WITec
focus innovations

Nanoscale Surface
Characterization

Atomic Force Microscopes

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WITec

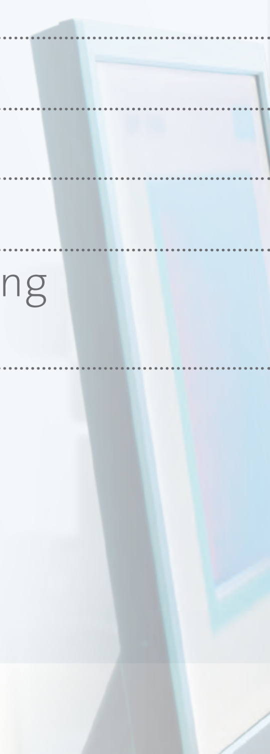
Atomic Force Microscopes

Nanoscale Surface Characterization

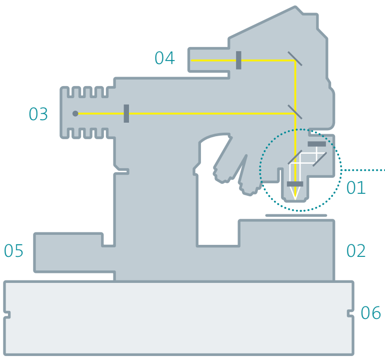
The WITec Atomic Force Microscope (AFM) module integrated with a research-grade optical microscope provides superior optical access, easy cantilever tip alignment and high-resolution sample survey. The WITec AFM objective provides a direct view of both sample and cantilever for straightforward and precise AFM tip positioning. All WITec Atomic Force Microscopes are designed for combination with other imaging techniques such as Raman spectroscopy, Scanning Near-field Optical Microscopy (SNOM), luminescence microscopy, polarization analysis and light-field/dark-field illumination. The user can change between imaging methods by simply rotating the (optionally motorized) objective turret.

Benefits

- Surface characterization on the nanometer scale
- Non-destructive imaging
- Optical and Atomic Force Microscope combination
- Convenient sample access from any direction
- Minimal, if any, sample preparation
- Ease of use in air and liquids
- Combinable with confocal Raman imaging and Scanning Near-field Optical Microscopy (SNOM)
- TrueScan™ controlled piezo-driven scanning stages with capacitive feedback loops:
  - 30 x 30 x 10 μm^3
  - 100 x 100 x 20 μm^3
  - 200 x 200 x 20 μm^3

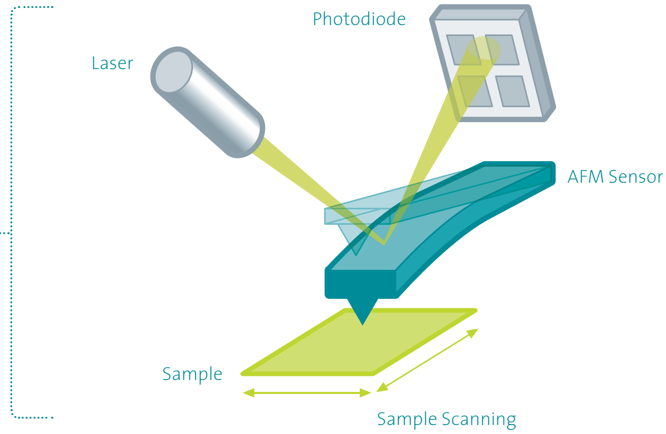


The AFM Microscope



01 Objectives
02 Piezo-driven scanning stage
03 Koehler white-light illumination
04 Color video camera
05 Z-stage for focusing
06 Active vibration isolation table

Beam Path



Working Principle

Atomic Force Microscopy (AFM) traces the surface contour of living and non-living samples by analyzing the interaction forces between the sample's surface and a sharp cantilever tip. The sample is scanned under the tip using a piezo-driven scanning stage and its topography is displayed as an image with up to atomic resolution.

The topography of a sample can be acquired while simultaneously extracting other properties such as the sample's adhesion, stiffness, viscosity, electrostatic potential and more.

Technology

Cantilever

- Inertial drive cantilever mount for AFM sensor positioning
- All commercially available AFM cantilever tips can be used

Resolution

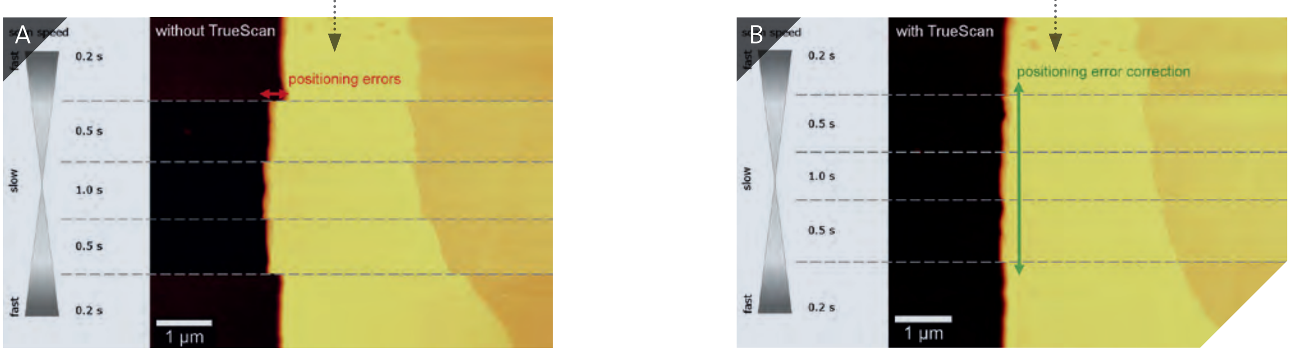
- Lateral resolution: Tip radius-dependent, down to 1 nm
- Depth resolution: Down to atomic scale on silicon, mica, HOPG etc.

TrueScan™

The piezo-driven scanning stage moves the sample forward and reverse during the measurement. The scanning stage's inertia leads to lag errors. This effect increases with scanning speed. While the incorporated capacitive feedback system determines the actual position of the stage, TrueScan™ can resolve the discrepancy between its actual and intended position, allowing high-resolution lag-free AFM imaging at high scanning speeds.

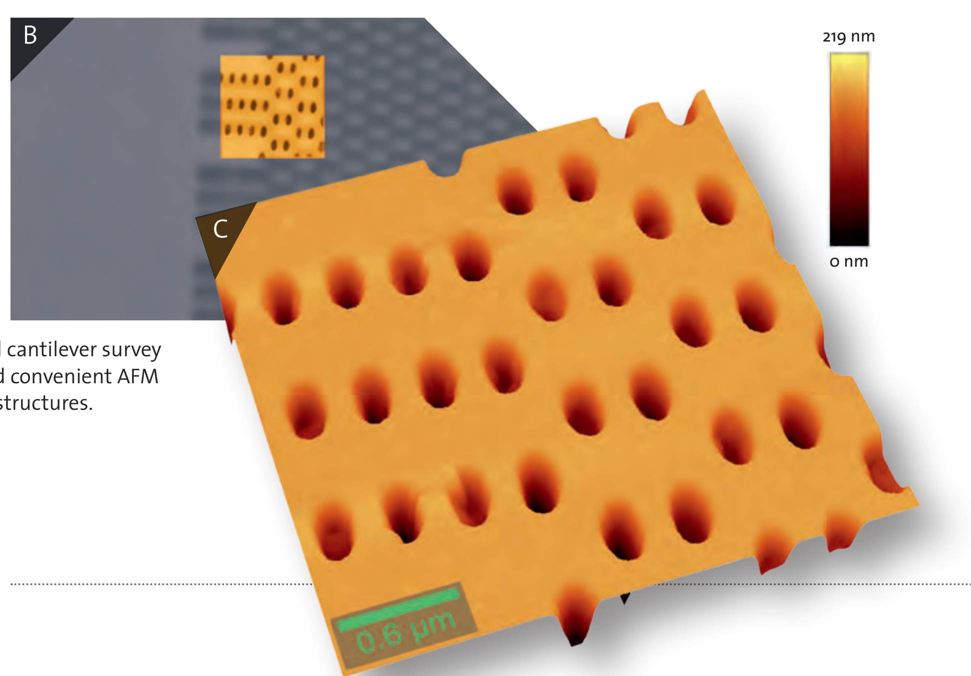
Computer Interface

- alphaControl: Digital controller for WITec microscope systems
- WITec Control software for instrument and measurement control
- Data evaluation and processing software included



Scan of the straight edge of a sample at speeds between 0.2 s and 1 s per scanning step. **(A)** Without TrueScan™ an increase in scanning speed leads to positioning errors. **(B)** TrueScan™ corrects the lag errors.

Simultaneous Cantilever and Sample Viewing for Easy Determination of the Measurement Position



The WITec AFM objective allows simultaneous sample and cantilever survey from above with high resolution. It facilitates accurate and convenient AFM tip alignment and positioning on even very small sample structures.
(A) Simultaneous cantilever and sample viewing.
(B) Overlay of the optical image with the AFM image (AFM image: AC mode, scan range: 3 x 3 µm²).
(C) 3D representation of the AFM image from B.

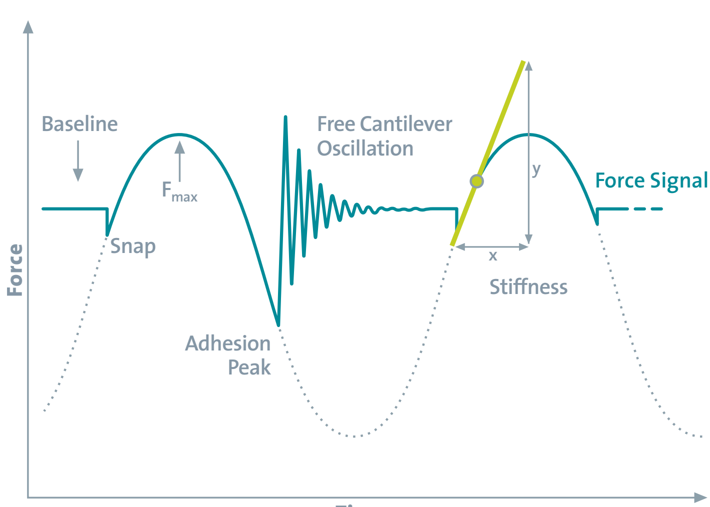
AFM Modes with DPFM

- **Contact Mode**
  The cantilever tip is kept in contact with the sample surface at a constant bending (force) while it is scanned, revealing the topography of the sample. The bending of the cantilever is monitored with a quadrant photodiode beam-deflection system. In this imaging mode large lateral forces may disturb loosely-bound particles.
- **AC Mode**
  In this approach, also called intermittent contact mode, the cantilever oscillates at its resonance frequency and is periodically in contact with the sample. Thus the technique is particularly well suited for delicate samples. When the tip comes close to the surface, sample-tip interaction causes forces to act on the cantilever which alter the phase of its oscillation. This phase shift can be recorded and depicted as a phase image.
- **Lift Mode™**
  This mode can be applied in combination with contact mode or AC mode. First, the sample is scanned in an imaging mode to trace the surface. Then Lift Mode™ is used to scan the sample again with a certain z-offset following the previously-recorded topography. Lift Mode™ is used in combination with EFM, MFM or Kelvin Probe Microscopy to reveal sample properties other than topography.
- **Kelvin Probe Microscopy**
  In this imaging mode the conductive AFM tip acts as a vibrating capacitor and measures in Lift Mode™ the differences in surface potential between the tip and the sample.
- **Digital Pulsed Force Mode™ (DPFM)**
  DPFM allows for the simultaneous acquisition of topography, adhesion, stiffness and other physical properties of the sample. For details see the next section.
- **Magnetic Force Microscopy (MFM)**
  This imaging mode uses a magnetic cantilever scanned in Lift Mode™ over the sample. During the z-offset scan, magnetic properties of the sample are revealed due to the magnetic interaction between the tip and the sample.
- **Electrostatic Force Microscopy (EFM)**
  In this imaging mode electrical properties of a sample surface are imaged. A DC voltage is applied between the tip and sample surface while scanning in Lift Mode™. When the tip is scanned at a z-offset over the sample, electrostatic charges lead to deflections of the cantilever resulting in an electrical properties map of the sample.
- **Nanomanipulation / Lithography**
  The lithography package "Da Vinci" enables the patterning of material surfaces on the nanometer-scale using tip-sample interaction forces.
- **Lateral Force Microscopy (LFM)**
  This imaging mode uses the twisting of the cantilever while scanning in contact mode to reveal surface friction characteristics.

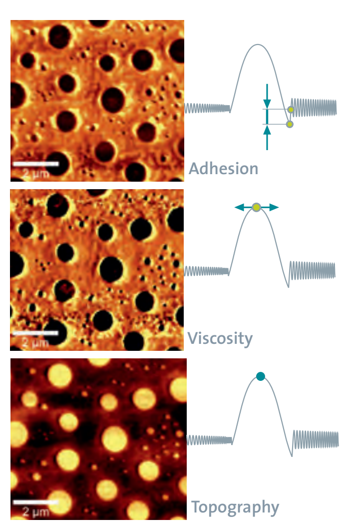
Digital Pulsed Force Mode (DPFM)

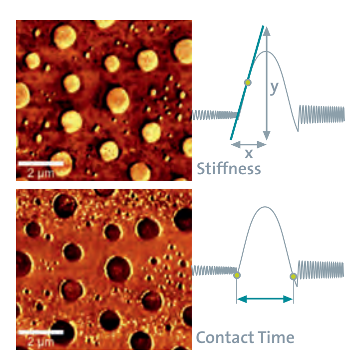
Pulsed Force Mode (PFM) is a non-resonant, intermittent contact mode for Atomic Force Microscopy that allows the characterization of material properties such as adhesion, stiffness and viscosity along with the sample topography. Additionally, lateral forces are virtually eliminated. Therefore high-resolution mapping of delicate samples in air and liquids is easily achievable while maintaining a scanning speed comparable to contact mode AFM. In contrast to most other intermittent contact techniques, the perpendicular forces on the sample (introduced by the AFM tip) are controlled by the feedback loop. The PFM electronics induce a sinusoidal modulation of the z-piezo of the AFM with an amplitude of 10-500 nm at a user-selectable frequency of between 100 Hz and 2 kHz: far below the resonant frequency of the cantilever. A complete force-distance cycle is carried out at this rate, resulting in the force signal as shown in the figure below.

Imaging of Surface Properties with DPFM



Pulsed Force Mode





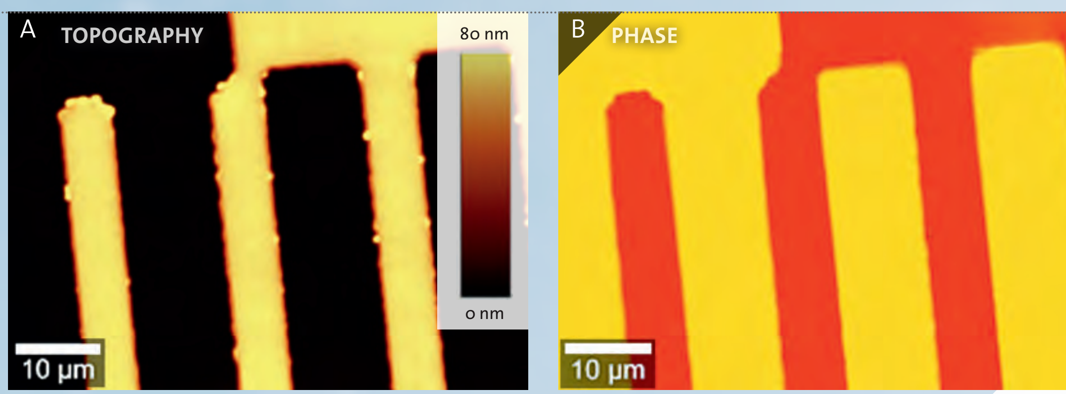
Digital Pulsed Force Mode (DPFM)

A selection of simultaneously-obtained images of a Ethyl-Hexyl-Acrylate/Polystyrene blend (EHA-PS) spin-coated onto a glass substrate. The corresponding curves show the analyzed Pulsed Force Mode properties. Dark areas correspond to low values. Scan range: 10 x 10 µm².

Applications

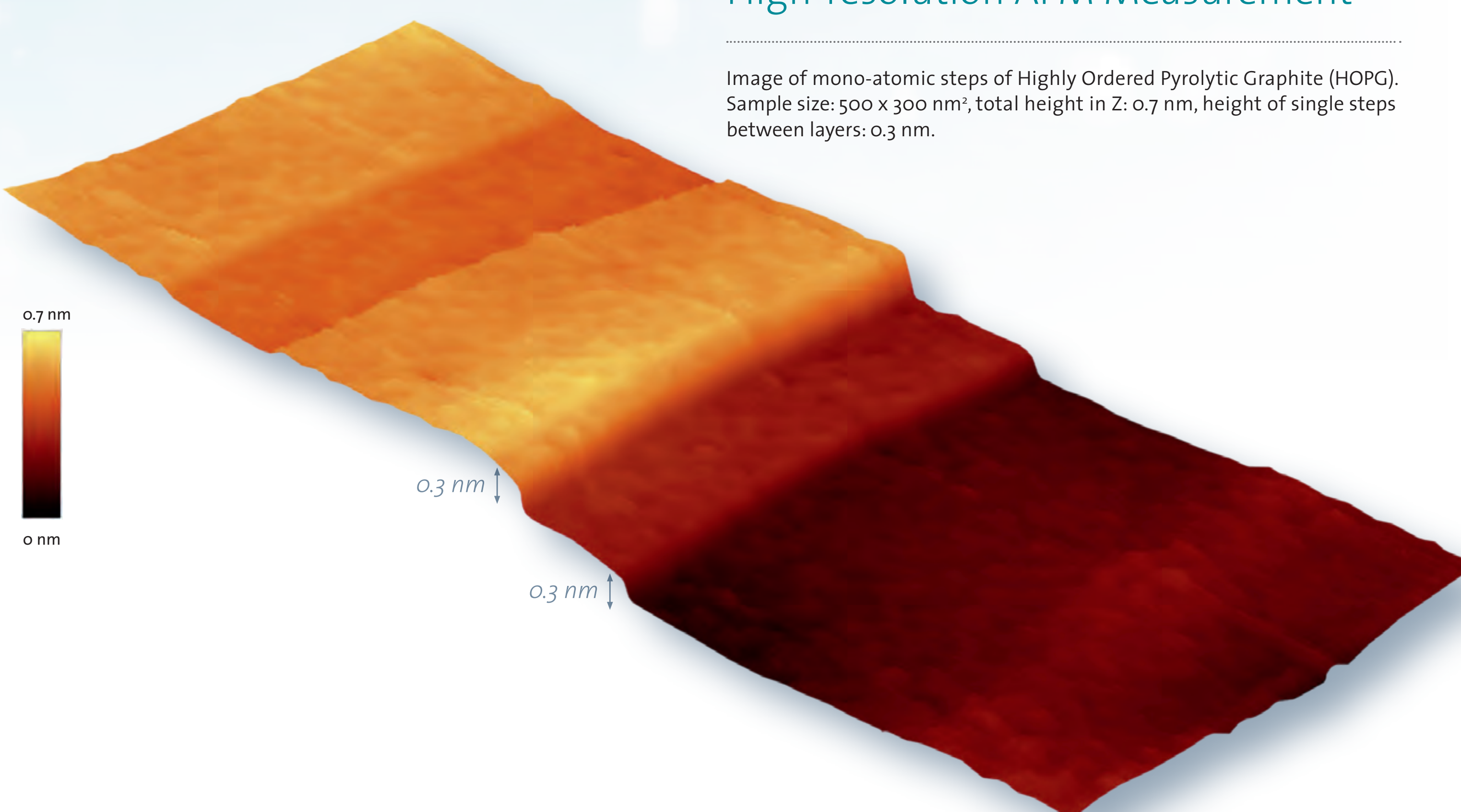
Investigation of Electrostatic Characteristics

AFM images of a gold structure measured in AC Lift Mode™ and with Electric Force Microscopy (EFM) revealing **(A)** topography and **(B)** conductive/nonconductive areas of the sample.

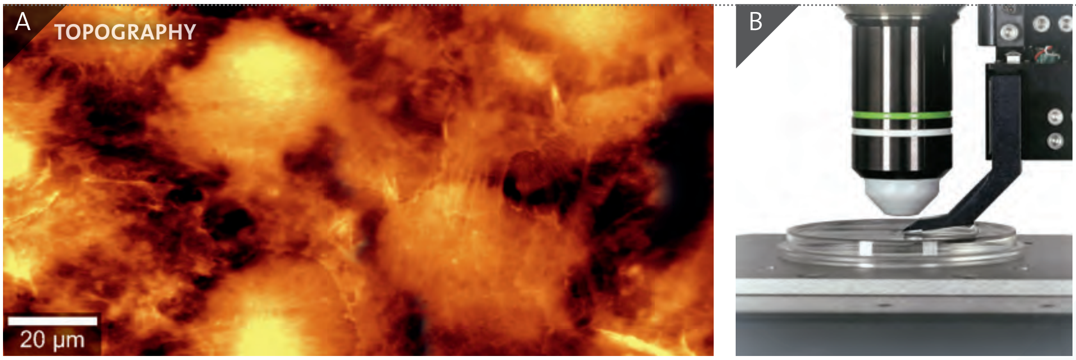


High-resolution AFM Measurement

Image of mono-atomic steps of Highly Ordered Pyrolytic Graphite (HOPG). Sample size: 500 x 300 nm², total height in Z: 0.7 nm, height of single steps between layers: 0.3 nm.

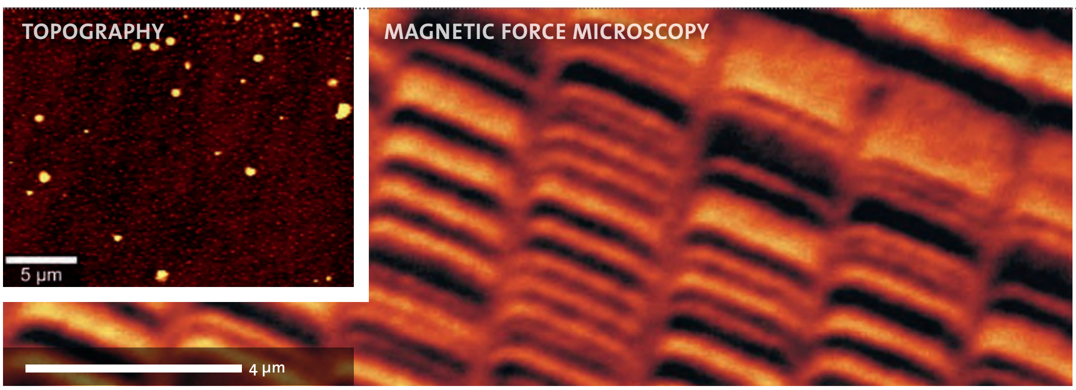


Large-area Measurements in Liquids



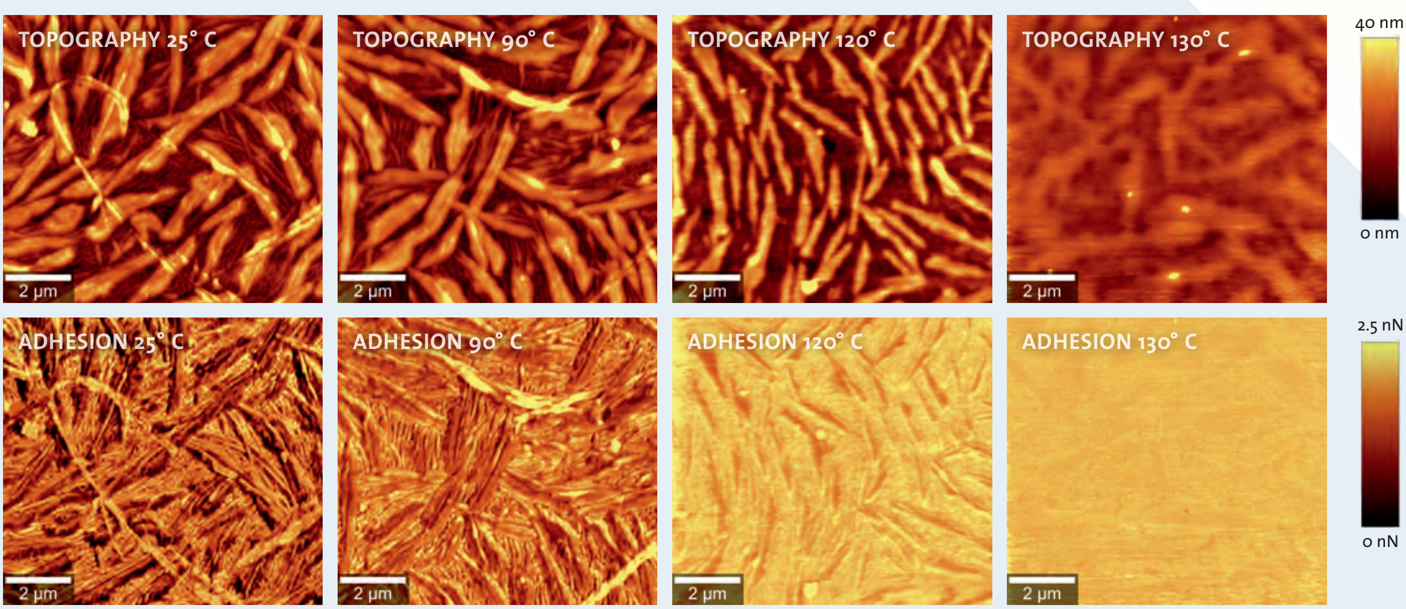
(A) 250 x 100 µm² large-area topography scan of a cell culture in liquid. Maximum measured height: 2.5 µm.
(B) Water immersion objective for AFM measurements in liquids.

Investigation of Magnetic Forces



Magnetic Force Microscopy (MFM) measurement of a hard drive. The measurements were performed using AC mode with magnetic tips. The topography is flat and uniform (see small image). The MFM image of the same sample area shows a clear magnetic contrast between magnetic hard drive domains (see large image).

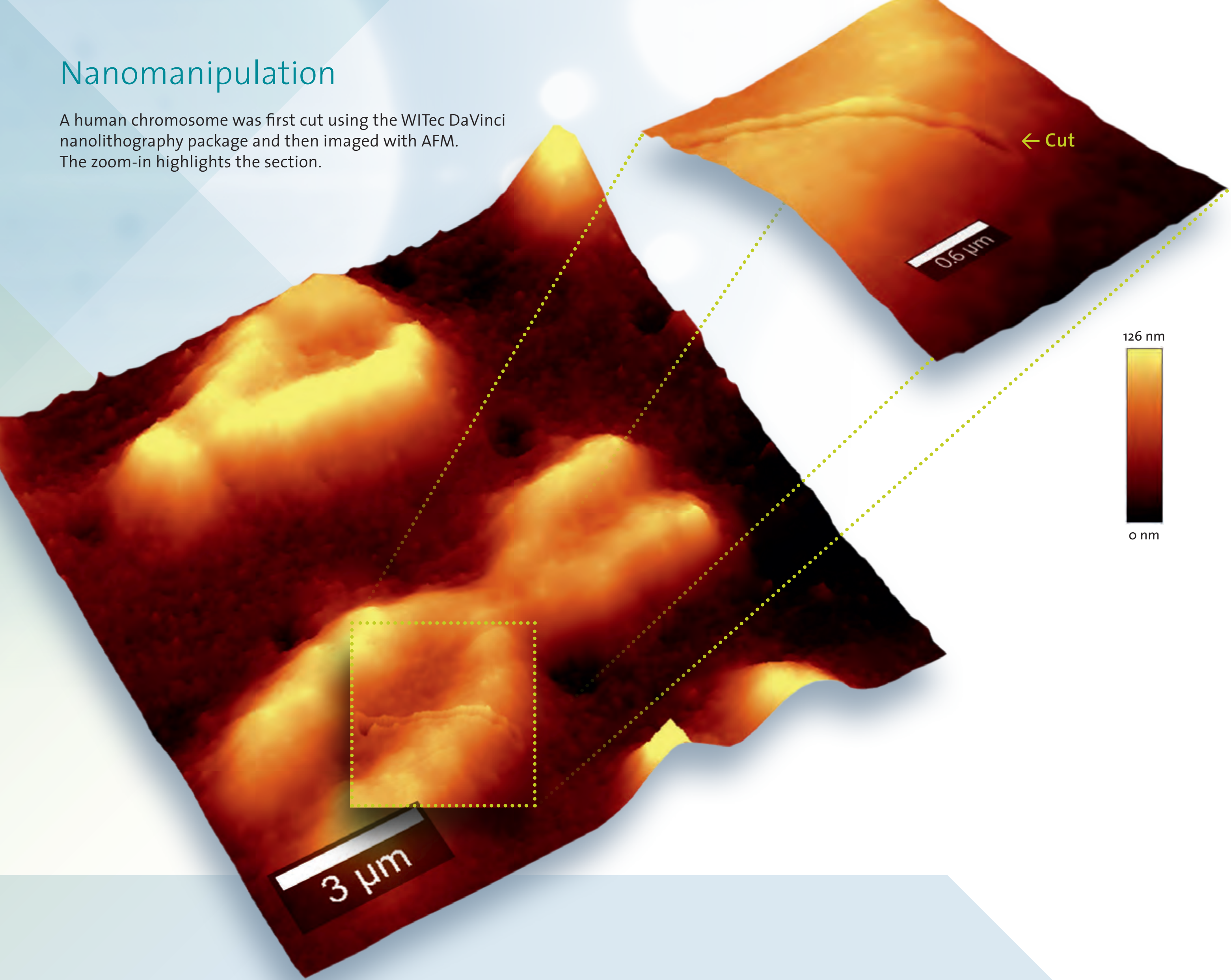
Temperature and Time Series



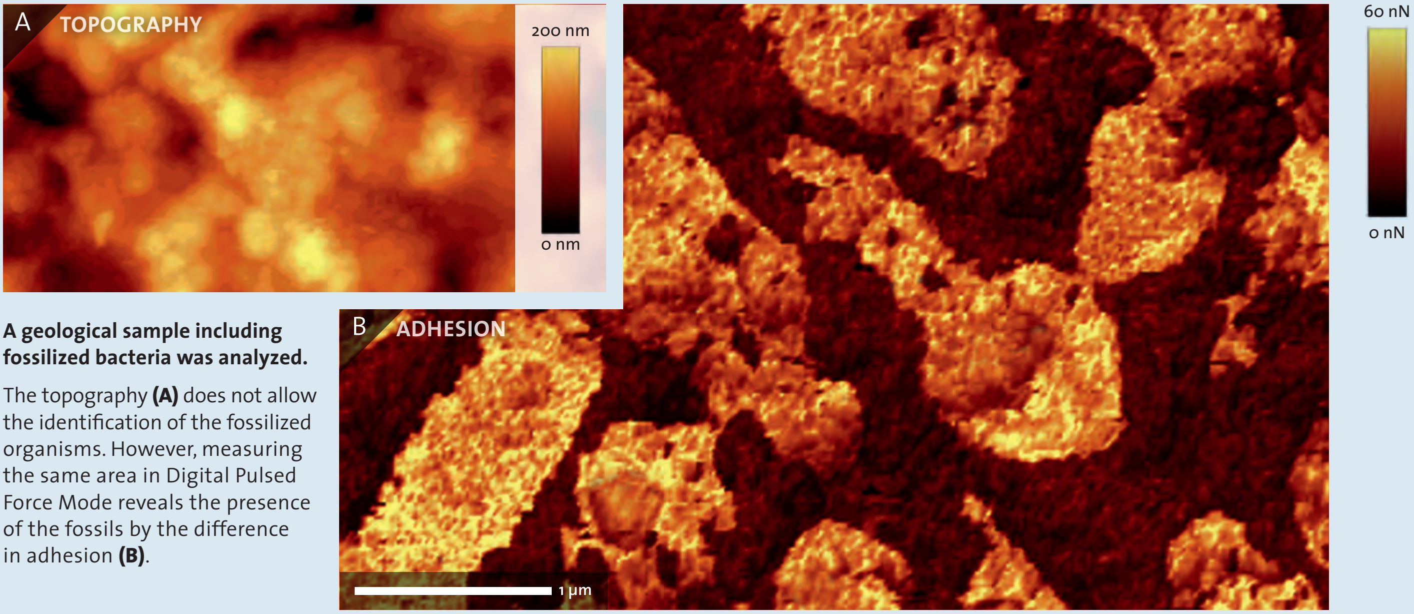
DPFM AFM images of heated paraffin at different temperatures.
Top row: Topography changes with rising temperature. At 130° C the topography is flattened due to melting processes.
Lower row: The adhesion increases while the temperature rises.

Nanomanipulation

A human chromosome was first cut using the WITec DaVinci nanolithography package and then imaged with AFM. The zoom-in highlights the section.



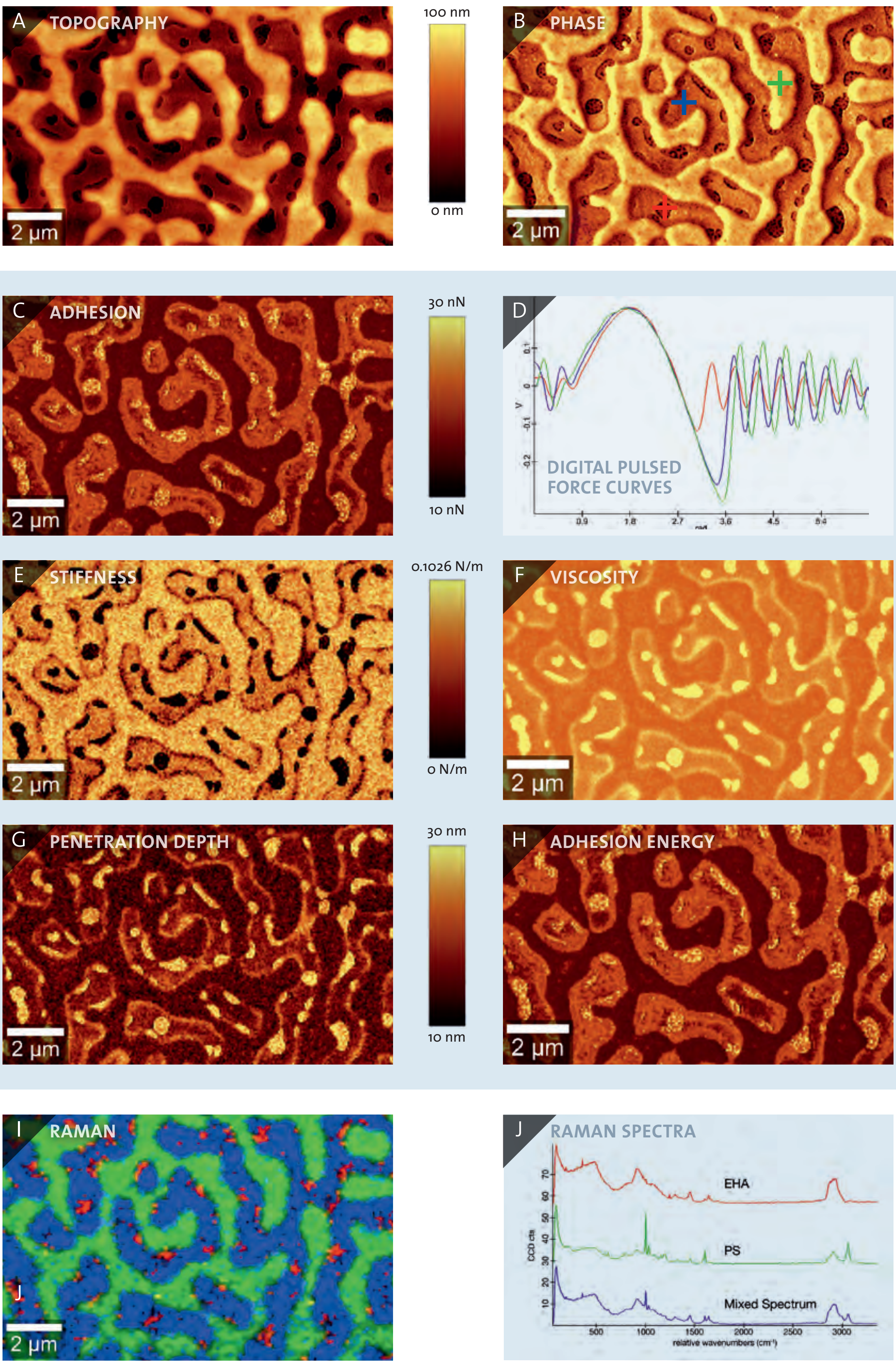
Applications of Different AFM Modes for Comprehensive Sample Characterization



A geological sample including fossilized bacteria was analyzed.

The topography **(A)** does not allow the identification of the fossilized organisms. However, measuring the same area in Digital Pulsed Force Mode reveals the presence of the fossils by the difference in adhesion **(B)**.

Correlative AFM and Raman Study of a Polymer Blend



Digital Pulsed Force Mode (DPFM) imaging demonstrates its most immediate advantage: topography and other physical information can be acquired simultaneously.

A mixture containing identical quantities of polystyrene, ethyl-hexyl-acrylate and styrene-butadiene-rubber was spin coated onto glass. The topography of the sample acquired in AC mode reveals a three-level structure **(A)**. The simultaneously recorded phase image shows the fine structure of the mixture **(B)**.

DPFM curves showing the force signal vs. time **(D)** were recorded from three areas as marked with the corresponding colored crosses in the phase image. For imaging, DPFM curves were measured at each pixel.

Differences in the DPFM curves generate contrasts in the images, showing adhesion **(C)**, stiffness **(E)**, viscosity **(F)**, tip penetration depth **(G)** and adhesion energy **(H)**.

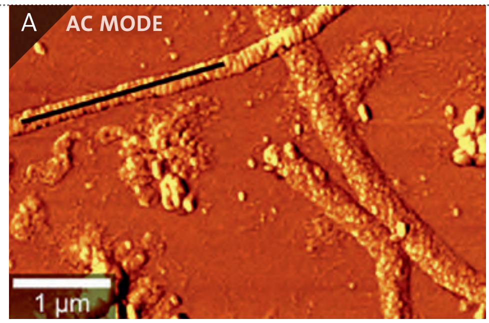
Concurrent with the AFM measurements the Raman spectra were recorded at each pixel using the alpha300 RA, a combined Raman-AFM instrument.

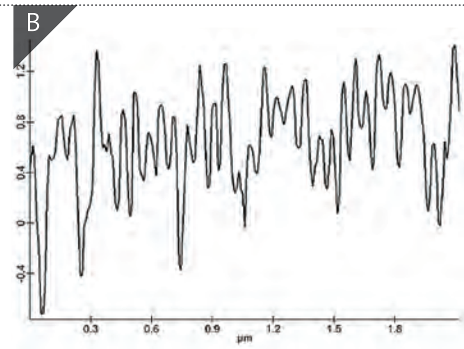
Spectral analysis reveals the three components of the blend, here displayed in red = EHA, green = PS and blue = mixed spectrum of EHA/SBR **(I)**. From all data a false color-coded Raman image was generated **(J)**. Correlating AFM and Raman images shows that the uppermost and stiffest features of the sample appear to be PS while the thinnest areas contain EHA only.

AFM Study of Collagen Fibers

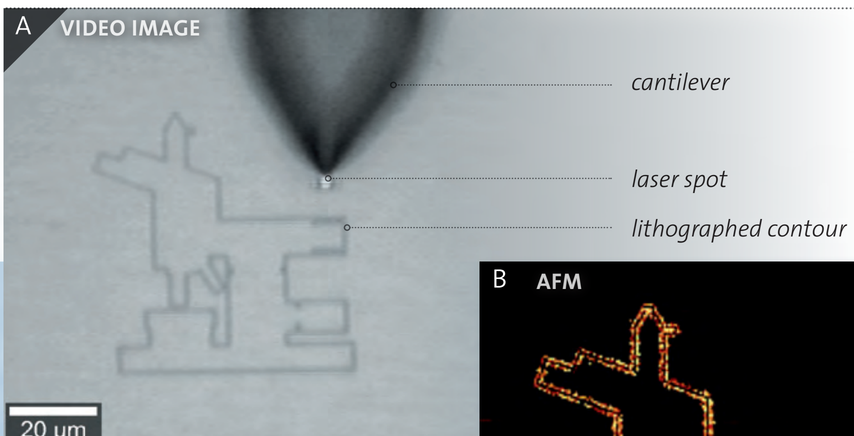
Type I collagen is present in many animal tissues such as bone. It is an elongated, highly organized structure consisting of many long molecules coiled around each other displaying a typical banding pattern.

(A) AC Mode AFM phase image of a collagen fiber. **(B)** Height profile of the fiber along the black line in **(A)**. The profile reveals regular bands 63 nm in width.



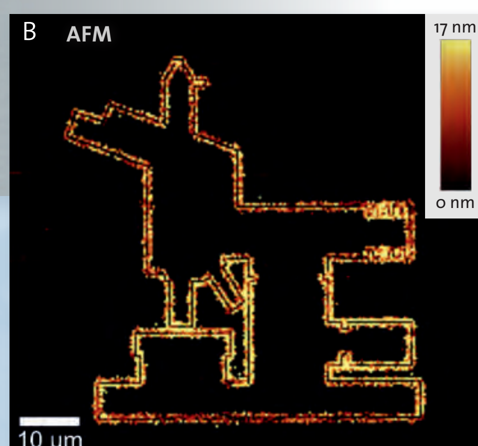


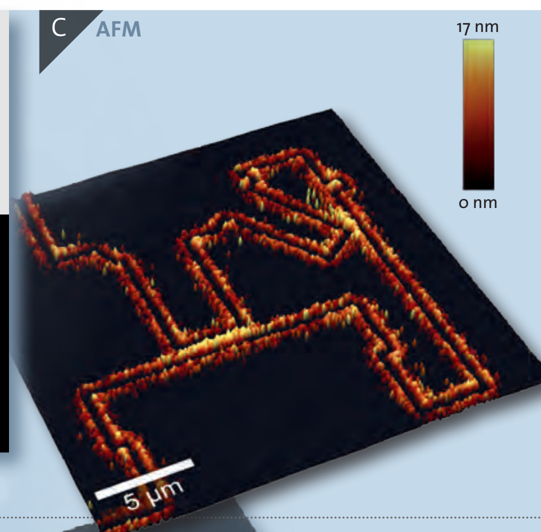
Simultaneous Raman-AFM Measurement

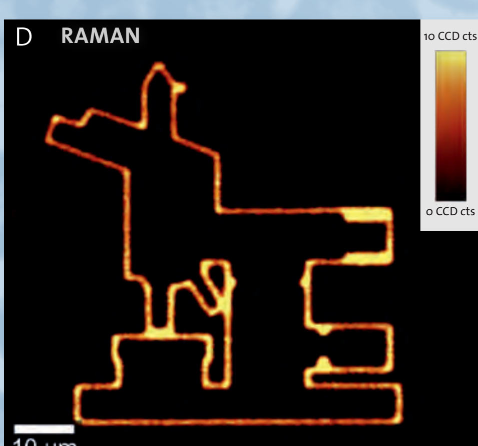


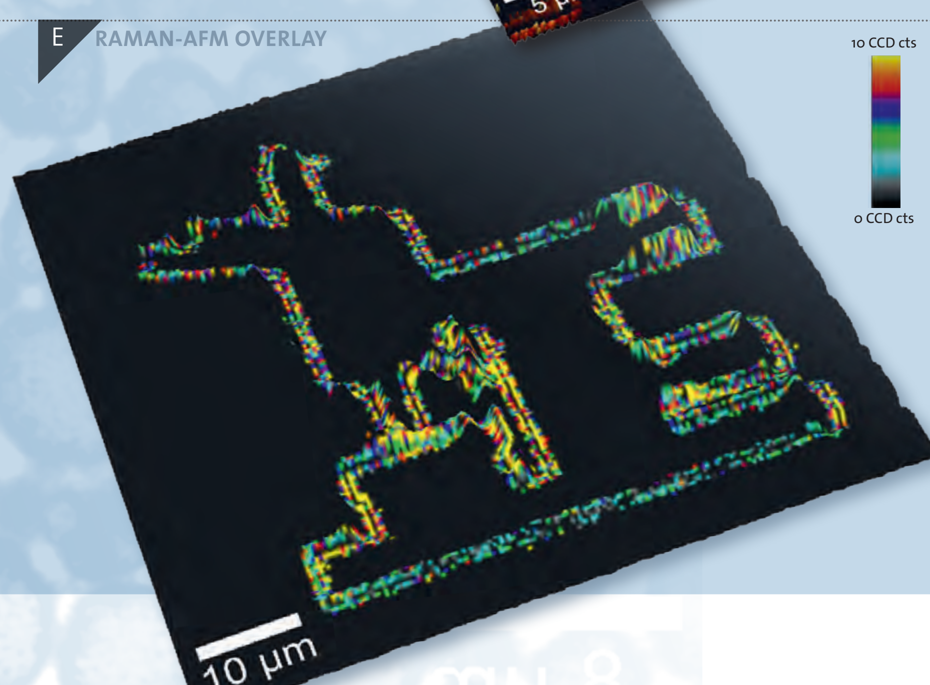
Nanolithography in GaAs simultaneously imaged by Raman-AFM.

(A) Video image with simultaneous cantilever, laser spot and sample view. **(B)** AFM topography image. **(C)** 3D representation of AFM topography zoom-in image. **(D)** Raman intensity image. Image parameters: 75 x 75 µm²; 200 x 200 pixels; 0.07 s integration time per spectrum. **(E)** 3D Raman-AFM image overlay.



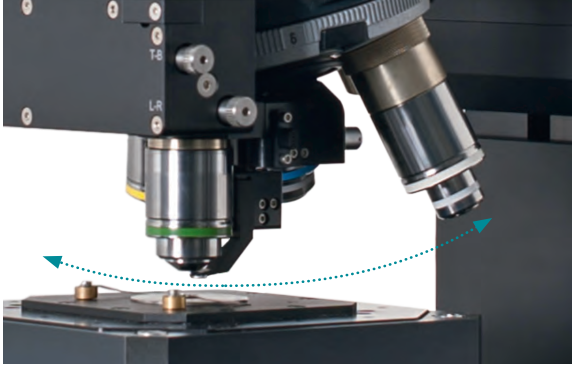




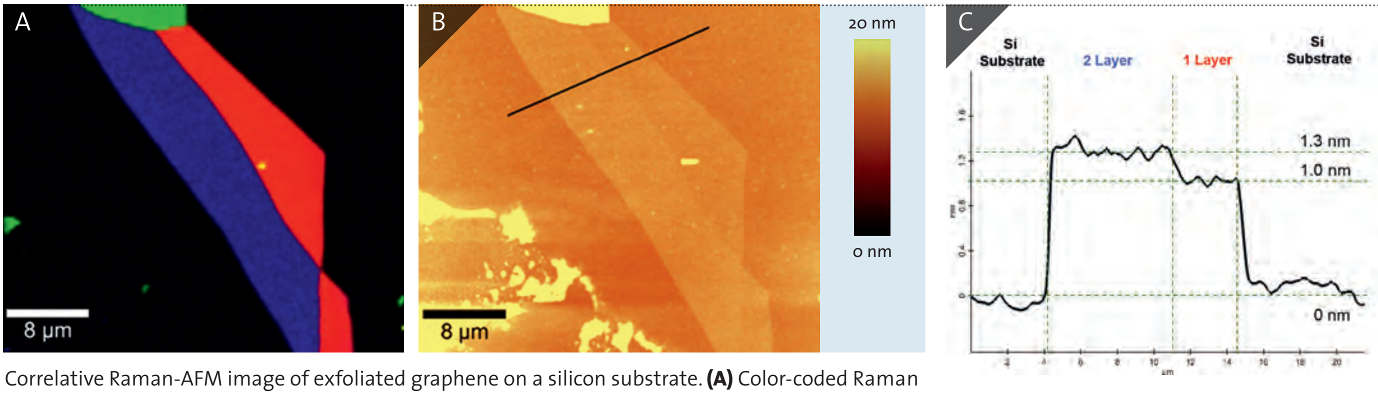


Combined Raman-AFM Measurement

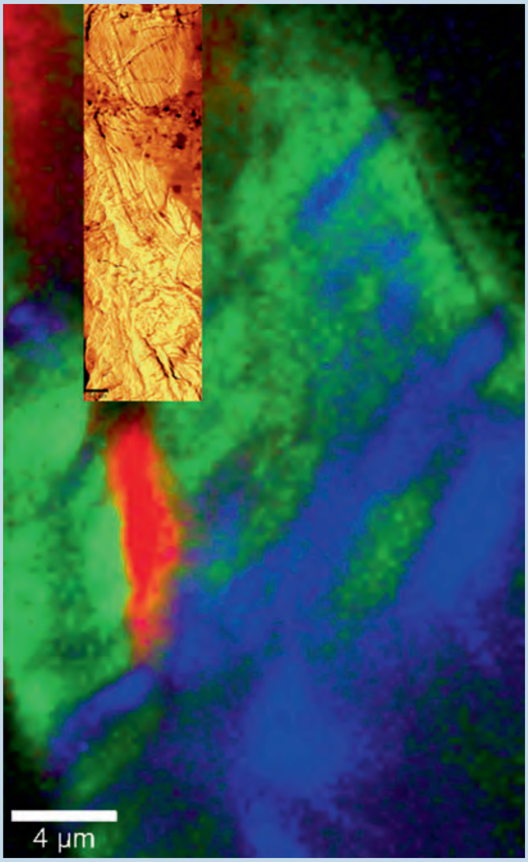
The modular and flexible design of the WITec alpha300 microscope series guarantees easy and cost-effective upgrade and extension possibilities. WITec's product line incorporates nearly all scanning probe and optical microscopy techniques to meet individual requirements. Each WITec alpha300 model can be equipped with new functionalities either as built-in features or as later upgrades. The WITec hardware and software environment is used for all features or upgrades, ensuring the best possible compatibility and ease of use.



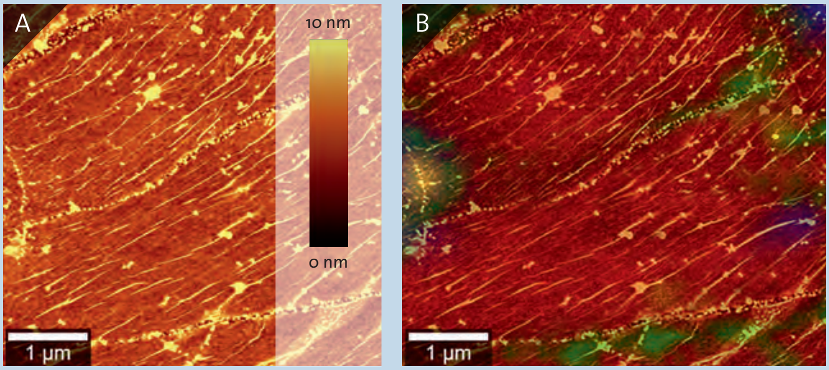
The objective turret is rotated to change from AFM to Raman imaging mode.



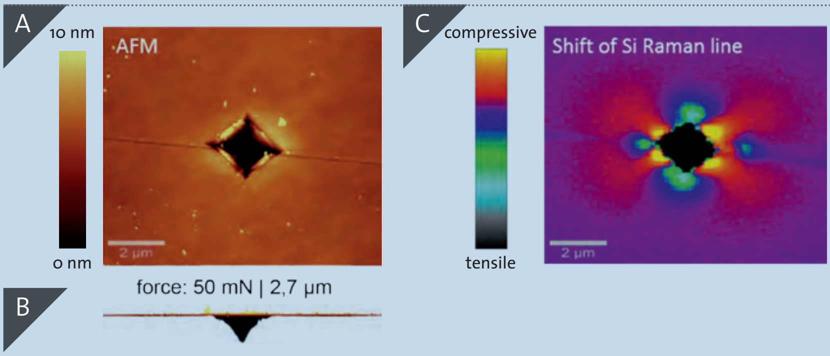
Correlative Raman-AFM image of exfoliated graphene on a silicon substrate. **(A)** Color-coded Raman image showing monolayer (red), bi-layer (blue) and multi-layer (green) graphene. **(B)** AFM topography image (AC mode). **(C)** Height profile of a cross section along the black line indicated in **(B)**. The height variation between one and two layers is approx. 0.3 nm.



Correlative Raman-AFM image of wood extractives. Color-coded Raman image of a cellulose fiber depicting cellulose (green, blue) and hexane extract (red). Inset: High-resolution AFM phase image.



Correlative Raman-AFM study of a CVD-grown graphene layer. **(A)** AFM topography image, 5 x 5 µm². **(B)** Raman image of the same area taken and overlaid with the AFM image. The different colors indicate layers and wrinkles in the graphene film.



Correlative Raman-AFM measurement investigating stress in silicon via Vickers indent. **(A)** 10 x 10 µm² AFM topography image around a Vickers indent. **(B)** AFM depth profile view. **(C)** Corresponding Raman image revealing the areas of stress in silicon.

WITec focus innovations

WE TAKE CARE
WITec uses environmentally friendly printed materials. While this policy is only a small contribution to a healthy environment, we at WITec believe that focusing on details can effect positive change in the world.

WITec alpha300 Series

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