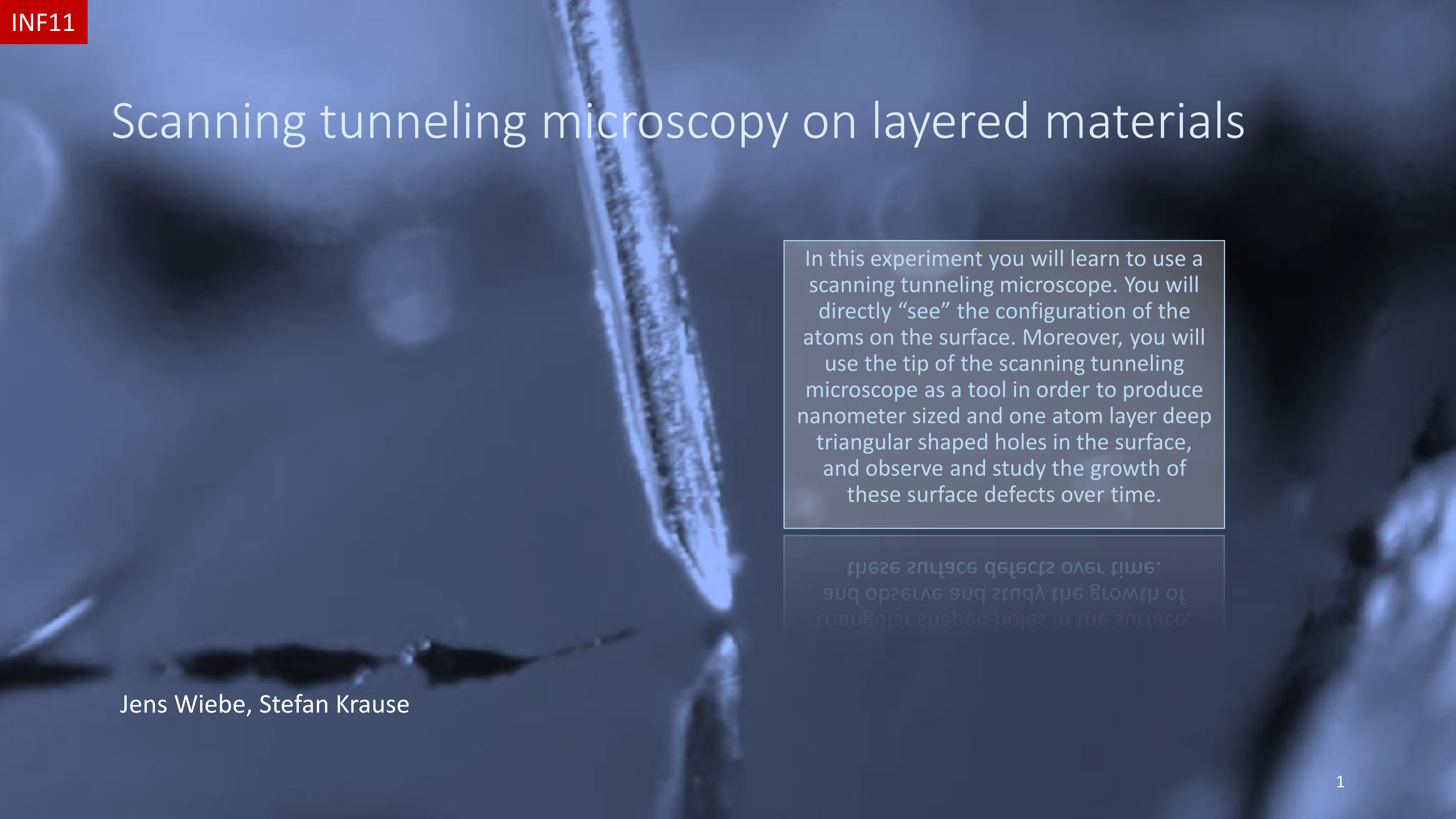


Scanning tunneling microscopy on layered materials

In this experiment you will learn to use a scanning tunneling microscope. You will directly "see" the configuration of the atoms on the surface. Moreover, you will use the tip of the scanning tunneling microscope as a tool in order to produce nanometer sized and one atom layer deep triangular shaped holes in the surface, and observe and study the growth of these surface defects over time.

Jens Wiebe, Stefan Krause

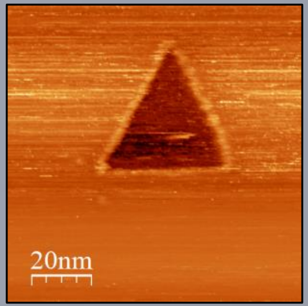
Scientific Content

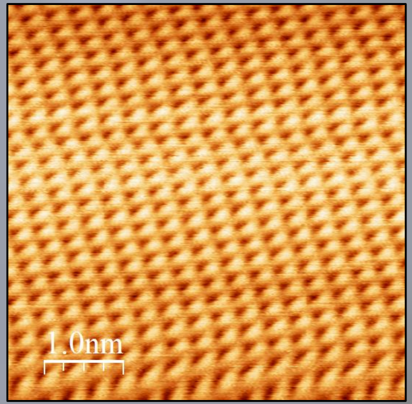
How is the experiment linked to the scientific bachelor program?

- STM connecting classical and quantum physics (Physics I-III lectures):
  - Measure electric current, coarse/fine positioning with piezo material, electric field from tip
  - Tunnel effect, atomic-scale precision for imaging and manipulation
- Strong connection to Physics IV lecture (solid state physics)

Added value of the experiment to the lectures

- Directly see the atomic lattice on HOPG
- Layered materials in WSe_2: Van-der-Vaals vs. Covalent bonding
- Experience tunneling effect and density of states:
  - Metal vs. Semiconductor
  - Schrödinger equation: Distance dependence
  - HOPG: two types of atomic coordination
- 2D Fourier transformation of a hexagonal lattice





Aim of the Experiment (1)

Have fun experimenting!

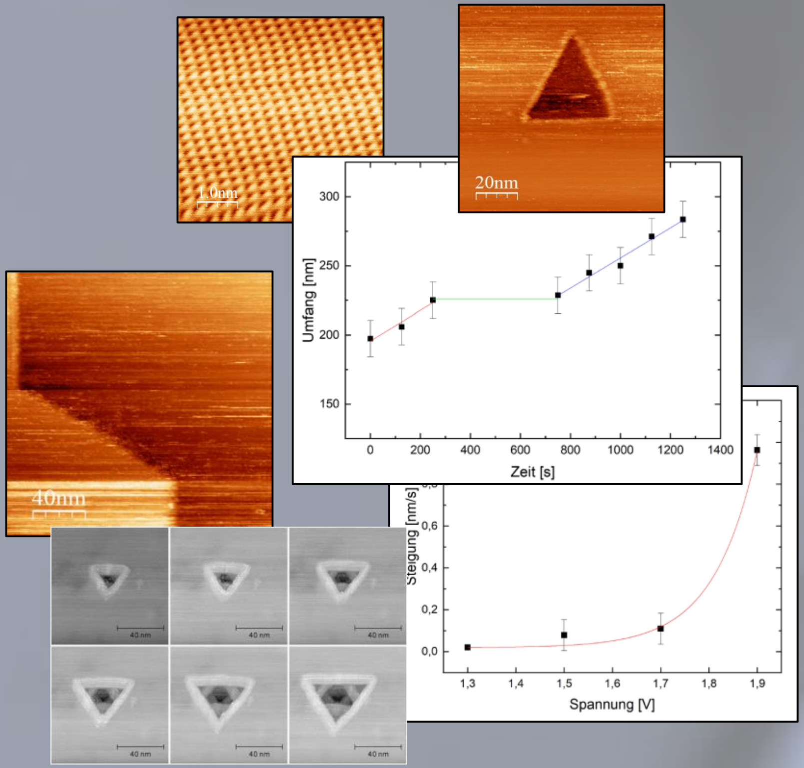
Calibration on HOPG:

- Real-space imaging with atomic resolution
- Observe and understand LDOS effects on the imaging process
- Use HOPG for the calibration of the STM

Manipulation and growth of atomic-scale defects on WSe_2:

- Electrochemical etching with STM tip
- Correlate triangular shapes with the crystal structure
- Observe and understand the growth of surface defects over time
  - Determine growth speed
  - Manipulate with electric field
- Use the new knowledge to “write” something into the WSe_2 surface

➔ Scanning tunneling microscopy as a standard tool for surface science



Aim of the Experiment (2)

Develop own experimental methods and ideas

- Reflect on a clever way to reach a goal with reasonable efforts (Don‘t waste time and efforts)
- What are the limits? Deal with parasitic effects (it is still physics!) (Thermal drift, Piezo creep, Noise)
- Every data point has an error bar:
  - What can be neglected, what is considered for error propagation? (Pixel resolution vs. STM resolution)
  - How to reduce the error bar of the quantity you are interested in? (Vary the parameters appropriately)
- There is much more in the raw data than the obvious or expected (Needs post-processing)

Get in touch with STM and the working group

- Spin-polarized STM, dynamics, multi-probe transport measurements
- Fundamental research for future quantum computers based on Spintronics, CNT, DNA, …
- Links to SFB925, Excellence Cluster “AIM”, EU Horizon Europe “3D-BRICKS”, …

Experimental Setup

Ready-to-use STM setup (under ambient conditions, no black box!):

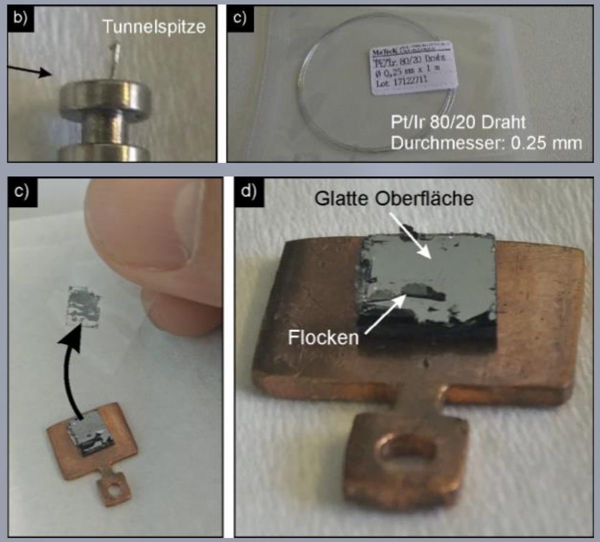
- How to position and move a scanning probe over a surface? (Piezo tube, Feedback-Loop)
- How to measure pA electrical currents? (Transimpedance amplifier with 10^9 gain)
- How to get rid of noise? (Mech. and acoustic vibration, 50Hz e-m fields)

On-site preparation:

- Quick and easy exchange of STM tips and samples
- Easy preparation of tips (cut wire) and sample surfaces (Scotch tape)

Data acquisition system:

- Interface between computer and instrument / user and sample
- How to process experimental data? Digitize physical quantities
- Calibration is fundamental!



Data Analysis: Tools and Methods

Free software tools for SPM data

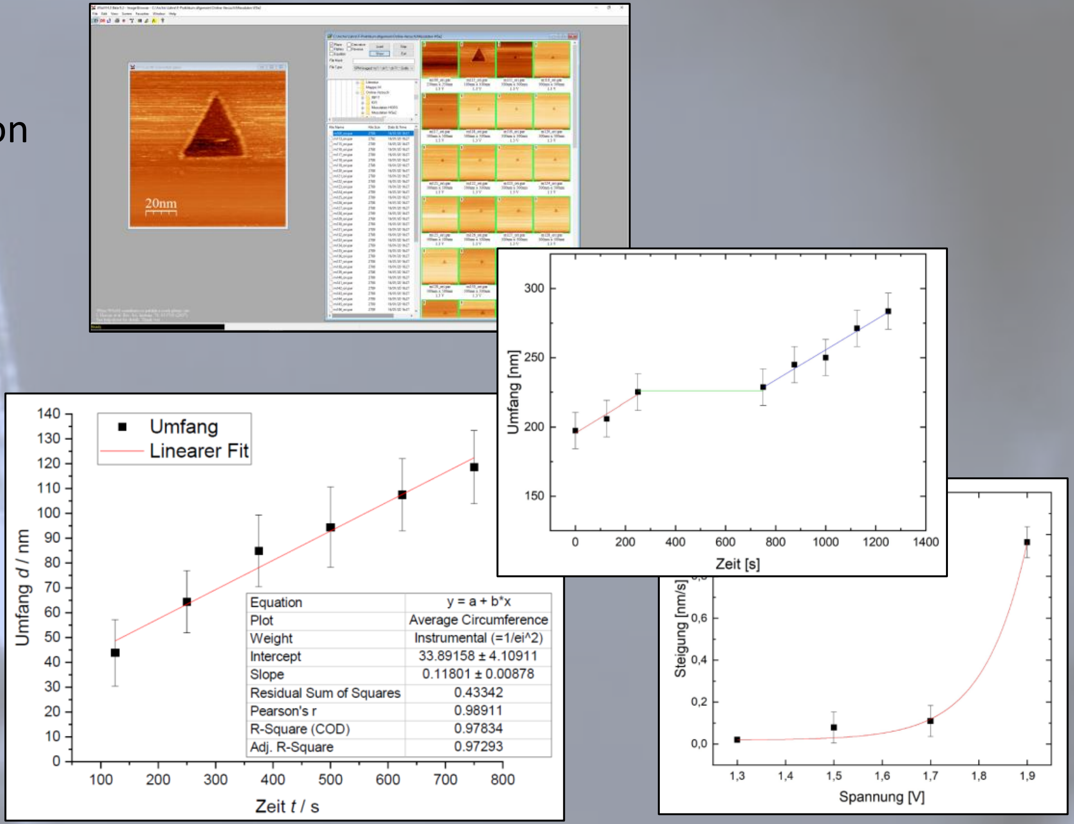
- WSxM and Gwyddion: Needed to read the STM data
- Possibility to export data and post-process, e.g. with Python

Real-space vs. reciprocal space

- What to use when
- Mind the error propagation!

On-the-fly data analysis

- Quick analysis during the experiment
- Adjust the experiment
- What and how much data is actually needed?
- Do not waste time collecting dump data, stay focused



Data Analysis: Teaching objectives

How to reduce error bars?

- Distance between two neighboring atoms
- Length of a chain of atoms
- Maximize data usage: Analysis in reciprocal space

How to find and deal with parasitic effects?

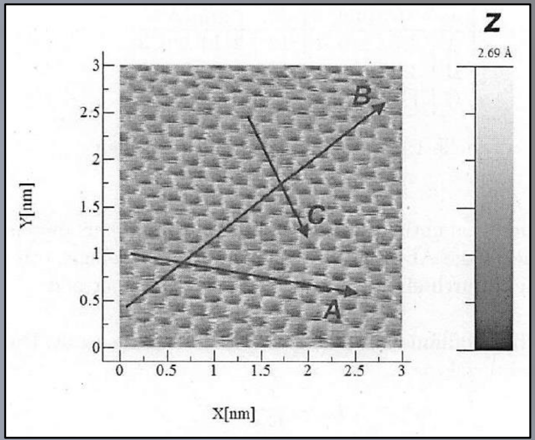
- Drift, creep, …: Use filters, or focus on sweet spots

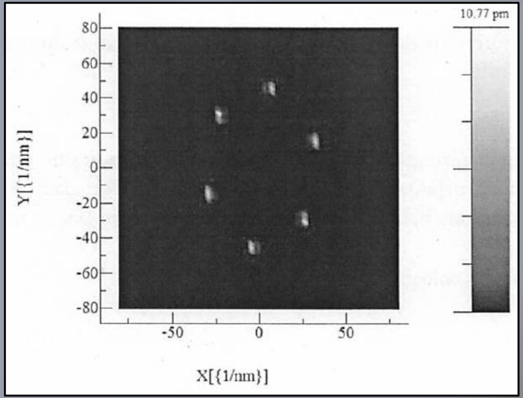
What periodic structure is real?

- Periodic noise, vibrations, …: Change scanning speed

What post-processing is necessary and valid?

- Plane and flatten filters: Benefits and limitations
- FFT filtering: Be careful!





Key Scientific Results

What do students learn from the experiment?

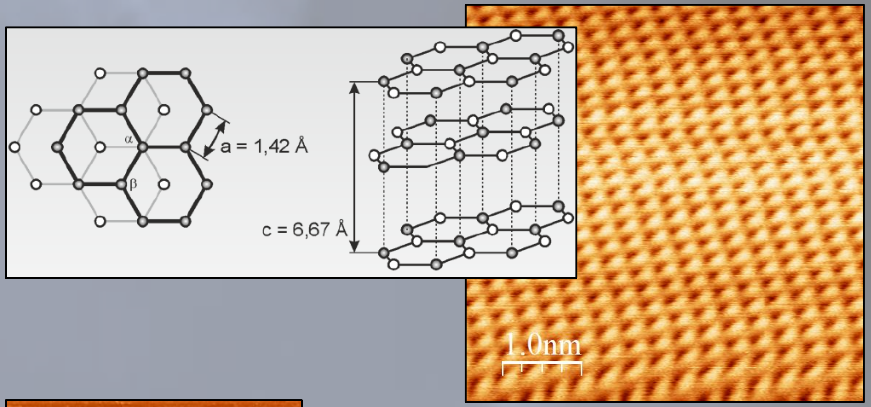
- HOPG
  - Two different types of atomic sites with different LDOS
  - Used as a standard for STM calibration
- WSe_2
  - Semiconductor material with triangular lattice geometry
  - STM tip used for nano-etching in water film
  - Etching speed grows with electric field

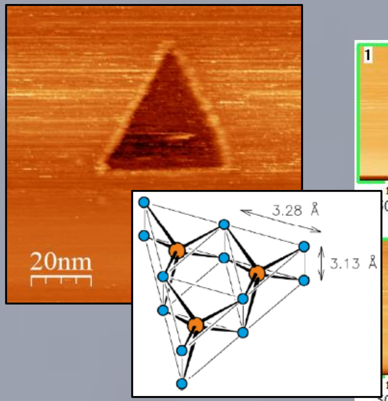
What is the emphasis of the experiment?

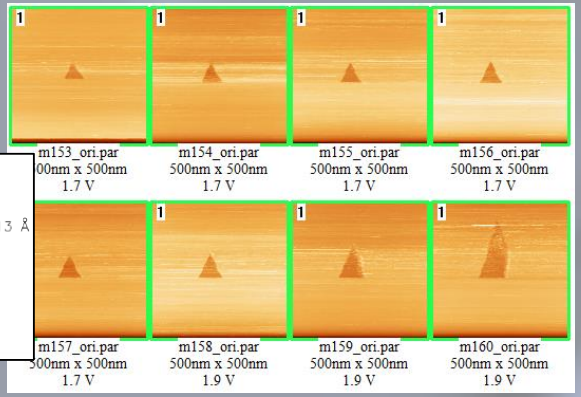
- Get in touch with STM, *see* and *interact* with the nano-world

Which skills do students learn?

- Perform a systematic STM study on the atomic-scale
- Fundamental STM data analysis methods in real and reciprocal space



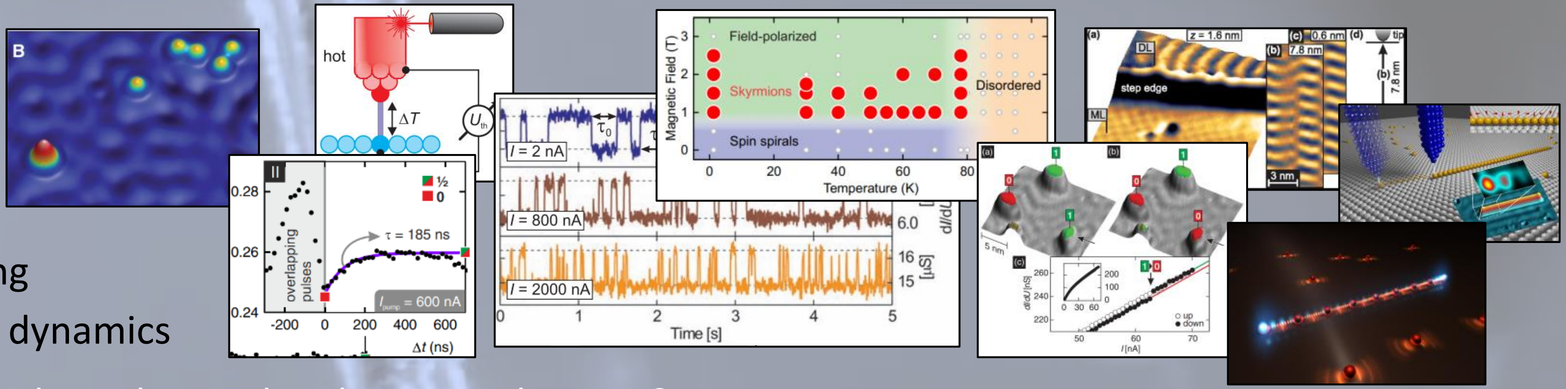




Link to Modern Research

What is the modern application of this technology/experimental method?

- STM is used for various atomic-scale experiments on surfaces:
  - Atomic magnets
  - Superconductivity
  - Magnetic skyrmions
  - Majorana physics
  - Magneto-Seebeck-tunneling
  - Electric/spin transport and dynamics



What skills do the students gain which can be used in the research group?

- Experimental skills (STM operation, design of dedicated experiments, data analysis, error treatment, …)

What is the difference between state-of-the-art equipment and the F-Praktikum setup?

- No UHV conditions, no low temperature, no magnetic field, *but* easy and quick access to the STM

Grade your Experiment

Complexity of aspects (1: high; 5: low)

- Theory / preparation: 1 2 **3** 4 5
- Setup / experimental: 1 2 3 **4** 5
- Data taking: 1 **2** 3 4 5
- Analysis: 1 **2** 3 4 5
- Protocol: 1 **2** 3 4 5

Scanning tunneling microscopy on layered materials

Thank you for your attention!

Jens Wiebe, Stefan Krause

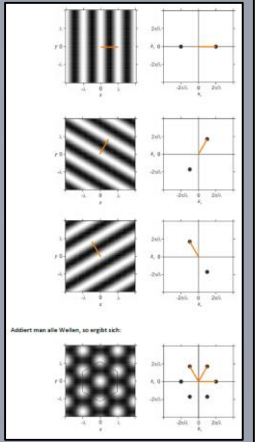
Course of the Experiment (1)

Supporting material:

- Theory of STM
- Technical aspects of building an STM
- Imaging techniques and artefacts
- Sample systems: HOPG & WSe_2
- Experimental setup
- Aims of the Experiment
- Tipps for writing the protocol
- Introduction to 2D Fourier transformation
- References and links
- Step-by-step instructions for the experiment

Allgemeine Hinweise für das Protokoll

- Der Theorie-Teil sollte nicht den größten Anteil des Protokolls bilden (nicht die Versuchsmappe kopieren!)
- Die einzelnen Experimente sollten motiviert werden (warum macht man welches Experiment? Welche Fragestellung wird damit bearbeitet?)
- Abbildungen brauchen eine Beschriftung (Figure caption), die beschreibt, was zu sehen ist
- Abbildungen sind in den Text einzubinden, sie dürfen inhaltlich nicht „losgelöst" und ohne Bezug zum Text stehen
- Abbildungen brauchen Pfeile, Markierungen, Beschriftungen u.ä., um verständlich zu sein
- RTM-Bilder brauchen nicht nur eine Längenskala (x,y), sondern auch eine Höhenskala (z, farbcodiert)
- RTM-Bilder sind im Kontrast anzupassen, damit zu sehen ist, was man zeigen will
- RTM-Bilder benötigen immer die Angabe der Tunnelparameter (U, I)
- Fehlerdiskussion ist unerlässlich
- Ablesefehler sind abzuschätzen und anzugeben
- Fit-Ergebnisse haben zu diskutierende Parameter mit Fehlergrenzen: Was bedeutet der Fit?
- Es ist klar zu dokumentieren, wie man von den unbehandelten Rohdaten zu den gezeigten Datensätzen kommt: Rohdaten -> Bearbeitung -> Ausmessen -> Ausgewertete Daten
- Wenn man immer wieder die gleiche Prozedur für mehrere Datensätze ausführt, dann sollte man diese einmal beispielhaft und detailliert zeigen
- Hypothesen sind mit Daten oder Skizzen zu belegen, um nachvollziehbar zu sein
- Experimentelle Ergebnisse brauchen eine Interpretation: z.B. erwartet / unerwartet?
- Resümee am Ende: Was hat man gelernt?



- Laden Sie den HOPG-Datensatz in das Analyseprogramm. Wenn nötig, aktivieren Sie den „plane"- bzw. „flatten"-Filter. Was würden Sie erwarten zu sehen? Was sehen Sie tatsächlich?
- Beachten Sie, dass nicht alle Oberflächenatome auf HOPG gleichwertig sind. Es gibt α- und β-Atome (s.o.). Welche Atome sehen Sie in dem Rastertunnelbild als helle Erhebungen? Und auf welchem Gitter liegen sie?
- Welchen Abstand erwarten Sie für die α-Atome untereinander, errechnet aus den bekannten Gitterparametern?
- Erstellen Sie ein Linienprofil entlang einer atomaren Reihe, um den Abstand zwischen den hellen Atomen (Periodizität) zu bestimmen. Überlegen Sie, wie Sie hierfür am besten vorgehen: Direkt den Abstand zweier Atome messen, oder die Länge einer Kette von ca. zehn Atomen messen und durch die Kettenlänge teilen? Welche der beiden Methoden führt zu einem geringeren Messfehler und ist daher die genauere Methode?
- Vergleichen Sie die gemessene Länge mit der erwarteten Länge und ermitteln so den Korrekturfaktor f_{corr}.
- Analysieren Sie atomare Reihen entlang der drei beobachteten Richtungen. Sind die gemessenen Abstände der Atome immer die gleichen, egal in welcher Richtung die Daten analysiert wurden? Entspricht dies den Erwartungen für ein HOPG-Gitter?
- Finden Sie mögliche Erklärungen für Abweichungen der Kalibrierungen entlang der unterschiedlichen Richtungen. Stichworte: Piezo-Creep, Thermischer Drift, ….

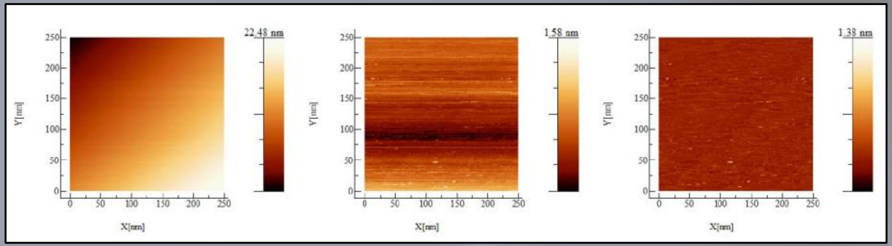
Diese Kalibrierung basiert auf der Analyse einzelner Linienprofile, die Sie aus den Daten der Messung gewinnen. Eine Methode, bei der die Gesamtheit aller gemessenen Datenpunkte genutzt wird, um periodische Anordnungen von Atomen zu analysieren, ist die Fourier-Transformation der Daten.

- Wenn Sie das perfekte HOPG-Gitter einer Fourier-Transformation unterziehen würden, was würden Sie als Ergebnis erwarten? Diskutieren Sie Richtungen und Längen der erwarteten Fourier-Komponenten.
- Führen Sie eine Fourier-Transformation der Daten in den reziproken Raum durch. Was beobachten Sie? Entspricht die Beobachtung der Erwartung?
- Analysieren Sie die Fourier-Spots, die zum beobachteten Gitter im Real-Raum gehören, und bestimmen Sie die Länge der k-Vektoren. Welche Richtungen im Realraum können den k-Vektoren im reziproken Raum zugeordnet werden?
- Berechnen Sie aus den k-Vektoren die entsprechenden Abstände im Realraum und kalibrieren hiermit das Rastertunnelmikroskop auf eine zweite Art (zusätzlich zur Kalibrierung im Realraum).

Course of the Experiment (2)

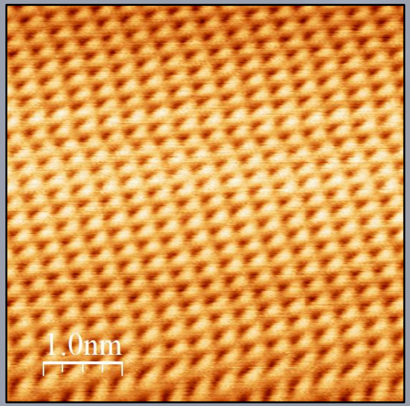
First contact with STM:

- In-depth discussion based on supporting material
- Introduction to Experimental Setup and Data Acquisition System
- Preparation of Tip and Sample
- First STM image: Understanding raw data, checking sample quality
- Postprocessing: Plane and Flatten filters and their limits



Calibration on HOPG:

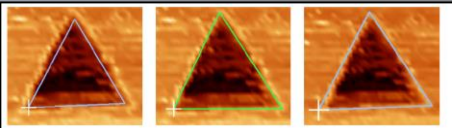
- First atomic-scale details: Noise, atoms, step edges, adsorbates, …
- Achieve atomic resolution on HOPG for calibration
- Check the data: Good enough for the calibration?
  - What is needed in real space?
  - What is needed in reciprocal space?



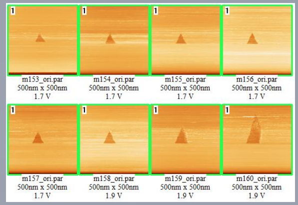
Course of the Experiment (3)

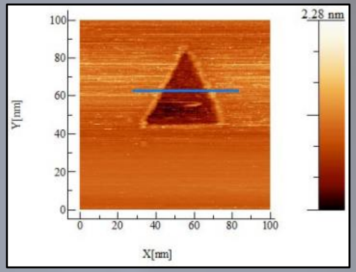
Nanostructuring on WSe_2:

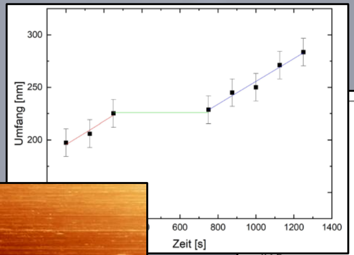
- Generate overview image to check sample quality
- Apply voltage pulses to the tunnel contact, change parameters until something happens
- Image the resulting hole, discuss shape and depth
- Discuss physics: electro-chemical etching, check with paper references
- Take image series at constant bias voltage, observe the growth of hole
- Quick-and-dirty data analysis: check perimeter as function of time (image #)
- Is the growth linear with time? Get growth speed from linear regression
- Determine growth speed as a function of bias voltage, and discuss
- Is the hole only growing when it is imaged? Develop an experiment to find out
- Use the new knowledge to „write something“ into the surface, and discuss results

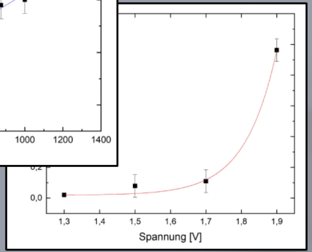


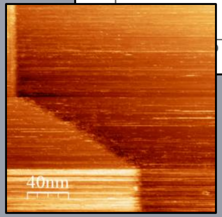
7. Darstellung des Vorgehens bei der Vermessung des Umfangs des Loches. Vermessung (1) innerhalb der Lochkanten (Links), (2) auf den Lochkanten (Mitte) und (3) außerhalb der Lochkanten (Rechts).











Course of the Experiment (4)

Stimulating additives

- Discussion about everyday's classical physics (transformer, 50 Hz noise, STM tip as antenna, …)
- Discussion about STM related experiments/physics and current STM projects in the group
- Lab Tour to see UHV chambers, Cryostats, Magnets, PhD students…

Data analysis and protocol

- In-depth analysis of the the data (~50 STM images, linear regression, Fourier transformation, error propagation, …)
- Create meaningful graphs, including highlighting with arrows, circles, letters, …
- Write protocol like a scientific paper
  - Present the chain of experiments
  - Provide motivation for each experiment
  - Experimental idea to answer specific questions
  - Results and discussion

Seminar presentation

Feedback from the Students

Supporting material

- Well balanced between theory, experimental setup and tasks for the experiment
- Very helpful general tips for writing the protocol
- Good to have the reference papers as part of the material
- Chance to get familiar with the analysis software at home

Supervision of the experiment

- Very useful to have the supervisor present during the whole experiment
- Fruitful discussions (from simple details to general questions)
- Good connections to the activities of the group

Protocol

- Helpful discussion right after the experiment on how to prepare the protocol
- Clear and timely feedback

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