arrays of barium and fluoride ions. The lattice constant of such structure is very close to that of the basal face of Ih ice. This structural coincidence has motivated the study ice nucleation on $BaF_2$ surfaces even at room temperature.[2] SPFM allows the study of liquid droplets and films on $BaF_2$ on a non-contact mode and simultaneously perform Kelvin Probe Force Microscopy (KPFM)[1]. Water adsorption on the surface was studied as a function of relative humidity. The formation of Ih ice patches predicted by infrared studies and theoretical calculations[2] was directly corroborated by SPFM and KPFM images.

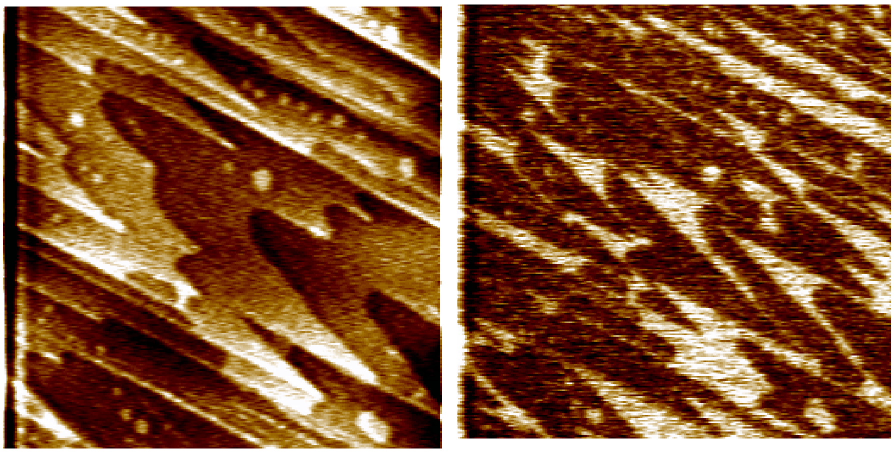

Fig 1: SPFM (left) and KPFM (right) images of a stepped BaF2(111) surface at room temperature and 50% relative humidity.. Ice-like patches on the surface can be observed as a bright contrast in the KPFM image.

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# Electric Dipoles of Surface Nano-Structures on Insulating Substrates

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Local charges on insulating substrate play important roles for wide range of applications such as catalysis, photocopy, nanoelectronics, biological processes and so on. Recently we found that potential and capacitance images of nano-scale adsorbates on *insulating* substrates can be obtained by FM-mode measurements. This finding is beyond the common understanding that the charges of nano-structure on insulating substrate should be almost negligible in comparison with that of bulk substrate. Figure 1 shows spatial distributions of the electric field just bellow a sharp tip apex calculated by finite element calculation. The results shows that the electric potential at the substrate surface remains ca. 10 % of the bias voltage applied to the electrode contacting with backside of the substrate. In case of sharp tip, the surface potential is mainly governed by filed gradients in the subsurface of substrate and in the space surrounding tip-surface gap. As a result, the variation range of surface potential is only 2 - 6.5 % with the change of tip-surface distance from 0.1 to 3 nm. The analysis of potential images using this calculation give dipole moments of 0.034 ± 0.018 D/nm$^2$ for double-stranded DNA, 0.022 ± 0.011 D/particle for Au-nanoparticles (AuNP) coated with citric acid, and 0.0017 ± 0.0009 D/molecule for transcription complex (protein-DNA complex) on mica substrates. These results demonstrate that surface potential microscopy provide comparable result with the measurement at conductive surface and scanning tunneling microscopy [ref1, ref2].

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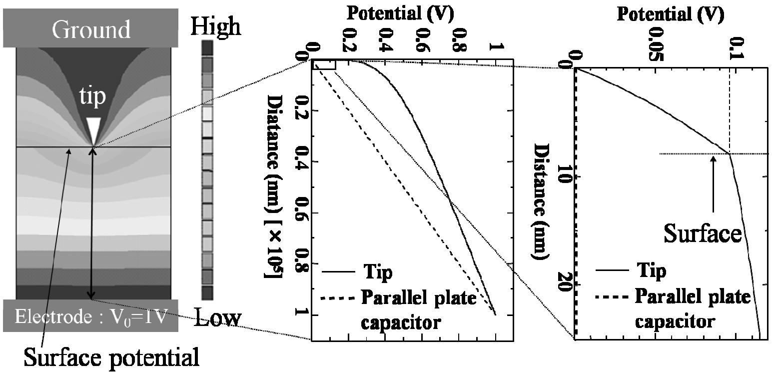
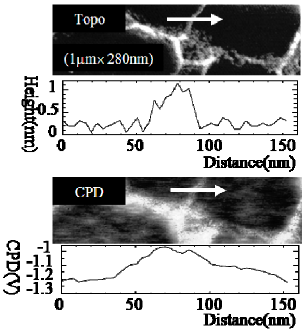


Fig1. Potential Calculation



$V_{(DNA-surface)}$ = 0.013 V
Dipole Moment = 0.034 ± 0.018 D/nm$^2$

Fig2. Potential Measurement of DNA on Mica

# Height measurements on organic ultra-thin films

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In Atomic Force Microscopy the topographic height is related to the applied force. This phenomenon is relevant in organic films, where the applied force can deform molecules. To study this effect we used ultra-thin films of sexithiophene (6T) grown on Si/$SiO_x$ substrates [1]. The morphology of the films, which is composed by islands, is dependent on the resistivity $\rho$ of the substrate. Low $\rho$ means sparse and dendritic islands, whereas high $\rho$ favors the formation of small, round-shaped islands (Fig. 1a). To investigate the relationship between applied force ($F$), molecules deformation and phase shift on these islands, we measure their height by tapping mode, exploiting cantilevers with different compliance and measuring the interaction regimes (attractive-AR or repulsive-RR) by phase shift [2]. For low $\rho$ (0.0015Ω·cm), the measured height is 2.3nm (AR, $F$=17nN), in agreement with the length of 6T (arranged orthogonal to the surface). In this case, the measured heights in AR and RR are comparable, with height differences ≈1-2 Å. Increasing $\rho$ (up to 8-12Ω·cm), the islands become smaller, rounded and higher, with height up to 3.7nm (Fig. 1b-AR, $F$=17nN). These measurements suggest a second layer grown on the islands, with molecules tilted with respect to the surface normal. Here, the island heights measured in AR and RR are markedly different. In RR, for $F$=20nN, the molecules of the second layer are compressed, leading to a height reduction of 10% (from ≈3.7nm to ≈3.2nm).

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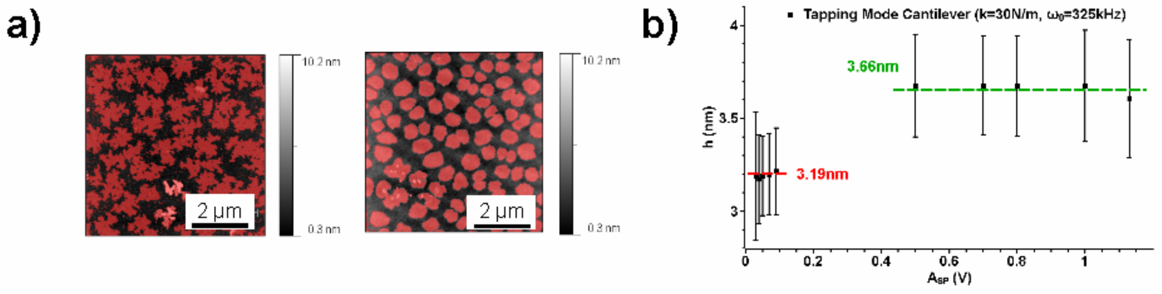

***Figure 1*** (a) AFM images of the 6T islands for different $\rho$; (b) Height difference in AR and RR.

# Adsorption and nucleation of PTCDA molecules on a nanostructured KBr (001) surface

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High-resolution SPM studies of organic molecules on insulating surfaces remain a crucial area in the development of practical molecular electronics. A great deal of focus has been placed on developing methods for controlling the growth of organic layers on the surface, particularly via surface templating. On insulating surfaces, irradiation is a powerful tool to engineer the surface and the treatment has been well characterized for alkali halides. In this work, we use irradiation to pattern the KBr (001) surface with regular pits, and then deposit 3,4,9,10-perylene-tetracarboxylic-dianhydride (PTCDA) molecules. NC-AFM measurements show that the majority of the resulting PTCDA islands conform to the rectangular shapes and nanometer-scale dimensions of the pits [1, 2]. Moreover, high-resolution images show a well-defined relationship between the molecules and the KBr lattice at kink sites.

In order to understand this we use first principles calculations to study the adsorption and diffusion of PTCDA on the ideal terrace, and step and kink sites on the KBr surface. As a base for the calculations we use the density functional theory as implemented in the SIESTA code, including full counterpoise corrections. In order to accurately model the interaction of an organic molecule with an insulating surface, we also include a first principles treatment of the van der Waals interaction. Our results show that PTCDA molecules will diffuse easily on the ideal terrace and will be pinned at kink sites, effectively acting as markers for contrast interpretation in atomically resolved images. Further, we show that the migration barrier for crossing step-edges is asymmetric, explaining why molecular islands form within pits.

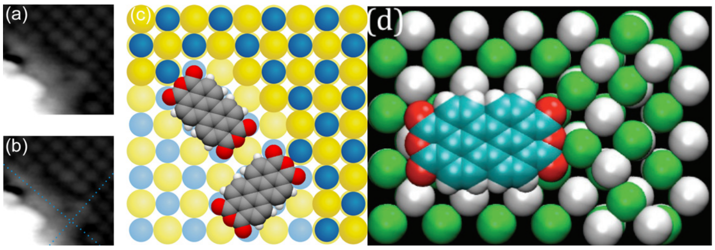

*Figure 1: (a-c) Atomically resolved AFM images of PTCDA adsorbed at kinks at pit edges on the KBr (001) surface, with a schematic of the structure. (d) Calculated geometry of molecules adsorbed at kinks.*

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# Small amplitude FM-AFM imaging of molecules using second flexural resonance mode

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In atomic force microscopy using the frequency modulation method (FM-AFM), interaction forces between an oscillating cantilever tip and the sample surface is detected as the resonance frequency shift of the cantilever. The frequency shift induced by the interaction forces becomes greater when the oscillation amplitude is small. However, if the oscillation amplitude is too small, the tip may jump into static contact. In order to avoid such 'jump to contact', one has to use a cantilever with a large spring constant for small-amplitude FM-AFM [1].

In this study, we performed small-amplitude FM-AFM imaging by utilizing the second resonance mode of the conventional cantilever (Nanosensor NCH) on lead phthalocyanine (PbPc) thin films deposited on $MoS_2$ (0001) substrate. Since the effective spring constant at the second resonance mode is much larger than that at the first one, we safely reduced the oscillation amplitude without 'jump to contact' [1]. We succeeded in sub-molecular-resolution imaging using the second resonance mode as shown in Fig. 1.

We also measured frequency shift versus distance curves using the first resonance mode and the second resonance mode. Force curves calculated from these frequency shift curves were shown Fig. 2. Theoretically these two force curves should overlap each other. We discuss the mechanisms of the discrepancy in detail.

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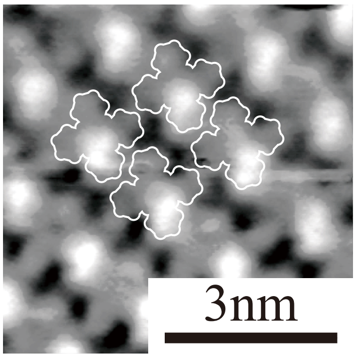

Fig.1 FM-AFM topographic images of PbPc multilayer film using second resonance mode. $f_2$ = 1.86 MHz. $\Delta f$ = -50 Hz. $A_{p\text{-}p}$ = 0.9 nm.

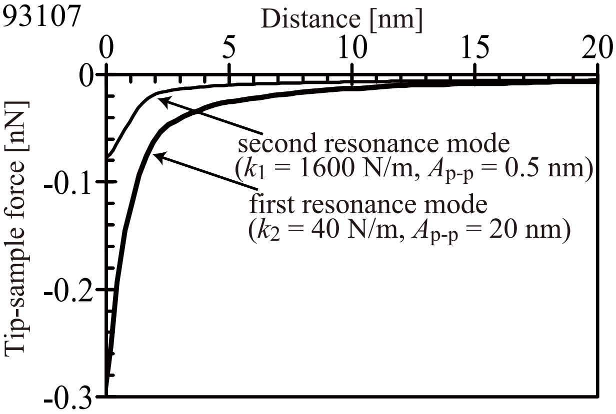

Fig.2 Force curves on PbPc with the first resonance mode and the second resonance mode.

# Molecular Deformations and Dissipation in FM-AFM on PTCDA/Ag(111)

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We investigate the origin of the energy dissipation processes in FM-AFM imaging with submolecular resolution of PTCDA molecules adsorbed on the Ag(111) surface [1]. To this end, we have performed large scale first principles simulations (using the FIREBALL code [2]) to study the adsorption of the organic molecules and the interaction of three different AFM tips with the PTCDA/Ag(111) system.

Regarding the analysis of the molecular adsorption and the contact formation, we have analyzed the structural and electronic properties of the interface, fully relaxing a large unit cell containing two PTCDA molecules in non-equivalent adsorption configurations forming the characteristic herring-bone pattern observed in the experiments. The bonding has a chemisorptive character and involves mainly the perylene core of PTCDA. The projected density of states that we obtain is in very good agreement with recent STM and photoemission experiments.

In order to study the origin of the submolecular resolution in the dissipation images we have performed large scale *ab-initio* simulations for the interaction of a Si nanoasperity with the PTCDA/Ag(111) system described above. We have studied the approach and retraction of the silicon tip located in different positions, ranging from top sites on oxygen atoms forming anhydride groups to carbon atoms on the perylene core. The analysis of the stable structures under the nanoasperity shows that deformation mechanisms are very local and vary strongly among the different positions. In particular, we have found that the Si tip induce a “flipping” of the anhydride groups, leading to an enhanced dissipation signal at the ends of the molecules, while the dissipated energy is significantly smaller on the center of the molecules. This effect suggests an explanation for the experimental energy-dissipation FM-AFM images in terms of adhesion hysteresis processes, similar to the ones recently identified as the dominant dissipation channel in FM-AFM on semiconductor surfaces [3].

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# Molecular assemblies grown between metallic contacts on insulating surfaces analyzed by nc-AFM and KPFM

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Porphyrin based molecules are promising candidates to form self assembled structures on insulating surfaces [1,2]. Their close similarity to hemoglobin and chlorophyll based molecules makes them promising candidates for functional building parts in molecular electronics. To analyze their opto-electronic activity, new measurement methods have to be applied. We investigated the formation of such molecular assemblies on ultrathin insulating films as well as on alkali halide single crystal substrates by means of a room temperature non-contact atomic force microscope (nc-AFM) in combination with a Kelvin probe force microscope (AM-KPFM). On those surfaces, the evaporated cyano-porphyrines are forming one and two dimensional structures. The simultaneously measured surface potential is clearly showing a material contrast between the molecules and the insulating substrate. We succeeded to obtain clear variations of the surface potential down to the molecular level [3].

Furthermore, we will show the possibility of contacting molecular assemblies with gold nanoclusters as can be seen in Fig. 1. At step edges gold clusters act as interrupting points of one dimensional molecular wires, while across terraces the clusters act as starting and endpoints for a two-dimensional growth between them.

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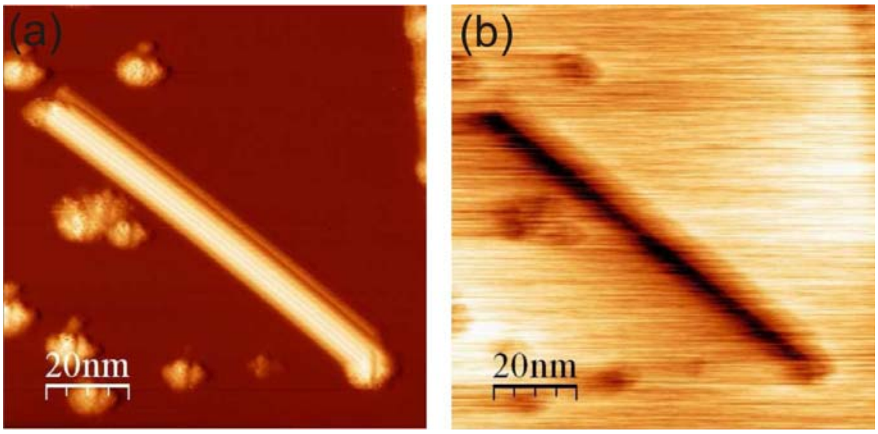

Fig. 1.: Topography (a) and LCPD (b) images of a porphyrin assembly formed between two gold clusters. For contrast enhancement the derivative of the topography was added to (a).

# Crystallites and Wires of Co-Salene on NaCl(001)

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The class of organic magnetic molecules is promising for the development of functionalized magnetic materials in which the genuine properties of the molecules are exploited. Chemistry allows us to customize the properties of the molecules for different applications like new contrast media for magnetic resonance imaging. For a deeper understanding of the ordering and growth of molecules on the nanometer scale, NCAFM is a powerful tool.

In our experiment, we studied the growth of Co-Salene molecules on NaCl(001). The structure of these molecules is predominantly planar with the Co atom in its center (Fig I). Since NaCl shows a large bandgap, the molecules can be investigated with negligible hybridization effects. During preparation, the molecules were evaporated from a homebuilt Knudsen cell to the substrate and a quartz crystal microbalance simultaneously. In this way, we were able to control the deposition rate during preparation.

At low coverages, the molecules typically decorate step edges of the NaCl surface. For higher coverages we observe two different growth modes. On the one hand we find growth of crystallites with heights on the order of some 10 nm and on the other hand wires of molecules with a height of about 2 nm are observed. Besides single wires we also find bundles and networks of wires. Within the bundles, single wires are aligned parallel to each other on the surface or on top of other wires.

One explanation for the two growth modes would be a difference in orientation of the planar molecules on the surface. In one orientation molecules lay flat on the surface and grow in stacks to crystallites while in the other one they stand upright on the surface and form wires.

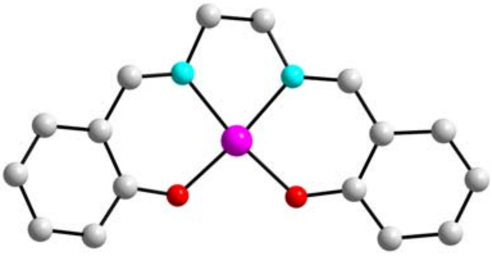

**Fig I** Chemical structure of salene molecules

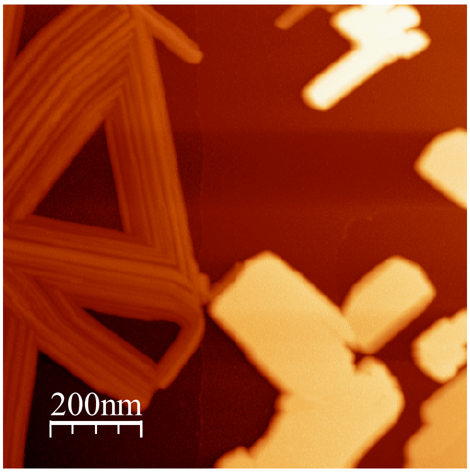

**Fig II** Crystallites and wires of Co-Salene on NaCl(001)

# Theoretical Modelling of Self-Assembly of Molecular Networks and their interaction with Au(111) surface

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The phenomenon of self-assembly of atomic and molecular superstructures on crystal surfaces has attracted an increasing interest in nanotechnology. Self-organised nano-templates where the self-assembled monolayer traps other molecules with selected functional properties, can be used as building blocks for larger nanoscale structures. Self-assembled superstructures can form chiral domains ranging from 1D chains to 2D monolayers.

In particular, there have been many scanning tunneling microscopy (STM) studies of self-assembly of melamine, perylene tetra-carboxylic di-imide (PTCDI) or perylene tetra-carboxylic di-anhydride (PTCDA) molecules on the Au(111) and Ag/Si(111) surfaces [1],[2][5]. STM images of these networks do not reveal the exact details of the intermolecular bonding and process of network growth. It is therefore the task of theory to determine the exact atomic structure of these networks.

We present a theoretical study of self-assembly of molecular networks based on melamine, PTCDA and PTCDI molecules. We propose a systematic approach to building molecular superstructures based on the notion of binding sites. First, we identify all possible sites for hydrogen bonding between molecules. Then we form molecular pairs and larger structures using all possible combinations of these binding sites. In this way, we constructed all possible dimers, chains and 2D monolayers of melamine, dimers and chains of PTCDA and PTCDI molecules. The energies of these structures are calculated using the density-functional theory SIESTA code. The strength of hydrogen bonding in various molecular arrangements is analysed. The theoretically predicted monolayer structures are in very good agreement with the results of STM measurements [4][5]. We also investigate the nature of interaction of melamine molecules and superstructures with the gold substrate and analyse the alignment of melamine networks with the Au(111) surface.

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# Imaging of a single molecule on insulator at room temperature

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Anchoring and NCAFM imaging of a single aromatic molecule at room temperature is quite a challenge due to considerably weak interactions between the adsorbate and the substrate. In the presentation we will present that patterning of the substrate together with the proper design of the molecule allows for solving that problem. In the experiment truxene molecules functionalized with three cyanobenzyl groups [1] were used. The cyanobenzyl groups have a big dipole moment facilitating anchoring the molecule at the surface of ionic crystal. As substrates (001) face of KBr crystals were used patterned by exposition to an electron beam, resulting in creation of rectangular pits of monolayer depth [2]. At very low coverage (approx 100 molecules/100nm$^2$) the molecules adsorb and remain stable at the kinks, whereas on the straight edges features that could be interpreted as mobile molecules are visible. That effect stems from the fact that the modulation of electrostatic potential is increased at the kink site, compared to the edge and to the flat surface. Consequenlty, the electrostatic interaction is strong enough to maintain a molecule under the scanning tip even at room temperature. Therefore, proper preparation of the substrate can provide a template on which kinks are scarce and thus allowing for insulating a single molecule for NCAFM imaging. At higher coverages, on the flat terraces the molecules form a bilayer with domains showing hexagonal ordering. Despite the lattices for different domains are identical, submolecular features resolved by NCAFM point at a large number of possible conformations of molecules.

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# What factors determine SPM imaging features of organic molecules?

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Recent progresses of scanning probe Microscopy techniques (SPMs), such as Scanning Tunneling Microscopy (STM) and Frequency-Modulation Non-contact Atomic Force Microscopy (FM-NCAFM), have been pushing forward the research activities of nanotechnology in device engineering using organic molecules. By means of SPMs, we can investigate various properties of individual small and fragile organic molecules on the substrate in sub-nanometer scale. Indeed, this method is very promising and powerful to treat single organic molecules as device components, however, the detailed mechanisms of their imaging features of organic molecules seems not fully understood and clarified yet. Because STM and FM-NCAFM signals originate from different properties of molecules, it seems possible to derive further detailed information concerning electrical and mechanical properties of molecules by analyzing their features.

In this paper, we present the differences of STM and FM-NCAFM images obtained from the same organic molecules on same substrates, and discuss their origin. Figure (a) shows a chemical structures of 5-(4-Methylthiophenyl)-10,15,20-tris(3,5 -di-t-butylphenyl)porphyrin (MSTBPP), which is specially tailored for this experiment. From the theoretical consideration, center ring of this molecule has conducting features, and slightly separated from the substrate by four non-conducting phenyl-based legs surrounded. Figure (b) and (c) show an images of individual MSTBPP molecules aligned along the terrace edge line of Au(111) surface obtained by FM-NCAFM and STM, respectively. In this geometrical configuration, MSTBPP molecules are directly connected to the terrace edge via a sulfur site of methylthiophenyl leg, which should affect tunneling current path between a molecules and substrate.

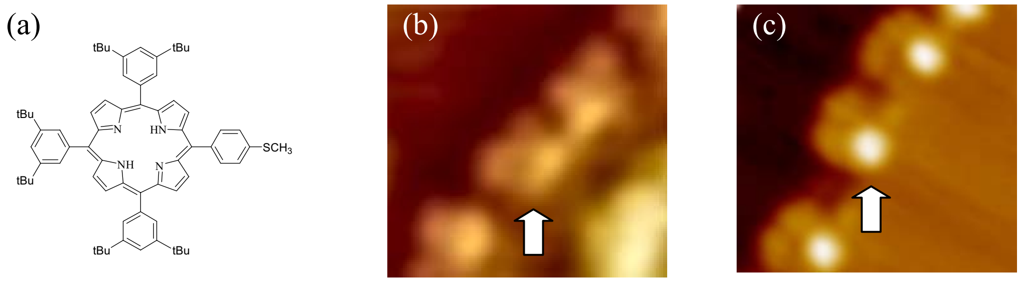

Figure (a) Chemical structures of 5-(4-Methylthiophenyl)-10,15,20-tris(3,5-di-t-butylphenyl)porphyrin (MSTBPP), (b) and (c) Images of individual MSTBPP molecules aligned along the terrace edge line of Au(111) surface obtained by FM-NCAFM and STM, respectively. Their can be seen slight difference in the contrast of three phenyl-based chemical groups.

# Modeling the adsorption and imaging of truxene molecules on the KBr (001) surface

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The adsorption, imaging and control of individual organic molecules on insulating surfaces is essential for the development of architectures for single molecular electronics and other molecular scale technologies. Based on recent experiments that have successfully imaged individual truxene molecules on the KBr (001) surface using non-contact atomic force microscopy, we have performed calculations to model the adsorption characteristics, dynamics and imaging of this system.

In order to accurately describe this large and complex system at the atomistic level, we developed a set of inter-atomic potentials for the interaction of the molecule with the KBr surface based on extensive quantum chemical cluster calculations, which were then combined with established force-fields for the molecule and surface. With this potential model it was then possible to determine the accessible adsorption configurations and corresponding energies, as well as barriers for diffusion, for the molecule adsorbed on the perfect surface, at step edges, and at various types of kink sites. In addition, the potential model was employed to simulate the imaging of the adsorbed molecule with various polar tip models.

# Nano-scale lithography with frequency-modulation atomic force microscopy

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Using atomic force microscope (AFM) one can draw nano-scale structures by repetitively applying voltage pulses between the probe tip and a substrate and deposit small amount of atoms from the tip apex by field evaporation. The lithographic method called AFM lithography has been studied extensively. The minimum width of the drawn lines achieved so far was ~25 nm. The reasons for the limitation are (1) relatively large tip radius of the probe since the tip was coated with the material used for the drawing and (2) AFM operation in the amplitude-modulation mode, which doesn't have enough control of the tip position. In this study, we employed quartz tuning fork [1] as a force sensor in the frequency-modulation (FM) mode, which has high force sensitivity and spatial resolution.

As a tip, a 10 μm diameter Au wire was glued on one of the two prongs of the sensor, and sharpened by focused ion beam in the method reported in ref. [2]. The tip radius characterized with TEM was ~15 nm, much sharper than Au-coated tips by a factor of 5 to 10. Using the tip, we performed the AFM lithography on a native-oxide covered Si substrate in a high vacuum and successfully obtained 10~20 nm diameter Au dots on the substrate (Fig.1). It turned out that the size and height depend on the applied voltage of the pulses; larger voltage produces larger size of dots. The results of the force curve and the force vs. bias voltage measurements indicated that the tip crash does not occur during the pulse application voltage because of high stiffness of the sensor.

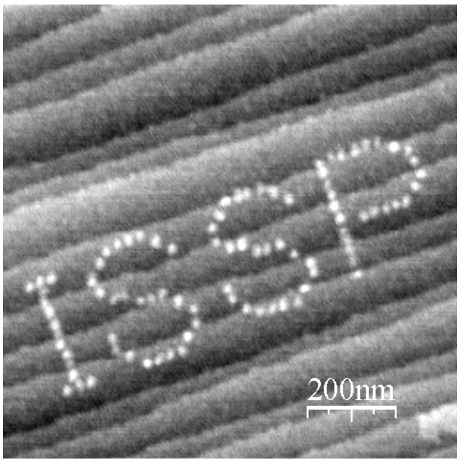

Fig. 1 FM-AFM image after AFM lithography

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# Measuring the charge state of an atom by nc-AFM

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Single metal atoms on a thin insulating layer, i.e. on 2 or 3 monolayer (ML) thick NaCl, on copper substrates can be reversibly switched between different charge states by pulsing the tip sample bias in an STM set-up.[1,2]

Using a qPlus tuning fork AFM [3] operated at 5K with oscillation amplitudes in the sub-Å regime we measure the frequency shift due to the charge switching of Au adatoms on 2 ML NaCl/Cu(111). The idea is to detect the electrostatic force of the negatively charged Au adatom ($Au^-$) with respect to the neutral adatom ($Au^0$). Due to ionic relaxations the tip-sample distance of $Au^-$ is increased with respect to $Au^0$ [2]. Nevertheless, we observe an increased attractive force when switching from $Au^0$ to $Au^-$ that we assign to the electrostatic force due to the single electron charge. This finding is confirmed by a shift on the order of +10mV in the $\Delta f(V)$ spectrum, i.e. in the respective minimum of the electrostatic force, in case of the negatively charged Au adatom.

These measurements demonstrate the ability of nc-AFM to determine single electron charges. This might be important especially on thicker insulating films, where STM is not feasible.

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# Transparent Scanning Kelvin Probe Force Microscopy

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Scanning Kelvin probe force microscopy (SKPM) is a powerful method to measure the local surface potential as the contact potential difference (CPD) in an atomic scale [1]. However, there are several cases where this method cannot be used directly. In the case of a surface immersed in an electrolytic solution, for instance, the surface potential measurement is impossible because of the onset of the electrolysis in the solution by the application the AC and DC voltage between the tip and the sample. In this paper, we report that the CPD of absorbed materials on an insulating thin film can be measured by SKPM across the film. The new method is called Transparent SKPM.

The present experiment was carried out with an environmental SPM (JEOL JSPM-5200) both in air and in vacuum. The sample was Au particles with 15 nm in diameter (BBInternational), whose particles were absorbed on a $Si_3N_4$ membrane window with 250×250 $\mu m^2$ in area and 30 nm in thickness (SPI Supplies). A conducting carbon layer was coated on the Au adsorbed side of the membrane window using a carbon sputter coater and from this side the dispersion of the Au particles was observed with an FE-SEM (JEOL JSM-7001F).

Fig. 1 shows simultaneously acquired topography (a) and CPD (b) images, respectively, across the membrane in an area of 10×10 $\mu m^2$ with the Transparent SKPM in vacuum. Fig. 2 shows SEM image of the same area as shown in Fig. 1. It is in fact a mirror image of the observed one. The topography image in Fig. 1(a) shows the deflection due to aggregates of the absorbed Au particles, which are observed in Fig.2. The CPD image in fig. 1(b) shows contrast at the same areas as in fig. 1(a). It seems that the CPD contrast reflected the work function difference between absorbed Au and coated C can be observed across the $Si_3N_4$ membrane at places where the Au particles adhered strongly to the membrane. In the present observation, the CPD image of the Au particles is broader than their individual size. Further improvement of this technique could be made by the use of a thinner and more uniform film. Better insulation of the film may also serve the improvement.

The newly developed Transparent SKPM substantially extends the applicability of SKPM to softer materials with high fluidity. Even living cells immersed in solution could be observed with this Transparent SKPM.

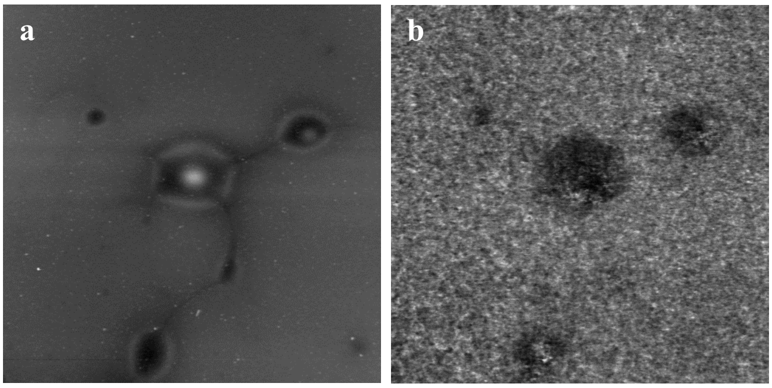

Fig. 1 Topography (a) and CPD (b) images with Au particles across $Si_3N_4$ thin film.

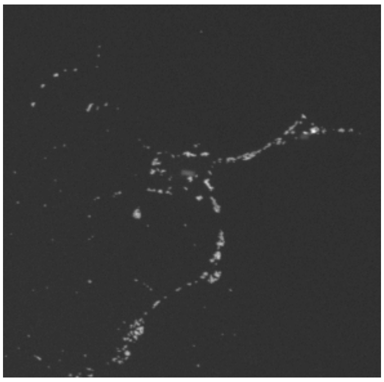

Fig. 2 SEM image from backside of thin film.

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# Cyber-infrastructure enabled Virtual Environment for Dynamic Atomic Force Microscopy

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## Abstract

Virtual Environment for Dynamic Atomic Force Microscopy (VEDA) [1] is a suite of free, state-of-the-art simulation tools deployed on the nanoHUB (www.nanohub.org) for the accurate simulation of tip motion in dynamic atomic force microscopy (dAFM) over organic and inorganic samples and in vacuum, ambient or liquid environments. VEDA takes advantage of nanoHUB's cyberinfrastructure to run high-fidelity dAFM tip dynamics computations on local clusters and the teragrid. Consequently, these tools are freely accessible and the dAFM simulations are run using standard web-based browsers without requiring additional software. A wide range of issues in dAFM ranging from optimal probe choice, probe stability, and tip-sample interaction forces, power dissipation, to material property extraction and scanning dynamics over heterogeneous samples can be addressed.

VEDA is already used by >350 users in dozens of countries who have run ~4000 jobs on the dynamic approach curve (DAC) tool and the amplitude modulated scanning tool (AMS). In September 2008, a new advanced applications tool will be released that simulates accurately multi-modal dynamics of microcantilever in liquid environments allowing for study of phenomena in liquids such as momentary excitation of higher eigenmodes on soft samples, higher harmonic imaging or the bi-modal/multi-modal imaging in liquids. In this poster, we demonstrate the different capabilities of VEDA and describe the iPod downloadable tutorials and learning modules that assist the new user in learning VEDA.

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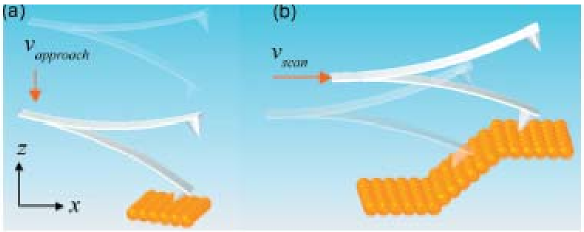

Fig. 1. Schematic of the DAC and AMS tools

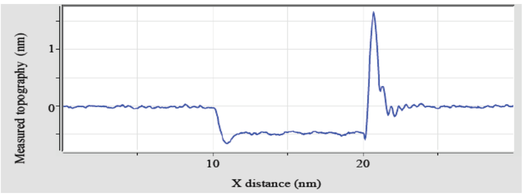

Fig.2. Simulated AMS topography on a flat sample with a patch of soft material.

# Poster Presentations

## Wednesday, 17 September

# Session II

## Wednesday,
## 17 September

# Step structures and dislocations on $CeO_2$(111)

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Oxide surfaces and metals dispersed on oxide supports are widely used in catalytic applications. The catalytic reactivity is strongly related to surface defects, where oxygen vacancies and step edges play a fundamental role, specifically for the nano-particles used in catalysis.

In this contribution, we present a thoughtful study on the morphology of step structures at the $CeO_2$(111) surface using dynamic scanning force microscopy (SFM). The surface exhibits hexagonal islands and pits [Fig. 1(a)] of O-Ce-O triple layer height, with steps mostly enclosing an angle of 120°. Atomically resolved images [Fig. 1 (b)] reveal that the (111) surface almost exclusively exhibits alternating steps having (001) and (110) facets [Fig. 1(c)]. Our results confirm the general notion that step formation energy on metal-oxides scales with the surface energy of the extended facet [1], and provide important insights into the understanding of the structure and reactivity of ceria nano-particles possessing mainly (111) surfaces [2]. Kink sites and missing oxygen atoms are identified to be the dominating defective sites at step edges and we demonstrate that low-coordinated oxygen atoms at step edges can be removed by the SFM tip. Furthermore, the structure of screw and edge dislocations that are frequently observed on the surface (see the arrows in Fig. 1(a)) will be discussed.

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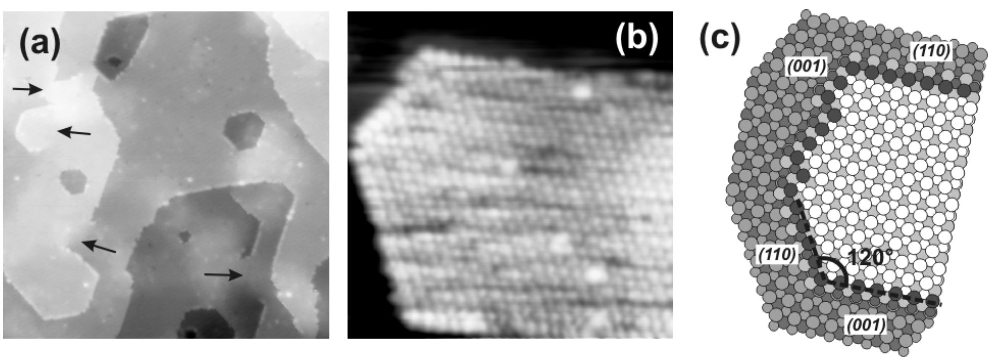

Figure 1.

# nc-AFM investigation of the air-exposed MgO(001) surface morphology

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The surfaces of ionic crystals are of large interest, due to their fundamental properties and with respect of a number of potential applications. Although for many materials a clean surface can certainly be achieved by preparation (e.g. cleavage) in an ultrahigh vacuum [1], the air exposure could be unavoidable for many applications in the forthcoming devices. However, significant chemical/morphological degradation of the surfaces has been observed in a few ionic crystals which had been exposed to the air [1, 2]. Despite of the importance that these effects have on future developments, only a few materials have been deeply investigated from these point of view [2]. In particular, despite preliminary results in the literature reported a granular structure induced on the MgO surface by air exposure [1], a detailed investigation of this degraded surface is still lacking.

Self organization of atoms on magnetically and chemically inert, nanopatterned substrates is a promising approach to prepare ordered arrays of metallic clusters of defined size, shape and distribution exhibiting new exciting properties, e.g. in the field of storage information and catalysis [3, 4]. MgO is an appealing material to be used as a substrate because it is a non magnetic insulator with a negligible electronic interaction with many overlayers, showing no significant oxidation-reduction reaction at the interface [5]. However the surface modification, induced by the air, makes difficult to use MgO as a nanopatterned support because many patterning procedures must be performed in the air and the small grains of the degraded substrate surface can compete with the pattern as template for a self-organized growth of an overlayer.
The morphology resulting by the air exposure of MgO(001) surface, for both cleaved and off-plane mechanically polished [6] single crystals, has been investigated. The main features of the granular structures on the surfaces have been studied by non-contact AFM, and the chemical and structural quality has been checked by means of X-Ray Photoemission Spectrocopy (XPS) and Low Energy Electron Diffraction (LEED). Preliminary results on the competition between the air-induced and the polishing-induced surface structures with respect to Fe nanoclusters nucleation will also be presented.

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# Atom-resolved AFM studies of the polar $MgAl_2O_4$ (001) surface

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Magnesium-aluminate spinel ($MgAl_2O_4$) has many interesting properties and applications, among other things the use as a support for catalysts. In particular, $MgAl_2O_4$ in a porous form is a support material for the important Ni-based catalyst used for steam reforming of methane, which today is the most common method of producing bulk hydrogen. A study of the clean spinel is thus of very great importance to understand the role of the support, and still almost no surface science studies on the $MgAl_2O_4$ surface have been presented due to the insulating nature of the material. Here we successfully use state-of-the-art atom-resolved nc-AFM, in combination with DFT calculations to study the termination and surface structure of the clean spinel $MgAl_2O_4$ (001) surface. However, when constructing a surface model we must consider the fact that the spinel (001) surface is polar, which means that the unit cell has a dipole in the direction perpendicular to the surface. This results in a diverging surface energy for the 'ideal' termination and the surface has to be modified somehow to cancel out or compensate for this effect. It is well known that the AFM tip can pick up material from the surface, and this material can then change the polarity of the nano-apex, which leads to variations in the AFM contrast seen in atom-resolved images. It was recently shown in detail [1,2] how the AFM tip functionalized in either a positive or negative terminated state, allowed imaging preferentially of the individual sub lattices. In this study we observe from atom-resolved AFM images that the $MgAl_2O_4$ surface may be imaged in two distinctly different contrast modes with either a square $5.72\times5.72Å^2$ unit cell (Mg-sub lattice) or a rectangular $2.86\times5.72Å^2$ (O-sub lattice). What we also see in our studies is that Mg-terminated patches are interrupted by small irregularities present on the surface, which could not be resolved with the AFM, but which could be Mg deficient and serve to reduce the surface dipole moment. Large-scale AFM images reveal a large step edge density which may also play a role in the compensation of the dipole moment of the surface. Additionally, metal-particle (Ni and Cu) deposition studies were carried out on the spinel surface to study the morphology, dispersion and stability of these metal clusters, as a model system for a catalyst.

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# Atomic scale imaging on $Fe_3O_4$(001) surface studied by Non-contact Atomic Force Microscopy

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## abstract

A (001) surface of $Fe_3O_4$ is expected to have unique magnetic features. However the crystallographic structure and stoichiometry of the $Fe_3O_4$(001) surface are still controversial. Two different kinds of terminations are possible to emerge on the $Fe_3O_4$(001) surface. One is called A-plane on which only Fe(III) cations are visible, and the other is B-plane that consists of Fe cations in different valence states(Fe(II) and Fe(III)) and oxygen anions. STM study has revealed reconstructed surfaces, a wave-like structure along <110> or <1-10> direction that were assigned to the B-plane[1]. Voogt et al.[2] suggests that an oxygen vacancy in each unit cells plays a key role to form the reconstructed structure. On the other hand Pentcheva et al. considered the Jahn-Teller effect as a possible reason to deform the topmost cations[3]. Experiments performed with STM could offer no information about exact position of oxygen anions and their vacancies on the surface. It must be important for understanding the origin of the surface reconstruction to identify existence of the oxygen vacancies. Noncontact atomic force microscopy (NC-AFM) is helpful for that study, since it is expected that NC-AFM is able to image both Fe ions and oxygen atoms. In this study we have performed NC-AFM measurements on the $Fe_3O_4$(001) to discuss its surface structures. $Fe_3O_4$(001) films were epitaxially grown on a MgO(001) substrate by using e-beam deposition method in an ultrahigh vacuum (UHV) chamber. The thickness of the films was 50 nm. After growing the thin films, the substrate was exposed in air to transport it to a different UHV chamber equipped with an NC-AFM system. The $(\sqrt{2}x\sqrt{2})$-R45° surface reconstruction is recovered by annealing and Ar ion sputtering processes. And then homo-epitaxial $Fe_3O_4$ growth was done in the chamber NC-AFM measurements were carried out in ultrahigh vacuum at room temperature, and a checkerboard patterned image with the same periodicity as the unit cell of the reconstructed structure(Fig.1).

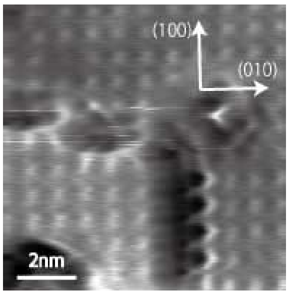

Fig.1 NC-AFM image of $Fe_3O_4$(001) surface.

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# nc-AFM investigations of Cu/α-$Al_2O_3$(0001) model catalyst

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Metal interactions specifically with alumina substrates (e.g. Cu/$Al_2O_3$) present an important area for study because of the vital role played by alumina in heterogeneous catalysis. In this model catalyst study, fully exploiting the potential of nc-AFM, we report on comparative studies between Cu growth on a clean surface exposing the Al-rich ($\sqrt{31}\times\sqrt{31}$)R±9° reconstruction [1] (fig. 1(a) & (b)) and a pre-hydroxylated surface of α-$Al_2O_3$(0001).

Scanning in the constant height mode, we obtained high quality images of the top facet of Cu nanoclusters revealing a clear hexagonal shape (fig. 1(c)). This equilibrium shape determined can be related to the adhesion energy, $E_{adh}$ by the equation derived from Wulff-Kaichew construction for supported nanoclusters [2]. It is evident from the equation that the nanoclusters become flatter when $E_{adh}$ increases, indicating a strong cluster-substrate binding. Our results for the Cu nanoclusters deposited on the hydroxylated surface corroborate this equation. This preferential Cu wetting clarifies that the bonding nature of Cu to the hydroxylated surface is substantially different from that on the clean surface. Moreover, our SFM images reveal that Cu nanoclusters are thermally stable only up to 400ºC, and at higher annealing temperatures there is a remarkable decrease in Cu cluster density on the two surfaces.

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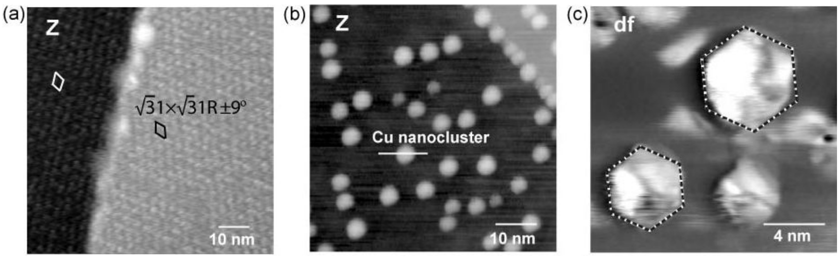

Fig.1. SFM images of (a) clean α-$Al_2O_3$(0001), (b) Cu on α-$Al_2O_3$(0001), (c) Cu cluster imaged in constant height mode.

# Measurement of adsorbed liquid films using Dynamic Force Spectroscopy

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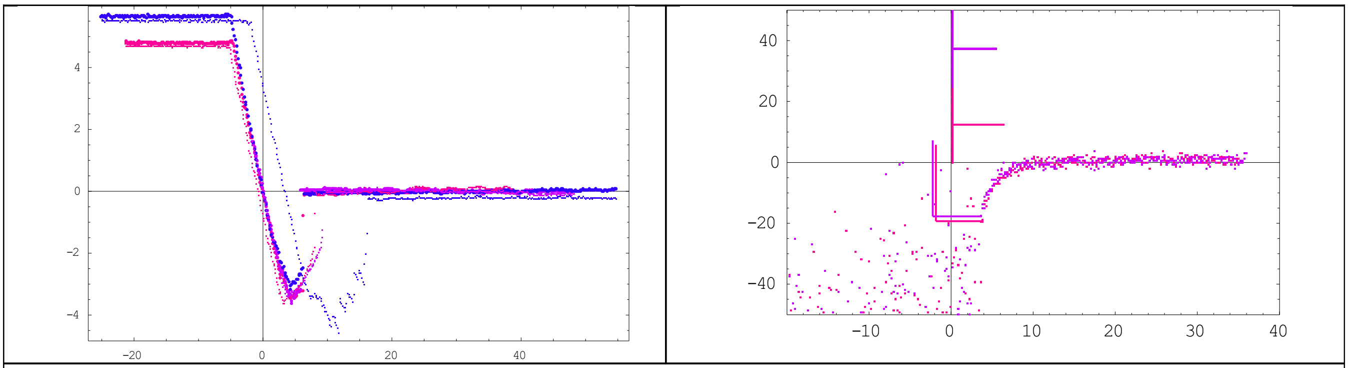
Tip-sample interaction is measured in ambient conditions on gold, glass and silicon at different relative humidity. As compared to other spectroscopic experiments, very small oscillation amplitudes (<100pm) are used to keep the tip-sample system in the linear regime, this simplifies data processing and data interpretation. Very high signal to noise ratio is obtained in spite of the small oscillation amplitude. The force, the force gradient and the quality factor are determined simultaneously as a function of tip-sample distance. In the non-contact regime of the interaction, essentially no force and no variation of the quality factor is measured. Until the jump to contact point is reached, the force gradient perfectly follows the relation predicted by pure Van der Waals interaction. Since force and force gradient are measured simultaneously, the tip-sample position can be determined either from the force vs. distance (FvsD) curve, or from the pole of the Van der Waals interaction. Interestingly, these two values do not coincide: the value obtained from the pole of Van der Waals interaction is consistently 1-2nm "higher" than the value obtained from the force data. Moreover, this difference increases with relative humidity. We therefore conclude that the difference in surface position is due to the liquid film adsorbed on tip and sample. In this interpretation, the force data would detect the "hard" surface (mechanical contact), while the pole would correspond to the "soft" surface of the liquid layer. For the figure shown, the jump to contact distance obtained from the FvsD curve (left) is 4.6nm while the pole of the Van der Waals interaction is 1.5nm over the position of the surface as determined from the FvsD curves.



Set of two FvsD curves (left) and force gradient vs. distance curves (right) measured on gold at low (3%) relative humidity with a 2N/m cantilever. In the left graph, the pole of the Van der Waals interaction is plotted at the position of the vertical axis, while the surface position as determined from the FvsD curve is shown as (short) vertical lines connected by a horizontal line (jump to contact distance) to the corresponding data points.

# Molecular Resolution Imaging of Cholesterol/Lipid Bilayer by Frequency Modulation Atomic Force Microscopy

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Frequency modulation atomic force microscopy (FM-AFM) can be employed to obtain atomic and molecular resolution images in liquid. The performance of FM-AFM in liquid suggests that the technique is ideal for the analysis of biological samples under physiological conditions. To date, hydration layers [1] and mobile ions [2] interacting with the lipid bilayer were investigated by FM-AFM, where the one-component lipid bilayer was used as a primitive model of biological membrane. Although these previous studies revealed novel information on the simplified biological membrane, they are inadequate to completely understand the behavior of biological membrane as the actual biological membranes are heterogeneous complexes consisting of various molecules (e.g., lipids, cholesterols and proteins).

In this study, we investigated the cholesterol/lipid bilayer complex as a heterogeneous model of biological membrane. The cholesterol molecules act as a key constituent in controlling membrane fluidity and permeability. However, the detailed structure of cholesterol/lipid bilayer complex has not been clarified. Figure 1 shows FM-AFM image of (a) dipalmitoyl-phosphatidylcholine (DPPC) bilayer and (b) DPPC bilayer/cholesterol complexes in phosphate buffer solution. The images reveal that the presence of the cholesterols increases the lateral spacing between the DPPC molecules. These results suggest that the cholesterol molecules are inserted between the DPPC molecules.

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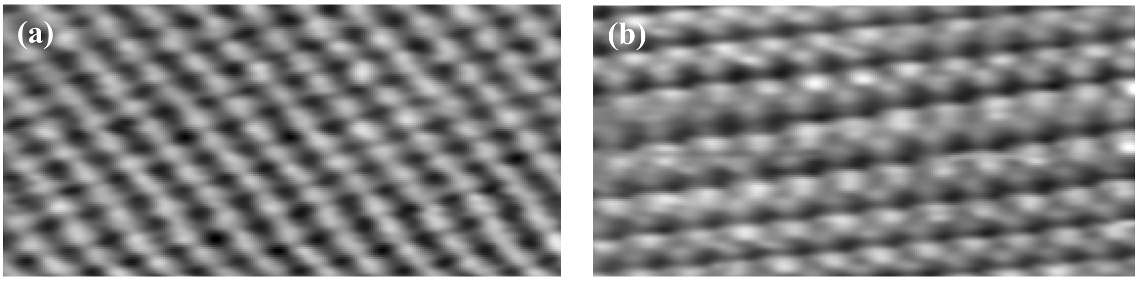

Fig. 1. FM-AFM images of the supported lipid bilayer on mica (4 nm x 8 nm).
(a) DPPC bilayer, (b) DPPC / 50% cholesterol

# Ultra-Low Tapping Amplitude AFM for High Resolution Imaging and Mechanical Mapping in Fluid

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Tapping Mode™ imaging in fluid at amplitudes from 0.1 nm to 0.5nm opens the possibility of using stiff silicon lever to image biological sample with low force and high resolution. In this report we have demonstrated resolution of single proton channel of membrane proteins with the ultra low tapping amplitude. The low amplitude operation was combined with HarmoniX where an offset tip was used to collect harmonic spectra. The mechanical properties of the DNA molecules, including stiffness, adhesion and peak interaction force, were mapped simultaneously throughout the imaging.

Other advantages of the stiffer levers are stability and clean harmonic oscillation in fluid. Cantilevers with stiffness up to 60 N/m can be tuned in fluid much easier compare to traditional silicon nitride levers. The laser optics drift due to bimorph bending was also substantially reduced, rendering much more stable operation. Yet another benefit of the low amplitude operation is the steady near field interaction between the tip and sample. Such interaction corresponds to a non-hysteretic phase function, which facilitates robust phase feedback for AFM control. We were able to achieve much quicker response of the system using phase feedback, as well as higher signal to noise ratio in the control loop. Applications extending to atomic resolution will also be presented.

# Force spectroscopy on DNA by FM-AFM

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## Body of abstract

In Non Contact mode, Frequency shift (Δf) versus separation (Z) curves lead to a detailed comprehension of the tip-sample interaction providing quantitative measurements of both force and interaction potential. [1, 2] We present Δf vs Z curves taken on DNA molecules deposited on different substrates (fig. 1). The microscope was developed in our laboratory for studying biological samples. [3] The high resolution force measurements on soft specimens like APTES functionalized mica and HOPG has returned adhesion forces less than 1 nN. The experiments on freely adsorbed DNA on HOPG permits to evaluate the net interaction between the sample and a sharp tip (fig. 2). The single point spectroscopy measurements indicate that FM-AFM can easily detect differences in interaction forces between sample and substrate opening the possibility to evaluate local interaction at the molecular scale.

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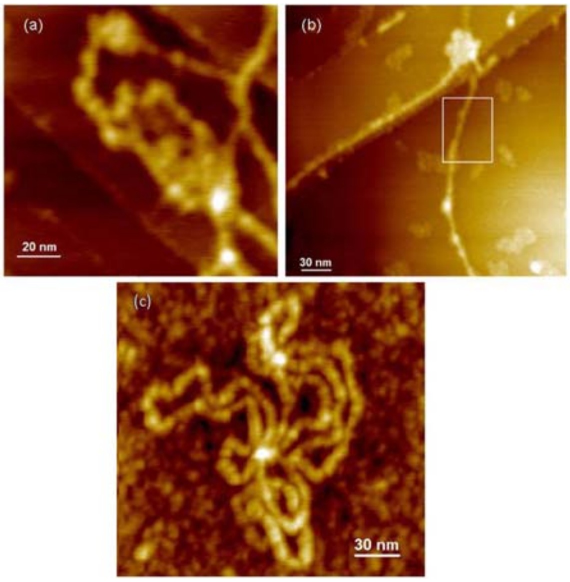

Fig. 1. Network DNA aggregates (a) and isolated filament (b) on HOPG. Single circular DNA molecule deposited on APTES modified mica (c).

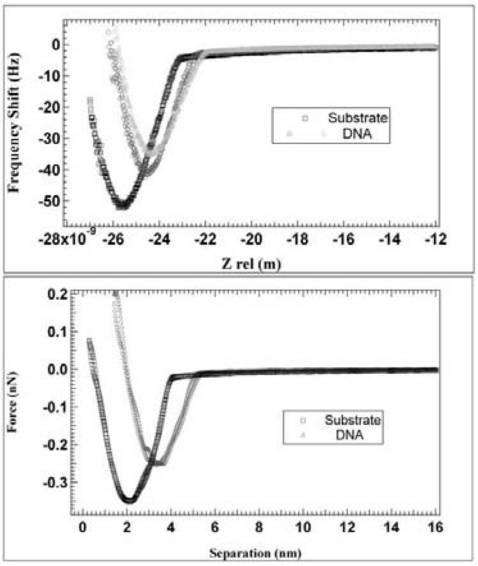

Fig. 2. Frequency shift and force vs distance curves taken on DNA. The data clearly indicate differences between curves taken on DNA compared to the substrate.

# Theory of Topography and Recognition Imaging by Dynamic Force Microscopy

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Although force spectroscopy (consisting in recording force vs. distance curves) represents an excellent method for studying molecular recognition, until recently it was impossible to record recognition maps at the same resolution and imaging speed as the conventional maps of topography, lateral force, phase, etc. The introduction of simultaneous Topography and RECognition (TREC) imaging [1,2] provided a simple and fast Dynamic Force Microscopy mode capable of simultaneously recording images of topography and specific recognition between the molecules at scanning tip and sample.

In this work, the dynamic response of the cantilever-tip system in water was simulated as a damped, driven harmonic oscillator with additional terms describing the interaction with the sample. Our results indicate that molecular recognition in TREC can be usefully modelled by a worm-like chain model. The origin of contrast in TREC phase images as well as its dependence on the driving frequency is analyzed.

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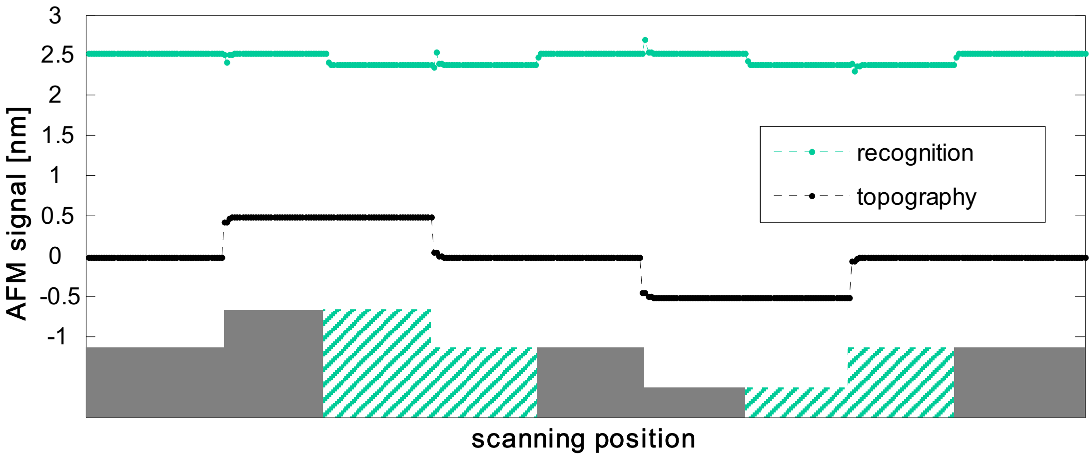

Fig. 1. Simulated scan line of an area containing both topographic and recognition features. The sketch at the bottom represents the sample scanned (striped portions are recognition sites).

# The force required to measure internal structure in block copolymers

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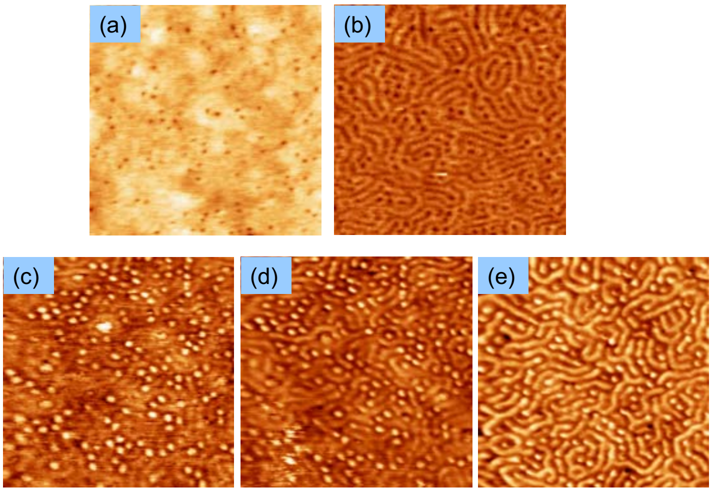
Block copolymers exhibit fascinated nanoscale structures as shown by atomic force microscope. This structure has been attributed to a combination of topography and mechanical properties [1]. In this work we apply frequency modulation AFM [2] (FM-AFM) and jumping mode (JM) [3] (also known as pulse force microscopy) to separate the contribution of both topography and mechanics in thin films of polystyrene-*block*-poly(ethylene/butylene)-*block*-polystyrene (SEBS) triblock copolymer supported on a silicon substrate. First we proceed by imaging the surface in frequency modulation AFM [2]. The images reveals a topography described by a smooth surface with small holes (<30 nm). The dissipation shows the characteristic worm-like structure that is known to be a consequence of the different components of the polymer. Topography and dissipation are quite independent of the working conditions and we do not observe a mix of both types of images. On the contrary, as is well-known, when using amplitude modulation the images evolve from hole like structures (for small amplitude reduction) to worm like structures (higher amplitude reduction). In order to obtain a direct measurement of the force required to pass from one to other structure we have used JM in water (acquiring images in liquids reduces the adhesion force) finding that for set point forces smaller than 0.35 nN we observe the same topography as in FM-AFM but reversed. As the force is increased the worm-like structure emerges and for set point forces higher than 1.2 nN it is the only visible one. We have confirmed this by acquiring force volume images.



**Imaging SEBS copolymers**. (a) Topography and (b) dissipation images taken in FM-modulation . (c-e) topography Jumping Force images in water taken at 0.35 nN, 0.65 nN and 1.2 nN. The size of the images is 1 × 1 $\mu m^2$

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# AFM simulation of large vacancy in aqueous environment

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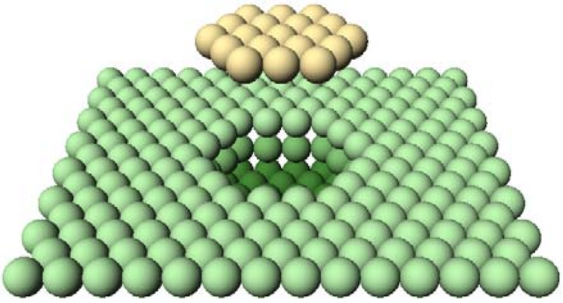
Many theoretical simulations of atomic force microscopy (AFM) have been performed so far to clarify the underlying physics in experimental AFM images which were difficult to understand intuitively. Most of the simulations use the models consisting of only tip and sample atoms and successfully reproduce experimental AFM images obtained in vacuo. However, those models are not suitable to simulate the AFM experiments performed in liquid or gaseous environments.

Reference Interaction Site Model (RISM) is used in our simulation to incorporate liquid effect. This method is based on statistical mechanics and has the advantage of low computational cost over all-atom molecular dynamical simulations. Moreover, the method keeps the information of the water molecule structure which is advantageous over the dielectric continuum model of water. The overall methodology for the AFM-RISM interface is referred to the studies of Koga et al.[1-3].

We investigated the effects of water molecules on images of vacancies on a surface using the model shown in the figure. Simulations are performed for models, each of which consists of differently charged and dented vacancy and differently charged tip. The images are also compared with the simulation results for the same models obtained in vauo.



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# Development of atomic force microscope with wideband magnetic excitation of cantilever in liquid

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Viscoelastic measurement of a single polymer chain is expected to provide knowledge on dynamics of biopolymer that is inaccessible with conventional ensemble-averaged measurements. In addition, it is recognized that dynamics of fluid confined in nanometer-scale space also play a crucial role in biological activities. In order to probe dynamical properties of these mesoscopic soft matter systems by measuring their viscoelastic response in a wide frequency range, a new atomic force microscope was developed. The well-known technique of magnetic cantilever excitation was intensified to incorporate excitation of the cantilever up to 1 MHz. An electromagnet with a small inductance of 4.5 μH was installed at the vicinity of the cantilever so that its position relative to the cantilever is finely adjustable to maximize the magnetic gradient force as shown in Fig. 1. A closed-loop voltage-to-current converter was developed to drive an AC current of ca. 1 $A_{p\text{-}p}$ within a bandwidth of 1 MHz irrespective of impedance variation of the electromagnet. Figure 2 shows a typical response of a commercial 0.1 N/m silicon nitride cantilever attached with a magnetized Ne-Fe-B sphere measured in distilled water. Three resonance peaks associated with a characteristic phase variation of ca. 180 deg. are clearly noticeable. A frequency-dependent viscoelasticity measurement of a single polymer chain was carried out at discrete frequencies by fitting of each peak with Lorenzian function.

This work was supported by Japan Science and Technology Agency (JST).

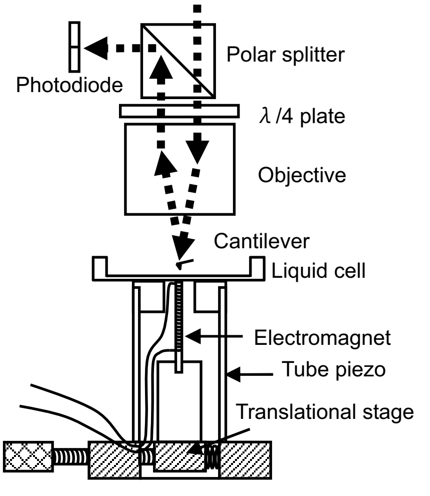

Fig. 1 Schematic view of electromagnet and optics of the developed AFM.

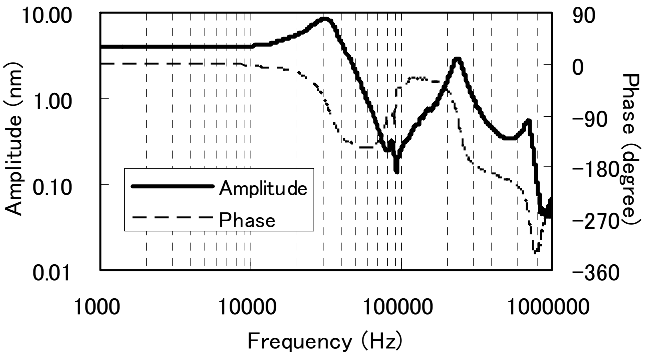

Fig. 2 Frequency response of magnetically excited 0.1 N/m cantilever measured in water.

# Local Deposition of Liquid Droplets using a Dynamic-Mode AFM Tip

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Local deposition techniques based on scanning probe methods such as the dip-pen nanolithography[1] and the AFM stencil method[2] have remarkable advantages over the normal lithography process in terms of simplicity and applicability. In particular, since most of the organic films cannot be patterned by the conventional lithography method using polymer resist films, these techniques are greatly useful for the patterning process of organic materials[3] including biomolecules. However, those techniques are based on the contact-mode AFM[4], which often causes severe damage to both tip and sample.

In this study we report a novel method for liquid droplet deposition with the size on the order of 10 nm using a dynamic-mode AFM cantilever. Droplets were deposited onto a substrate surface from an aperture fabricated adjacent to the apex of the probe tip of the cantilever by applying high-electric field between the aperture and the substrate. After the deposition the surface was imaged by the same cantilever. Furthermore, patterning process was carried out by vector-scanning of the aperture. In this study, using ionic liquid (1-butyl-3-methyl imidazolium tetrafluoborate) we made an array of 8×10 droplets on a Pt substrate, as shown in figure 1.

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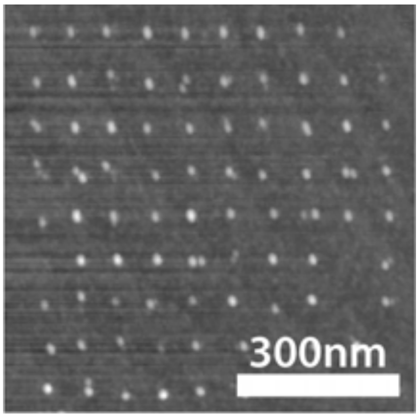

Fig. 1. Tapping mode AFM image of an array of 8×10 deposited droplets. The average- and the minimum-size of droplets are 20 nm and 16nm in diameter, 4 nm and 1 nm in height, respectively. The minimum volume is 200 yoctoliter. A 9.9 volt pulse with a duration of 1 sec was applied for each deposition.

# Dissipation due to adhesion hysteresis: chemical bond rupture and capillary condensation in dynamic AFM

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Amplitude-modulation AFM (AM-AFM) has been shown to be a powerful tool for dissipation mapping. The analysis of the dissipated power as a function of the cantilever oscillation amplitude has been proposed as a way to identify specific energy-dissipation processes behind the compositional contrast [1]. In air ambient condition, when the tip approaches the sample, water condensation from the humidity can induce the formation of a nanometer-sized water bridge and dissipation can be strongly influenced by capillary forces [2].

In this work we present AM-AFM dynamic-dissipation experiments performed on GaAs samples as a function of the relative humidity (RH). At low RH, the AM-AFM dissipation spectra (Fig.1-left) are consistent with contact adhesion hysteresis [3]. Our results at high RH (Fig.1-right) are in agreement with recent theoretical predictions on capillary induced dissipation [2]. The transition from low to high RH will be discussed in detail.

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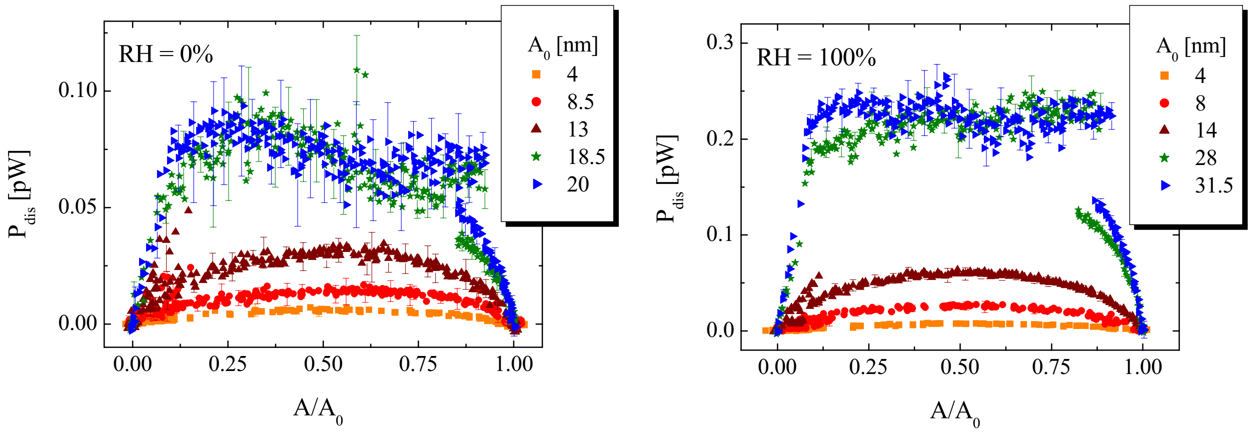

Fig. 1. Experimentally determined dissipated power versus normalized oscillation amplitude for a relative humidity of 0% (left) and 100% (right) on GaAs. The symbols correspond to different free oscillation amplitudes $A_0$. The data were smoothed (average over 10 data points), the standard deviation is shown.

# About Local Conformation within Conjugated Supramolecular Assemblies by Non-Contact Atomic Force Microscopy

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Supramolecular self-assembly is ubiquitous in biological systems and underlies the formation of a wide variety of complex nanostructures. Inspired by biology, self-assembly is also emerging as a new strategy in materials science [1], with the potential of generating functional nanostructures. In particular, the concept of supramolecular electronics relies on self-assembly of $\pi$-conjugated building blocks for the controlled formation of organic semiconducting structures with specific optoelectronic properties [2]. To optimize the properties of supramolecular assemblies, it is essential to determine their structure on the local scale, *i.e.* not by averaging over ensembles but examining each single object.

While Scanning Tunnelling Microscopy is extremely powerful for determining the structure of adsorbed monolayers, it is hardly applicable to thicker deposits of non-conducting materials or to more complex structures. Dynamic force microscopies provide valuable information on the size and shape of the nanostructures but with no details on the inner structure of the assemblies [3]. In this context, here we show that Non Contact Atomic Force Microscopy (NC-AFM) applied on soft matter at room temperature provides information with unprecedented resolution on the molecular conformation within conjugated organic supramolecular assemblies in the solid state.

This brings important new insight on the intimate supramolecular organization in functional assemblies on surfaces, and more specifically on the presence of local variations in the molecular conformation. Extension to other types of organic or bio nanostructures can be expected to provide valuable information on the conformational properties on the local scale with a direct impact on the functional properties.

## Acknowledgements

The collaboration between Grenoble and Mons was carried out in the framework of the CNRS/FNRS-CGRI Tournesol Programme. The work in Grenoble is partly supported by the RTB program “Post-CMOS moléculaire 200 mm” of CEA-Grenoble. The work in Mons is partly supported by the Belgian Federal Science Policy Office (PAI 6/27), the European Commission, the Region of Wallonia (ETIQUEL Project) and the Belgian National Fund for Scientific Research (FNRS/FRFC). Ph. L. is Chercheur Qualifié of the FRS-FNRS (Belgium).

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# FM-AFM in liquid using high-Q fiber tips

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SPM tips made of tapered optical fibers represent the most successful class of tips for scanning near-field optical microscopy (SNOM), a technique which combines topographic measurements and sub-diffraction limited optical imaging [1]. Although usually operated in a shear-force mode, using a quartz tuning fork as the force transducer, the fiber itself can be used as the force transducer by using a bent geometry [2]. Bent fiber probes are compatible with laser beam deflection sensors and enable SNOM on conventional, commercial atomic force microscopes, in particular integrated atomic force microscopes (AFM) and inverted optical microscopes or so-called bio-AFM's.

Due to the intrinsic stiffness of bent fiber probes, contact and AM feedback modes are difficult to employ for live cell imaging. We have pursued FM feedback in a liquid environment as an alternative [3]. Due to the nature of the optical near field of the SNOM probe, small tip-sample separations are not needed to obtain high optical resolution. [4] Additionally, the size of the SNOM tip precludes high topographic resolution. Thus, we could expect to operate at low forces by maintaining a relatively large tip-sample separation and small frequency shift. This also presents the advantage of limiting the effect of vibrations on frequency shift noise, which can be a prominent source of noise on a bio-AFM, in particular for combined AFM and fluorescence measurements.

We have employed a method to ensure a stable resonant frequency and Q-factor while operating in liquid with a bent fiber probe using a diving bell approach [5]. The diving bell also allows high Q in liquid and values higher than 100 have been measured. Using the diving bell we can stably image glass surfaces in liquid and measure frequency shift-distance curves of sufficient signal-to-noise to enable the force to be computed. Results to date show that biologically relevant forces are accessible with bent fiber probes using FM mode in liquid. This opens up live cell imaging to simultaneous high resolution optical microscopy and force spectroscopy.

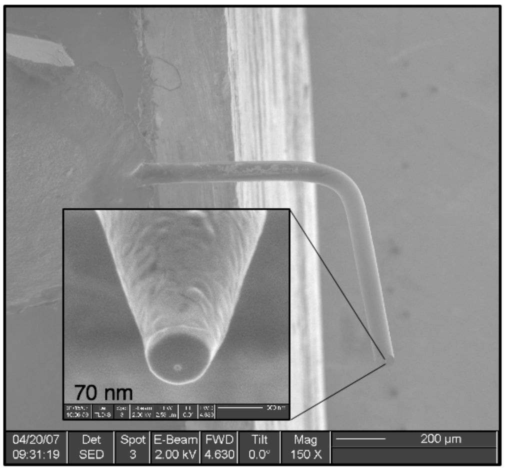

Fig.1. Example of a bent fiber tip. The inset shows the 70 nm subwavelength aperture created by FIB milling.

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# Development of High-speed Phase Modulation AFM in CA mode in Liquid

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High-speed AFM is a useful new tool for imaging the dynamic behavior of biomolecules such as protein motor in liquid. The conventional high-speed AFMs are operated in amplitude modulation (AM) mode. In order to develop the next generation high-speed AFM, the higher force sensitivity to observe soft biomolecules at lower tip-sample interaction force and the multi-functionalization to understand the dynamics are necessary. Recently, as a new operation mode, we have proposed phase modulation (PM) AFM in constant amplitude (CA) mode [1]. This mode has higher force sensitivity than AM-AFM and can operate stably without the instability of a cantilever dynamics [1]. These advantages suggest that the high-speed PM-AFM in CA mode should be established instead of the conventional high-speed AM-AFM [2]. In this study, we develop the high-speed PM-AFM in CA mode to simultaneously image the topography and the energy dissipation in liquid.

Figure 1 shows a block diagram of developed high-speed PM-AFM in CA mode. The photothermal excitation is adopted to excite the cantilever without the spurious vibrations. Amplitude and phase shift of the oscillating cantilever are detected for one cycle by the high-speed detector using sample & hold technique with the bandwidth of 1MHz. The high-speed AGC circuit maintains the oscillating amplitude at a constant level. The topography can be imaged from the feedback signal which is maintained the constant phase shift. The energy dissipation can be measured from the driving amplitude. We demonstrate the topography and the energy dissipation images of DNA on Mica and polymer-blend film with the scan speed of 10frame/s in liquid (Fig. 2). Thus, for the first time, we succeeded in developing the second generation multi-functional high-speed AFM in liquid.

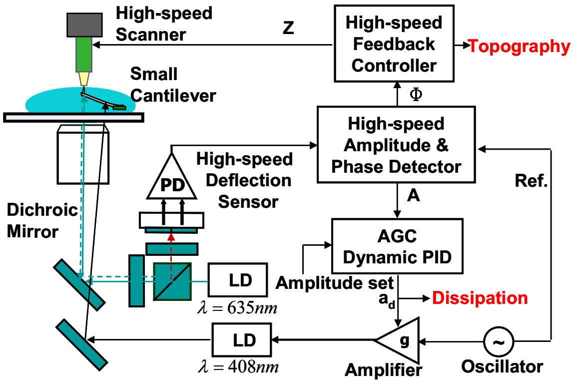

Fig.1 The block diagram of developed high-speed AFM.

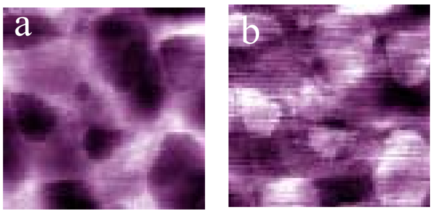

Fig.2 (a) Topographic and (b) energy dissipation images of polymer film. Image speed is 10 frames/s.

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# Dynamic Strength of the Interaction between Concanavalin A and Neoglycoconjugates of α-D-Mannosides.

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This work is centered on Atomic Force Microscopy (AFM) experiments in order to study the interactions between Concanavalin A (ConA) and neoglycoconjugates of α-D-(oligo)mannosides. ConA is a non-specific lectin, but it exhibits specificity towards α-D-mannopyranosides, α-D-glucopyranosides and α-*N*-acetyl-D-glucosaminides.[i] Many cellular recognition events are med carbohydrates.[ii] The interactions between lectins and cell-surface carbohydrate ligands are multivalent and their high-affinity is based on the oligomeric nature of these proteins and on the branched structure of cell glycolipids and glycoproteins.[iii]

To study these kind of interactions, we constructed self-assembled monolayers (SAMs) of mannosides to present carbohydrate ligands in a multivalent way.[iv] The adhesion of ConA on the tip was carried out by chemical treatment. In a first stage, the tip was functionalized with amino groups by treatment with(3-aminopropyl) triethoxysilane.[v] Glutaraldehyde was used to bind streptavidin.[vi,vii] Then the recognition surface was produced by incorporating biotin-labelled ConA via streptavidin. Once the recognition system formed the interactions forces could be studied (Fig. 1) in a buffer solution.

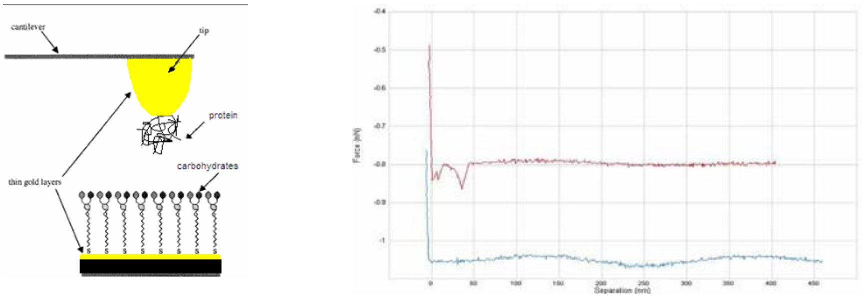

**Fig 1. SAMs on gold vs. AFM modified tip and the force distance curves obtained with and without carbohydrates.**

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# Advances in the AFM characterization of silkworm and spider silk fibers

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The study of silks represents a meeting point of some of the most active areas of scientific research in the last decades such as Materials Science, Biotechnology and Fluid Mechanics. Beside the academic interest in the characterization of these fibers, the possibility of reproducing their outstanding properties, such as their unrivalled capacity to absorb and dissipate mechanical energy in artificial bioinspired fibers [1], has been the main thrust behind the field. However, mimicking over 400 million years of evolution is proving a formidable task.

One of the major difficulties that must be overcome is the very limited knowledge on some of the basic aspects of the natural material, especially those regarding its microstructure. Despite intense efforts and the use of the most advanced characterization techniques, including Raman microscopy [2] and Nuclear Magnetic Resonance [3], the hierarchical microstructure of silk fibers has shown elusive, even in some of its gross features.

The contribution of atomic force microscopy to the field is still pending due to the lack of images of sufficient resolution. In this work we show how the repulsive regime in the dynamical mode yields AFM images of nanometer resolution (Fig.1). This technique has been used to compare the microstructure of silkworm and spider silk. It has been found that both silks share a basic microstructural design but each one is finely tuned to fulfill their biological functions.

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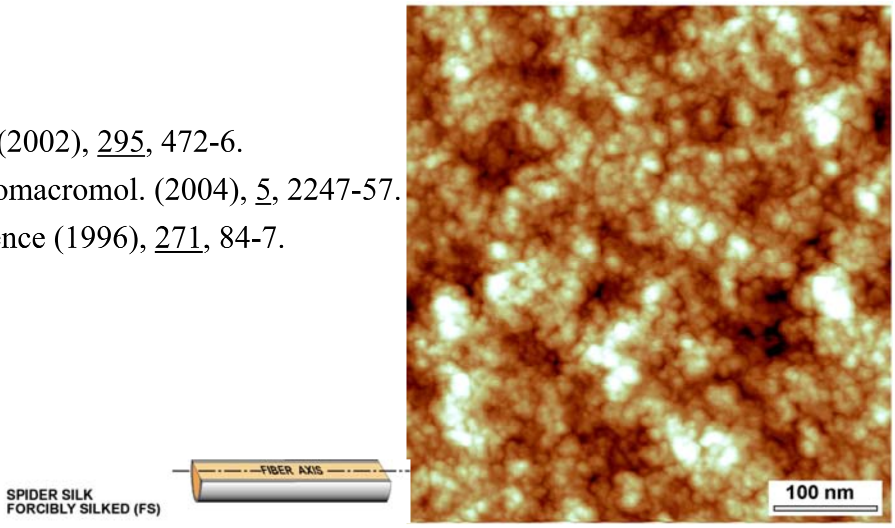

Fig. 1. Longitudinal section of an *Argiope trifasciata* major ampullate gland silk fiber.

# Organic Films Observations by TF-AFM in Liquid Environment in NC-AFM

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The study of the organization at the nanometer scale of organic molecules on an insulating substrate requires using of the AFM. In a liquid environment, operation in dynamic AFM with a cantilever necessitates to immerse the sensor in order to minimize capillarity effects. The consequence is a dramatic loss in the quality factor Q prohibiting the use of NC-AFM as in this case, the limit of sensitivity to the force gradient varies in $1/\sqrt{Q}$.

In the past decade, the use of quartz crystal tuning forks has been successfully introduced in the field of NC-AFM (Qplus sensor). Though mostly operated under vacuum, TF-AFM appears in a liquid environment as a prospective alternative to conventional cantilever based AFM due to its numerous advantages:

- high Q values by only immerging the end of the tip;
- enhanced detection allowed by low oscillation amplitudes;
- limited jump to contact due to the higher stiffness and easier coarse approach thanks to the absence of optical components;
- use of etched tips similar to STM.

Recently, Phase-Mode AFM has been developed [1]. As it directly uses the phase shift signal instead of using a PLL frequency demodulator, this technique should enhance stability and speed. We will present the results obtained with a custom TF-AFM setup working in NC-AFM, dedicated to liquid observations of molecular films. This microscope in spite of its simplicity displays an extremely high sensitivity to the formed films at the solid-liquid interface. Comparison between FM-AFM and PM-AFM will be discussed.

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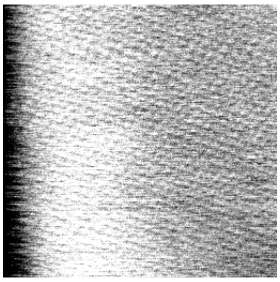

Fig. 1 Topography of cleaved (001) Mica in tetradecane (repulsive PM-AFM).
Scan= $(15,4\text{nm})^2$

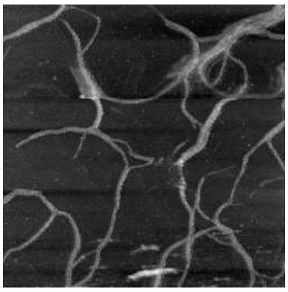

Fig. 2 Topography of an oligomer film fomed on (0001) α-Al2O3. The image is comparable to Tapping Mode AFM.
Scan $(3,3\mu\text{m})^2$

# Torsional noise of a colloidal probe interacting with a polymer layer

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The interaction forces between microparticles and surfaces determine the stability of dispersions and the adhesion of particles to the container wall. An atomic force microscope (AFM) was converted into a sensing apparatus for these colloidal force by attaching a microsphere to the cantilever. The principle of force measurement is the same as in a conventional AFM. Thus, the colloidal probe AFM can be used to measure compressive and frictional forces by analyzing the cantilever deflection or lateral force signal, respectively. A noise analysis of the torsional modes allows for identifying in-plane viscoelastic properties [1]. For colloidal probe experiments this means that flexural oscillations essentially probe viscoelastic properties vertical to the surface while torsional oscillations probe the response to a shearing motion. In an aqueous environment an analysis of torsional oscillations has the advantage that the hydrodynamic interaction between particle and wall is smaller for a shearing motion than for a vertical motion. Thus, the analysis of Brownian torsional oscillations seems well suited to dynamically probe the interaction between particles and various surface coatings.

The thermal noise analysis reveals the lateral confinement potential and lateral dissipative effects as they can occur due to layer interpenetration or due to the contact between the polymer brush and the surface. The observed increase of energy dissipation can be explained by an increased viscosity of the compressed polymer film. Notably, for the interaction between a coated sphere and a coated surface the viscosity of the contact only slowly increases with the load although the resonant frequency and thus the lateral elastic coupling increase linearly.

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# Multifrequency Fluxgate-Magnetic Force Microscopy

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**Abstract**— We consider a multifrequency imaging method which uses first resonant flexural mode for topography signal acquisition and second resonant flexural mode for measuring magnetic field interaction simultaneously. As in fluxgate magnetometer, modulation of magnetic moment of thin films of nickel attached on the apex of AFM tip can be used to couple or decouple the interactions.

A multifrequency MFM approach have not been shown yet. LiftTM mode MFM technique is generally used [1]. But it needs two passings and may suffer from thermal drifts or topographical artifacts. Also calibration of the cantilever tips coated with evaporation of magnetic materials depends on the charactheristic decay length of the calibration samples.

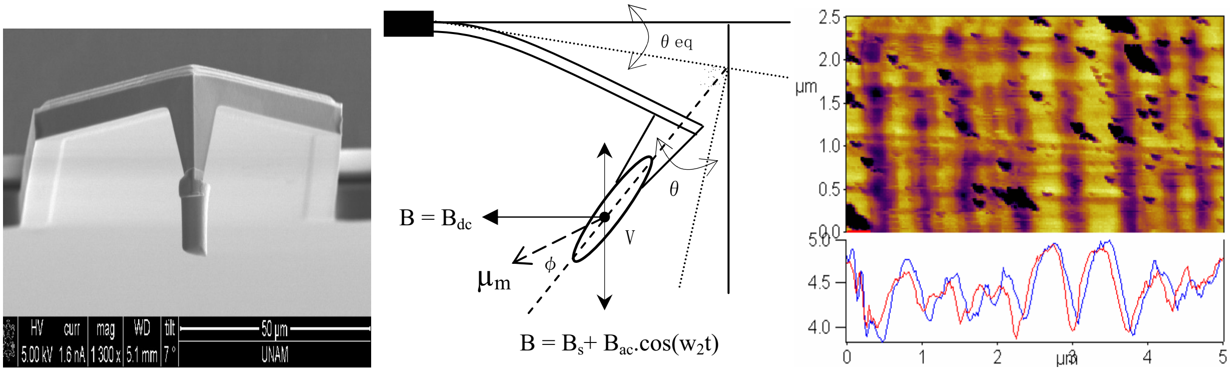

Figure: (a) Micrograph of AFM tip with thin film of nickel, modified using FIB (b) When cantilever deflects at some angle $\theta$, vertical magnetic field applies a torque on tip which can also be used to drive 2nd flexural mode (c) Amplitude of 2nd mode showing coupling between topography and magnetic interaction on a harddisk sample.

By using cantilever magnetometry, not only magnetic particles but also thin films can be magnetically characterized and can be used in MFM imaging [2]. It is observed that new tip prepared by FIB shows considerable magnetic contrast when used in standard Lift-Off imaging methode . In this work, by modulation of magnetic force applied on nickel thin film attached at the apex of tip, we showed that coupling of signals from various channels can be achieved. Coupling strength can be varied by changing coil current or setpoint parameters of MFM system. Magnetic force modulation as a direct drive on cantilever can be effectively used in multifrequency imaging[3].

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# Surface electron spectroscopy based on nc-AFM with changing bias voltage

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We introduced nanomechanical surface electron spectroscopy measuring the shift in resonance frequency ($\Delta f$) of a cantilever caused by changing bias voltage between a tip and a sample, based on noncontact atomic force microscopy (nc-AFM), while holding the tip position over the sample surface intermittently on scanning. We termed it bias-voltage noncontact atomic force spectroscopy (bias-voltage nc-AFS) [1]. The $\Delta f$-bias voltage ($V$) curves (bias-voltage nc-AFS spectra) obtained at atomically close tip-sample separations exhibited peaks on the background of a parabolic behavior due to electrostatic force between a Si tip and a Si(111)-7x7 sample. This indicates that the energy levels of the tip apex and the sample surface can be tuned electrostatically by changing the bias voltage, leading the resonance integral in quantum chemistry to be at maximum. This corresponds to formation of covalent bonding. Furthermore, we simultaneously measured electric current $I$ -$V$ curves [2]. The peak voltage of a $\Delta f$-V curve coincided with that of a $I$-$V$ curve simultaneously measured at a close separation: this behavior was different from electron tunneling, indicating that an electric conductance channel opens on forming of covalent bond with decreasing tip-sample separation under an appropriately tuned bias voltage. Note that the electrostatic force between two pieces of condensed matter involves the difference in the Fermi levels controlled by the bias voltage, the contact potential difference and the change in charging states at a tip and a sample induced by charge transfer between them, resulting in a shift, a curvature change and a deviation of the parabolic $\Delta f$-V curve.

On the other hand, by examining the dependence of $\Delta f$-V and $I$-$V$ curves on the tip-sample separation, we can attain a clue to decompose the interaction into some origins, i.e., van der Waals force, electrostatic force and covalent bonding. In addition, the collapse of potential barrier can be estimated; in general, tunneling current increases exponentially on the separation, and the increase in current should saturate in a ballistic conduction regime. Metallic bonding would also be sorted by analyzing the detailed change with the barrier collapse. We will present the dependence and discuss their behavior at atomically close separation using this spectroscopy.

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# Magnetic dissipation in Variable Field Magnetic Force Microscopy

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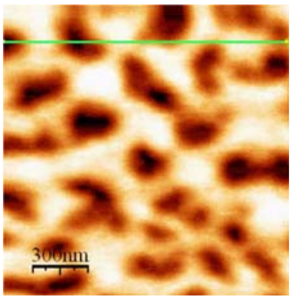
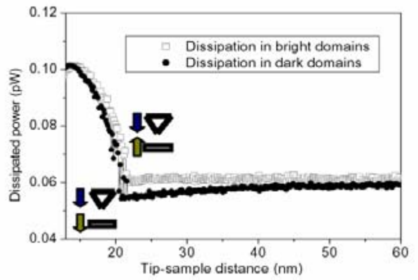
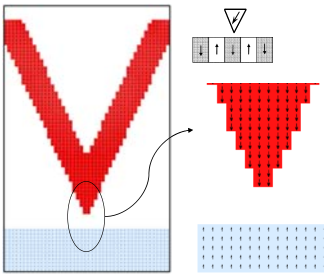
Standard magnetic force microscopy (MFM) images display the magnetic pole density on the surface [1]. The analysis of energy dissipation maps by using different dynamic force microscopy modes [2,3] can provide useful information of magnetic dissipation processes [4] at the nanoscale.

In this work, CoNi multilayers have been characterized by MFM. The sample presents high perpendicular anisotropy i.e. a distribution of domains with the magnetization pointing in up and down direction can be observed (Fig 1a). The MFM signal recorded is the frequency shift measured at a retrace distance of few tenths nanometers. In addition, the amplitude variation is recorded simultaneously i.e. the dissipated power can be calculated. In order to distinguish the magnetic effects from other dissipation origins, we have evaluated the dissipation varying the distance, the ambient humidity [5] and the external magnetic fields. The observed magnetic contrast in energy dissipation maps (Fig. 1b) is in good agreement with numerical micro-magnetism calculations (Fig.1c).







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# Initial stages of the contact between a metallic tip and carbon nanotubes.

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Hertz's model is a good approach to describe the mechanical contact between solids. At this scale, and for low mechanical loads, only the elastic properties of the bodies are taken into account. Understanding the formation of a contact between nano-objects requires an accurate description of the relevant interactions at the nanometer scale: van der Waals (VdW) and electrostatic forces for distance greater than 0.5 nm, and chemical forces for smaller distances. In this work we combine theoretical and experimental results to describe the contact formation in the model system formed by a gold tip, a carbon nanotube and a macroscopic silicon substrate. We use an atomic force microscope (AFM) (1) to measure both the tip nanotube interaction and the electrical contact resistance at low mechanical load. Density Functional Theory (DFT) (2), complemented with pairwise atom-atom VdW potentials (3), and conductance (3) calculations, reproduce the experimental data and shows the plastic deformation of the gold tip as it contacts the extremely hard single walled carbon nanotube and the variation of the contact resistance.

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# Ultra-Low Temperature, High Magnetic Field AFM: Design and Operation

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We have designed and constructed an atomic force microscope (AFM) capable of operation at milliKelvin temperatures and extremely strong magnetic fields. This microscope is mounted to the mixing chamber of a standard dilution refrigerator and has recently shown to be fully operational at 4 K and in magnetic fields of up to 15 Tesla. The results presented document the highest-field AFM yet reported in the literature. Significant design challenges are inherent in this type of experiment. Materials, wiring, and electronics must withstand both extreme temperatures and high fields, and must be compatible with the layout of the dilution refrigerator. As the force sensor, we use a commercially available piezoelectric quartz tuning fork ($f_0 \sim 32$ kHz) with a metallic tip glued to the free tine. To reduce acquisition times resulting from high quality factors of the tuning fork (~100,000 at 4K), a frequency-modulation (FM-AFM) detection scheme employing a phase lock loop has been utilized. We envision using this microscope to explore various electronic systems and exotic quantum phenomena that only arise when temperatures approach absolute zero.

Fig 1. Photograph of the AFM detached from the dilution unit showing the scanning piezotube assembly and sample holder.

# Improved characterization of MFM-tips for separating electrostatic and magnetic forces in multiferroics

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Single phase multiferroics like the transition metal oxides $BiFeO_3$ (BFO) and $BiCrO_3$ (BCO) [1] combine (anti-)ferroic properties of magnetic and dielectric nature within one unit cell. Tuning the coupling between electrostatic and magnetic forces favours these thin film oxides as being prospective candidates for novel oxidic nanoelectronic applications, such as memories, interface transport and spin controlled devices.

Recently, we showed that electrostatic and magnetic forces in these thin film multiferroics can be clearly disentangled [2] and mapped at the thermal noise limit by means of a low-temperature non contact AFM (ncAFM) operated with Kelvin Probe Force Microscopy (KPFM) in combination with Magnetic Force Microscopy (MFM). While KPFM can achieve a resolution below the 1 nm length scale [3], MFM needs improved tips as well as a thorough characterization of their magnetic behaviour.

Beyond imaging we present here a novel way to MFM tip characterization that has been tested with two different types of cantilever tips, i.e. a whisker and a Co-alloy coated MFM tip. Clear spectroscopic fingerprints are observed when sweeping the external magnetic field while recording frequency shift, damping, and static bending signals from such cantilevers. This enables us to specify suitable regimes of proper MFM and KPFM operation with the highest possible resolution.

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# Time-resolved electrostatic force detection by frequency shift mode

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Dynamics of photoexcited charges are fundamental to wide range of scientific and technological issues including optoelectronic devices, light harvest, photocatalysis, and electrophotography. However, there is no effective method to observe time-dependent behavior of photoexcited charges with nanometer scale resolution. In this paper, we report the methodological development for time-resolved electrostatic force detection using frequency shift mode. In the dynamic-mode operation, the tip interacts with the surface at only the moment when the tip closes to the sample surface. The interactive duration is estimated 1μs or less. Taking advantage of this short duration, submicrosecond-class time-resolved force detection has been achieved by the coincidence between photo-irradiation and cantilever motion.

Figure 1 shows a block diagram of time-resolved EFM. Since the frequency of cantilever oscillation is changed by the tip-sample force variation during the scan, it is necessary that the timing of each laser pulse should be synchronized by each stroke of cantilever motion. Figure 2(a) shows the relationship between frequency shift and the laser-pulse delay with various sample bias voltage. The sample was a Si(111) flushed in UHV condition. Under the pulsed laser irradiation, the oscillation amplitude is dramatically changed as a function of both delay time and bias voltage. This result indicates that the transient charge generated by pulsed laser irradiation can be detected with μs-class resolution. Furthermore, in Figure 2(b), the time course of frequency shift indicates immediate response within 1 ms to the discontinuance of laser pulse irradiation. These data suggests that this method has a potential to obtain time-resolved electrostatic force image reflecting photoexcited charge distribution.

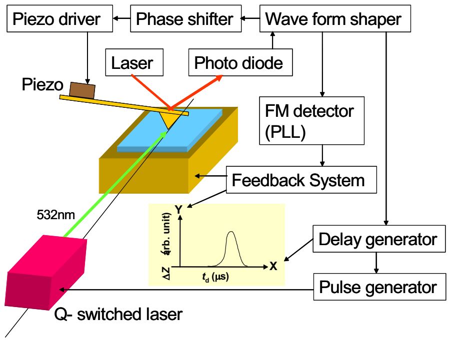

Fig. 1. Set up of apparatus for time-resolved scanning force microscopy.

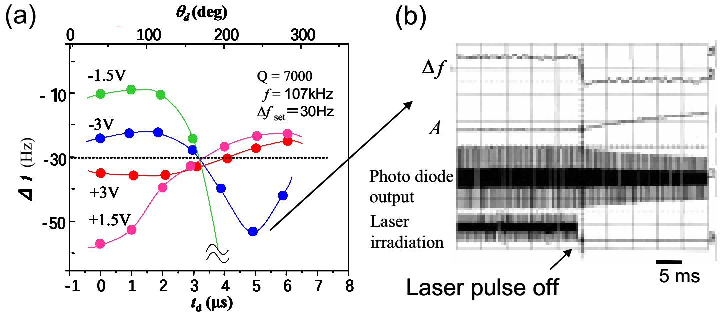

Fig. 2. (a) Freauency shift vs. delay time with various sample bias voltage. (b) Time course of frequency shift and amplitude at the discontinuance of laser pulse irradiation.

# The mechanical-diode ultrasonic regime: monitoring nanoscale events in the ultrasonic time-scale from the quasi-static cantilever mechanical response

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Mechanical-diode (MD) based Ultrasonic Atomic Force Microscopies provide a useful means for nanoscale ultrasonics [1, 2]. The MD approach for the detection of surface ultrasonic vibration relies on the nonlinearity of the tip-sample interaction forces, and on the inertia of the cantilever. It is based on the measurement of the quasi-static response of an Atomic Force Microscopy (AFM) cantilever when the forces actuating upon the tip vary nonlinearly in the ultrasonic time scale. When using the MD mode, the inertia of the cantilever allows us to detect ultrasound without monitoring any particular cantilever resonance. The MD effect has been described by introducing ultrasonic-amplitude-dependent tip-sample force-distance curves [3]. We have further extended the model in [3] by considering the ultrasonic-amplitude-dependent potential energy curves [4]. Those demonstrate that in the case that energy is dissipated during contact formation and breaking at each ultrasonic cycle, the tip encounters distinct quasi-static minimum-energy states separated by an energy barrier for certain ultrasonic excitation amplitudes. By means of the heterodyne ultrasonic force mode [5], nanoscale dissipative events in the ultrasonic time scale can be detected with high sensitivity from phase variations in the MD cantilever response. The MD cantilever response can also be monitored while realizing ultrasonic tip-induced manipulation of nanoparticles (NP) [6, 7]. In the presence of surface ultrasonic vibration, the tip-induced NP motion can be controlled by varying the ultrasonic excitation amplitude. In this case, the study of the MD cantilever response may provide information about the tip-NP-surface interactions and the NP dynamics during manipulation.

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# Intermodulation Atomic Force Microscopy

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We demonstrate the use of intermodulation to generate information in frequency space about the nonlinear tip-surface interaction in dynamic AFM. Our method employs two drive frequencies. The freely oscillating cantilever, far away from the surface, has a highly linear response. In this linear regime, when the cantilever is driven with two frequencies, it will respond with oscillation at only these two frequencies. However, when the cantilever with two drive frequencies is brought close to the surface, the nonlinear tip-surface interaction causes the generation of intermodulation products (IMPs), or new frequencies in the response, which are not present in the drive [1]. With appropriate choice of the two driving frequencies, many intermodulation spectral peaks can be generated within the mechanical bandwidth of one eigenmode of cantilever oscillation. Each of the IMPs in the response spectrum has a unique dependence on probe-surface separation, indicating that the IMPs contain information about the tip-surface interaction. Measurement of the tip-surface interaction, traditionally a slow process done in a quasi-static AFM mode by means of a force-distance curve, can be done rapidly by analysis of the spectrum of IMPs measured while scanning in dynamic imaging mode. The low order IMPs are very responsive to small changes in probe-surface separation, demonstrating that the technique is as sensitive as amplitude or phase imaging modes AFM. Intermodulation AFM is very efficient in its use of the mechanical bandwidth of the cantilever, and in comparison to tapping mode AFM, it delivers far greater information content for the same measurement bandwidth.

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# Nanopillar growth of Ge on a Si tip in inert gas under atmospheric pressure

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The shape of a tip apex of SPM plays a crucial role in depicting images with high resolution. In addition the chemical property of the tip seriously affects results of chemical analysis using SPM. Thus, it is needed to establish techniques to prepare sharp SPM tips with well-defined chemical properties. One possibility to realize it is in situ Si nanopillar growth performed using ultra-high vacuum nc-AFM [1]; we brought a clean Si tip in contact with a heated Si substrate with AFM, and retracted it slowly in a controlled manner until a neck between the tip and the substrate was broken, resulting in a sharp nanopillar consisting of Si atoms. To extend the technique to various applications, we examine Ge nanopillar growth on Si tips in an environmental controlled chamber filled with an inert gas under atmospheric pressure.

Commercial Si cantilevers were used in this study, which were set in an nc-AFM and heated to remove contaminants from them in the chamber filled with pure Ar or $N_2$ gas, or pumped to a conventional vacuum. Afterwards a Ge substrate on a sample stage of the nc-AFM was heated and vaporized above 500 °C to deposit Ge on Si tips. Next the tips were brought in contact to the substrate for about 20-30 s, and slowly retracted. The procedure was controlled by monitoring current passing between tip and sample. Figure 1 shows SEM images of a Ge nanopillar on a Si tip. Dark spots on the tip sides correspond to Ge clusters (Fig. 1(a)), and the contrast of tip apex changed dark, attributed to Ge coverage (Fig. 1(b)), with a tip radius less than 10 nm. The change in quality of nc-AFM images and bias voltage-$\Delta f$ curves using the tips will be presented.

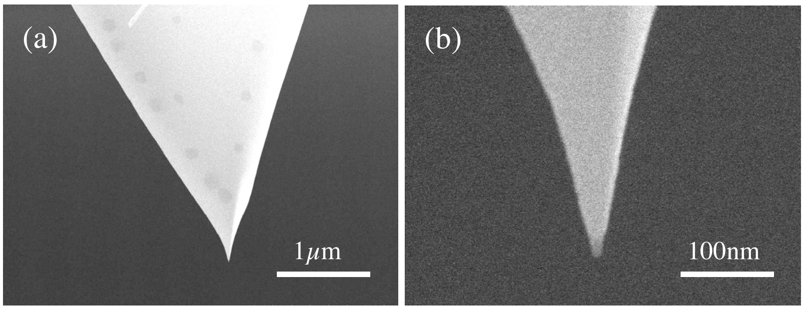

Fig. 1 SEM images of a Ge nanopollar grown on a Si tip.

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# Rapid Multidimensional Data Acquisition in Scanning Probe Microscopy: Applications to Quantitative Dissipation Measurements

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## Abstract

Quantitative energy dissipation measurements in force-based SPM is key to understanding fundamental mechanisms for energy transformations on nanoscale, molecular, and atomic levels. To date, these measurements are invariably based on the phase and amplitude detection in constant frequency mode (Cleveland method), or amplitude detection in frequency-tracking mode. The data analysis in both cases implicitly assumes that the driving force exciting the cantilever is constant, and hence the amplitude of oscillations at the resonance is inversely proportional to the Q-factor of the cantilever. Hence, these approaches are not applicable when the driving force is position dependent, as is the case for virtually all SPMs including mechanical, electromechanical, and Kelvin probe measurements. This limitation stems from the fact that all current SPM sample a single frequency in the Fourier domain of the system, limiting the amount of obtained information. In other words, only two out of three parameters (response at the resonant frequency, resonant frequency, Q-factor) defining cantilever dynamics can be determined independently. Here, we developed and implemented a new approach for SPM detection, based on excitation and detection of signal having a finite amplitude density in a selected region in Fourier domain. This allows simultaneous measurement of all three parameters defining resonance. The applications of this band excitation method allows one to obtain local spectral response at ~10ms/pixel rate, compatible with fast imaging, and is illustrated for force-distance spectroscopy and magnetic and elastic imaging. The BE method thus represents a new paradigm in SPM, beyond traditional single-frequency excitation and provides rapid acquisition of multidimensional spectroscopic information.

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# Theory of Multifrequency Atomic Force Microscopy

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The need for higher compositional resolution and sensitivity at the molecular level has lead to explore atomic force microscopy imaging with higher harmonics as well as with other flexural or torsional cantilever modes. More recently, the AFM is experiencing the evolution from single to multifrequency excitation and detection schemes of the cantilever-tip motion. Here we develop a theory that explains the origin of the high force sensitivity observed in multifrequency force microscopy experiments. The ability of the microscope to extract complementary information on the surface properties is increased by the simultaneous excitation of several flexural cantilever modes. The analytical model identifies the virial and the energy dissipated by the tip-surface forces as the parameters responsible for the material contrast. The agreement obtained among the theory, experiments and numerical simulations validates the model.

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Fig. 1 (Courtesy of Ramses V. Martinez)

# Measuring and optimising quality-factors Q of cantilevers used in dynamic force microscopy

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## Abstract

We describe a test-setup for fast quality control regarding the Q-factor of commercial cantilevers prior to their use in an ultra-high vacuum non-contact atomic force microscope. The detection of cantilever motion is based on the beam-deflection method.

We examine a variation of the Q-factors for the same cantilever type fabricated from the same wafer where the resonance frequency does not exhibit strong fluctuations. To further elucidate the influence of defects and contamination of the cantilever on the Q-factor, we analyze the influence of ex-situ annealing experiments in different atmosphere on the Q-factor.

In another set of experiments, we study the effect of different cantilever support mechanisms on the Q-factor. For cantilevers mechanically clamped to the excitation stage in our test setup, we observe a decrease of Q-value after gluing them onto standard cantilever holders of different AFM set-ups (Fig. 1). Additional variations of the measured Q-Values can be caused by the contact between the cantilever holder and the excitation stage.

Finally, methods to determine the cantilever spring constant by known cantilever dimensions [1, 2] are compared to a method which determines the spring constant by measuring the thermal excitation of the cantilever in the AFM set-up [3] (Fig. 2).

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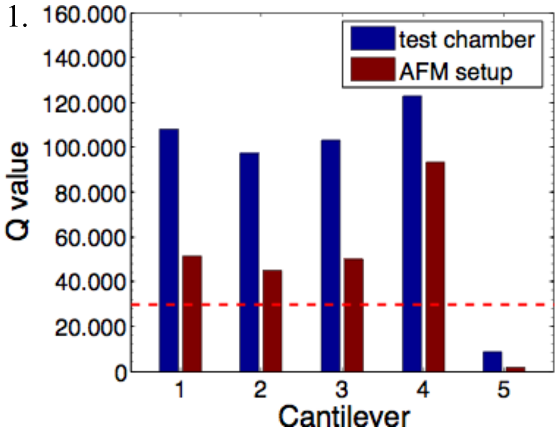
Fig. 1.



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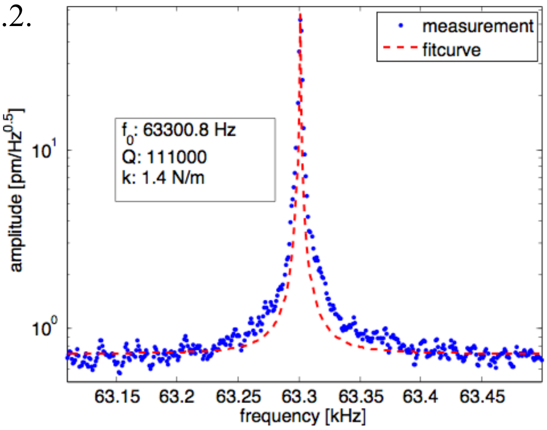

# Significant performance improvement of a commercial dynamic scanning force microscope

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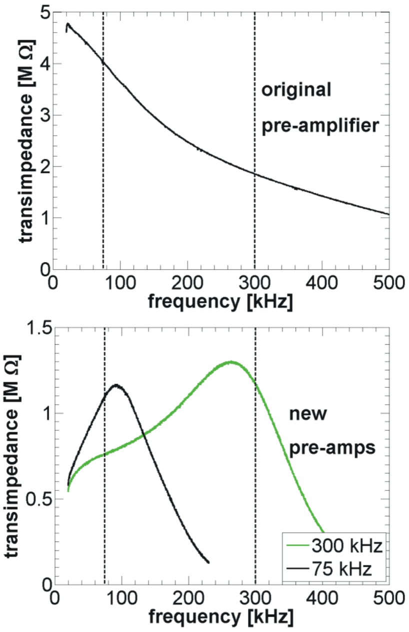
## Abstract

We report on a modification of the optical beam deflection set-up and the pre-amplifier of a commercial ultra-high vacuum scanning force microscope (Omicron UHV AFM/STM) yielding a decrease of the spectral noise density from 2757 fm/$\sqrt{Hz}$ to 272 fm/$\sqrt{Hz}$ [1]. The major part of the noise reduction is achieved by an exchange of the originally installed light emitting diode (LED) by a laser diode (LD) placed outside the vacuum, where the light is coupled into the ultra-high vacuum chamber via an optical fibre. The set-up is further improved by the use of pre-amplifiers having a band pass characteristics tailored to the cantilever resonance frequency. Fig. 1 shows the frequency dependence of transimpedance for the originally installed preamplifier in comparison to the new preamplifiers optimized for 75 kHz and 300 kHz. Fig. 2 shows the difference in root spectral noise density before and after the modification of the set-up. The enhanced signal to noise ratio is demonstrated by a comparison of atomic resolution images on $CeO_2$ (111) obtained before and after the modification.

**Reference** S.Torbrügge et al., Rev. Sci. Instrum., submitted

Fig. 1.



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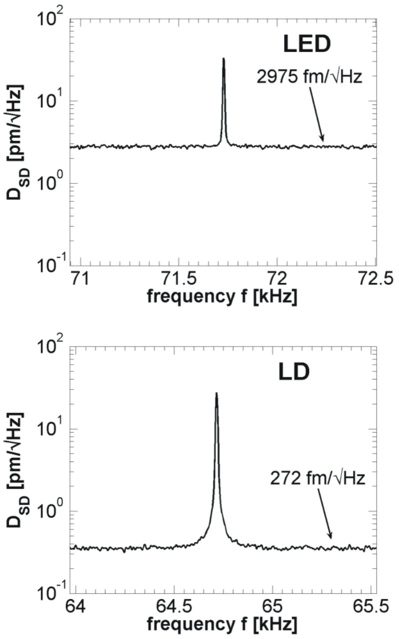

# Decoupled Control of Piezoelectric Tube Actuator Based AFM Scanning Using the MIMO Disturbance Observer

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## Abstract

Quartered piezoelectric tubes used in nano-scale AFM scanning (Fig. 1) present undesired coupled motion of their axes which reduces the accuracy of the scan due to the inevitable eccentricity of their inner and outer diameters during manufacturing. Here, we propose a method called the MIMO disturbance observer, referred to the work of Güvenç [1], applied to this problem to decouple the x and y axes of lateral motion which causes image distortions. A simulation study is used to demonstrate the success of this method. An accurate analytical model governing the coupling of the axes is used referred to the work of El Rifai [2].

The controller architecture is presented in Fig. 2. Image distortions are prevented by appropriate selection of the filter Q and the nominal plant $P_n$.

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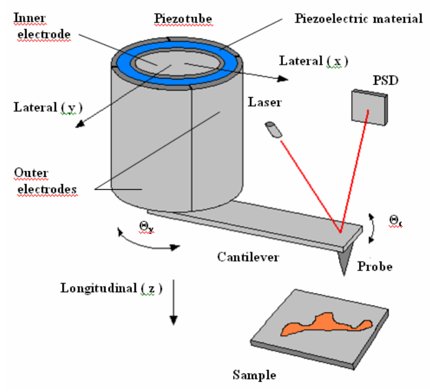

Fig. 1. A quartered piezotube AFM scanner setup.

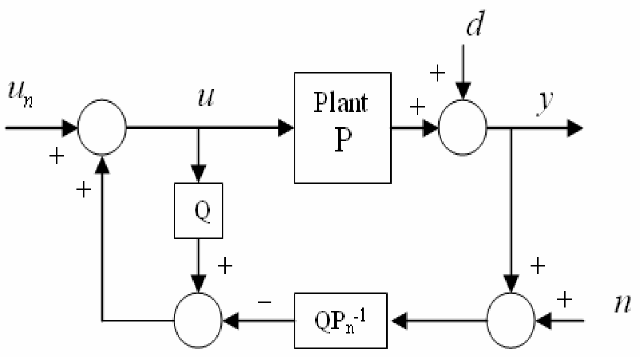

Fig. 2. MIMO disturbance observer controller architecture.

# Eddy Current Microscopy of Nanostructures in UHV and under ambient conditions

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Eddy current microscopy (ECM) provides a unique method to gain qualitative insight into the local electrical conductivity of nanostructures. ECM is based on the well established method of dynamical force microscopy [1]. The basic principle is as follows: The time-dependent magnetic field of an oscillating magnetic probe induces eddy currents within conducting materials [2], resulting in a detectable dissipation signal. We will present results from ex situ [3] as well as from in situ measurements on various samples in comparison with previous measurements. As an example, figure 1 shows the dissipation signal measured at irradiated $CaF_2$ [4,5] .

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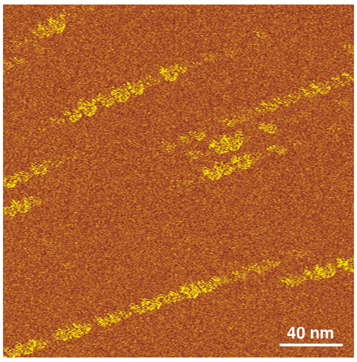

Fig. 1: Dissipation image of irradiated $CaF_2$ measured under UHV conditions [5].
The scale bar runs from 0.5 to 2.5 eV/cycle.

# Frequency and amplitude modulation torsional resonance atomic force microscopy

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Scanning probe shear force imaging allows for the characterization of in-plane surface properties. We realized a scanning shear force microscope for operation under ambient conditions which was based on a torsional resonance mode atomic force microscope [1]. The system was equipped with a frequency modulation unit ensuring sensitive surface tracking. In close proximity to the surface we observed a positive detuning. However, a negative frequency shift could be achieved by slightly tilting the specimen with respect to the plane of oscillation [2]. Also the in-plane mechanics of polymers can be probed by integrating frequency modulation and torsional resonance mode atomic force microscopy [3].

In order to test the capabilities of the system a freshly cleaved mineral surface was investigated. The mineral chlorite exhibits brucite-like and talc-like surface areas. The surface was characterized in constant amplitude mode in order to measure topography and local energy dissipation. Energy dissipation revealed a slight contrast between the brucite-like and talc-like surfaces [4].

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# XANAM[II] – X-ray Induced Force Change Image -

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Development of nanoscale elemental analysis methodology is the next target in the SPM technology. We have proposed a new SPM for chemical analysis called as X-ray Aided Non-contact Atomic Force Microscopy (XANAM). The XANAM utilizes the NC-AFM and the energy tunable synchrotron radiation which provides us chemical information. The XANAM relies on the novel phenomenon we found previously. When a cantilever tip of NC-AFM was fixed above an Au island and the X-ray energy was scanned around the Au $L_3$ X-ray absorption edge to measure force spectrum, we found a peak in the attractive force between the tip and surface at a certain energy [1].

In this paper, we have succeeded in obtaining a 2 dimensional chemical image by scanning the NC-AFM tip under the X-ray irradiation before and after the Au $L_3$ X-ray absorption edge. The experiments were carried out at the BL7C in KEK-PF in Tsukuba, Japan. After obtaining a normal NC-AFM image on an Au deposited surface (Fig. 1a), we observed the same surface under X-ray irradiation with changing its energy from lower to higher than that of the absorption edge. We obtained an image according to the X-ray induced force change as shown in Fig. 1b). The attractive force is enhanced under the X-ray irradiation with higher energy than that of the absorption edge. The change is corresponding to the 0.1~0.2 nm height difference. We proposed a XANAM mechanism for such a large contrast change as follows. The X-ray induced alteration of force interaction results from the increase of covalent electron density in the bonding orbital between the tip and the surface caused by the transition from the core electron to the bonding orbital. We are now improving signal sensitivity and spatial resolution by adopting quartz tuning fork which has a sharper tip apex than our present system of the PZT thin film cantilever.

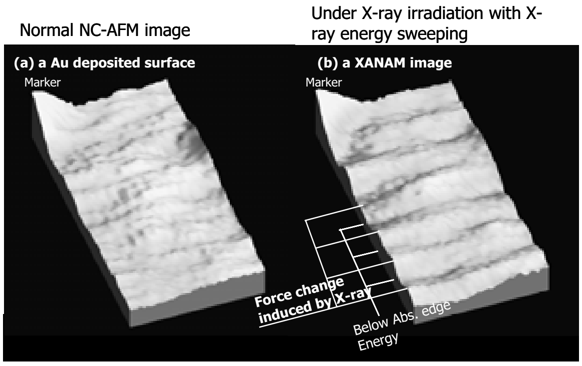

Fig. 1 a) a normal NC-AFM image of a Au deposited surface. b) a NC-AFM image of the same area under X-ray irradiation, 153x78 μm$^2$, $f_0$=101.7 kHz, $\Delta f$= -5 Hz A= 1.0 nm.

This work is supported a Grant-in-Aid for young scientists (A) from the JSPS.

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# Experimental study on stabilization of chaotic cantilever oscillation in AM-AFM by time-delayed feedback control

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It has been recently reported that cantilevers in the amplitude modulation atomic force microscopy (AM-AFM) may exhibit irregular and non-periodic oscillation due to nonlinear tip-sample interaction force [1-3]. The oscillation is called *chaotic oscillation* and often observed when a soft cantilever is excited with a large amplitude. Some of the authors have thus proposed application of the time-delayed feedback control (TDFC) [4] to stabilize chaotic oscillation [5].

In order to demonstrate the applicability of TDFC experimentally, we implemented a TDFC controller using a digital delay line. We employed a magnetic excitation method and a home-built deflection sensor for minimizing phase delay in the feedback loop. We used magnetically coated cantilevers (Agilent, Type I MAC levers of nominal spring constant and resonance frequency: 0.6 N/m and 75 kHz, respectively). The sample was a polyimide film. An experimental result is shown in Fig. 1. As shown in Fig. 1(a), irregular and non-periodic oscillation was observed in the close proximity of the sample surface. Fig. 1(b) shows the stabilization of the oscillation achieved after control input was activated. It was confirmed that the control input converged to nearly zero volt. These facts suggest that an unstable periodic orbit is successfully stabilized by adding small perturbation to the excitation signal.

**Acknowledgment** This work is partly supported by Grant-in-Aid for Young Scientists (B) No. 20760238, Japan Society for the Promotion of Science (JSPS).

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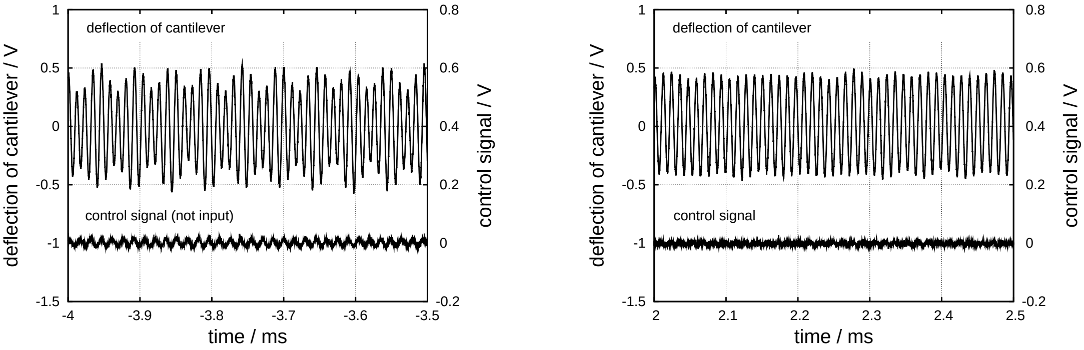

(a) without control

(b) with control

Figure 1: Stabilization of cantilever oscillation by time-delayed feedback control. (a) Irregular and non-periodic oscillation observed without control. (b) A periodic oscillation was recovered using time-delayed feedback.

# Understanding FM-AFM images on Ceria with ab initio simulations

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Ceria ($CeO_2$) is a rare-earth oxide with important industrial applications in catalysis and solid-oxide fuel cell technology. Acting as a noble metal supporting substrate, ceria enhances the water-gas shift reaction (WGS, $CO + H_2O \rightarrow H_2 + CO_2$), leading to the massive production of hydrogen [1]. FM-AFM represents the perfect tool for improving our understanding of the microscopic pathways in the WGS reaction [2]: it offers different channels of information and does not require the particular doping and high-temperature conditions that are necessary for the use of the STM in this wide-gap material. A confirmation of the possibilities offered by this methodology comes from recent work where a combination of FM-AFM topography and dissipation images provide the basis to locate sub-surface oxygen vacancies and to show convincingly their local ordering [3].

We have performed an extensive set of ab initio simulations in order to contribute to the understanding of the image contrast, the identification of the observed defects (including oxygen vacancies, adsorbed water molecules and hydroxyl groups) and the interpretation of recent manipulation experiments. Our calculations involve the characterization of the stoichiometric and the reduced surfaces, and the calculation of the tip-sample interaction for different tip models. These calculations are based on approximate DFT+U methods (as implemented in VASP [4]), where the corresponding functional (LDA, GGA) is corrected with the addition of an effective repulsive interaction fitted to reproduce accurate results from hybrid functionals [5].

We thank Oscar Custance, Michael Reichling, Veronica Ganduglia-Pirovano, Matthew Watkins and Alexander Shluger for sharing with us their recent results on this system.

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# 11 Proceedings

The organizing committee of NCAFM-2008 and IOP Publishing have agreed with that two separate and distinct publications will be produced.

- a Topical Issue of the journal *Nanotechnology* *(http://www.iop.org/journals/nano)*, and
- an open access proceedings in *Journal of Physics: Conference Series* *(http://jpcs.iop.org)* (JPCS)

The objective is to create a proceedings that record work presented at NCAFM-2008 and a Topical Issue whose scope will build on NCAFM-2008, reaching out into the wider community, to reflect many of the cutting edge and exciting developments in our field. Specific publication details are as follows:

## Topical Issue "High-resolution Non-contact Atomic Force Microscopy" of the journal *Nanotechnology*

Papers appearing in the Topical Issue are required to have the high standards regarding scientific content and novelty of *Nanotechnology*. The objective is to create an editorially balanced, high quality journal issue that reflects the most interesting and innovative work in the field.

Authors that want to contribute a paper to the Topical Issue should send an email to the organizers (ncafm2008@uam.es) including the tentative title, a short summary of the content (few sentences) as well as the expected length (not exceeding 5-7 journal pages). Given that the number of papers that can be included in the Topical Issue is limited, contributions accepted as talks at the conference will be given first priority if too many submissions are committed. Both the journal *Nanotechnology* and the conference organizing committee will work together to prepare the final list of authors that will be invited to contribute.

### Submitting to the Topical Issue

Authors invited to contribute a paper will be contacted with submission details and links to the appropriate templates and guidelines.

### Submission deadline

The submission deadline for the Topical Issue is 10 weeks after the conference (December 1st 2008). Publication is expected in Winter-Early Spring 2009.

# Proceedings

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Designed specifically to capture just the work presented at NCAFM-2008, the JPCS proceedings offer all conference participants the possibility to contribute an article. All papers published in JPCS are fully citable and upon publication will be free to download.

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# Advertisement

Nanonis designs and manufactures SPM Control System for highest demands with a strong emphasis on flexibility with track record reliability. Our goal is to provide innovative products and services to help researchers increase their efficiency and optimize routine SPM work.

Thanks to our adaptation kits and integration know-how, most commercial and home-built systems can be seamlessly connected to a Nanonis controller, thus often opening new techniques and field of applications not previously possible. The fully digital and modular architecture combined with a wide range of add-on modules let the user decides what is most suited to its needs. For instance, our Oscillation Controller (OC4) allows for all dynamic AFM modes and includes a fully digital PLL, a FFT and lock-in up to 5MHz.

And for our most demanding customers, we provide a LabVIEW programming interface that programmatically controls the Nanonis software for custom designed experiments. Instead of using a simple scripting language, the Nanonis system provides full access to the LabVIEW programming environment, with its well-known user-friendliness and widespread use.

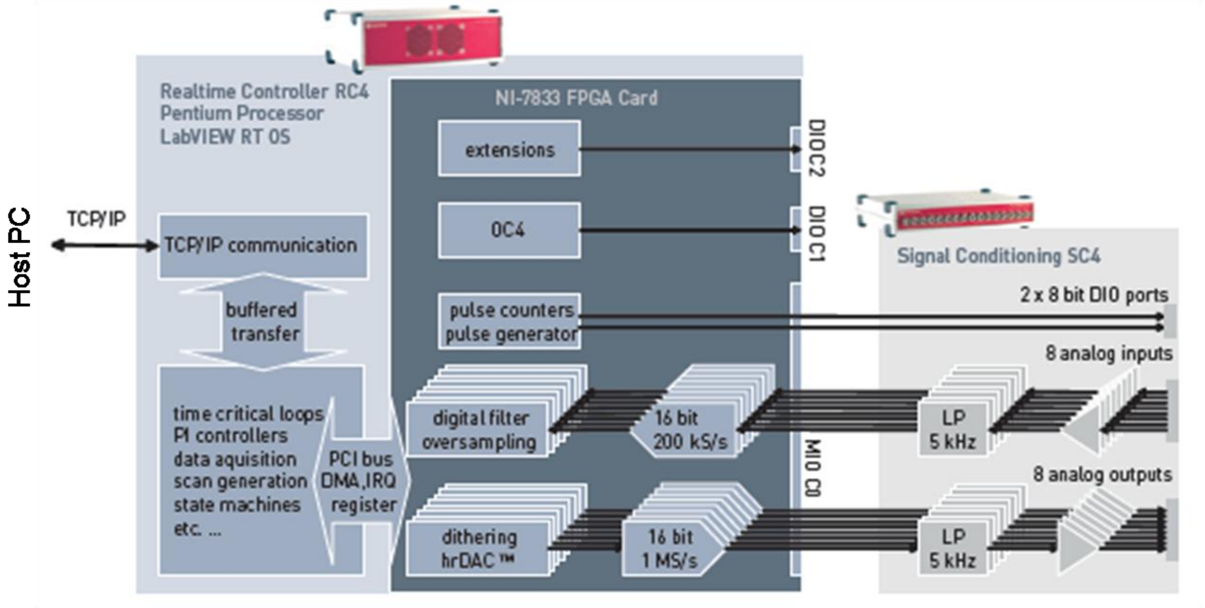

**Fig: fully digital SPM Controller with 8 independent inputs, 8 independent outputs and 8 internal signals.**

Visit Nanonis at www.nanonis.com

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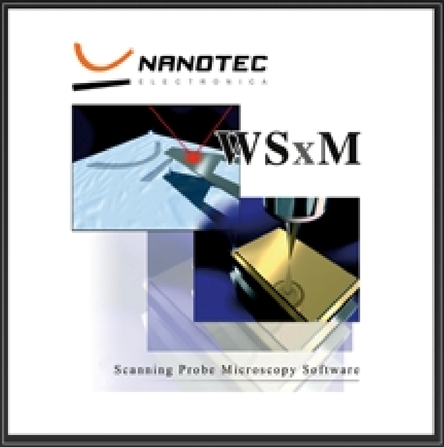
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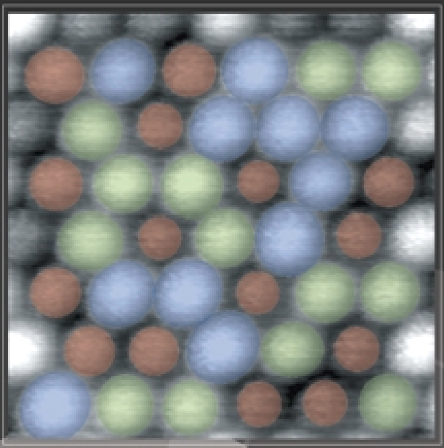
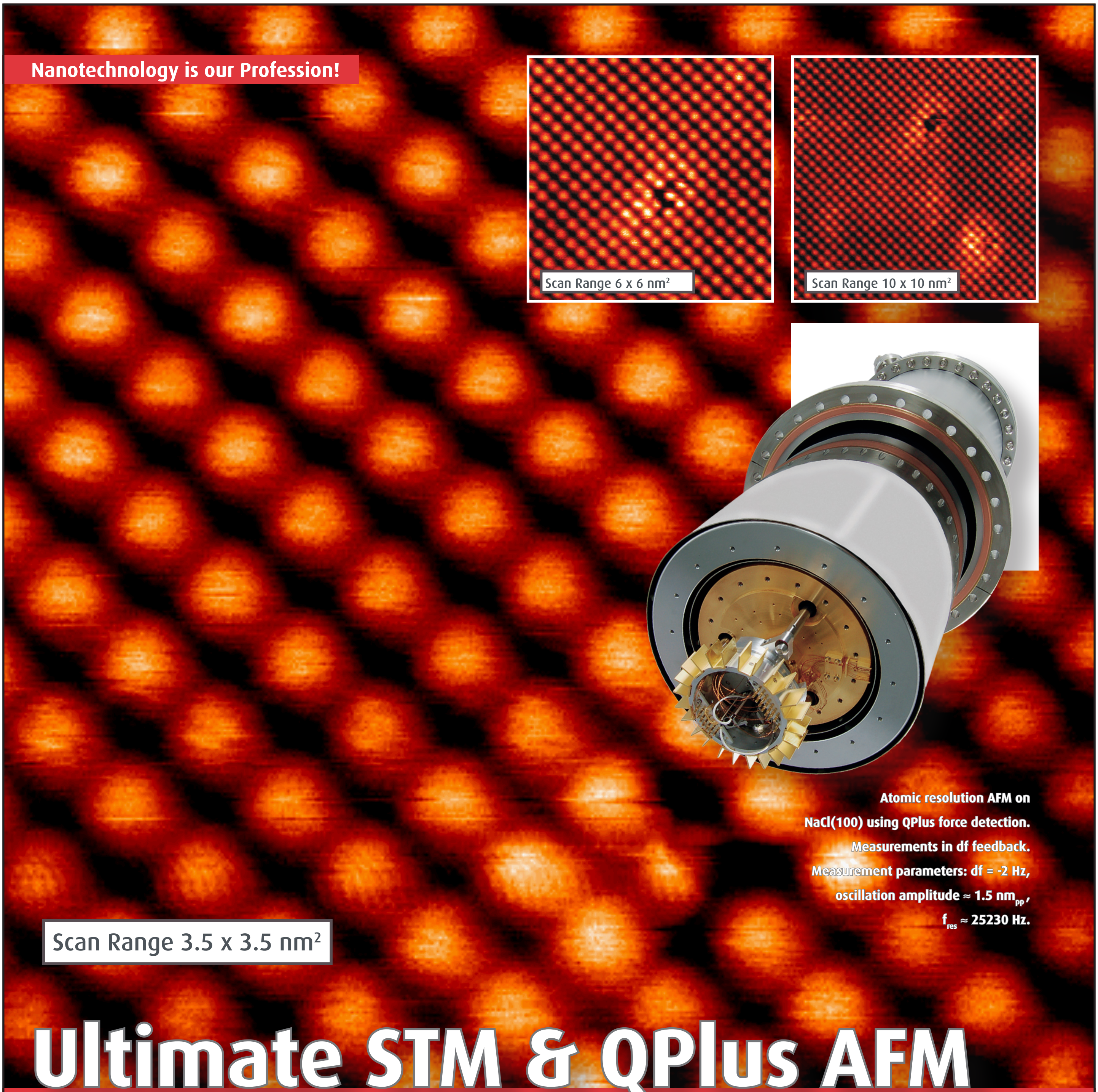
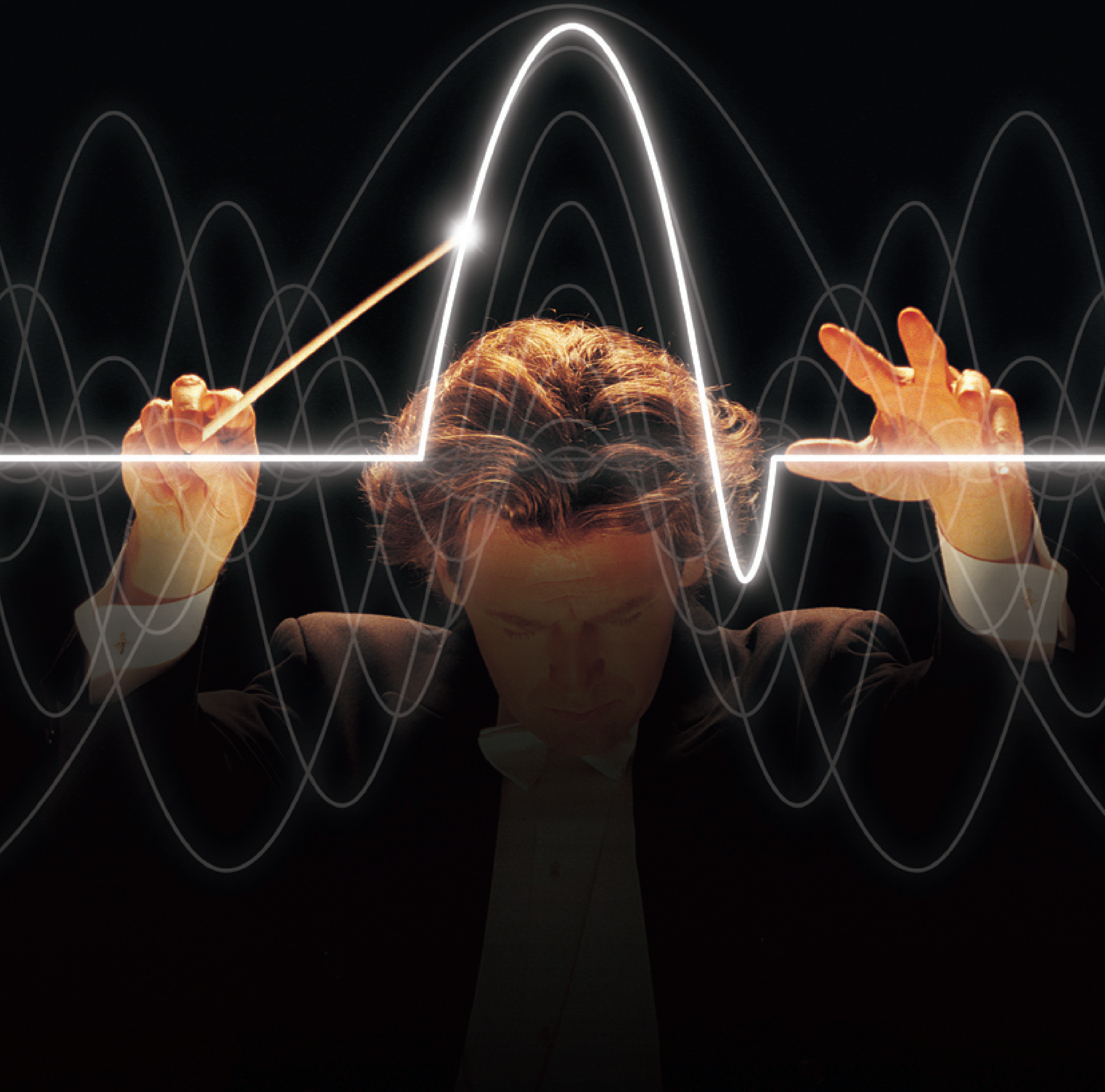
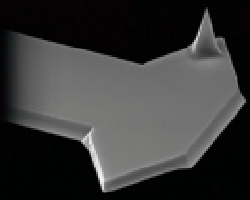
WSxM
Scanning Probe Microscopy Software

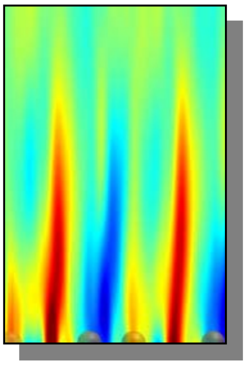








Veeco HarmoniX Probe



# NC-AFM 2009

## 12th International Conference on Noncontact Atomic Force Microscopy

Yale University, New Haven, CT, USA

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## *First Announcement:*

## 12th International Conference on Noncontact Atomic Force Microscopy

**Venue:** Campus of Yale University, New Haven, CT, USA
*(easy to reach from New York City airports)*

**Date:** August 25-28, 2009 *(tentative)*

| | |
|---|---|
| Conference Chair: | Udo D. Schwarz |
| Local Organizing Committee: | Eric I. Altman<br>Hong X. Tang<br>Woo-Joong Kim |

**Webpage:** A link to the future conference site will be posted on
***http://www.eng.yale.edu/nanomechanics***

The conference will overlap with a *Satellite Workshop on Casimir Forces and Their Measurement* organized by Steve K. Lamoreaux on August 24-25

**Sponsors:** *(preliminary list)*

- Yale Center for Research on Interface Science and Phenomena (CRISP)
- Yale Institute for Nanoscience and Quantum Engineering (YINQUE)
- Office of the Provost, Yale University
- Department of Mechanical Engineering, Yale University
- Sloane Physics Laboratory, Dept. of Physics, Yale University

# NC-AFM 2008 Sessions:

- Force Spectroscopy I:
  Tuesday 16, 9:20 – 10:40
- KPFM I:
  Tuesday 16, 11:20 – 12:40
- Manipulation:
  Tuesday 16, 14:30 – 15:50
- Oxides:
  Wednesday 17, 9:00 – 10:40
- KPFM II:
  Wednesday 17, 11:20 – 12:40
- Liquids and biomolecules I:
  Wednesday 17, 14:30 – 15:50
- Magnetic and electronic properties:
  Thursday 18, 9:00 – 12:00
- Liquids and biomolecules II:
  Thursday 18, 12:00 – 13:00
- Insulators and molecules:
  Friday 19, 9:00 – 10:40
- Force Spectroscopy II:
  Friday 19, 11:20 – 12:40

| | **Sunday**<br>14, Sept. | **Monday**<br>15, Sept. | **Tuesday**<br>16, September | **Wednesday**<br>17, September | **Thursday**<br>18, September | **Friday**<br>19, September |
|---|---|---|---|---|---|---|
| *8:30* | ***1st Multi-Frequency AFM Conference*** | ***1st Multi-Frequency AFM Conference*** | *Registration* | *Registration* | *Registration* | *Registration* |
| **9:00** | | | Opening Remarks | M. Reichling | A. Schwarz | T. Filleter |
| 9:20 | | | M. Ashino | J. V. Lauritsen | H. Holscher | S. Gauthier |
| 9:40 | | | S. A. Ghasemi | M. Heyde | G.Rubio-Bolllinger | P. Rahe |
| 10:00 | | | U. D. Schwarz | H. P. Pinto | Y. Miyahara | S. Burke |
| 10:20 | | | Y. Sugimoto | R. Bechstein | S. Huant | S. Torbrugge |
| *10:40*<br>*40 min* | | | *Coffee Break* | *Coffee Break* | *Coffee Break* | *Coffee Break* |
| **11:20** | | | S. Sadewasser | Ch. Loppacher | P. Jelinek | R. Hoffmann |
| 11:40 | | | Th. Glatzel | E. Tsunemi | B. Uder | O. Custance |
| 12:00 | | | S. Watanabe | P. Milde | H. Yamada | M. Abe |
| 12:20 | | | A. Baratoff | R. Bennewitz | W. Hofbauer | F. J. Giessibl |
| ***12:40*** | | | ***Lunch***<br>*110 min*<br>*(12:40-14:30)* | ***Lunch***<br>*110 min*<br>*(12:40-14:30)* | E. Sahagun | Closing Remarks |
| *13:00* | | *14:00-19:00*<br>***KPFM Workshop*** | | | ***Lunch***<br>*(13:00- )* | |
| **14:30** | | | Y. Sugawara | T. Fukuma | | |
| 14:50 | | | P. Pou | N. Oyabu | | |
| 15:10 | | | A. Foster | N. F. Martinez | | |
| 15:30 | | | T. Trevethan | M. Tsukada | | |
| **15:50**<br>\|<br>**18:00** | ***1st Multi-Frequency AFM Conference*** | | Poster Session<br>I<br>*Coffee Service*<br>*(15:50-18:00)* | Poster Session<br>II<br>*Coffee Service*<br>*(15:50-18:00)* | | |
| | | ***19:00 - 20:00***<br>*Registration*<br>*Welcome Party* | | | ***20:00 - 23:30***<br>*Banquet* | |