Exercise M1
Characterization of nanostructures with atomic force microscopy

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Requirements

Physical background of AFM: Students should understand the nature of the piezoelectric effect, distinguish between direct and inverse piezoelectric effects, know which materials exhibit piezoelectric properties, and comprehend how piezo elements are used in AFM for precise positioning of the probe relative to the sample in three dimensions. They should understand the behavior of a driven harmonic oscillator, including the dependence of amplitude and phase on excitation frequency, and the significance of parameters such as quality factor (Q), resonance width, and damping in the context of dynamic AFM operation (non-contact and intermittent contact modes). Special attention should be paid to understanding the nature of the forces acting between the probe tip and the sample surface — students should know the attractive nature of van der Waals interactions and the repulsive forces resulting from the overlap of electron orbitals (Pauli exclusion principle). They should understand that the net interaction as a function of tip–sample distance can be graphically represented by the Lennard-Jones potential, which illustrates the presence of an energy minimum and regions of attractive and repulsive force dominance. Students should also be aware of other interaction types relevant under real conditions, including: capillary forces, electrostatic forces, chemical forces, magnetic forces.

Operation of the AFM: Students should understand the force detection mechanism using an optical system composed of a laser and a quadrant photodetector. They must be able to explain how cantilever deflections are converted into electrical signals and interpreted in different AFM operating modes. They should understand the function of the piezoelectric scanner (typically a tube scanner) that enables movement in X, Y, and Z directions, including non-linearities, hysteresis, and drift issues that can lead to topographical distortion. Understanding the feedback loop, which maintains a selected parameter (e.g., contact force, oscillation amplitude) at a constant value, is essential. Students should know how feedback settings affect stability and image quality and how incorrect settings may cause image artifacts. They should also understand the properties of AFM probes — how tip radius affects image resolution, the importance of cantilever stiffness depending on the mode, and the risks of sample damage or image distortion due to improper probe selection.

Operating modes: Students must be able to explain the working principles of AFM in: contact mode (continuous contact with the surface), intermittent contact mode (tapping mode, periodic contact), non-contact mode (probe oscillates near the surface without touching it). They should understand the advantages and limitations of each mode, particularly in terms of image quality, tip–sample interaction, and potential sample damage. Additionally, students should be prepared to work with advanced techniques used during the exercise: conductive AFM (C-AFM) – simultaneous topography and local conductivity mapping at the contact point, magnetic force microscopy (MFM) – detection of resonance disturbances in a magnetic probe caused by interaction with local magnetic fields.

Objective of the exercise

The aim of this exercise is to acquire practical skills in operating an atomic force microscope (AFM) and to understand the principles underlying its operation. The task involves using AFM to examine nanostructured surfaces through basic topographical imaging and selected advanced modes: Conductive AFM (C-AFM) for studying local conductivity, and Magnetic Force Microscopy (MFM) for imaging magnetic domain structures. Students will learn how to perform measurements, analyze image quality, and interpret data in relation to physical properties of the materials under study.

Theoretical introduction

Atomic Force Microscopy (AFM) is one of the fundamental techniques within the broader class of Scanning Probe Microscopy (SPM), alongside Scanning Tunneling Microscopy (STM). Unlike STM, which requires conductive surfaces and relies on the quantum tunneling of electrons, AFM can be applied to the investigation of both conductive and non-conductive surfaces.

Due to its exceptionally high spatial resolution, AFM has found widespread application across numerous scientific and industrial fields. In materials science, it enables detailed analysis of surface topography and mechanical properties (e.g., stiffness, adhesion) of various substrates, thin films, and nanostructures. In electronics and nanotechnology, it is employed for the characterization of functional materials and micro- and nanoscale structures, as well as for localized measurements of electrical conductivity and ferroelectric or piezoelectric properties. In the fields of biology and medicine, AFM is used to image biological structures (such as cells, membranes, proteins, and nucleic acids) under conditions close to their native environment, facilitating the analysis of morphology, elasticity, and molecular interactions.

The versatility of AFM stems not only from its ability to probe a wide range of materials but also from its adaptability to different measurement environments (vacuum, air, liquid) and the broad spectrum of operational modes available, each tailored to specific physical or chemical properties of the investigated samples.

1. Operating principle of the AFM microscope

The fundamental operating principle of an Atomic Force Microscope (AFM) is based on the measurement of interaction forces between an extremely sharp probe and the surface of the sample under investigation. The probe is mounted on a thin, flexible cantilever, which bends or twists in response to interatomic forces as the probe approaches the sample. The typical radius of curvature of the probe tip ranges from a few to several tens of nanometres, allowing for high spatial resolution.

During imaging, the sample is scanned in the x–y plane relative to the probe (or vice versa), while the interaction forces that induce cantilever deflection are continuously monitored. Cantilever deflection is detected by a laser beam reflected off the back side of the cantilever and directed onto a quadrant photodiode. Even minute changes in the deflection angle cause a shift in the laser spot position on the detector, enabling highly sensitive measurements of the force acting between the probe and the sample. In order to maintain a constant interaction force (or another control parameter, such as oscillation amplitude), the AFM system employs a feedback loop that continuously adjusts the vertical position of the scanner (Z-axis). This adjustment compensates for surface height variations and allows the selected control parameter to remain stable. The changes in scanner position are recorded and used to reconstruct the surface topography.

Precise positioning of the probe (or the sample) in all three spatial dimensions is achieved using a piezoelectric scanner. The use of piezoelectric materials allows for extremely fine movements, with positioning accuracy on the order of tenths of a nanometre. Such precision enables atomic-scale surface mapping, depending on the selected operating mode, sample properties, and the measurement environment.

2. Forces in AFM tip–sample interactions

During AFM measurements, various physical forces arise between the probe tip and the sample surface. The nature and magnitude of these forces depend on the tip–sample distance, environmental conditions, and the material properties of both the probe and the sample. The main types of interactions are outlined below:

- Van der Waals forces – these are attractive intermolecular forces that dominate at distances ranging from a fraction of a nanometre to several tens of nanometres, depending on the geometry of the tip–sample system and the nature of the interacting materials. In typical AFM configurations, they are relevant within the range of approximately 0.2–10 nm. Van der Waals forces consist of three

components: induced dipole interactions, dispersion forces (London forces), and orientation forces. They act between electrically neutral molecules and are always present in atomic force microscopy.

- Repulsive (quantum-mechanical) forces – these arise at very short distances, when the electron orbitals of atoms in the tip and sample begin to overlap. According to the Pauli exclusion principle, two fermions cannot occupy the same quantum state, resulting in a rapidly increasing repulsive force as the overlap intensifies.
- Capillary forces – these occur during ambient measurements, when a thin water layer (moisture film) forms on the sample surface. A water meniscus may develop between the tip and the sample, generating an attractive force. This force can become dominant under conditions of elevated humidity.
- Chemical bonding forces – these emerge when chemical bonds are formed between atoms of the probe and the sample. These are short-range interactions, significantly stronger than van der Waals forces, and occur only when the tip is in near-direct contact with the sample.
- Electrostatic forces – these result from the presence of electric charges on the probe or sample surface. They may arise from natural surface potential differences, ionic contamination, or triboelectric charging (e.g., due to contact or friction between materials with different triboelectric properties).
- Magnetic forces – these are relevant only if the probe or the sample exhibits magnetic properties (e.g., ferromagnetism). In such cases, additional forces arise from magnetic dipole interactions or external magnetic fields.

The force–distance relationship can be illustrated using the Lennard-Jones potential, which shows that attractive van der Waals forces dominate at longer distances, while strong repulsive forces emerge at very short distances due to orbital overlap. At the equilibrium point, these forces cancel out, corresponding to the system's minimum potential energy.

In practice, the total interaction potential between the tip and the sample is a complex superposition of all the aforementioned forces, which contribute in a nonlinear and inseparable manner. As a result, AFM cannot experimentally isolate individual force components — only the net force acting between the probe and the sample can be analysed.

3. AFM operating modes

Atomic Force Microscopy enables surface topography imaging using three primary operating modes:

a) contact mode,
b) tapping (intermittent contact or semicontact) mode,
c) non-contact mode.

Each of these modes is based on interatomic interactions between the probe and the sample surface. When the probe is positioned far from the surface, no interaction occurs. As the tip approaches the sample, attractive forces (mainly van der Waals) emerge, followed by a sharp increase in repulsive forces due to orbital overlap. By analyzing the shape of the potential energy curve as a function of the tip–sample distance (i.e., interatomic distance), one can identify the characteristic regimes in which different AFM modes operate (Figure 1).

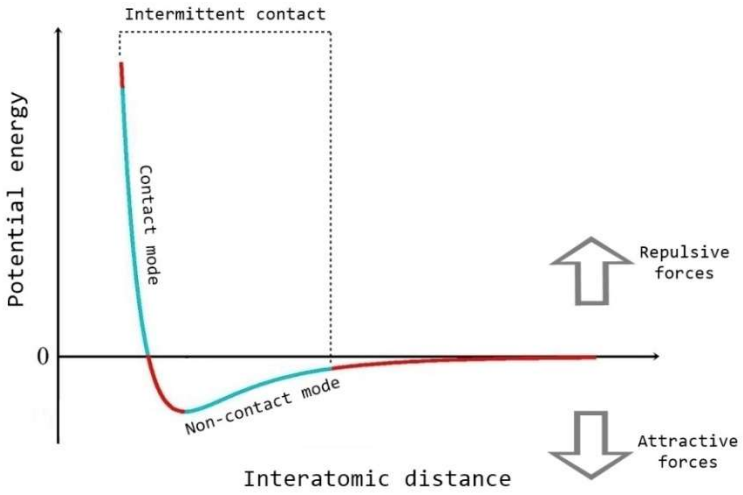


Figure 1. Potential energy as a function of interatomic distance, indicating the operating regimes of AFM modes.

Contact mode

In contact mode, the probe remains in continuous, direct contact with the sample surface. The dominant force is repulsive, arising from short-range quantum-mechanical interactions. The cantilever deflection is measured by monitoring the position of a reflected laser beam, and the interaction force is described by Hooke's law:

$$F = -k \cdot D \tag{1}$$

where: F – tip–sample interaction force [N], k – cantilever spring constant [N/m], D – cantilever deflection [m]

Contact mode provides high resolution and is primarily used for imaging hard materials such as metals, minerals, and composites. It also enables mechanical property analysis of surfaces. However, due to the continuous contact, it may cause damage to soft or delicate samples, especially when using stiffer cantilevers.

Tapping mode (Intermittent contact / Semicontact)

In tapping mode, the cantilever is driven to oscillate near its resonance frequency (typically tens to hundreds of kHz). The tip periodically touches the surface, but only briefly during each oscillation cycle. The mechanical contact occurs within the repulsive force regime, while attractive forces (e.g., van der Waals interactions) act during the approach phase, before the tip physically touches the surface. Changes in the oscillation amplitude due to tip–surface interaction are detected and compensated by a feedback loop, which adjusts the vertical position of the piezoelectric scanner.

Tapping mode is less invasive than contact mode and is suitable for probing mechanically sensitive materials while maintaining high image quality. Due to the reduced lateral (frictional) forces during scanning, tapping mode is currently the most widely used AFM operation mode.

Non-contact mode

In non-contact mode, the tip oscillates a few nanometres above the surface, avoiding physical contact. Detection is based on measuring shifts in amplitude or resonance frequency resulting from long-range attractive forces (mainly van der Waals). A feedback loop maintains the tip at a constant height, enabling surface topography mapping.

This mode is particularly suitable for imaging delicate, easily damaged structures—such as biological samples, polymers, or thin films—offering minimal mechanical interference with the sample.

Each AFM mode utilizes different ranges of tip–sample interactions, allowing for optimal adaptation to the nature of the surface and the measurement objectives. Contact mode operates in the repulsive force regime, non-contact mode in the attractive regime, while tapping mode spans both, offering a balance between imaging resolution and measurement delicacy. The overall tip–sample interaction potential—typically described by the Lennard-Jones curve—forms the theoretical basis for selecting the appropriate AFM mode.

4. Practical relevance and measurement preparation

During the laboratory session, students will gain hands-on experience with the operation of an atomic force microscope (AFM), including probe preparation, measurement parameter setup, sample scanning, and interpretation of acquired topography images. A solid understanding of the feedback loop mechanism and the appropriate selection of the imaging mode is essential for accurate data acquisition.

The primary objective of the exercise is to develop practical skills related to the operation of the AFM, as well as to understand the key factors that influence image quality.

Course of the exercise

The AFM laboratory exercise is conducted over a four-day cycle, combining theoretical instruction with practical training. The aim is to familiarize students with the operation of the atomic force microscope, its control system, and the acquisition and analysis of measurement data using the **NT-MDT Nano Solver** instrument.

The first day begins with a discussion of the theoretical background listed in the Requirements section. Topics covered include:

- operating principles of the AFM;
- nature of tip–sample interactions;
- description of AFM operating modes (contact, intermittent contact, non-contact);
- factors affecting image quality;
- the role of the Lennard-Jones potential in interpreting force-distance relationships and operating regimes.

This is followed by a practical introduction to the NT-MDT Nano Solver system, including:

- overview of microscope components;
- mounting and alignment of cantilever with probe;
- calibration procedures for the scanning system;
- setting operational parameters (resonance frequency, drive amplitude, scan range);
- guidelines for selecting scan regions (surface cleanliness, flatness, stability).

Students observe real-time imaging of a sample surface, during which:

- the feedback loop and its influence on image stability are explained;
- the impact of scan speed and resolution is demonstrated;
- adjustment strategies for varying sample characteristics are discussed.

Following the demonstration and practical setup procedure, each student independently acquires topographic data on an epitaxial graphene sample in intermittent contact mode, learning to control imaging parameters and interpret results. The first measurement day concludes with an introduction to the AFM image processing and analysis software, which is used to visualize, process, and interpret acquired data.

On the remaining days, students perform measurements using advanced AFM modes tailored to their selected task. They may choose between two specialized exercises:

Exercise 1: Characterization of electrode materials using conductive AFM (C-AFM)

The session begins with topography imaging in intermittent contact mode to identify suitable regions of interest. Students then switch to contact mode using a conductive probe to measure electrical properties.

Objectives:

- acquire surface topography and correlate it with conductivity;
- generate spatially resolved current maps;
- record local current–voltage (I–V) characteristics to analyze charge transport behavior.

Students learn how to:

- establish electrical contact between the sample and the system;
- select appropriate conductive probes;
- interpret transport mechanisms and identify electrical inhomogeneities.

Exercise 2: Magnetic domain imaging using magnetic force microscopy (MFM)

This task involves magnetic characterization of data storage media such as VHS tape and HDDs of varying capacities. Students use magnetically coated probes and perform dual-pass MFM scanning.

Objectives:

- detect and image magnetic domain structures;
- compare domain sizes and densities across different storage technologies;
- understand the influence of magnetic tip-sample interaction range.

Students analyze:

- differences in domain organization between VHS and high-density HDD media;
- limitations of MFM resolution and the influence of tip magnetization;
- best practices for setting lift height and minimizing electrostatic artifacts.

Report guidelines

Each student is required to prepare a detailed report documenting the completed laboratory exercise. The report must include the following sections:

1. **Objective and summary**
   A concise description of the objective of the selected exercise and a summary of the tasks performed. This section should specify the AFM operating mode used and the physical quantity analyzed (e.g., topography, local conductivity, magnetic interactions).
2. **Theoretical background**
   A brief introduction highlighting the measurement technique used and the type of sample investigated. The focus should be on the principles and mode of AFM operation relevant to the chosen exercise (C-AFM or MFM).
3. **Measurement method, instrumentation, and sample description**
   A comprehensive description of how the measurements were performed, including:
   - operating mode and measurement environment;
   - type of AFM probe used (e.g., conductive or magnetic, stiffness, coating);
   - step-by-step measurement procedure and parameter settings;
   - sample type and preparation method.
4. **Results and analysis**
   Presentation and interpretation of the acquired data:
   - include representative AFM images and, if applicable, current maps or MFM phase images;
   - analyze key features observed in the data;
   - identify and discuss any visible image artifacts (e.g., noise, drift, tip convolution);
   - evaluate the measurement reliability and data quality.

   Not all acquired images need to be included — only those that best illustrate the measurement results. Reports should demonstrate the student's understanding of both the measurement technique and the physical phenomena being investigated.
5. **Conclusions**
   Summarize the main findings, discuss the effectiveness of the measurement method, and reflect on the experimental limitations or potential improvements.

Evaluation criteria

The primary criterion for evaluation is the quality and completeness of the obtained results, particularly the clarity and resolution of AFM images and the depth of data analysis. The report will be assessed based on the following aspects:

- appropriate selection and presentation of representative data;
- detailed description of the experimental procedure and parameters;
- proper use of terminology and physical interpretation;
- identification and explanation of image artifacts and potential sources of error;
- clarity, conciseness, and structure of the written report.

Literature and supporting materials

1. P. Eaton, P. West, *Atomic Force Microscopy*, Oxford University Press, 2010.
   A comprehensive guide covering both theoretical and practical aspects of AFM measurements.
   Online access: https://academic.oup.com/book/7224
2. M.L. Mironov, *Fundamentals of Scanning Probe Microscopy*, NT-MDT, 2004.
   A foundational manual presenting the principles of operation for SPM techniques, with practical focus on NT-MDT instruments.
3. E. Meyer, H.J. Hug, R. Bennewitz, *Scanning Probe Microscopy: The Lab on a Tip*, Springer, 2004.
   Academic-level reference on scanning probe techniques including AFM and MFM.
   Online access: https://link.springer.com/book/10.1007/978-3-662-09801-1