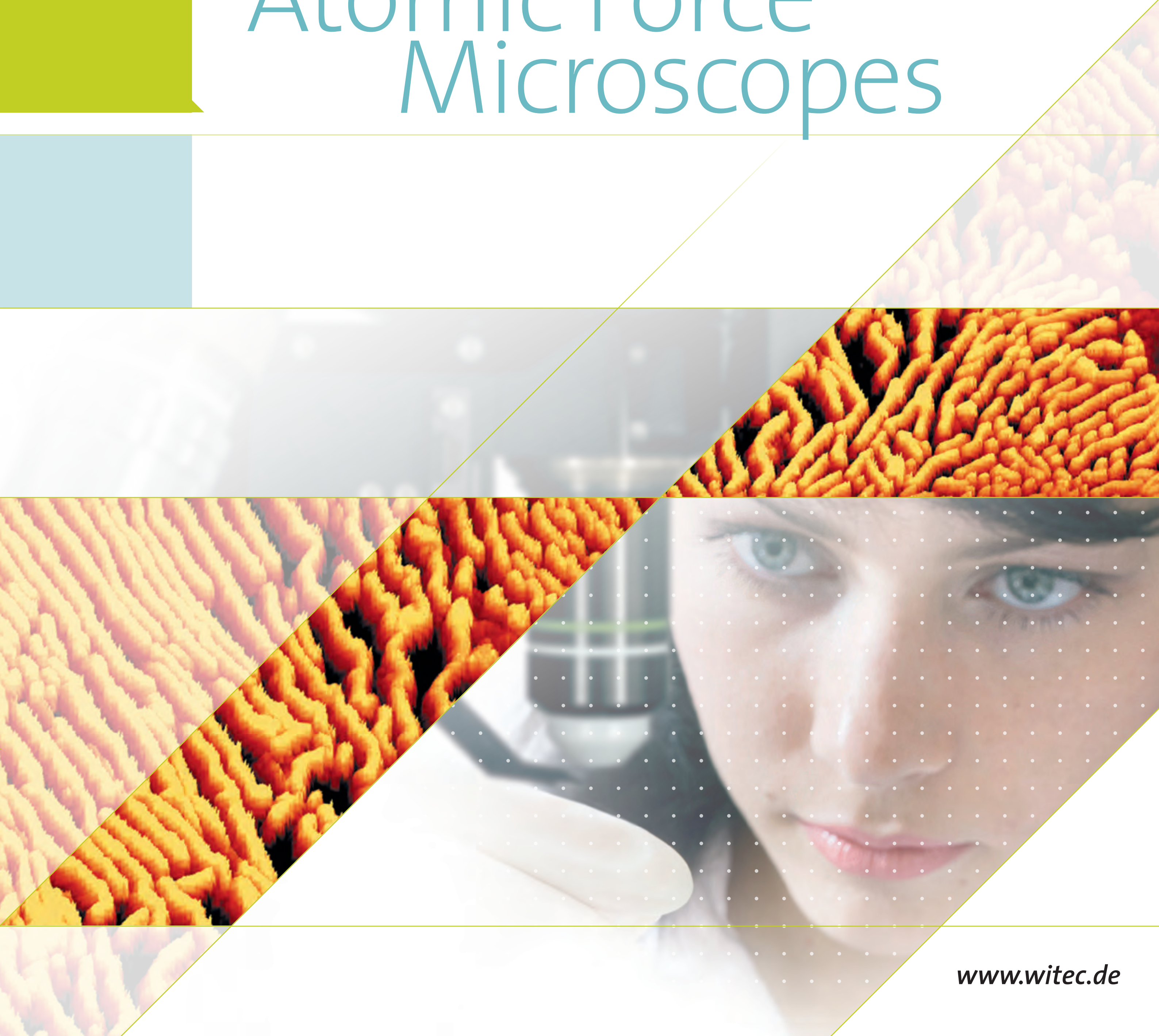
WITec
focus innovations

Nanoscale Surface Characterization

Atomic Force Microscopes



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01

WITec
Atomic Force Microscopes
Nanoscale Surface Characterization

The WITec Atomic Force Microscope integrated with a research-grade optical microscope provides superior optical access, easy cantilever alignment and high-resolution sample survey.

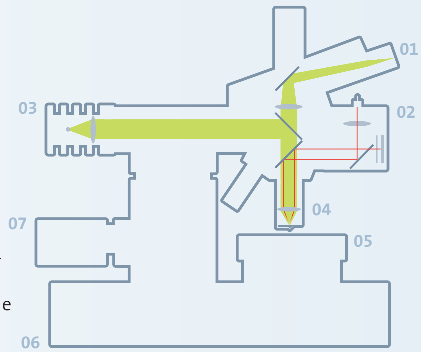
All WITec Atomic Force Microscopes are developed and designed for combination with other imaging techniques such as Raman spectroscopy, SNOM, luminescence, fluorescence, polarization analysis, bright-field and dark-field. By simply rotating the microscope turret the user can switch between the various imaging techniques.

BENEFITS

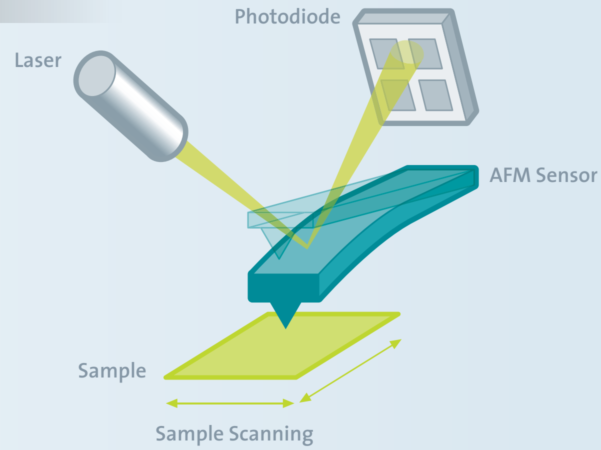
- Surface characterization on the nanometer scale
- Non-destructive imaging technique
- Optical and Atomic Force Microscope combination
- Convenient sample access from any direction
- Minimal, if any, sample preparation
- Ease of use in air and liquids
- Combinable with confocal Raman imaging and Scanning Near-field Optical Microscopy (SNOM)
- Precise TrueScan™ controlled scan stages with capacitive feedback loop, individually selectable:
  - 30 x 30 x 10 µm³ direct-drive scan stage
  - 100 x 100 x 20 µm³ scan stage
  - 200 x 200 x 20 µm³ scan stage

THE AFM MICROSCOPE

01 Video camera system
02 Beam deflection unit
03 LED white light illumination
04 AFM objective with cantilever
05 Scan-stage
06 Active vibration-isolation table
07 Z-stage for focusing



Beam Path



Working Principle

Atomic Force Microscopy traces the topography of samples with extremely high resolution by recording the interaction forces between the surface and a sharp tip mounted on a cantilever.

The sample is scanned under the tip using a piezo-driven scanning-stage and the topography is displayed as an image. Simultaneously either lateral forces or phase images are acquired. Atomic Force Microscopy provides spatial information parallel and perpendicular to the surface with resolution in the nm or sub-nm range.

In addition to topographic high-resolution information, local material properties such as adhesion and stiffness can be investigated by analyzing the tip-sample interaction forces.

02

Technology

Cantilever

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

Resolution

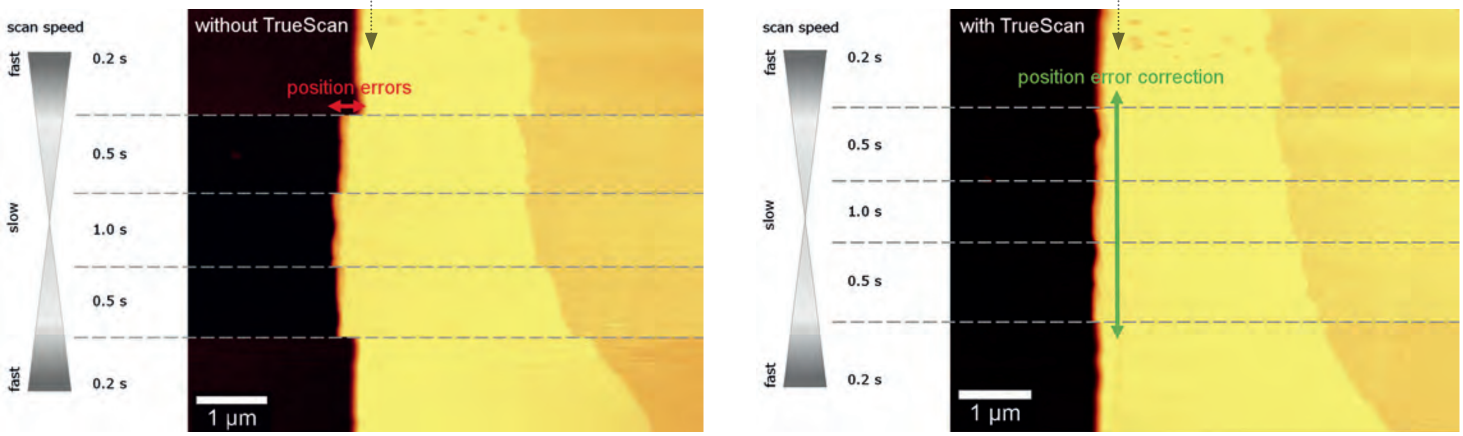
- Lateral resolution: Tip-radius dependent, down to 1 nm
- Depth resolution: Less than 0.3 nm for individual steps

TrueScan™

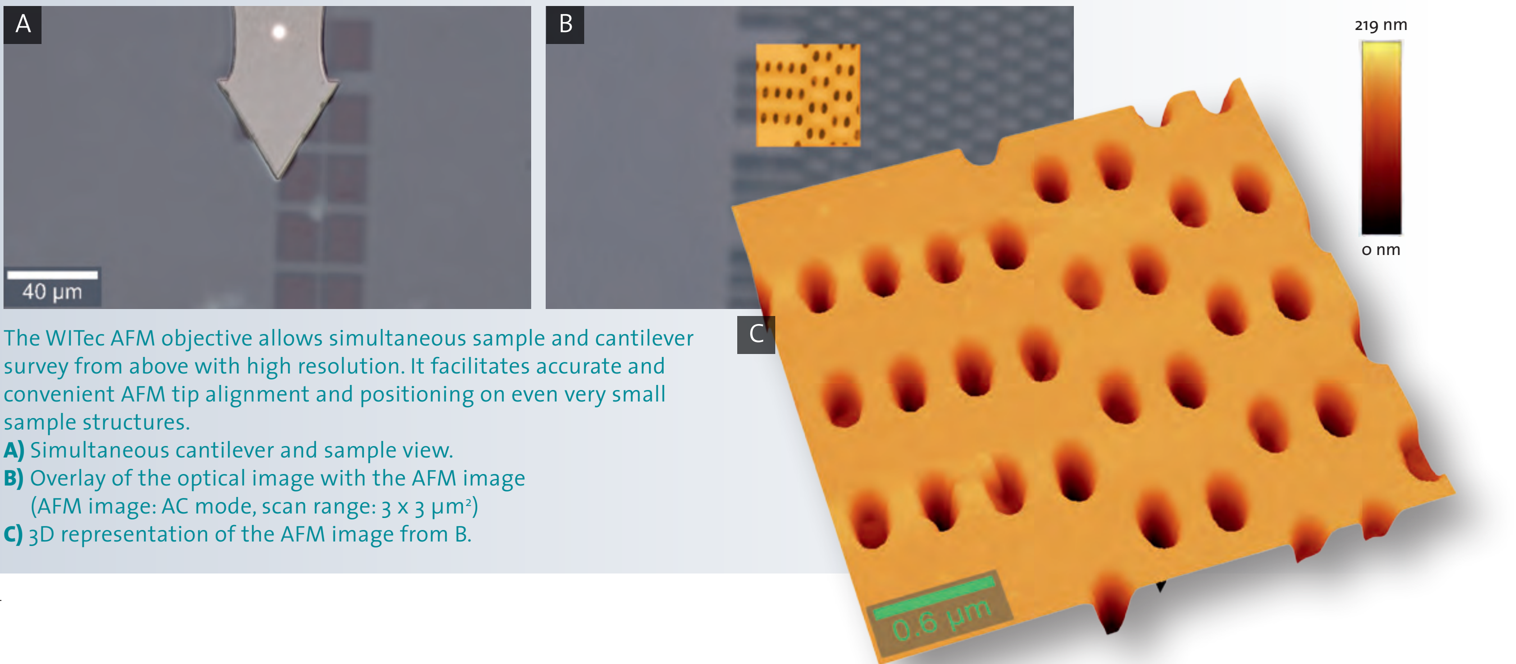
- Dynamic position error correction
- Continuous scanning without time gaps and script-based nanolithography for accurate high-resolution AFM imaging at high scan speeds.

Computer Interface

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



SIMULTANEOUS CANTILEVER AND SAMPLE VIEW FOR AN 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 view.
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.

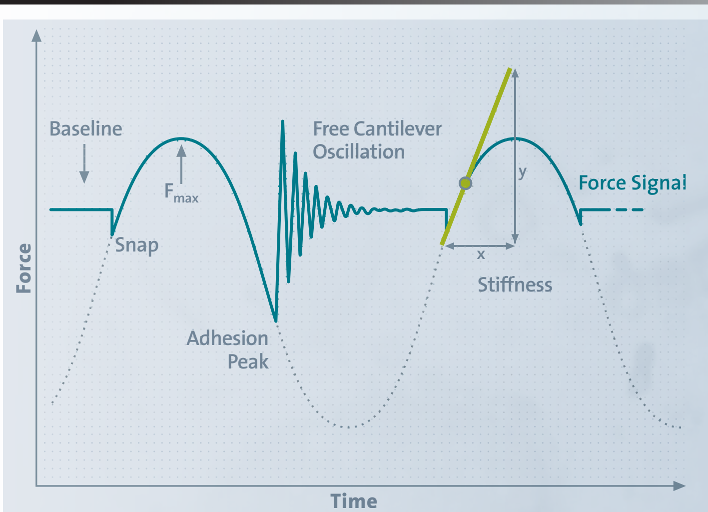
AFM Modes

- **Contact Mode:**
  The sample is scanned in direct contact with the AFM tip. The surface is recorded through deflection of the cantilever.
- **AC (Tapping™) Mode:**
  Also called intermittent mode. The cantilever oscillates at its resonance frequency and is not in constant contact with the sample. Thus the technique is particular well suited for delicate samples. When the tip comes close to the surface, sample-tip interactions cause forces to act on the cantilever which alter the oscillation.
- **Lift Mode™**
  Lift Mode can be applied in combination with Contact or AC Mode. First, the sample is scanned in an imaging mode such as contact 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 can be also combined with other AFM modes.
- **Digital Pulsed Force Mode (DPFM)**
  See information below.
- **Magnetic Force Microscopy (MFM)**
  Non-contact mode to measure the magnetic field above a sample.
- **Electrostatic Force Microscopy (EFM)**
  Non-contact mode to probe the electrostatic forces.
- **Phase Imaging (Tapping Mode™)**
  Recording and imaging of the phase shift signal in intermittent-contact mode (Tapping Mode™).
- **Nano-Manipulation/Lithography**
- **Lateral Force Microscopy (LFM)**
  Contact mode imaging that reveals the surface friction characteristics.
- **Chemical Force Microscopy (CFM)**
  Contact or intermittent mode to measure chemical forces such as Van-der-Waals forces.
- **others optional**

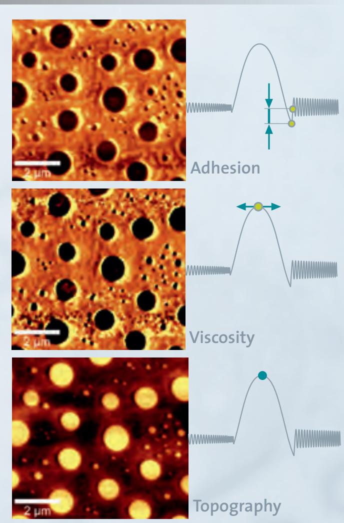
Digital Pulsed Force Mode (DPFM)

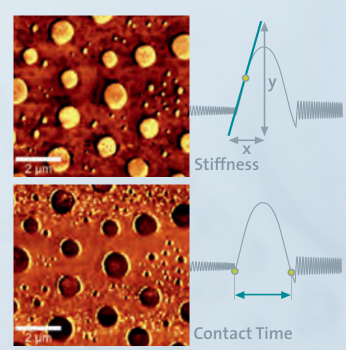
Pulsed Force Mode (PFM) is a non-resonant, intermittent contact mode for Atomic Force Microscopy that allows the characterization of material properties such as adhesion, stiffness, viscosity, energy dissipation, contact-time and long range forces along with the sample topography. Additionally, lateral forces are virtually eliminated. Therefore high-resolution mapping of delicate samples in air and fluids is easily attainable while maintaining a scanning speed comparable to contact-mode AFM. In contrast to most other intermittent contact techniques, the normal forces on the sample (introduced by the AFM tip) are controlled by the feedback loop. The PFM electronics introduce a sinusoidal modulation to 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



Pulsed Force Mode (PFM)





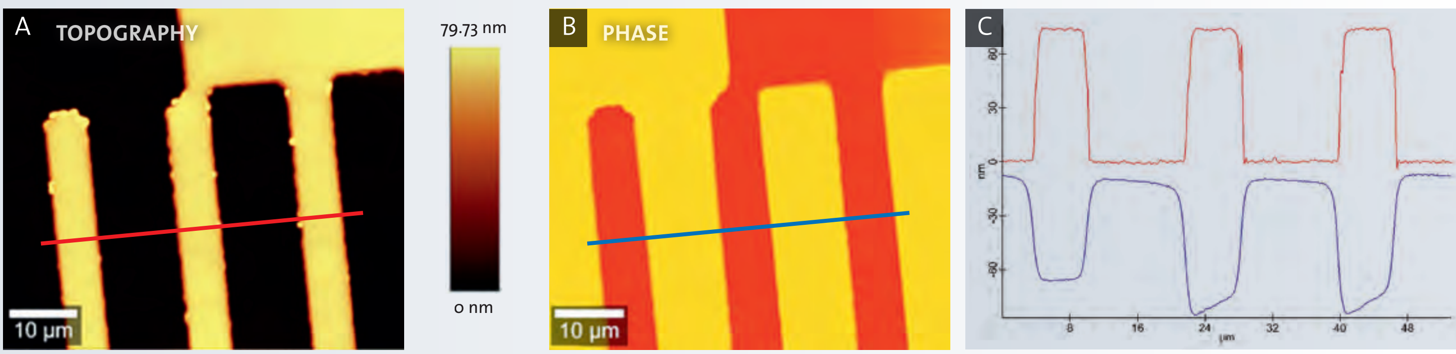
Digital Pulsed Force Mode (DPFM)

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

03

Applications

INVESTIGATION OF ELECTROSTATIC CHARACTERISTICS

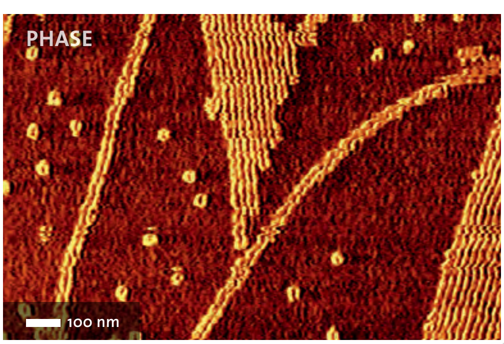


Lift Mode™ & Electric Force Microscopy (EFM) of a gold structure (with an applied potential of 3V). **A)** Topography image **B)** Phase image collected in Lift Mode. The contrast is a result of the voltage difference between the sample and the tip. **C)** Cross sections along the red and the blue line in A) and B). The red graph represents the height diferences. The blue graph shows the phase shift caused by electrostatic tip-attractions. It indicates that strong electrostatic sample-tip interactions occur in the red areas in B.

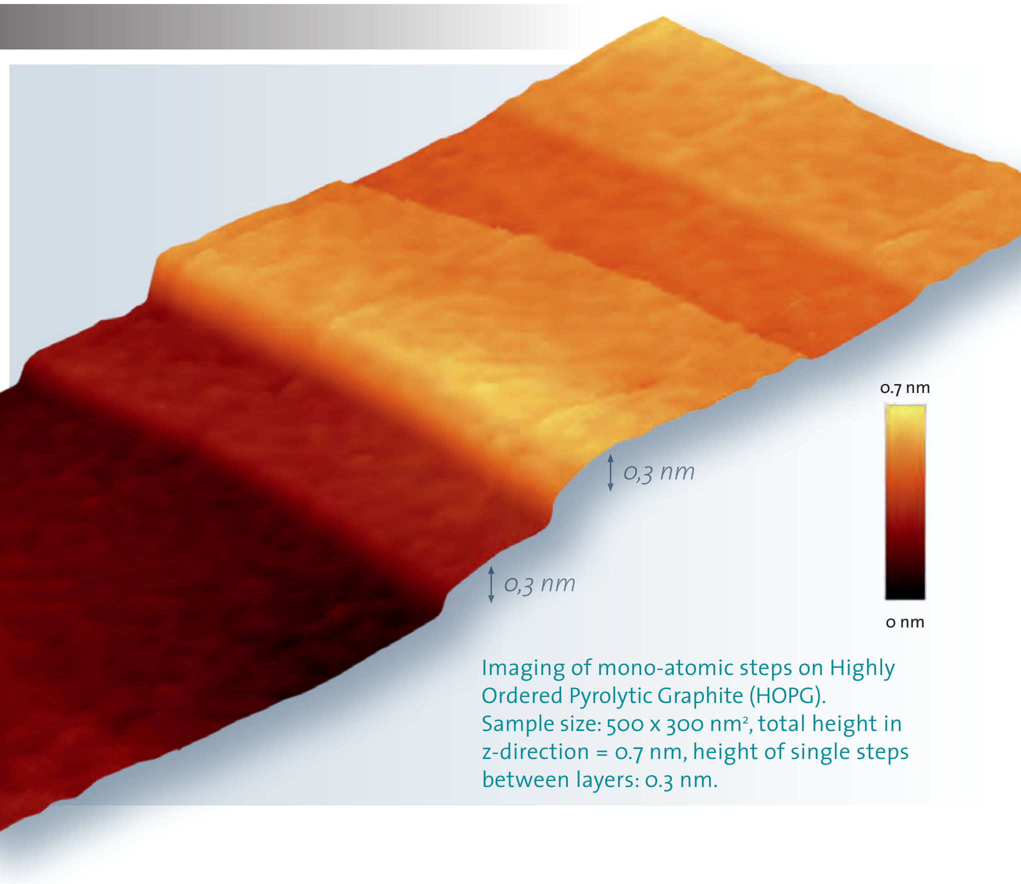
Further reading

- F. Sharifi, R. Bauld, G. Fanchini, Acridine orange as a biosensitive photovoltaic material. Journal of Applied Physics 114, (2013)10.1063/1.219614810.1063/1.4824181).
- F. Rubio-Marcos, A. Del Campo, R. López-Juárez, J. J. Romero, J. F. Fernández, High spatial resolution structure of (K,Na)NbO3 lead-free ferroelectric domains. Journal of Materials Chemistry 22, 9714-9720 (2012)10.1039/c2jm30483j).

HIGH-RESOLUTION AFM MEASUREMENTS

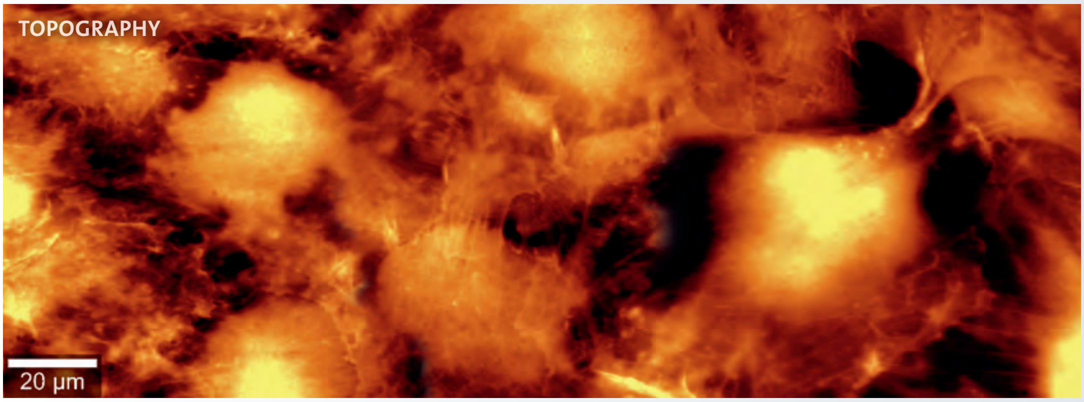


High-resolution AFM phase image of fluoro-alkane on a Si-substrate.

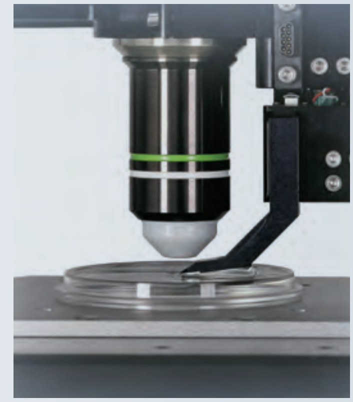


Imaging of mono-atomic steps on Highly Ordered Pyrolytic Graphite (HOPG). Sample size: 500 x 300 nm², total height in z-direction = 0.7 nm, height of single steps between layers: 0.3 nm.

LARGE-AREA MEASUREMENTS IN LIQUIDS

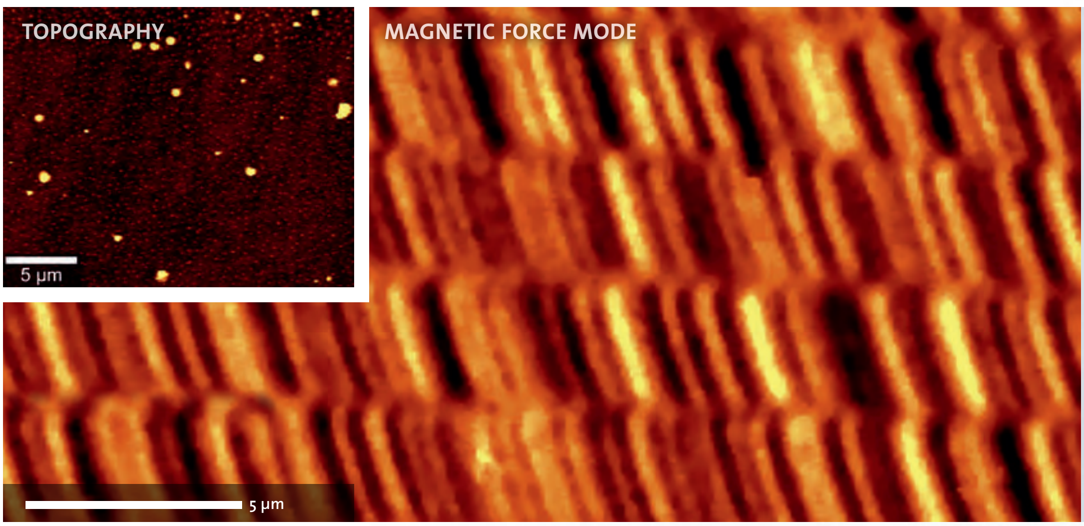


250 x 100 µm² large-area topography scan of a cell culture in fluid. Maximum measured height: 2.5 µm



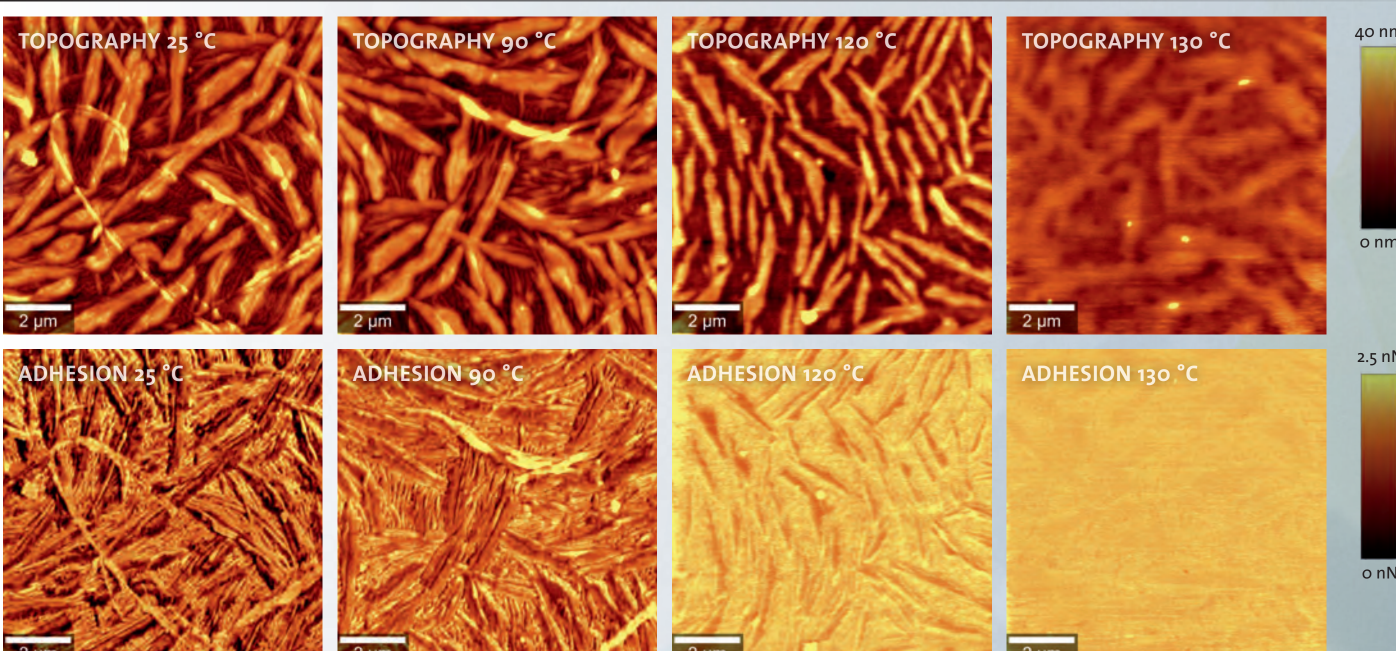
Water objective for AFM measurements in liquids.

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 at the magnetic hard drive domains (see large image).

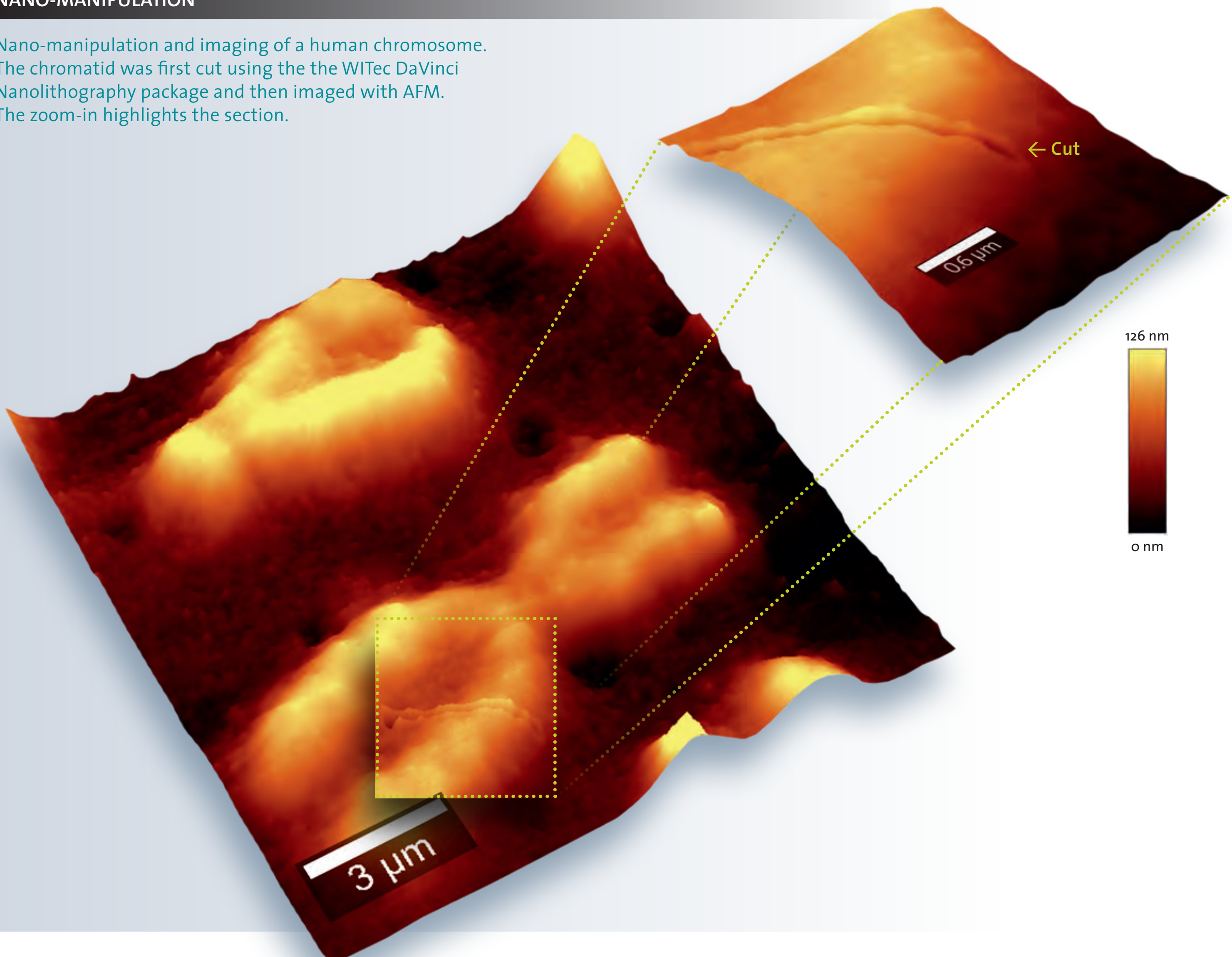
TEMPERATURE AND TIME SERIES



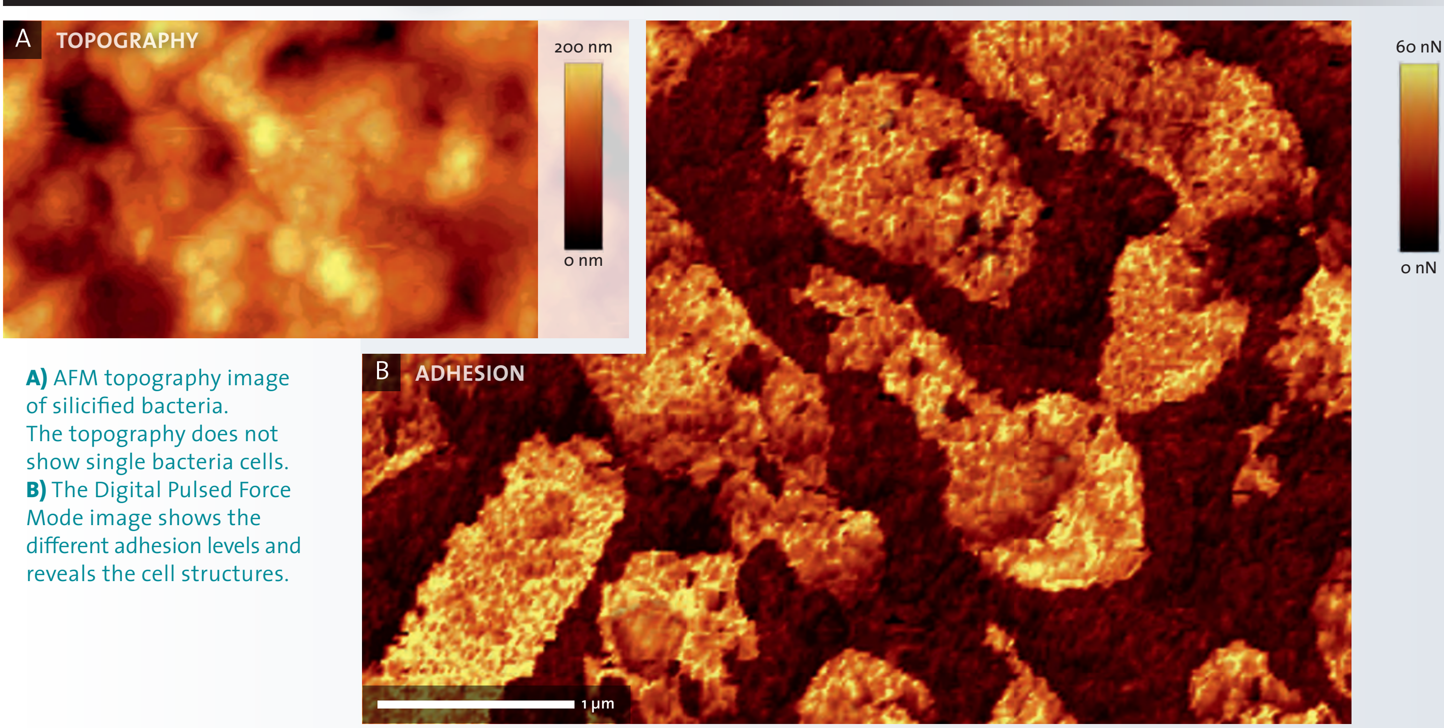
DPFM AFM image of heated paraffin at different temperatures. Top row: Topography changes with rising temperature. At 130 °C the topography is flattened due to melting processes. Lower row: The adhesion increases while the temperature rises.

NANO-MANIPULATION

Nano-manipulation and imaging of a human chromosome. The chromatid was first cut using the the WITec DaVinci Nanolithography package and then imaged with AFM. The zoom-in highlights the section.

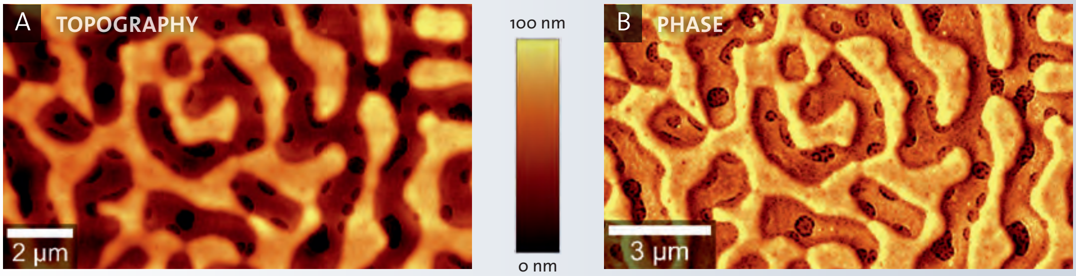


APPLICATION OF DIFFERENT AFM MODES FOR COMPREHENSIVE SAMPLE CHARACTERIZATION

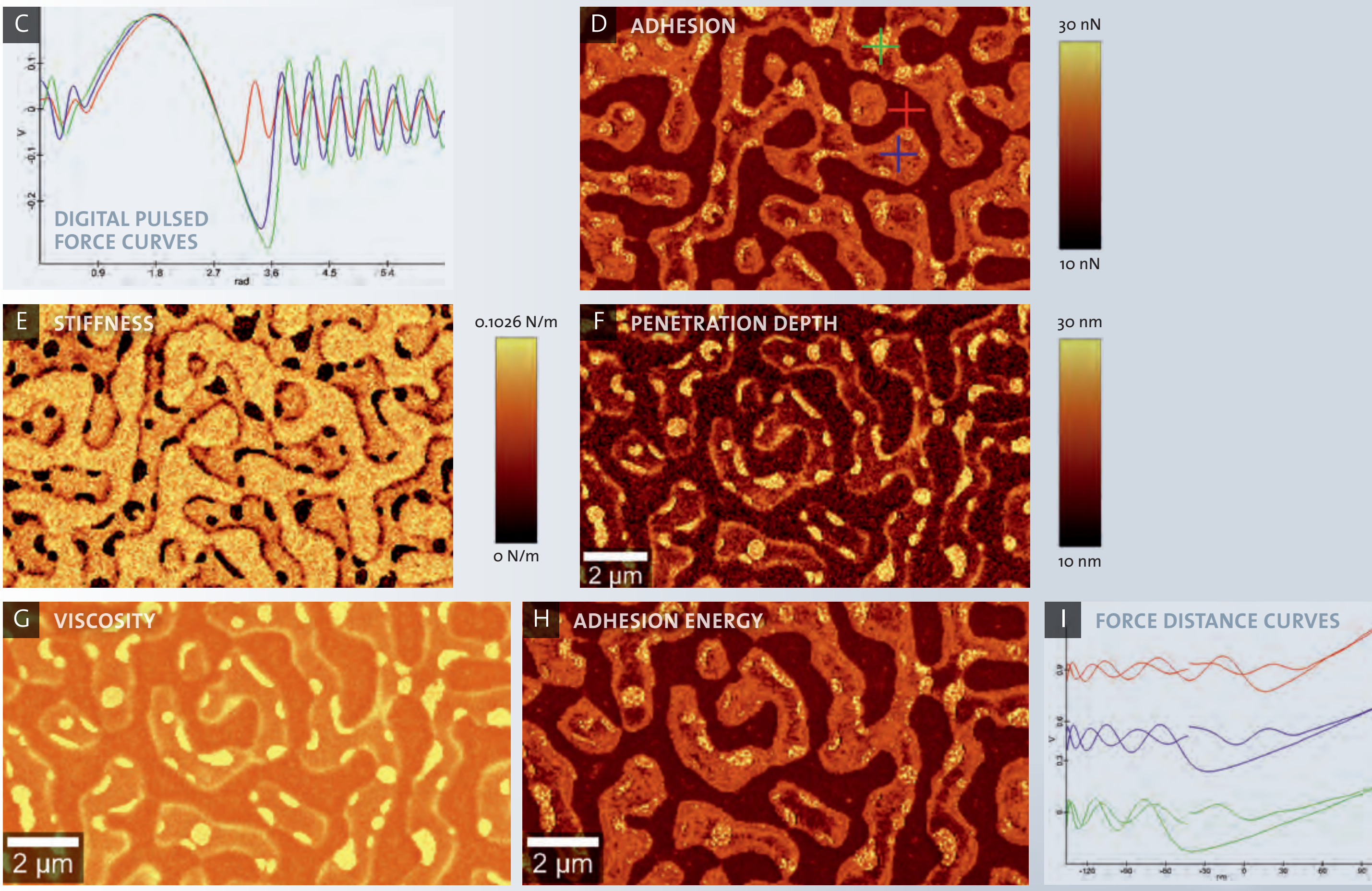


A) AFM topography image of silicified bacteria. The topography does not show single bacteria cells.
B) The Digital Pulsed Force Mode image shows the different adhesion levels and reveals the cell structures.

COMPREHENSIVE AFM-RAMAN STUDY OF A POLYMER BLEND (Polystyrene:Ethyl-Hexyl-Acrylate:Styrene-Butadien-Rubber; 1:1:1)

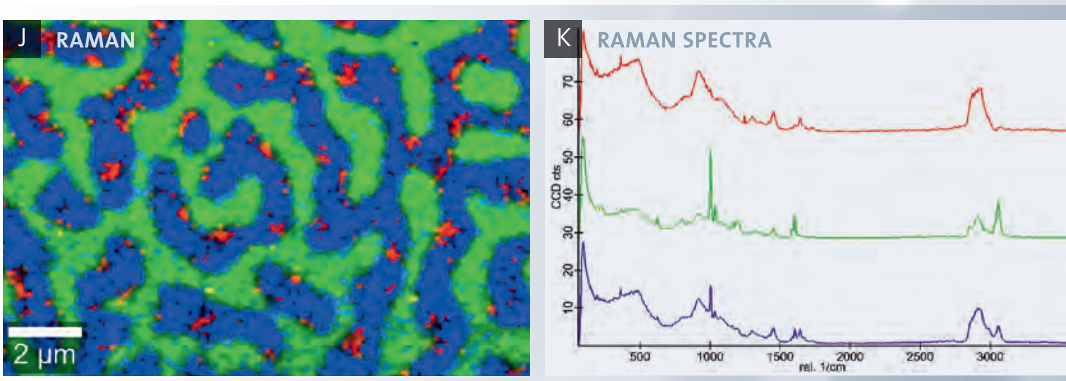


A) Topography image acquired in AC mode revealing a three level fine structure.
B) Simultaneously recorded phase image showing different viscoelastic properties at each topographic level.



Digital Pulsed Force Mode (DPFM) images: Simultaneous acquisition of topography and corresponding DPFM curves at each image pixel.

C) Characteristic DPFM curves from areas with different viscoelastic properties: The positions at which the three graphs were acquired are marked color-coded in D. The three DPFM curves show clear differences in the slope and adhesion peaks leading to the contrast in the adhesion (D) and stiffness (E) images. Further images can be evaluated from the DPFM curves: **F)** tip penetration depth, **G)** viscosity and **H)** adhesion energy. Representations of DPFM curves as standard force distance curves (cantilever approached to and retracted from the sample) from the same spots marked in image (D) are shown in **(I)**.

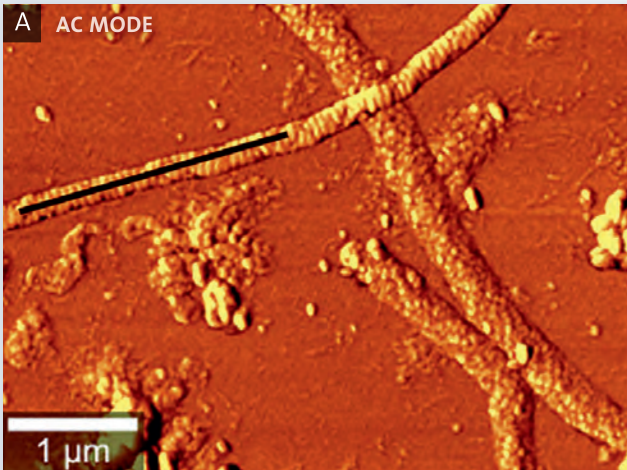


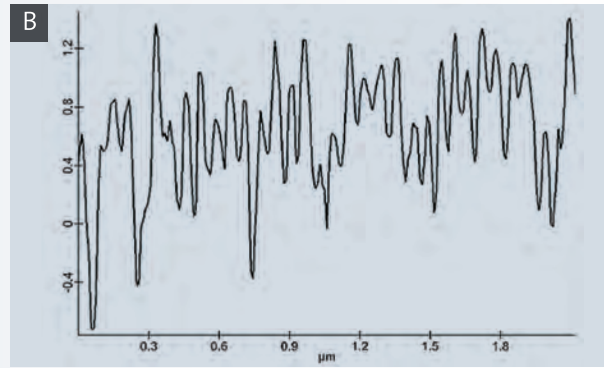
J) Confocal Raman image from the same sample area with corresponding Raman spectra
K) Green spectrum: Polystyrene; Red spectrum: Ethyl-Hexyl-Acrylate; Blue spectrum: mixed spectrum of Ethyl-Hexyl-Acrylate and Styrene-Butadien-Rubber.

Further reading

Schmidt et al., Confocal Raman Imaging of Polymeric Materials; From: Confocal Raman Microscopy; T. Dieing, O. Hollricher, J. Toporski (eds.); Springer Series in Optical Sciences, Springer, Berlin, Heidelberg, 2010.

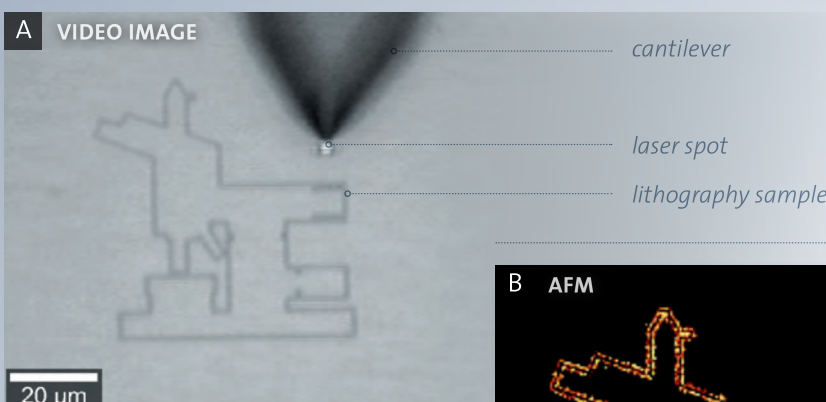
GENERATION OF CROSS SECTIONS





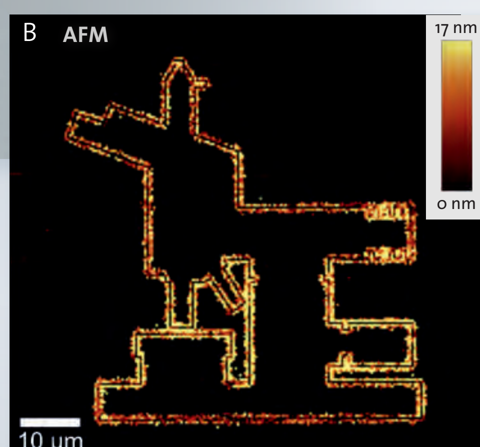
A) AC tapping mode AFM phase image of a collagen fiber. **B)** The cross section along the black line in A) reveals single peptide domains with a periodicity of 63 nm.

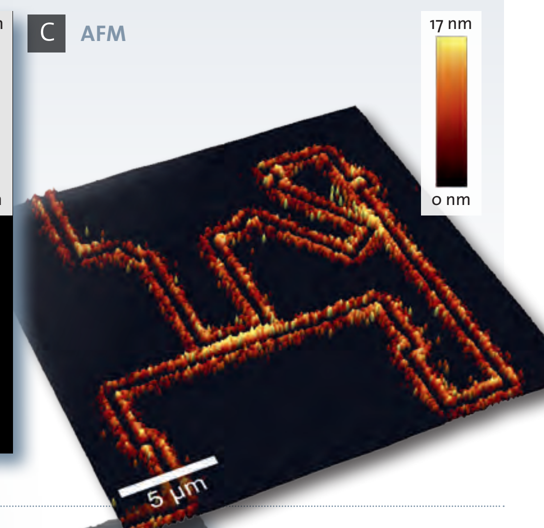
SIMULTANOUS RAMAN-AFM MEASUREMENT

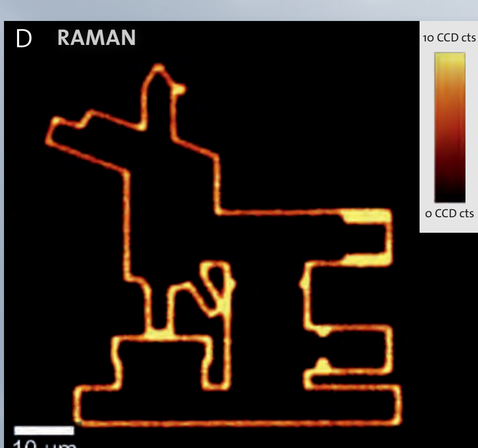


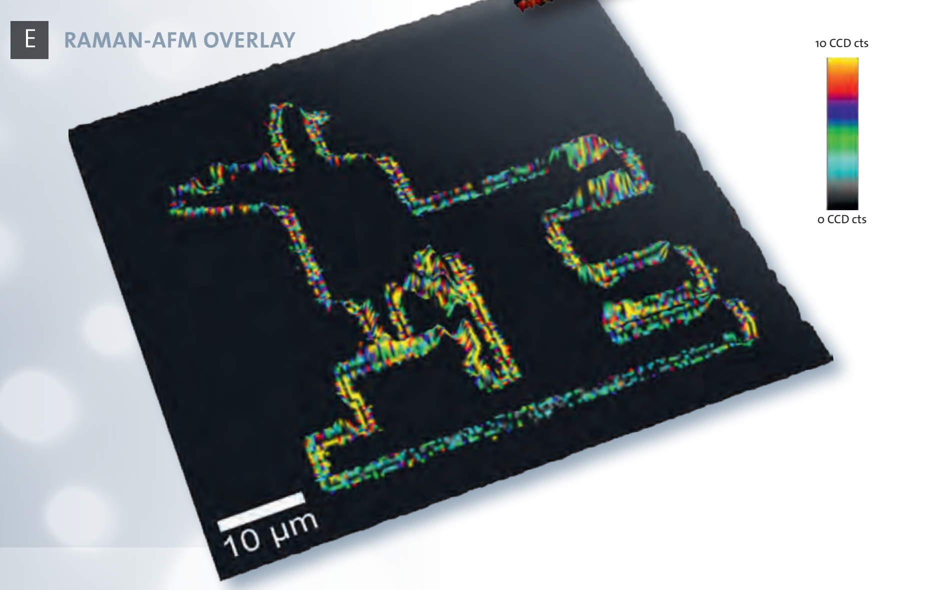
Nano-lithography in GaAs simultaneously imaged by Raman-AFM.

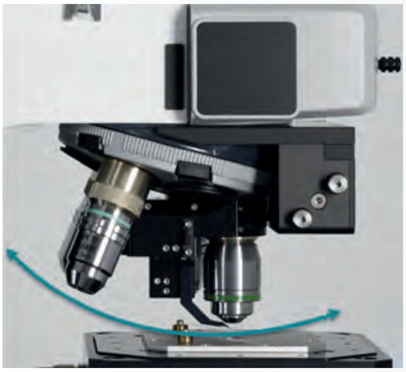
A) Video image with simultaneous cantilever, laser spot and sample view. **B)** AFM topography image. **C)** 3D representation of AFM topography zoom-in image. **D)** Raman intensity image, image parameters: 75 x 75 µm²; 200 x 200 pixels; 0.07 s integration time per spectrum. **E)** 3D Raman-AFM image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





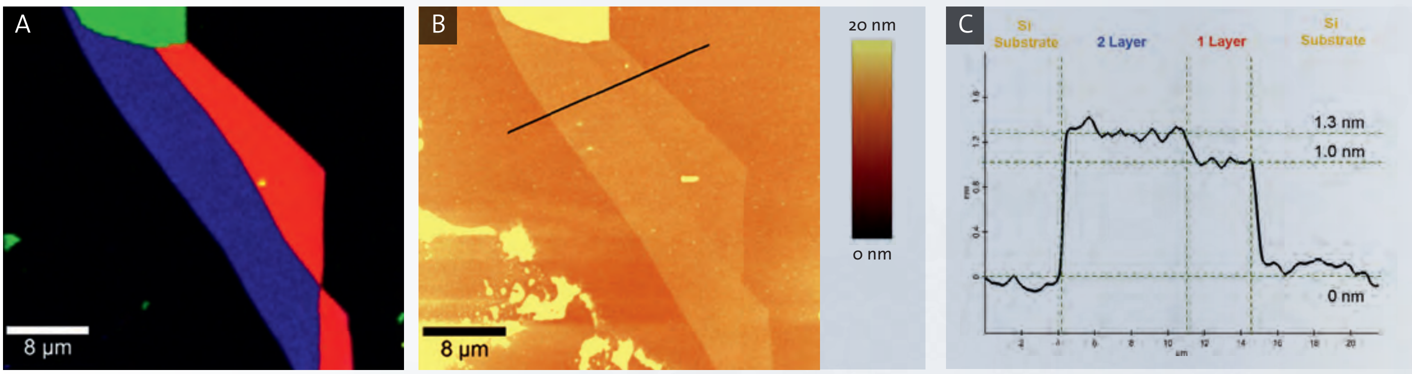




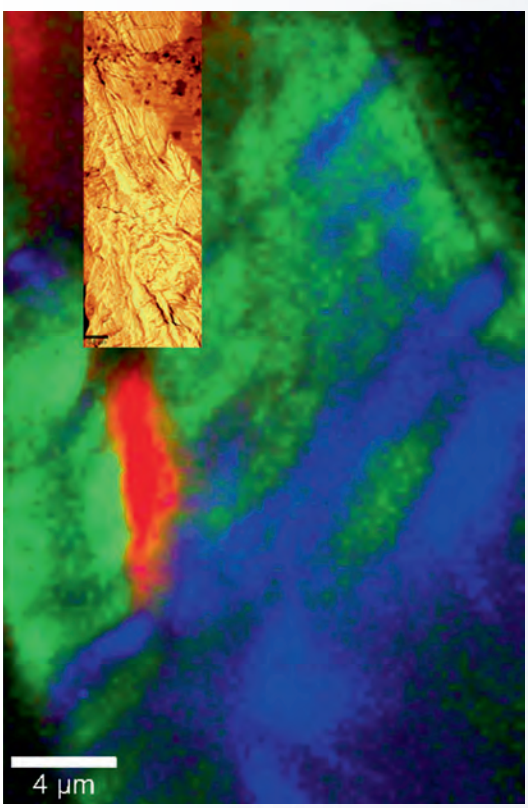


The modular and flexible design of the WITec alpha300 and alpha500 microscope series guarantees easy and cost-effective upgrade and extension possibilities. WITec's product line incorporates nearly all scanning probe and optical microscopy techniques to meet new individual requirements. Each WITec microscope model can be equipped with new functionalities either as a built-in feature or as a later upgrade. The WITec hardware and software environment is used for all new features or upgrades, securing the best possible compatibility and ease of use.

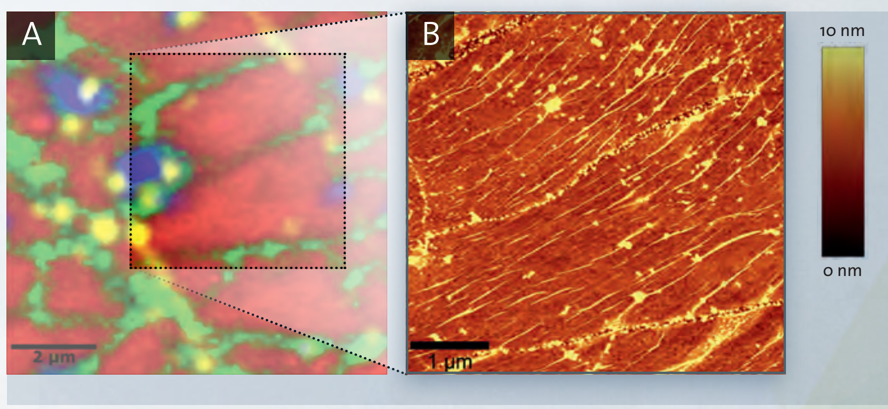
The objective turret is rotated to change from AFM to Raman imaging mode.



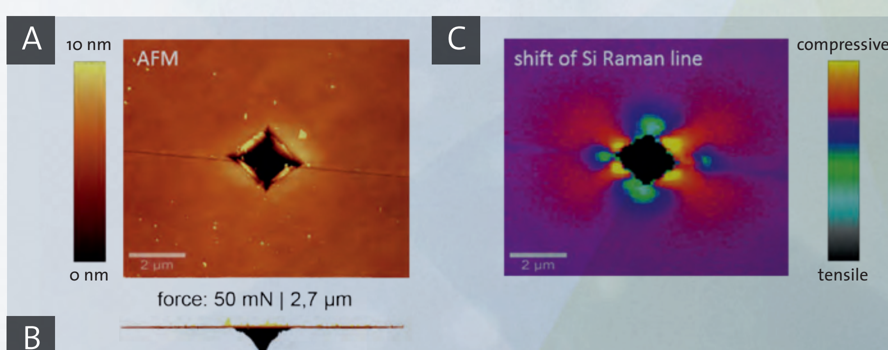
Combined Raman-AFM measurement from the same sample area of exfoliated graphene on a silicon substrate. **A)** Color-coded Raman image (Red: Monolayer; Blue: Bi-layer; Green: Multi-layer). **B)** AFM topography image (AC mode). **C)** Height profile of a cross section along the black line indicated in B). The height variation between one and two layers is only approx. 0.3 nm.



Confocal Raman-AFM image of traces of wood extractives on cellulose fibers. Color-coded Raman image of a cellulose fiber (image area 20 x 40 µm², 80 x 160 pixels, integration time: 100 ms/spectrum (Green and blue: Cellulose; Red: Hexane extract) Inlay: High-resolution AFM phase image.



Combined Raman-AFM investigation of defects of a CVD graphene layer. **A)** 10 x 10 µm² color-coded Raman image. The different colors indicate layers and wrinkles in the graphene film. **B)** 5 x 5 µm² AFM topography image of the same sample area.



Combined 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 of A). **C)** Corresponding Raman image revealing the stress areas in silicon.

List of scientific papers including results acquired with a WITec AFM:

- D. Dobrovolskas, J. Mickevičius, G. Tamulaitis, H. S. Chen, C. P. Chen, Y. L. Jung, Y. W. Kiang, C. C. Yang, Spatially resolved study of InGaN photoluminescence enhancement by single Ag nanoparticles. Journal of Physics D: Applied Physics 46, 145105 (2013)10.1088/0022-3727/46/14/145105).
- M. Potara, E. Jakab, A. Damert, O. Popescu, V. Canpean, S. Astilean, Synergistic antibacterial activity of chitosan-silver nanocomposites on Staphylococcus aureus. Nanotechnology 22, 135101 (2011); (10.1088/0957-4484/22/13/135101).
- S. Fuentes, R. A. Zarate, E. Chavez, P. Munoz, D. Díaz-Droguett, P. Leyton, Preparation of $SrTiO_3$ nanomaterial by a sol-gel-hydrothermal method. Journal of Material Science 45, 1448-1452 (2010).
- d. C. Silva, A. L. F. de Souza, R. A. Simão, L. F. Brum Malta, A Simple Approach for the Synthesis of Gold Nanoparticles Mediated by Layered Double Hydroxide. Journal of Nanomaterials 2013, 1-6 (2013)10.1155/2013/357069).
- M. A. Cejas, W. A. Kinney, C. Chen, J. G. Vinter, H. R. Almond, Jr., K. M. Balss, C. A. Maryanoff, U. Schmidt, M. Breslav, A. Mahan, E. Lacy, B. E. Maryanoff, Thrombogenic collagen-mimetic peptides: Self-assembly of triple helix-based fibrils driven by hydrophobic interactions. PNAS 105, 8513-8518 (2008); (10.1073/pnas.0800291105).

Combined Raman-AFM Measurements:

- M. Pilarczyk, A. Rygula, L. Matheuszuk, S. Chlopicki, M. Baranska, A. Kaczor, Multi-methodological insight into the vessel wall cross-section: Raman and AFM imaging combined with immunohistochemical staining. Biomedical Spectroscopy and Imaging, (2013)10.3233/bsi-130048).
- Meier, C. Peike, T. Kaltenbach, K.-A. Weiß, paper presented at the 28th European PV Solar Energy Conference, Paris, France, 2013.
- V. Carozo, B. Fragneaud, L. Gustavo Cançado, C. M. Almeida, Bede, P. M., B. S. Archanjo, C. Alberto Achete, The role of interference and polarization effects in the optical visualization of carbon nanotubes. Journal of Applied Physics 113, 084314 (2013)10.1063/1.4794007).
- K. B. Biggs, K. M. Balss, C. A. Maryanoff, Pore networks and polymer rearrangement on a drug-eluting stent as revealed by correlated confocal Raman and atomic force microscopy. Langmuir 28, 8238-8243 (2012); (10.1021/la300808z).
- J. Y. Chou, J. L. Lensch-Falk, E. R. Hemesath, L. J. Lauhon, Vanadium oxide nanowire phase and orientation analyzed by Raman spectroscopy. Journal of Applied Physics 105, 034310 (2009)10.1063/1.3075763).
- D. L. Wetzel, Y.-C. Shi, U. Schmidt, Confocal Raman and AFM imaging of individual granules of octenyl succinate modified and natural waxy maize starch. Vibrational Spectroscopy 53, 173-177 (2010)10.1016/j.vibspec.2010.03.011).
- X. Zhao, N. O. Petersen, Z. Ding, Comparison study of live cells by atomic force microscopy , confocal microscopy , and scanning electrochemical microscopy. Canadian Journal of Chemistry 183, 175-183 (2007)10.1139/V07-007).

Combinations:

apyron for Raman-AFM combinations

alpha 300 RA for Raman-AFM combinations

alpha300 RAS for Raman-AFM-SNOM combinations

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