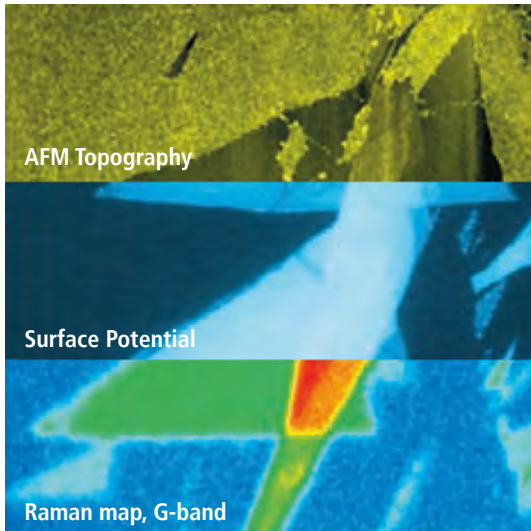




***AFM - Raman - SNOM - TERS***

Graphene flakes on gold, 30 x 30  $\mu\text{m}$



# NTEGRA SPECTRA

Atomic Force Microscopy  
Confocal Raman / Fluorescence Microscopy  
Scanning Near-Field Optical Microscopy  
Tip Enhanced Raman Scattering



***Your AFM & Raman Company***

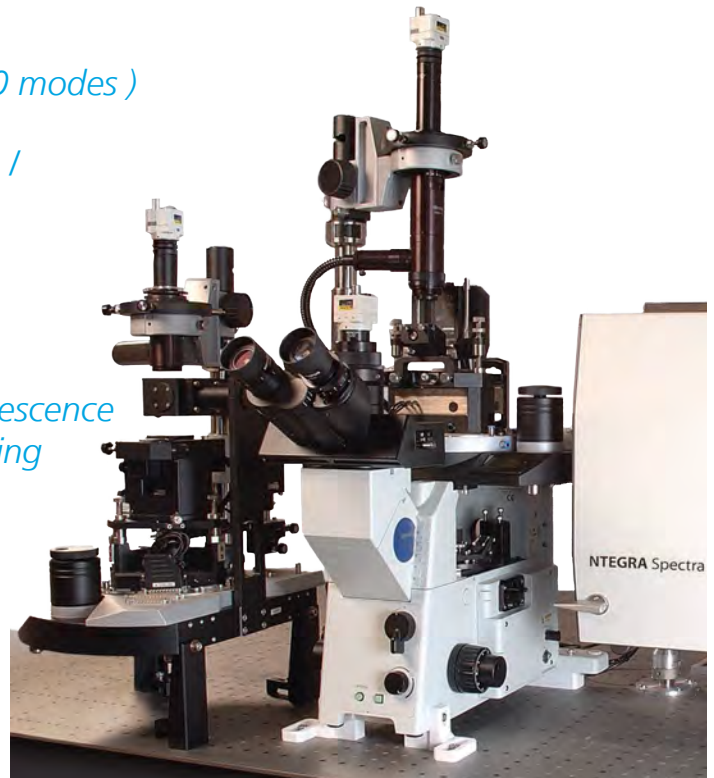
# AFM - Raman - SNOM - TERS

*Atomic Force Microscopy (> 30 modes)*

*Confocal Raman / Fluorescence /  
Rayleigh Microscopy*

*Scanning Near-Field Optical  
Microscopy (SNOM)*

*Tip Enhanced Raman and Fluorescence  
(TERS, TEFS, TERFS) and scattering  
SNOM (s-SNOM)*



*Configuration with NT-MDT confocal spectrometer*

## ■ *NTEGRA Spectra, the most versatile, fully integrated AFM- Raman-SNOM-TERS instrument*

Since 1998 NT-MDT has been integrating its open, modular AFM with various optical microscopy and spectroscopy techniques. Professional upright and inverted optical microscopes, confocal microscopes, Raman spectrometers, fluorescence, lifetime imaging microscopes – this is only a partial list of optical devices that have been fully integrated with NT-MDT AFM.

More than 30 general and advanced AFM modes are supported by the NT-MDT AFM providing extensive information about the sample's physical properties. **Simultaneous** optical measurements of the same sample area provide the widest range of additional information about the sample – thus combining the best of all techniques.

Integration of AFM with confocal Raman/fluorescence microscopy is of a special interest. Simultaneously measured AFM and Raman maps of exactly the same sample area provide complementary information about sample physical properties (AFM) and chemical composition (Raman).

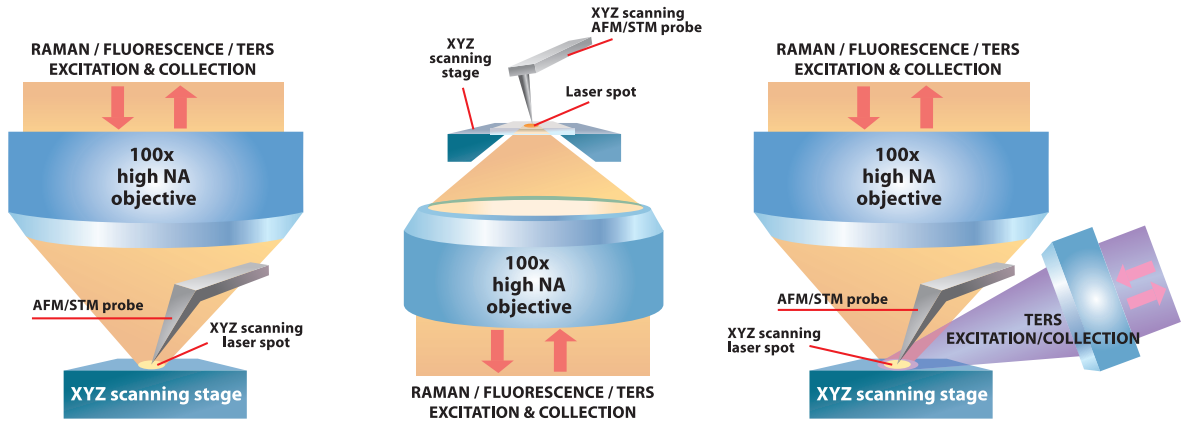
Specially prepared AFM probes (nanoantennas) can be used to enhance and localize light at the nanometer scale area near the end of the tip. Such nanoantennas act as a “nano-source” of light giving possibility of optical imaging (Raman, fluorescence etc) with nanometer scale resolution. Tip Enhanced Raman Spectroscopy (TERS) maps with spatial resolution reaching down to 10nm have been successfully obtained and reported using NT-MDT systems.

Scanning near-field optical microscopy (SNOM) is another approach to obtain optical images of optically active samples with resolution below diffraction limit.

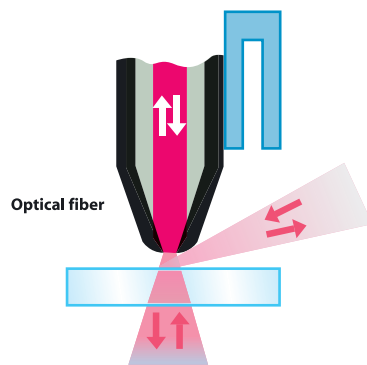
With hundreds systems installed worldwide, the constantly developing and improving NTEGRA Spectra platform has become the best-selling device in the AFM-Raman-SNOM-TERS market. The instrument has been awarded the R&D 100 award.



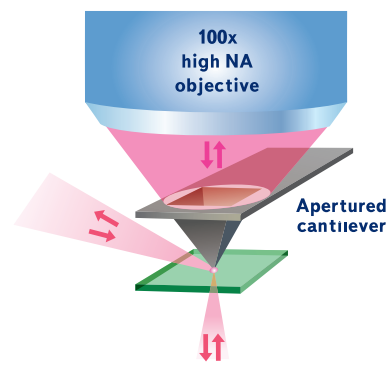
## Solution for all possible excitation / detection and TERS geometries



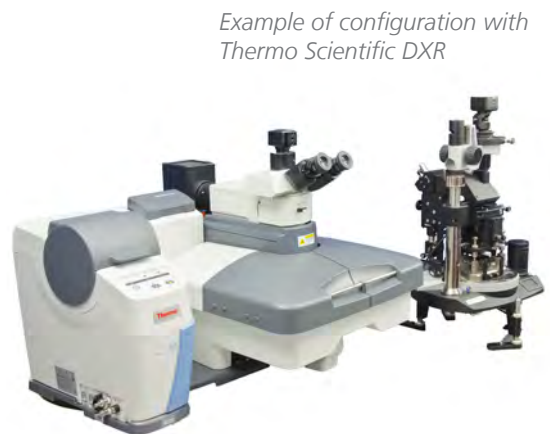
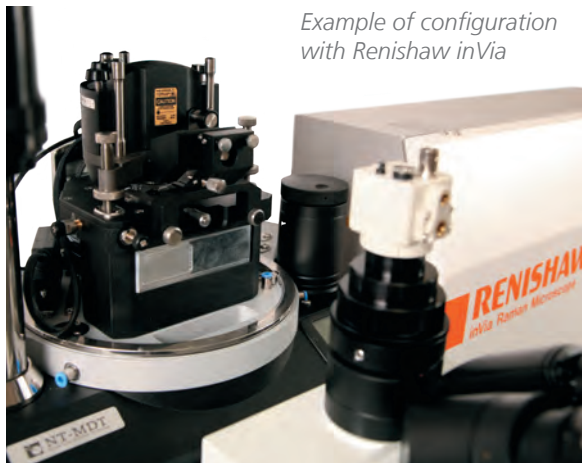
## Scanning Near-Field Optical Microscopy



Based on quartz SNOM fiber, shear-force feedback



Based on silicon cantilevers with nanofabricated aperture



### Inverted setup:

- Optimized for transparent samples
- Highest optical resolution achievable (<200 nm) **simultaneously** with AFM
- Highest efficiency of Raman/fluorescence photon collection (with immersion optics) **simultaneously** with AFM
- Probe scanning in addition to sample scanning (important for TERS)
- Equipped with heating stage, temperature controlled liquid cell and environmental chamber
- Fits most commercial inverted microscopes, supporting advanced imaging modes

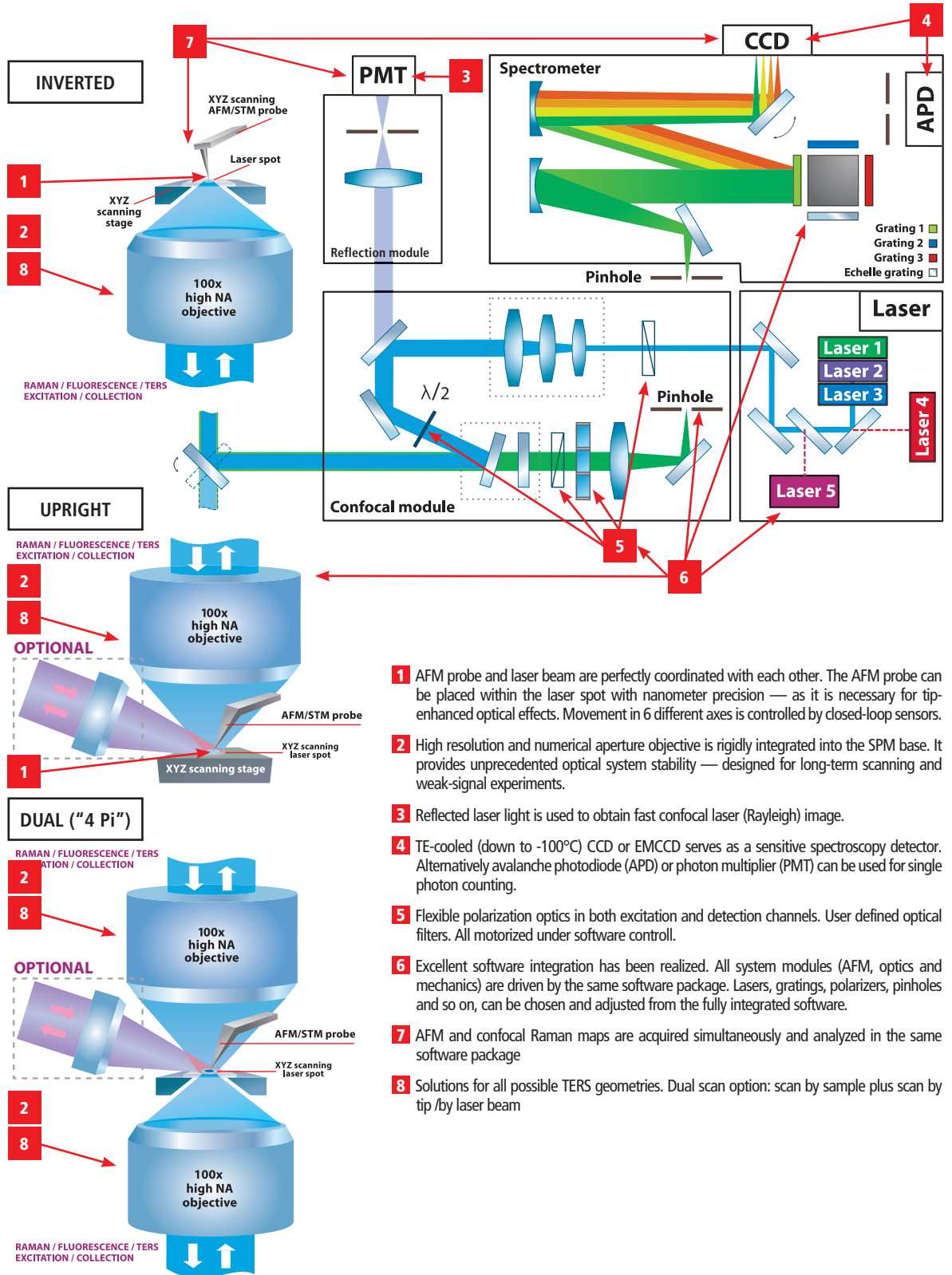
### Upright setup:

- Optimized for opaque samples
- Highest optical resolution (280–400 nm) **simultaneously** with AFM
- Highest efficiency of Raman/Fluorescence photon collection **simultaneously** with AFM
- Beam scanning in addition to sample scanning (necessary for TERS)
- Equipped with heating stage, environmental chamber

Work both with cantilevers (contact, intermittent contact and other modes: more than 30) and with metal tips (STM mode, shear force mode, normal force mode)



# AFM / Confocal Raman & fluorescence/SNOM/TERS optical scheme\*



- 1 AFM probe and laser beam are perfectly coordinated with each other. The AFM probe can be placed within the laser spot with nanometer precision — as it is necessary for tip-enhanced optical effects. Movement in 6 different axes is controlled by closed-loop sensors.
- 2 High resolution and numerical aperture objective is rigidly integrated into the SPM base. It provides unprecedented optical system stability — designed for long-term scanning and weak-signal experiments.
- 3 Reflected laser light is used to obtain fast confocal laser (Rayleigh) image.
- 4 TE-cooled (down to  $-100^{\circ}\text{C}$ ) CCD or EMCCD serves as a sensitive spectroscopy detector. Alternatively avalanche photodiode (APD) or photon multiplier (PMT) can be used for single photon counting.
- 5 Flexible polarization optics in both excitation and detection channels. User defined optical filters. All motorized under software control.
- 6 Excellent software integration has been realized. All system modules (AFM, optics and mechanics) are driven by the same software package. Lasers, gratings, polarizers, pinholes and so on, can be chosen and adjusted from the fully integrated software.
- 7 AFM and confocal Raman maps are acquired simultaneously and analyzed in the same software package
- 8 Solutions for all possible TERS geometries. Dual scan option: scan by sample plus scan by tip /by laser beam

## Modes:

- AFM (mechanical, electrical, magnetic properties, nanomanipulation etc.)
- White Light Microscopy and Confocal Laser (Rayleigh) Imaging
- Confocal Raman Imaging and Spectroscopy
- Confocal Fluorescence Imaging and Spectroscopy
- Scanning Near-Field Optical Microscopy (SNOM)
- Tip Enhanced Raman and Fluorescence Microscopy (TERS, TEFS, TERFS)

## Controlled Environment:

- Temperature
- Humidity
- Gases
- Liquid
- Electrochemical environment
- External magnetic field

\* Corresponds to NT-MDT confocal spectrometer

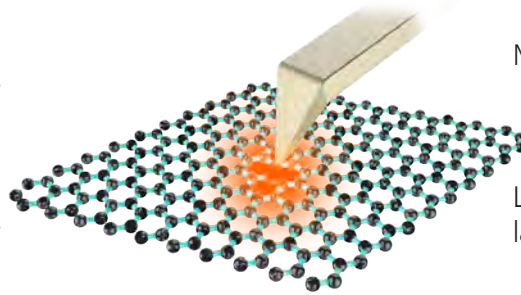
Atomic force microscopy: mechanical, electrical, magnetic properties and nanomanipulations

Confocal fluorescence: imaging and spectroscopy

Near-field optical microscopy

Confocal Raman: imaging and spectroscopy

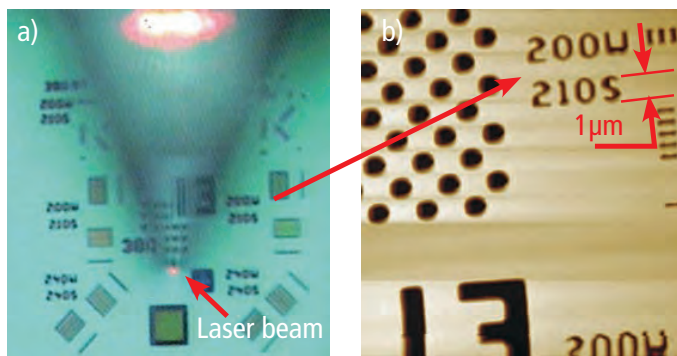
Light microscopy and reflected laser confocal imaging



Tip enhanced Raman and fluorescence microscopy

All techniques can be applied to the same sample

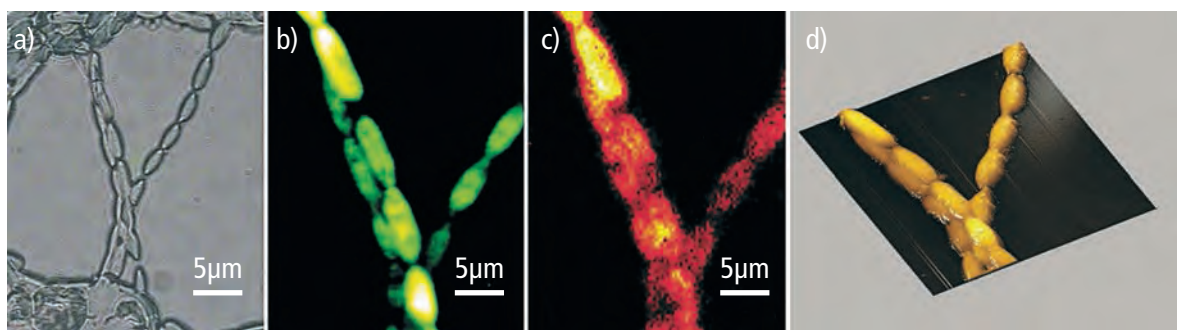
### ■ AFM working simultaneously with 400nm resolution upright optics



"AFM + confocal microscope" with high magnification optics in upright configuration. Note extremely high imaging resolution of 100x objective as seen on 1µm height characters on Si substrate a) Due to the high numerical aperture (0.7) of the objective, opaque silicon AFM probe looks "transparent" on the image. The very end of the

tip can be seen. AFM scanning b) can be obtained simultaneously with both white light and confocal Raman/fluorescence imaging. Thanks to the additional beam scanning option, a tightly focused laser spot can be positioned exactly at the apex of the AFM probe — as required for TERS experiments.

### ■ Comprehensive analysis of biological structures



Algal cells visualization by different techniques. a) Bright field overview. b) Confocal Raman map at 1524 cm<sup>-1</sup> (beta-carotene line). c) Confocal image of autofluorescence at 492–513 nm. d) AFM image.

Data from Don McNaughton, Monash University, Australia) and Pavel Dorozhkin, NT-MDT



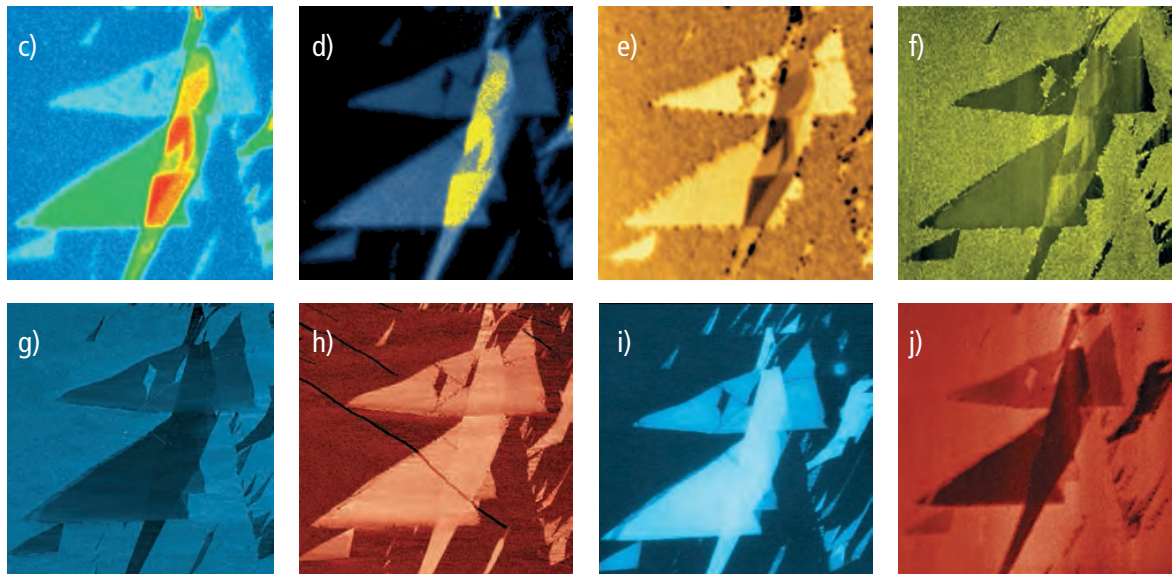
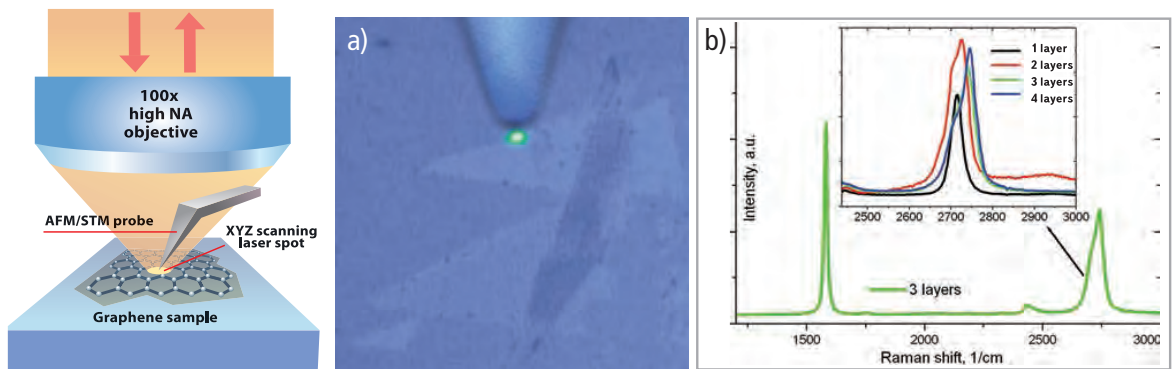
■ *Graphene studied by various optical, AFM and spectroscopy techniques*

Combination of AFM, confocal Raman / Fluorescence / Rayleigh microscopy and Scanning Near-Field Optical Microscopy provides unique opportunities for graphene investigation. Different AFM techniques allow studying of mechanical, electrical, magnetic and even elastic properties of graphene flakes. Studies of local work function, conductivity, capacitance, piezoresponse and many other surface properties are available. At the same time, Raman microscopy (available simultaneously with AFM) provides information about flake

thickness, structural uniformity, presence of impurities and defects etc. Additionally, Rayleigh imaging and SNOM measure local optical properties of the sample providing further information about flake structure.

Importantly, most of the measurements can be performed under environmental control: at variable humidity and temperature, in controlled atmosphere, in liquid and even (in some configurations) in electrochemical environment and with external magnetic field.

**RAMAN / FLUORESCENCE / TERS  
EXCITATION & COLLECTION**



a) White light image of graphene flakes with AFM tip and Raman laser spot  
 b) Raman spectra of flakes with different thickness  
 c) Raman map: G-band intensity  
 d) Raman map: 2D (G') band mass center  
 e) Rayleigh light intensity

f) AFM: Height (topography)  
 g) AFM: Lateral force (friction)  
 h) AFM: Force modulation (elastic properties)  
 i) AFM: Kelvin probe (surface potential)  
 j) AFM: Electrostatic force (charge distribution)

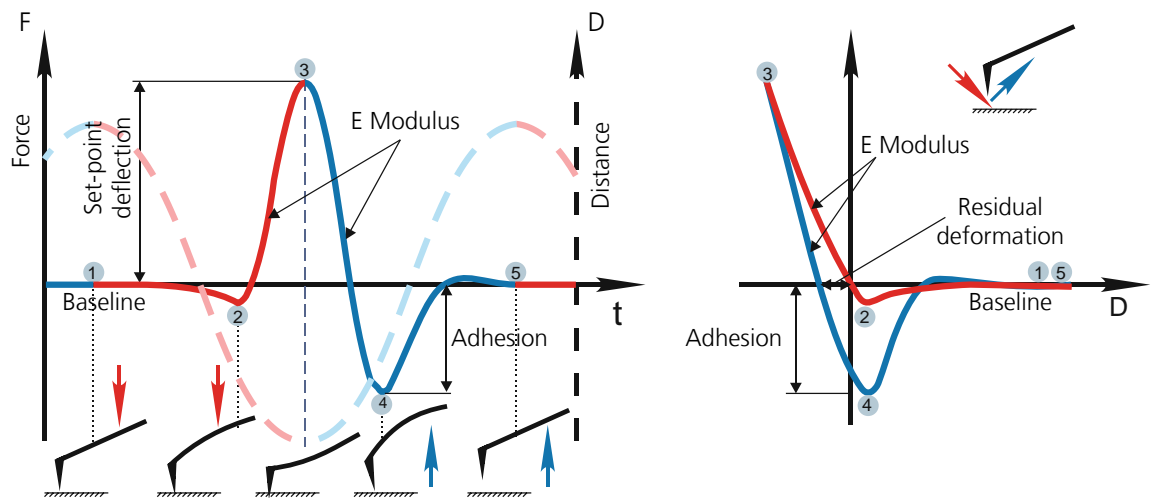
# HybriD-Mode™

## ■ HD-AFM™ for nanomechanical properties complemented with Raman for chemical imaging

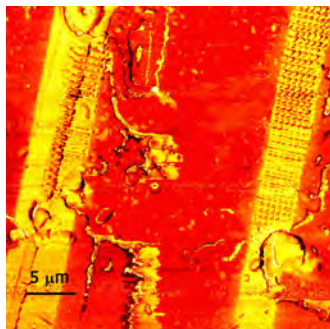
In HybriD Mode™ the tip-sample distance is modulated according to the quasi-harmonic law. Thus tip enters a force interaction with the sample thousands of times per second. Force-distance curve analysis enables maps of topographical, mechanical and electrical properties of the sample to be extracted with high spatial resolution.

High-performance electronic components and unique algorithms provide superb level of real-time signal processing and analysis.

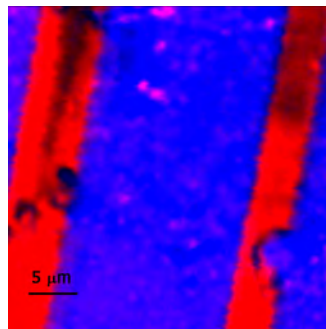
HybriD Mode™ provides a wealth of data within a single experiment cycle, eliminates lateral forces, and provides high stability for long-term experiments.



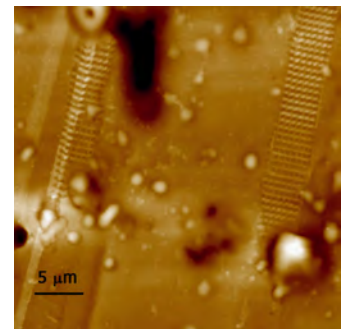
Ntegra Spectra equipped with the new electronics and software allows HD-AFM and Raman imaging of exactly the same area within single measurement session.



Stiffness map of HDPE/LDPE polymer sandwich cut by microtome



Overlap of Raman maps: HDPE (red), LDPE (blue)

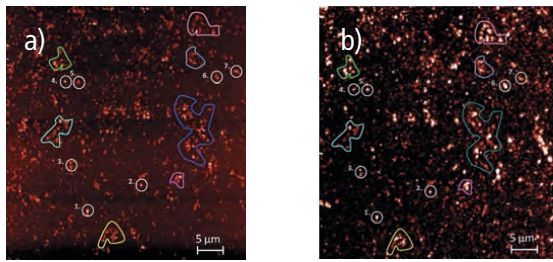


AFM topography

Data from M. Yanul, S. Magonov, P. Dorozhkin, NT-MDT.



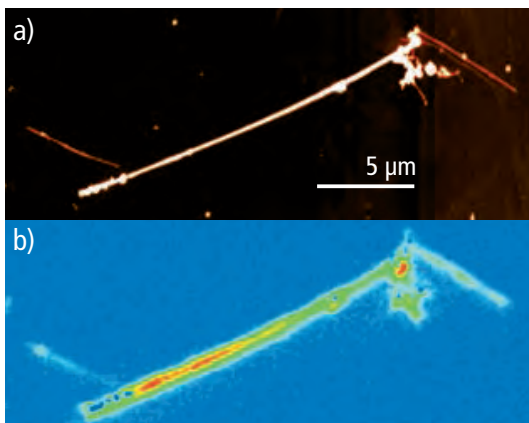
■ Nitrogen-vacancy (NV) color centers in nanodiamonds



Observation of nitrogen-vacancy (NV) color centers in discrete detonation nanodiamonds. a) AFM topography image; smallest particles observed are discrete isolated nanodiamonds of  $\sim 5\text{nm}$  size. b) Confocal fluorescence map of the same sample area; nitrogen-vacancy luminescence from isolated nanodiamonds is clearly seen.

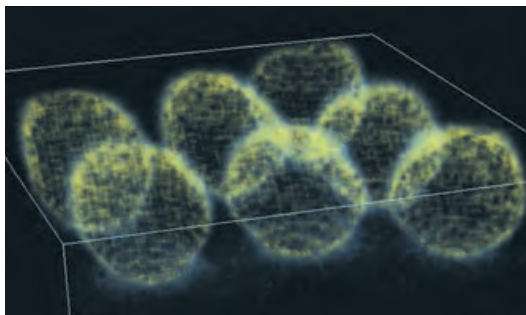
*A/Prof. James Rabeau, Quantum Materials and Applications group, Department of Physics and Astronomy, Macquarie University (Sydney, Australia). For more details see: C. Bradac et al., Nature Nanotechnology 5, 345 - 349 (2010)*

■ More applications

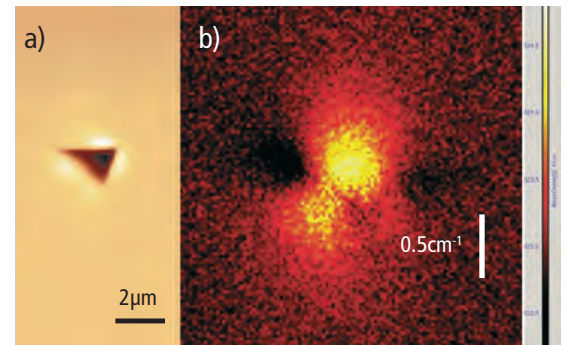


a) AFM topography and b) confocal Raman map (spectral shift of  $520\text{cm}^{-1}$  Si band) of individual silicon nanowire

*Data from P. Dorozhkin, NT-MDT, M. Bloomfield, Renishaw*

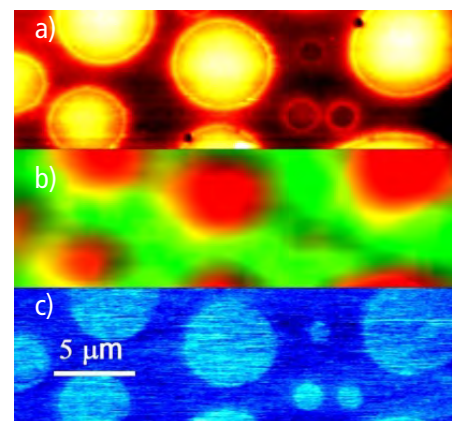


■ Stress mapping in silicon structures



a) AFM topography of indentation in silicon substrate. b) Center of mass shift of  $520\text{cm}^{-1}$  silicon Raman band is showing stress distribution around the indentation. Spectral resolution is better than  $0.1\text{cm}^{-1}$

*Data from S. Timofeev, S. Leesment, A. Shelaev, NT-MDT*



a) AFM topography of PS-PVAC polymer blend thin film; b) Overlap of Raman maps, PS (green) and PVAC (red), c) Surface potential by KFM.

*Data from A. Shelaev, S. Mitko, S. Magonov, P. Dorozhkin, NT-MDT*

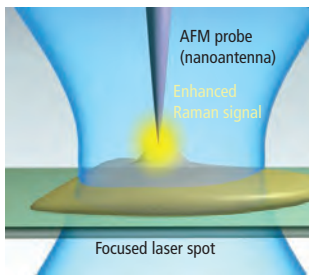
3D confocal Raman image of polystyrene microspheres. Scan size:  $10 \times 10 \times 14 \mu\text{m}$ . Full Raman spectrum was recorded in each point of 3D map, further software analysis allowed to build 3D Raman maps based on any selected Raman band.

*Data from S. Timofeev, NT-MDT*



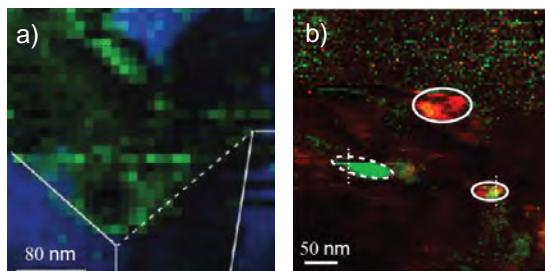
# Tip Enhanced Raman Scattering (TERS)

- **Tip Enhanced Raman Scattering (TERS).** Chemical (spectroscopic) imaging with ultra high sensitivity and spatial resolution down to 10 nm.



A specially prepared AFM probe (usually metal coated cantilever or etched metal wire) acts as a “nanoantenna” localizing and enhancing excitation laser light near the apex. The nanoantenna effectively performs as a “nano-source” of light. Scanning a sample across the nanoantenna results in spectroscopic imaging of the sample (Raman scattering / TERS, fluorescence etc.) with spatial resolution down to 10nm - ~30 times below diffraction limit.

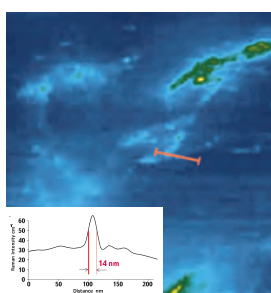
## ■ Graphene



- a) TERS maps of single layer CVD Graphene on copper substrate. Green color: areas of pristine graphene (2D band intensity). Blue color: CH-terminated graphene areas (CH-bands intensity).
- b) TERS map of mechanically exfoliated single layer graphene on Au substrate. Green color: 2D band intensity. Red color: D-band intensity (areas with strong defects). **Spatial resolution of all nano-Raman (TERS) maps is <12 nm**

J. Stadler, T.Schmid, and R. Zenobi, *Nano Letters* (2010), 10, 4514-4520

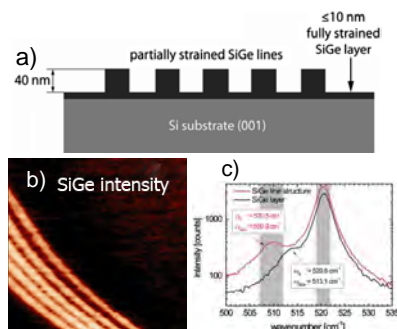
## ■ Carbon Nanotubes



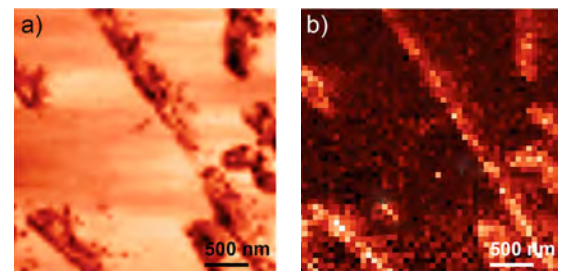
Nano-Raman” (TERS) map of nanotube bundle aggregate (G-band intensity). **Spatial resolution of TERS map is <14 nm**

A. Chan & S. Kazarian, *Nanotechnology* 21, 445704 (2010)

## ■ Semiconductors



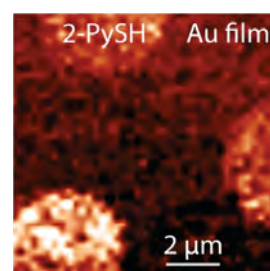
## ■ Peptide Nanotapes



- a) STM image of individual self-assembled peptide nanotapes.
- b) TERS map of the aromatic ring marker band. **Spatial resolution of nano-Raman (TERS) map is <80 nm. Sensitivity: individual peptide nanotape**

Melissa Paulite, Carolin Blum, Thomas Schmid, Lothar Opilik, Klaus Eyer, Gilbert C. Walker, and Renato Zenobi, *ACS Nano*, 2013, 7 (2), pp 911–920

## ■ Thiol monolayer



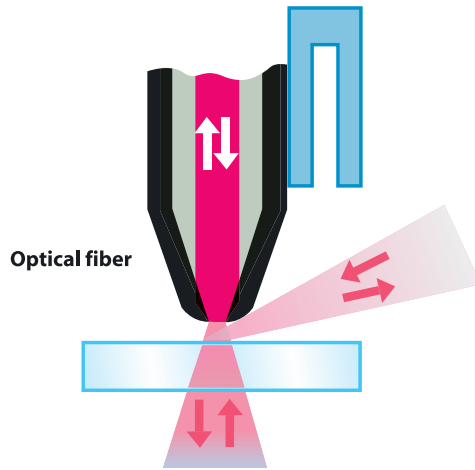
TERS map of two isometric thiols in a self-assembled monolayer (SAM) on a gold surface. SAM pattern was produced by micro-contact printing. **Sensitivity of Raman (TERS): single monolayer**

J. Stadler, T.Schmid, L. Opilik, P. Kuhn, P.S. Dittrich, and R. Zenobi, *Beilstein J. Nanotechnology* (2011), 2: 509-515

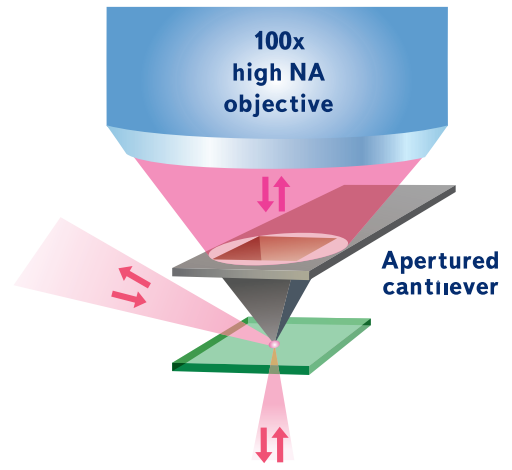
- a) Periodic SiGe line structure on Si substructure. b) TERS map of SiGe band intensity. c) Comparison of TERS spectra from a SiGe line (red) and the remain thin SiGe area a few um away from the line structure. Peak position of SiGe band shifts depending on the material stress. **Spatial resolution of nano-Raman (TERS) map is <50 nm**

P. Hermann, M.Hecker, D. Chumakov, M. Weisheit, J. Rinderknecht, A. Shelaev, P. Dorozhkin, L. M. Eng, *Ultramicroscopy* 111(2011) 1630-1635

# Scanning Near-field Optical Microscopy (SNOM)

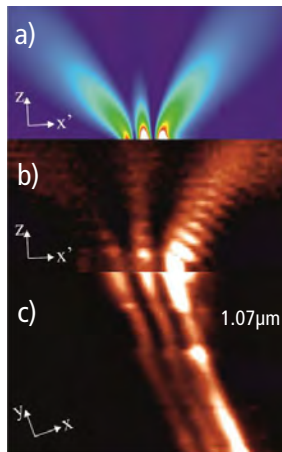


Based on quartz SNOM fiber, shear-force feedback



Based on silicon cantilevers with nanofabricated aperture

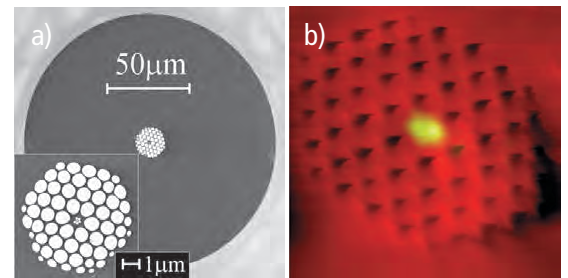
## Lasers



a) Simulation of XZ intensity distribution of the light emitted by  $1.07\mu\text{m}$  laser diode. b) XZ intensity distribution measured by cantilever based SNOM, c) XY intensity distribution of light emission measured at the laser surface in SNOM collection mode.

Data from A. Shelaev, M. Yanul, P. Dorozhkin, NT-MDT; A. Ankudinov, S. Slipchenko, A. Podoskin, I. Tarasov, Ioffe Physical Technical Institute.

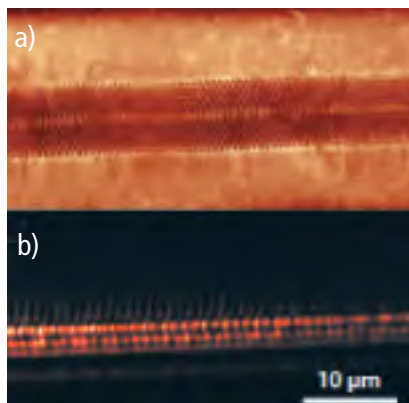
## Optical fibers with photonic crystals



a) SEM image of the optical fiber cross-section, showing photonic crystal structure in the fiber core. b) Overlay of topography map (red palette) and light intensity (SNOM collection) image (green palette) taken from the fiber section. Light propagating in the fiber is perfectly localized in the center of the photonic crystal structure.

Data from Yinlan Ruan, Heike Ebendorff-Heidepriem, Tanya M. Monro. Centre of Expertise in Photonics, School of Chemistry & Physics, University of Adelaide, and S. Shikin, P. Dorozhkin, NT-MDT

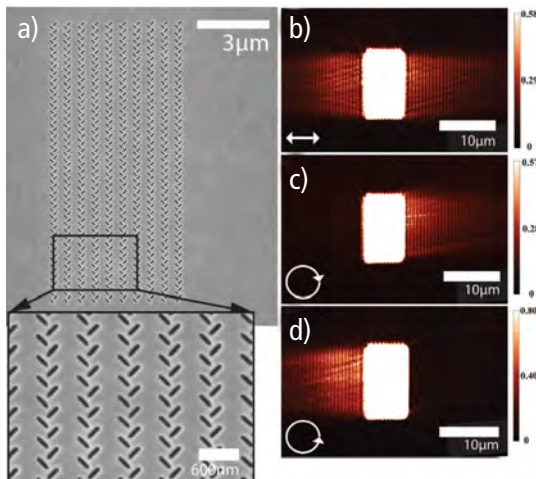
## Photonic crystals



Light propagation in a one-line-defect photonic crystal (PhC) waveguide patterned into a 450nm thick free-standing lithium niobate membrane. SNOM topography a) and optical near-field b) images recorded above the surface of the PhC waveguide. The Bloch wave vectors of the PhC waveguide can be retrieved from optical near-field images.

R. Geiss, S. Diziain, N. Janunts, APPLIED PHYSICS LETTERS 97, 131109 (2010)

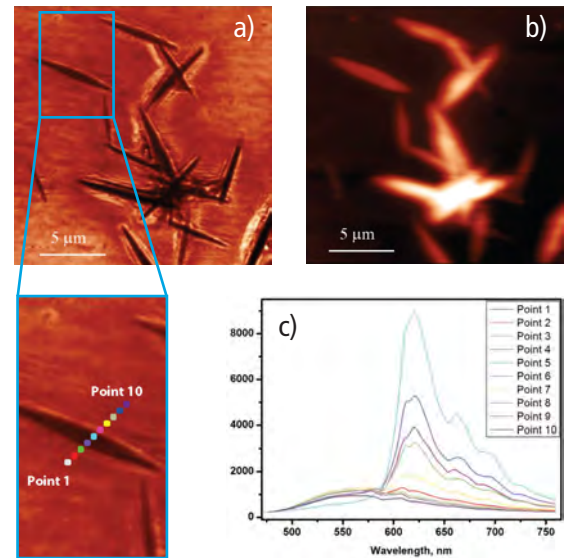
## ■ Plasmonics



Surface plasmon polaritons (SPP) formed by coupler structure. a) SEM image of a structure fabricated in a gold film for operation at  $L=633$  nm. SNOM images of the structure under illumination from the back by light with different polarization: b) linear, c) right circular, d) left circular.

Jiao Lin, J. P. Balthasar Mueller, Qian Wang, Guanghui Yuan, Nicholas Antoniou, Xiao-Cong Yuan, Federico Capasso, *SCIENCE*, Vol. 340, 331-334 (2013)

## ■ Polymer

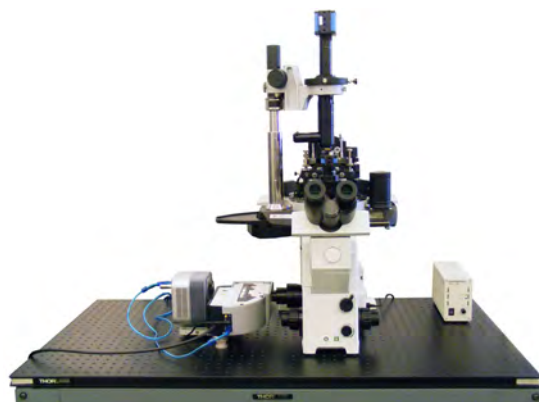


a) SNOM transmission image of Perylene embedded in Polyfluorene (PFO). b) AFM topography image, c) Fluorescent spectra for 10 points of section from SNOM image. The peak at 620 nm corresponds to beta-phase rhombus perylene.

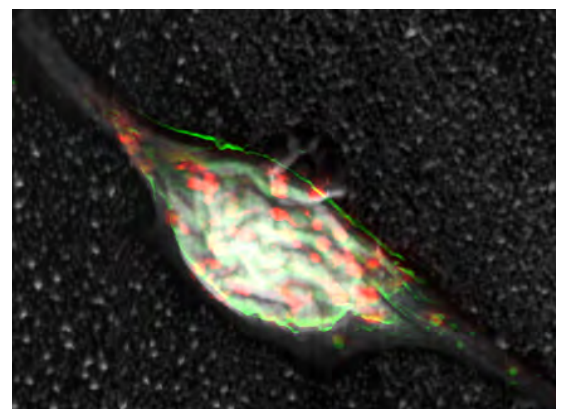
Data from E. Kuznetsov, S. Timofeev, P. Dorozhkin, NT-MDT; Xinping Zhang, Beijing University of Technology.

## ■ AFM integration with high speed confocal microscopy

Additional capabilities for studies of labeled biological samples arise from integration of AFM with high speed confocal microscopy. NT-MDT and ANDOR Technology introduce our new instrument with fully integrated multiple imaging capabilities. Taking advantage of a combination of NT-MDT AFM with the 3D high quality imaging of Andor DSD confocal unit, this instrument offers a new, exciting imaging solution. Complete software and hardware integration has been realized, making the instrument easy to use for biologists.



Integrated "AFM + Spinning Disc Confocal Microscope" instrument



Overlay of AFM topography (grey color) and confocal fluorescence images (red & green color) of a labeled cell.

Data from B. Combettes, ANDOR Technology; S. Lemesko, M. Yanul, NT-MDT.





# Specifications

## Confocal Raman / fluorescence microscopy

Confocal Raman / fluorescence / Rayleigh imaging runs simultaneously with AFM (during one sample scan)

Diffraction limited spatial resolution: <200nm in XY, <500nm in Z (with immersion objective)

True confocality; motorized confocal pinhole for optimal signal and confocality

Motorized variable beam expander / collimator: adjusts diameter and collimation of the laser beam individually for each laser and each objective used

Full 3D (XYZ) confocal imaging with powerful image analysis

Hyperspectral imaging (recording complete Raman spectrum in every point of 1D, 2D or 3D confocal scan) with further software analysis

Optical lithography (vector, raster)

## AFM / STM: Integration with spectroscopy

Upright and inverted optical AFM configurations (optimized for opaque and transparent samples correspondingly); side illumination option

Highest possible resolution (numerical aperture) optics is used simultaneously with AFM: 0.7 NA for Upright, 1.3–1.4 NA for Inverted

AFM/STM and confocal Raman / Fluorescence images are obtained simultaneously (during one scan)

All standard SPM imaging modes are supported (>30 modes) — combined with confocal Raman / Fluorescence

Low noise AFM / STM (atomic resolution)

Vibrations and thermal drifts originating from optical microscope body are minimized due to special design of optical AFM heads

Focus track feature: sample always stays in focus due to AFM Z-feedback; high quality confocal images of very rough or inclined samples can be obtained

## Software

Seamless integration of AFM and Raman; all AFM / Raman / SNOM experiment and further data analysis is performed in one and the same software

Powerful analysis of 1D, 2D and 3D hyperspectral images

Powerful export to other software (Excel, MatLab, Cytospec, etc.)

## Spectroscopy

Extremely high efficiency 520 mm length spectrometer with 4 motorized gratings

Visible, UV and IR spectral ranges available

Echelle grating with ultrahigh dispersion; spectral resolution: 0.007nm (< 0.1 cm<sup>-1</sup>)\*\*

Up to 3 different detectors can be installed:

- TE cooled (down to -100°C) CCD camera. EMCCD camera is optional — for ultrafast imaging
- Photon multiplier (PMT) or avalanche photodiode (APD) in photon counting mode
- Photon multiplier for fast confocal laser (Rayleigh) imaging

Flexible motorized polarization optics in excitation and detection channels, cross-polarized Raman measurements

Fully automated switching between different lasers — with a few mouse clicks

## Scanning Near Field Optical Microscopy (SNOM)

Two major SNOM techniques supported: (I) based on quartz fiber probes, (II) based on silicon cantilever probes

All modes are supported: Transmission, Collection, Reflection

All SNOM signals are detected: laser intensity, fluorescence intensity, spectroscopy

SNOM lithography (vector, raster)

## Optimized for Tip Enhanced Raman Scattering (TERS) and other tip-related optical techniques (S-SNOM, TEFS, STM-LE)

All existing TERS geometries are available: illumination/ collection from bottom, from top or from side

Different SPM techniques and TERS probes can be used: STM, AFM cantilever, quartz tuning fork in tapping and shear force modes

Dual scan (for Hot Point Mapping in TERS): scan by sample AND scan by tip / by laser spot

Motorized polarization optics to produce optimal polarization for TERS

■ AFM-Raman measurements can be run in air, controlled atmosphere or in liquid — all with variable temperature

Some features listed are optional — not included into basic system configuration

NT-MDT AFM can be integrated with NT-MDT, Renishaw inVia or Thermo Scientific DXR spectrometers. Specification is given for the former. Other specifications are available upon request.

\*\*Exact value of spectral resolution highly depends on how "resolution" is defined



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