

AFM-Raman, SNOM and TERS: Recent Advances and Applications

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Product Line

AFM

AFM-Raman / IR / TERS



Integration with Horiba Scientific spectrometers Sonora University,



Suzhou University, CAS



Integration with Thermo Fisher Scientific DXR spectrometer







Integration with Renishaw inVia spectrometer







NT-MDT + Renishaw integrated software

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NT-MDT + Renishaw data analysis software



Data is exported to Renishaw Wire 3.x



EXACTLY THE SAME DATA in two programs: NT-MDT & Wire-3

All scans can be seen and analyzed in any software: AFM (height, phase etc) & Raman maps. Data from NT-MDT Nova was exported into WIRE-3

Integration with NT-MDT spectrometers







NTEGRA Spectra II in Upright, Inverted and Side illumination configuration





AFM – Confocal Raman / Fluorescence – SNOM – TERS

Spectra optical scheme



Spectrum Instruments

Optical scheme of Spectrometer



• True confocal design. Motorized confocal pinhole.

- Diffraction limited resolution guaranteed (e.g. 200 nm for blue laser, immersion optics)
- •Extremely high optical throughput (~70-80 % for spectrometer, ~40-50% sample-to-detector)
- •Fully motorized laser change (up to 3 / 5 lasers). UV VIS IR region
- Fully motorized: polarization optics, zoom beam expander, pinhole, 4 gratings
- •Can be equipped by fastest and most sensitive detectors available (FI/BI CCD, EMCCD, DD-CCD etc.)
- •Zoom beam expander to guarantee diffraction limited laser spot to every objective
- Three optical ports for detectors: two in monochromator, one in separate channel



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Sensitivity: 4th order of Si Raman band is clearly resolved





Low wavenumbers Raman spectra



488 nm laser, 1800 lines/mm grating.



Defining number of 2H SnS2 layers



Optical access from Top, Bottom and Side

Access for **Mitutoyo** long working distance objective for top illumination

> Access for bottom Illumination objective





Excitation-collection configurations





Top optical access to the AFM probe with 100x objective



AFM probe over a structured Si substrate. View through 0.7NA 100x objective Apex of opaque Si tip looks transparent on the image! This unique observation is due to high aperture (0.7 NA) of the imaging objective



MoSe2 flakes



Spectrum Instruments

Wolfgang Mertin, Gerd Bacher, Artem Shelaev, Sergey Lemeshko, Universität Duisburg & NT-MDT

CdS-Polyaniline nanowires: AFM – Raman mapping



473 nm excitation.



KPFM-Raman Studies of Polymer Blends

Polymer Blend PS-PVAC: Thick Film on ITO glass













Cyanobacteria biofilm: AFM and Raman mapping



Combined study of cyanobacteria biofilm by means of atomic force microscopy and confocal Raman microscopy. AFM image in phase contrast (left) gives an image with nm resolution, however, does not contain any chemical information. Raman map (right) corresponds to the distribution of beta-carotene. Resolution Raman map is limited by the optical limit and is 400-500 nm. Beta-carotene is the pigment contained in cyanobacteria which perform photosynthesis. Overlay of two images (center) provides the chemical identity and relate it to the AFM image of high resolution.

Data courtesy: Thomas Schmid, Pawel L. Urban, Andrea Amantonico, Renato Zenobi ETH Zurich, Switzerland



Topography and FLIM image of e-coli

AFM Topography

Lifetime map 525-540 nm

Fluorescence 525-540 nm

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Topography (left) and FLIM mapping of 525-540 nm band (center) and fluorescence intensity (right). Decay curve on the bottom left image and fluorescence spectrum on the bottom right one. Different FLIM signals come from different fluorescent proteins, which produced by e-coli genetically modified in different ways. Spectrum shape is very similar. Intensity and lifetime is different. AFM + Spectrometer + FLIM provides sufficient information to identify different proteins in bacteria.

Biodegradation of carbon nanotubes



AFM image showing the capture of a single nanotube by living cell of immune system (neutrophils).

Raman spectra of neutrophil with IgG-nanotubes after 2 hours (b) and 8 hours (c). Reducing the intensity of G- and D-bands of carbon nanotube indicates biological degradation of single nanotubes. Thus, neutrophils were successfully processed and excreted objects such as carbon nanotubes

Y. Volkov et all // Carbon nanotubes degraded by neutrophil myeloperoxidase induce less pulmonary inflammation, Nature Nanotechnology, 2010



DLC Protective Layer of Hard Disk Drive



AFM topography Nearly parallel scratches in DLC protective layer are produced by low-flying magnetic head. Bumps are signatures of erosion of Co magnetic layer. 40x40 µm



Raman map, sp3 (diamond-type) bonding fraction ωG = 1580.



Raman map, ID/IG ratio Increased fraction of sp2 bonds and defects.





Simultaneous PFM and Raman mapping



PFM mapping and Raman mapping of 553-599 1/cm band Lithium Niobate. Changes of Raman bands intensities appears on the border of domains

> 100x0,7 objective used. ~8 mW of 473 nm laser used. Exposure time 1 s/points and 50x50 points scan size.



4000

2000

500



L. Eng, A. Shelaev & NT-MDT

Photocurrent mapping under localized optical excitation



Spectrum Instruments

Martin Ledinský, Antonín Fejfar, Aliaksei Vetushka and Artem Shelaev

Solar cell diagnostics by combination of Kelvin probe microscopy with local photoexcitation

	Ga _{0.99} In _{0.01} As-n ⁺	500 nm
	Al _{0.51} In _{0.49} P-n	30 nm
2	Ga _{0.51} In _{0.49} P-n	50 nm
3-	Ga _{0.51} In _{0.49} P-p	680 nm
	Al _{0.25} Ga ₀₂₅ In ₀₅ P-p	50 nm
	Al _{0.4} Ga _{0.6} As-p ⁺⁺	15 nm
	GaAs-n ⁺⁺	15 nm
	Al _{0.51} In _{0.49} P-n	30 nm
	Ga _{0.51} In _{0.49} P-n	100 nm
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	Ga _{0.51} In _{0.49} P-p	100 nm
	Al _{0.25} Ga ₀₂₅ In ₀₅ P-p	30 nm
	Al _{0.4} Ga _{0.6} As-p ⁺⁺	30 nm
	GaAs-n ⁺⁺	30 nm
	Al _{0.53} In _{0.47} P-n	50 nm
	Ga _{0.99} In _{0.01} As-n	1000 nm
	Ga _{0.53} In _{0.47} P-n	100 nm
1-+	Ge-substrate (n doped)	~ 300 nm
1-	Ge-substrate (p	doped)

Multijunction solar cell structure. Digits 1,2,3 show p-n junctions. Blue: n-type layers, pink: p-type layers, yellow: highly conductive layers



Individual p-n junction is locally excited by a 400 nm laser spot. Variation of surface potential is measured by cantilever (Kelvin prove microscopy)



A. Ankoudinov, A. Shelaev Ioffe Institute & NT-MDT

Solar cell diagnostics by combination of Kelvin probe microscopy with local photoexcitation





Surface potential variation for local excitation by 473 nm laser (left row: a,b,c) and 785 nm laser (right row: d,e,f).

Different individual p-n junctions are locally excited.

Experimental results correspond well to numerical simulation

A. Ankoudinov, A. Shelaev loffe Institute & NT-MDT



Nitrogen vacancy 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 ~5 nm size. (b) Confocal luminescence map of the same sample area; nitrogen-vacancy luminescence from isolated nanodiamonds is clearly seen. (c) Luminescence spectrum of individual NV center in a 5 nm crystal host.

C. Bradac et al., Nature Nanotechnology 5, 345 - 349 (2010)



Focus track feature of integrated AFM - confocal Raman/fluorescence instrument



(a)Integrated AFM-Raman instrument and its "focus track" feature. Sample surface always stays in focus due to AFM feedback mechanism. This provides true information about sample chemical composition even for very rough surfaces.

(b) Standard confocal Raman/fluorescence imaging – sample is scanned in X&Y directions; Sample gets out of focus, providing incorrect data about optical properties of the surface.



Focus track feature of integrated AFM - confocal Raman/fluorescence instrument





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C. Bradac et al., Nature Nanotechnology 5, 345 - 349 (2010)



Graphene, AFM + Confocal Raman



Real time simulation. Scanning area of 100x100 pixels, 50 sec scanning time



Graphene, AFM + Confocal Raman

1 layer

2 layers



Lateral Force Microscopy (friction)



Capacitance Microscopy



Raman Map, 2D Band position



Electrostatic Force Microscopy (charge distribution)



AFM Topography Size: 30*30 μm



Confocal Rayleigh Microscopy



Force Modulation Microscopy (elasticity)



Scanning Kelvin Probe Microscopy (surface potential)



Raman Map, G-band Intensity



Resolution and capabilities of different techniques





Optical techniques (color imaging, physical & chemical analysis)

Scanning probe microscopy (topography, mechanical, electrical, magnetic and other properties of the surface)

AFM (STM) + Optical techniques = Dramatic increase of resolution and sensitivity



Super-resolution imaging using scanning optical antennas





Optical antenna: a device designed to efficiently convert free-propagating optical radiation to localized energy, and vice versa.

- L. Novotny, N. van Hulst, Nature photonics 5, 89 (2011)
- P. Bharadwai, B. Deutch, L. Novotny, Adv. In Opt. Phot. 1, 438 (2009)
- Pohl D. W., Optics, Principles and Applications (World Scientific, 2000).



Localized surface plasmon resonance in metal nanoparticles (0D geometry)



$$\alpha = 4\pi R^3 \frac{\varepsilon(\omega) - \varepsilon_d}{\varepsilon(\omega) + 2\varepsilon_d}$$

Nanoparticle polarizability

Resonant interaction with light at:

 $\mathcal{E}(\omega_{res}) = -2\mathcal{E}_d$ (Fröhlich mode)

metal spherical nanoparticle in dielectric medium Drude model:

$$\mathcal{E}'(\omega) = 1 - \omega_p^2 / \omega^2$$

$$\omega_{pe} = \sqrt{\frac{4\pi n_e e^2}{m}} \quad \omega_{res} = \omega_p / \sqrt{3}$$



Localized EM field in macroobjects



Video of plasma created by EM irradiation of grapes in a commercial microwave oven in the traditional manner.



Proceedings of the National Academy of Sciences Mar 2019, 116 (10) 4000-4005; DOI:10.1073/pnas.1818350116



TERS: Importance of light polarization

"Z-polarized" light (with electrical field polarized along the tip axis) light experiences the largest enhancement at the tip apex



Fig. 1 Calculated field distribution at a sharp Au tip with a diameter of 5 nm. (a) Field distribution for an incident electric field vector parallel to the tip shaft showing localization of the electric field at the tip apex. (b) Field distribution for an incident electric field orientated nonparallel to the tip shaft. The field is no longer confined to the tip apex.

Taken from: N. Anderson, A. Hartschuh, L. Novotny, Materials Today (2005)



TERS enhancement in different geometries (1D, 2D, 3D)



Far field to near field volume ratio decreases with decreasing dimension – lower dimensions are more advantageous for TERS



TERS enhancement factor as function of tip-sample distance Fullerene thin film

Laser: 633 nm Tip: Au etched wire & Au coated cantilever Mode: Shear force & non-contact mode

Enhancement factor: ~ 5–10x



Data courtesy of S. Kharintsev, J. Loos, G. Hoffman, G. de With, TUE, the Netherlands



TERS on carbon nanotubes All data – using NT-MDT instrument

Tip Enhanced Raman Scattering ("nano-Raman") imaging

NT-MDT



TERS on carbon nanotubes All data – using NT-MDT instrument



M. Salakhov J. Phys. D: Appl. Phys. 46

(2013) 145501

175701 (2011)

M. Zhang, J. Wang, Q. Tian, Optics Communications 315, 164 (2014)



TERS spectroscopy of single DNA molecules

modifying freshly cleaved mica with Mg2+

modifying freshly cleaved mica with 3-aminopropyltriethoxysilane



Lipiec, E., Japaridze, A., Szczerbiński, J., Dietler, G. & Zenobi, R. Preparation of Well-Defined DNA Samples for Reproducible Nanospectroscopic Measurements. *Small* **12**, 4821–4829 (2016).



TERS spectra and images of mono/bilayer WS₂ flake



C. Lee *et al.*, "Tip-Enhanced Raman Scattering Imaging of Two-Dimensional Tungsten Disulfide with Optimized Tip Fabrication Process," *Sci. Rep.*, vol. 7, no. September 2016, p. 40810, Jan. 2017.



STM TERS of monolayer WS₂



Lee, C. *et al.* Unveiling Defect-Related Raman Mode of Monolayer WS2 via Tip-Enhanced Resonance Raman Scattering. *ACS Nano* **12**, 9982–9990 (2018).



Hybrid mode of feed back loop





TERS on Graphene Oxide AFM TERS cantilevers, HYBRID regime









Typical TERS resolution with AFM TERS probes: $\sim 20 - 40$ nm.



TERS map of single defects in Graphene oxide (GO). HybriD mode.



TERS of single defects in GO flakes. Distance between point 1 and 2 is ~25 nm. Spectra from 1 and 2 points are shown on the graph



TERS ("nano-Raman") on periodic Si-Ge structure resolution ~50 nm



P.Hermann, M. Hecker, D. Chumakov, M. Weisheit, J. Rinderknecht, A.Shelaev, P. Dorozhkin, L.M.Eng, Fraunhofer CNT & AMD, Dresden; NT-MDT. Ultramicroscopy 111, 1630 (2011)



TERS on periodic Thiols structure as a reference sample resolution ~10 nm



Sacco, A., Imbraguglio, D., Giovannozzi, A. M., Portesi, C. & Rossi, A. M. Development of a candidate reference sample for the characterization of tip-enhanced Raman spectroscopy spatial resolution. *RSC Adv.* **8**, 27863–27869 (2018)

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TERS in the liquid environment, HD mode



In the experiment, in an optical AFM head a polarized exciting laser (λ exc = 632.8 nm) was focused on the tip of the AFM TERS probe through a 60× immersion objective (working length: 2 mm) with a high numerical aperture (N.A. = 1). The enhancement of Raman scattering signal was about 200x **NT-MD**

Spectrum Instruments

Tip Enhanced Raman Scattering

Various types samples. Proven by multiple publications by NT-MDT customers.



Carbon nanotubes Resolution: ~10 nm Nanotechnology, 2011 & ~10 other papers



DNA molecule Resolution: ~15 nm Ang. Chem. Int., 2014, E. Lipiec et. al



Si/SiGe structures Resolution: <50 nm Ultramicroscopy, 2011 P. Hermann et al.



Graphene Resolution: ~12 nm ACS Nano, 2011 R. Zenobi et. al.



Graphene Oxide Resolution: ~15 nm A. Shelaev, et. al., 2014



Thiol monolayers Resolution: ~10 nm Beilstein J. Nano, 2011 R. Zenobi et. al.

Laser on tip Laser on background (C)

Thin molecular layers Resolution: ~15 nm NanoLett., 2010 R. Zenobi et. al.



Amyloid fibrils Resolution: ~50 nm Plasmonics, 2012 E. Di Fabrizio et. al.



Peptide nanotapes Resolution: ~50 nm ACS Nano, 2013 R. Zenobi et. al.



Polymers Resolution: ~50 nm Macromol., 2011 G. Hoffmann et al.



More than 100 publications.

Super-resolution imaging using scanning optical antennas

localized surface plasmon ω_{VIS} , $\omega_{VIS} - \Delta \omega$, ω_{VIS} Tip enhanced near-field optical microscopy Light localization and enhancement by localized surface plasmon



Optical antenna: a device designed to efficiently convert free-propagating optical radiation to localized energy, and vice versa.

- L. Novotny, N. van Hulst, Nature photonics 5, 89 (2011)
- P. Bharadwai, B. Deutch, L. Novotny, Adv. In Opt. Phot. 1, 438 (2009)
- Pohl D. W., Optics, Principles and Applications (World Scientific, 2000).



Two major types of SNOM



(laser signal)





Generating unidirectional SPP beams



Required shape

Numerical simulation for calculated Delta-shape structures

Experiment, SNOM data

Profiles at different distances

You, O., Wang, Q., Bai, B., Wu, X. & Zhu, Z. A simple method for generating unidirectional surface plasmon polariton beams with arbitrary profiles. *Opt. Lett.* **40**, 5486 (2015).

Plasmons Generation

Experiment, SNOM data





Zhang, C. et al. Polarization-to-Phase Coupling at a Structured Surface for Plasmonic Structured Illumination Microscopy. Laser Photonics Rev. 12, 1–7 (2018).



SPP interference studied by SNOM



Numerical simulation



Kvapil, M. *et al.* Imaging of near-field interference patterns by aperture-type SNOM – influence of illumination wavelength and polarization state. *Opt. Express* **25**, 16560 (2017).



NT-MDT cantilever SNOM: contact AND non-contact probes

1) Lever sizes and the pyramid position:



Pyramid LxWxH = 20x20x13 (70 deg)

	Spring Constant (N/m)		Frequency (kHz)		Length (micron)		Width (micron)			Thickness (micron)					
	Nominal	Min	Max	Nominal	Min	Max	Nominal	Min	Max	Nominal	Min	Max	Nominal	Min	Max
NonContact	16.5	5.9	39.0	130	88	180	200	190	210	55	54	57	4	3	5
Contact	1.01	0.41	2.30	20.8	15	27	500	490	510	55	54	57	4	3	5

Probe	Resolution	TR@ 473			
1 contact	150 nm	~3*10-4			
1 contact	???	0.3*10-4			
1 noncontact	110 nm	~0.16*10-4			
2 noncontact	120 nm	~0.5*10-4			
3 noncontact	135 nm	~0.7*10-4			
4 noncontact	100 nm	~0.2*10-4			
5 noncontact	150 nm	~1.6*10-4			

2) Tip shape and aperture size:



Pyramid (SiO2) thickness 400-600 nm

3) Coating: Al, about 100 nm, coating from bottom side. Bottom FIB milling is done after coating. Typical aperture diameter about 170 ± 25 nm.





SNOM of InP/GaInP quantum dots with GaInP cap layer



Spectrum Instruments

A. Mintairov, A. Ankudinov, A. Shelaev, P. Dorozhkin, Ioffe Institute & NT-MDT, 2016

QD SNOM spectroscopy and topography

SNOM PL, 750-780 nm

AFM Topography



Shelaev A. V., Mintairov A. M., Dorozhkin P. S., and Bykov V.A. Scanning near-field microscopy of microdisk resonator with InP/GalnP quantum dots using cantilever-based probes // J. Phys. Conf. Ser. 2016. Vol. 741. P. 12132.

Confocal map, 750-780 nm



Whispering gallery light modes in microdisks with InP/GaInP self-organized quantum dots



A. Mintairov, A. Ankoudinov, A. Shelaev, P. Dorozhkin, Ioffe Institute & NT-MDT, 2016

Laser emission in 3D studied by SNOM





Aperture SNOM applications (NT-MDT instrumentation)



Spectrum Instruments

Physical and chemical characterization at the nanoscale: experimental approaches utilizing AFM probe

Co-localized AFM-Raman

- Comprehensive simultaneous physical (AFM) and chemical (Raman) sample characterization.
- Spatial resolution: AFM ~1 nm; Raman ~200-400 nm.
- Various excitation and collection geometries.
- Tip Enhanced Raman Scattering (TERS)
- Signal enhancement for weakly scattering samples.
- ~10 nm spatial resolution in Raman (chemical) imaging.
- Graphene and other carbon nanomaterials, polymers, thin molecular layers, semiconductor nanostructures, biological structures, DNA molecules etc.
- Advances in production of reproducible TERS probes (STM, tuning fork, AFM cantilevers).

> Aperture scanning near-field optical microscopy (SNOM)

- $\sim \lambda/10$ spatial resolution (~100 nm for NIR).
- Advances in cantilever SNOM manufacturing: contact & non-contact probes, improved signal collection efficiency.
- Plasmonics and nanophotonics structures, metamaterials, lasers, optical fibers, photovoltaics, QDs, etc.

Controlled environment: liquid, gases, temperature, electrochemistry, magnetic field





Thank you for your attention!

www.ntmdt-si.com



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