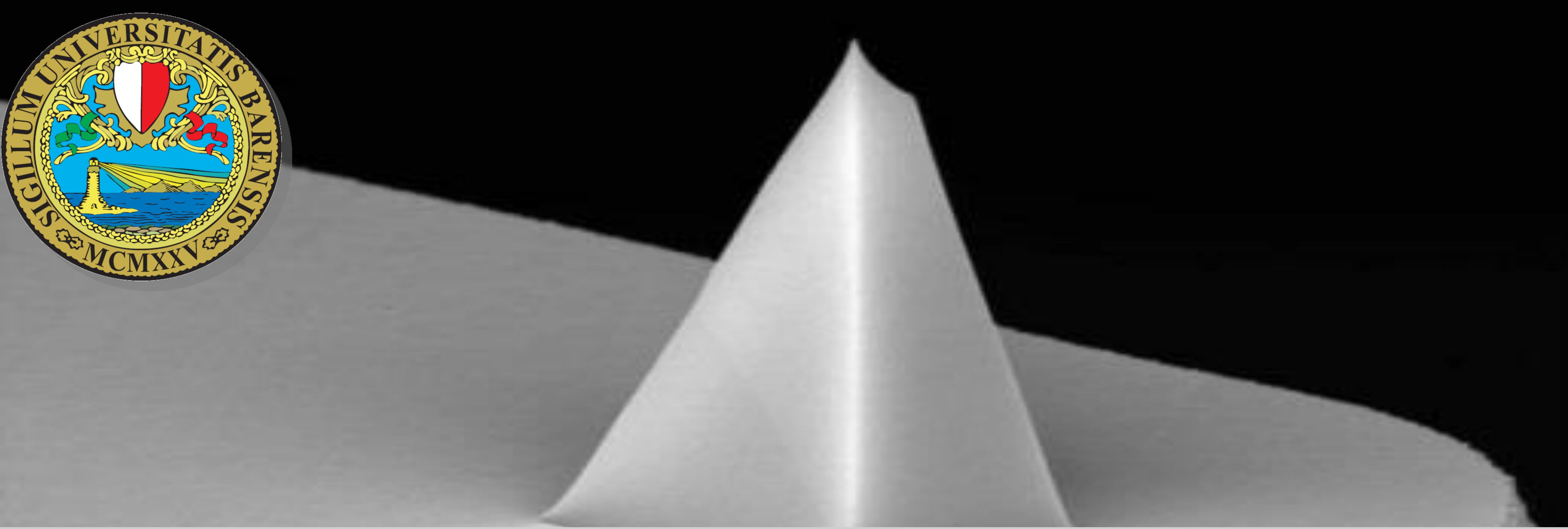

UNIVERSITÀ DEGLI STUDI DI BARI "ALDO MORO"

Atomic Force Microscopy (AFM) + Raman Spectroscopy: Instrumentation and applications

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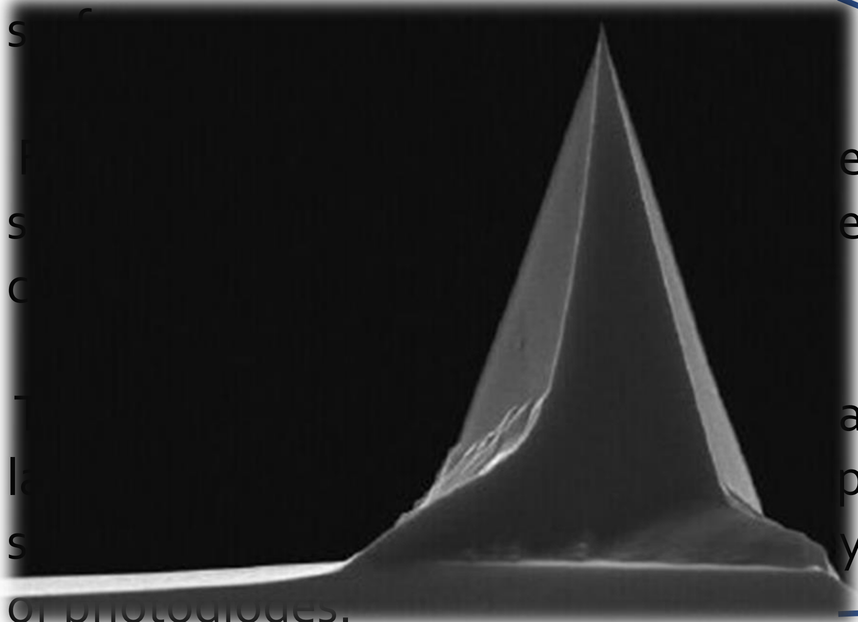
55 Cs	56 Ba	57-71 Lanthanides	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89-103 Actinides	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 Uut	114 Fl	115 Uup	116 Lv	117 Uus	118 Uuo

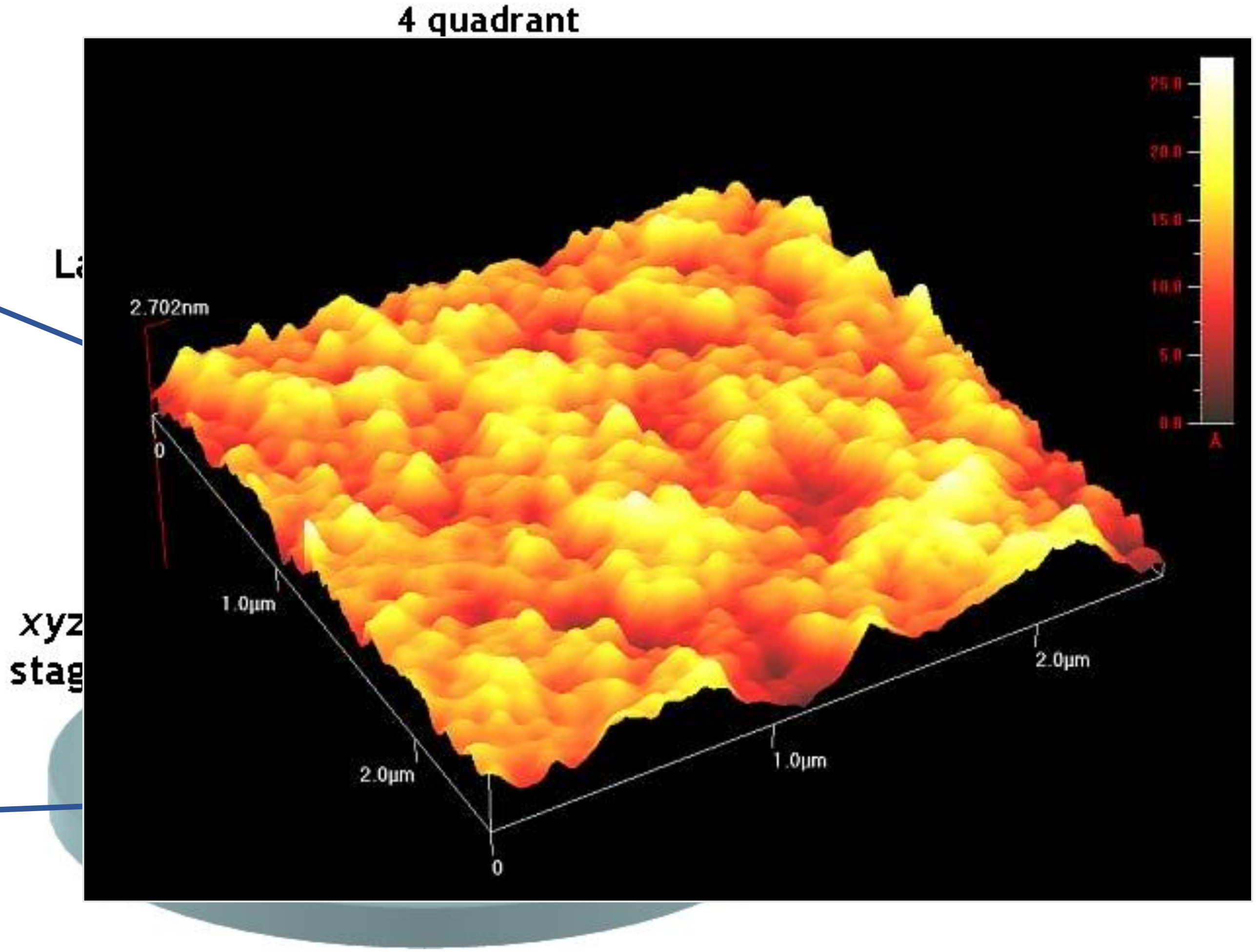
Introduction to Atomic Force Microscopy (AFM)

1. The cantilever with a sharp tip (probe) is brought into proximity of a sample s

2.

3. of photodiodes.

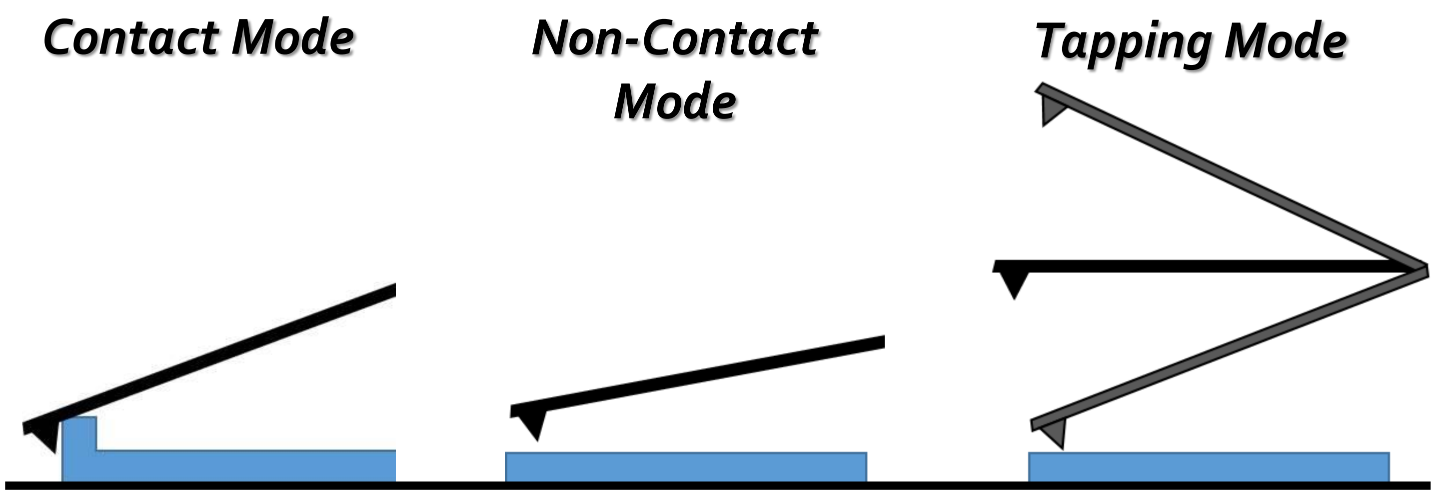




55 Cs $[Xe]6s^1$	56 Ba $[Xe]6s^2$	57-71 Lanthanides	72 Hf $[Xe]4f^{14}5d^26s^2$	73 Ta $[Xe]4f^{14}5d^36s^2$	74 W $[Xe]4f^{14}5d^46s^2$	75 Re $[Xe]4f^{14}5d^56s^2$	76 Os $[Xe]4f^{14}5d^66s^2$	77 Ir $[Xe]4f^{14}5d^76s^2$	78 Pt $[Xe]4f^{14}5d^96s^1$	79 Au $[Xe]4f^{14}5d^{10}6s^1$	80 Hg $[Xe]4f^{14}5d^{10}6s^2$	81 Tl $[Xe]4f^{14}5d^{10}6s^2p^1$	82 Pb $[Xe]4f^{14}5d^{10}6s^2p^2$	83 Bi $[Xe]4f^{14}5d^{10}6s^2p^3$	84 Po $[Xe]4f^{14}5d^{10}6s^2p^4$	85 At $[Xe]4f^{14}5d^{10}6s^2p^5$	86 Rn $[Xe]4f^{14}5d^{10}6s^2p^6$
87 Fr $[Rn]7s^1$	88 Ra $[Rn]7s^2$	89-103 Actinides	104 Rf $[Rn]5f^{14}6d^27s^{2*}$	105 Db $[Rn]5f^{14}6d^37s^{2*}$	106 Sg $[Rn]5f^{14}6d^47s^{2*}$	107 Bh $[Rn]5f^{14}6d^57s^{2*}$	108 Hs $[Rn]5f^{14}6d^67s^{2*}$	109 Mt $[Rn]5f^{14}6d^77s^{2*}$	110 Ds $[Rn]5f^{14}6d^97s^{1*}$	111 Rg $[Rn]5f^{14}6d^{10}7s^{1*}$	112 Cn $[Rn]5f^{14}6d^{10}7s^{2*}$	113 Uut $[Rn]5f^{14}6d^{10}7s^27p^{1*}$	114 Fl $[Rn]5f^{14}6d^{10}7s^27p^{2*}$	115 Uup $[Rn]5f^{14}6d^{10}7s^27p^{3*}$	116 Lv $[Rn]5f^{14}6d^{10}7s^27p^{4*}$	117 Uus $[Rn]5f^{14}6d^{10}7s^27p^{5*}$	118 Uuo $[Rn]5f^{14}6d^{10}7s^27p^{6*}$

G. Binnig et al., *Phys. Rev. lett.*, **1986**, 56, 930–933

Introduction to Atomic Force Microscopy (AFM)



- ✓ **Contact mode**: the cantilever is "dragged" across the surface of the sample and the contours of the surface are measured directly using the deflection of the cantilever.

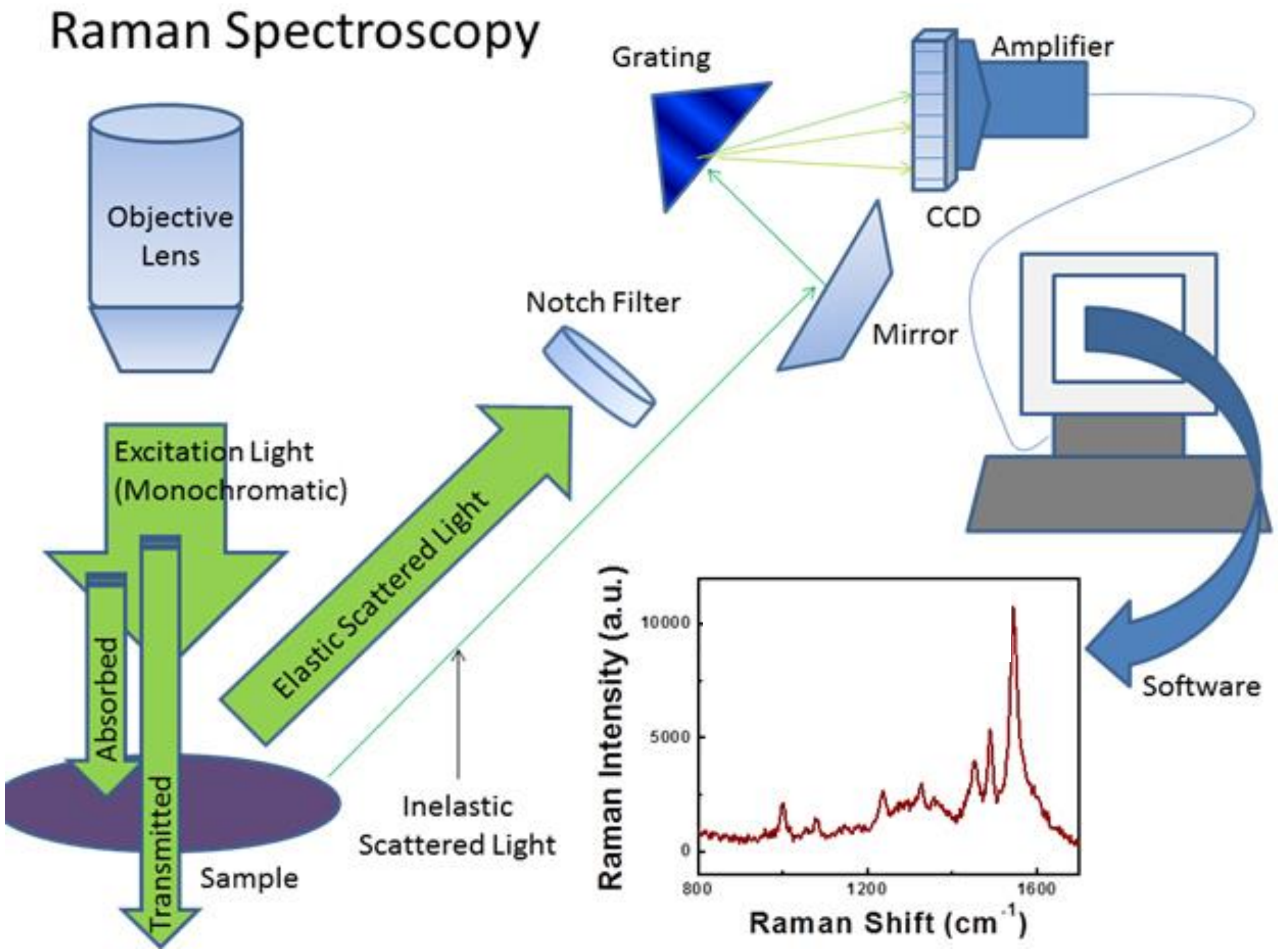
- ✓ **Non-contact mode**: the tip of the cantilever does not contact the sample surface and is instead oscillated at its resonant frequency or just above it. The van der Waals forces act to decrease the resonance frequency of the cantilever.

- ✓ **Tapping mode**: the cantilever is driven to oscillate up and down at near its resonance frequency by a small piezoelectric element mounted in the AFM tip. The interaction of forces acting on the cantilever when the tip comes close to the surface cause the amplitude of this oscillation to decrease as the tip gets closer to the sample.

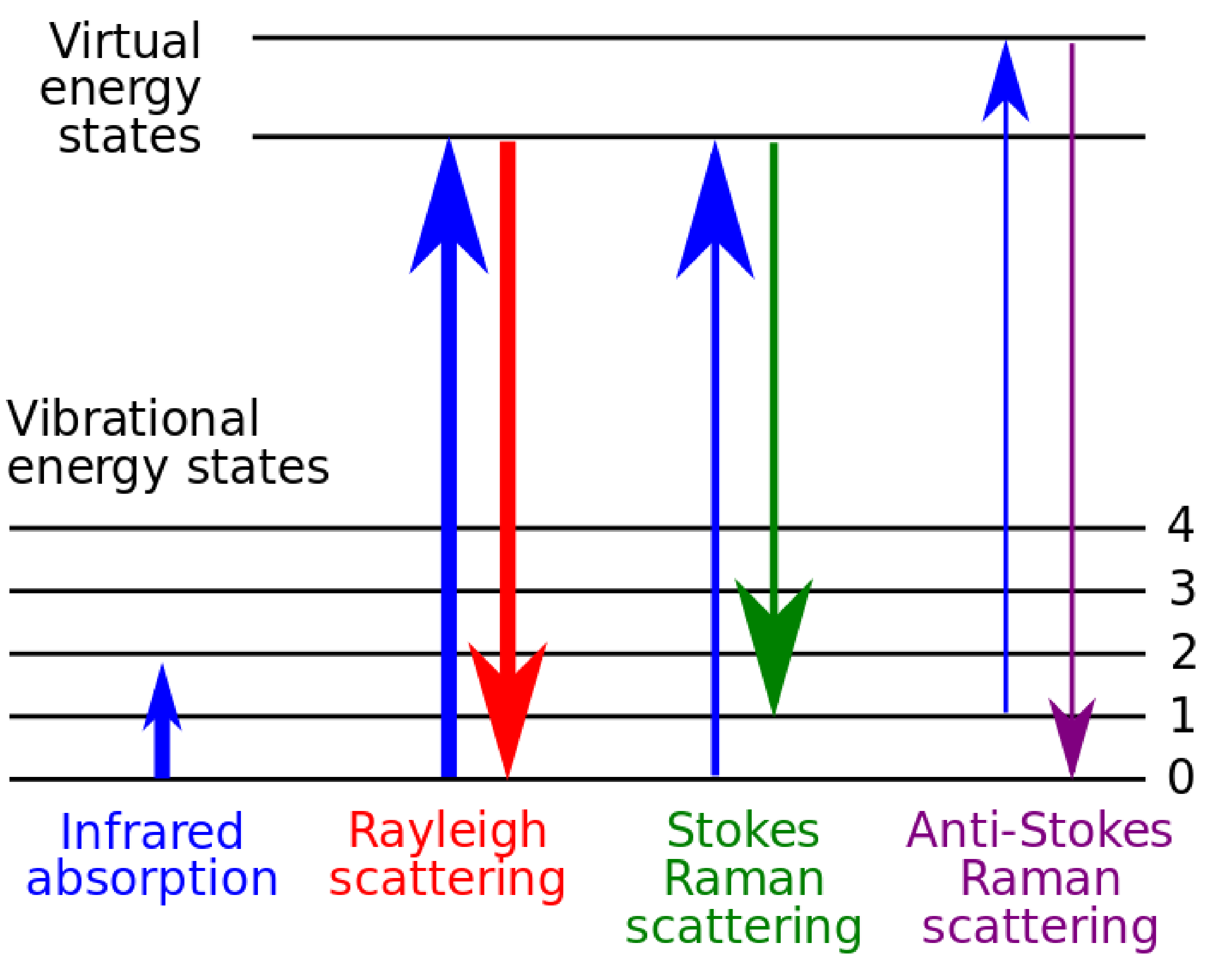
G. Binnig et al., *Phys. Rev. lett.*, **1986**, 56, 930–933

Introduction to Raman Spectroscopy

- ✓ It relies on inelastic scattering of monochromatic light, usually from a laser;
- ✓ The laser light interacts with molecular vibrations, phonons or other excitations in the system, resulting in the energy of the laser photons being shifted up or down;
- ✓ The shift in energy gives information about the vibrational modes in the system, complementary to IR informations.



Introduction to Raman Spectroscopy



- ✓ Raman scattering is typically very weak → the main difficulty of Raman spectroscopy is separating the weak inelastically scattered light from the intense Rayleigh scattered laser light;
- ✓ A change in the molecular polarization in respect to the vibrational coordinate is required for a molecule to exhibit a Raman effect. The amount of the polarizability change will determine the Raman scattering intensity;
- ✓ Vibrational informations are specific to the chemical bonds and symmetry of molecules. Therefore, it provides a fingerprint by which the molecule can be identified.

E.V. Efremov et al., *Analytica Chimica Acta*, **2008**, 606, 119–134

Combining the two techniques

Atomic Force Microscopy

- ✓ Morphological characterization of nanoscale objects;
- ✓ High spatial resolution, up to few nanometers;
- ✓ Physical properties of the sample surface can be studied: surface potential, work function, capacitance…

Lack of chemical informations

AFM + Raman

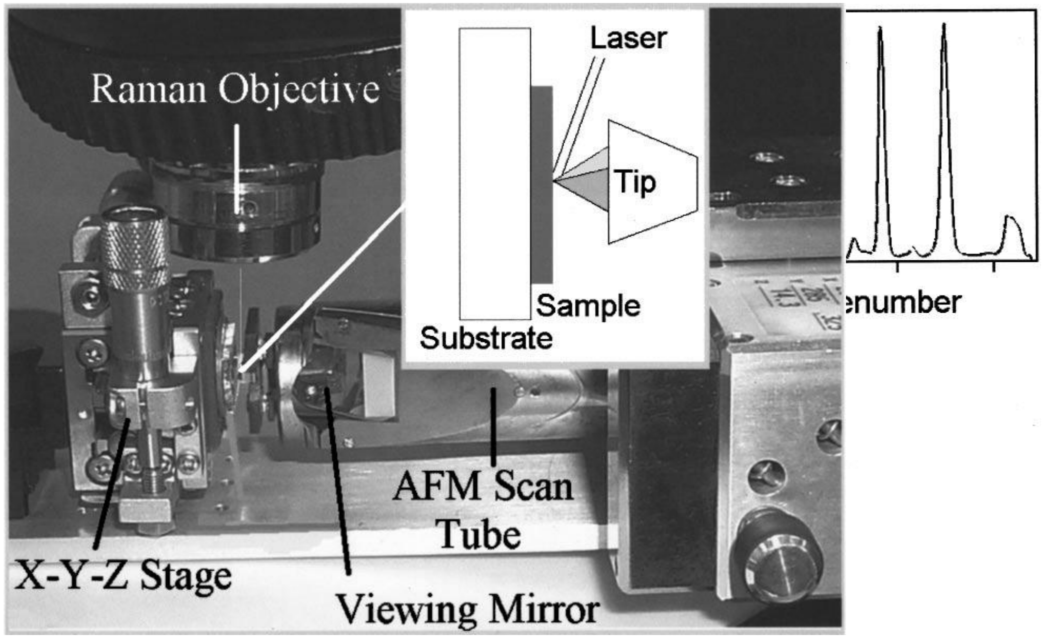
Imaging and chemical speciation of exactly the same sampled area, with the same sample scan.

Combining the two techniques

REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 73, NUMBER 3 MARCH 2002

A Raman-atomic force microscope for apertureless-near-field spectroscopy and optical trapping

Mark S. Anderson[a] and William T. Pike



Problems

- ✓ Positioning the high-resolution objective lens near the AFM cantilever without compromising AFM performance;
- ✓ Laser spot and AFM probe being spatially correlated with high precision and temporal stability, to provide AFM and spectroscopy measurements from exactly the same sample area.

55 Cs $[Xe]6s^1$	56 Ba $[Xe]6s^2$	57-71 Lanthanides	72 Hf $[Xe]4f^{14}5d^2 6s^2$	73 Ta $[Xe]4f^{14}5d^3 6s^2$	74 W $[Xe]4f^{14}5d^4 6s^2$	75 Re $[Xe]4f^{14}5d^5 6s^2$	76 Os $[Xe]4f^{14}5d^6 6s^2$	77 Ir $[Xe]4f^{14}5d^7 6s^2$	78 Pt $[Xe]4f^{14}5d^9 6s^1$	79 Au $[Xe]4f^{14}5d^{10}6s^1$	80 Hg $[Xe]4f^{14}5d^{10}6s^2$	81 Tl $[Xe]4f^{14}5d^{10}6s^2p^1$	82 Pb $[Xe]4f^{14}5d^{10}6s^2p^2$	83 Bi $[Xe]4f^{14}5d^{10}6s^2p^3$	84 Po $[Xe]4f^{14}5d^{10}6s^2p^4$	85 At $[Xe]4f^{14}5d^{10}6s^2p^5$	86 Rn $[Xe]4f^{14}5d^{10}6s^2p^6$
87 Fr $[Rn]7s^1$	88 Ra $[Rn]7s^2$	89-103 Actinides	104 Rf $[Rn]5f^{14}6d^2 7s^{2*}$	105 Db $[Rn]5f^{14}6d^3 7s^{2*}$	106 Sg $[Rn]5f^{14}6d^4 7s^{2*}$	107 Bh $[Rn]5f^{14}6d^5 7s^{2*}$	108 Hs $[Rn]5f^{14}6d^6 7s^{2*}$	109 Mt $[Rn]5f^{14}6d^7 7s^{2*}$	110 Ds $[Rn]5f^{14}6d^9 7s^{1*}$	111 Rg $[Rn]5f^{14}6d^{10}7s^{1*}$	112 Cn $[Rn]5f^{14}6d^{10}7s^{2*}$	113 Uut $[Rn]5f^{14}6d^{10}7s^2 7p^{1*}$	114 Fl $[Rn]5f^{14}6d^{10}7s^2 7p^{2*}$	115 Uup $[Rn]5f^{14}6d^{10}7s^2 7p^{3*}$	116 Lv $[Rn]5f^{14}6d^{10}7s^2 7p^{4*}$	117 Uus $[Rn]5f^{14}6d^{10}7s^2 7p^{5*}$	118 Uuo $[Rn]5f^{14}6d^{10}7s^2 7p^{6*}$

P. Dorozhkin et al., *Micr. Today*, **2010**, 11, 28-32;
M. Anderson et al., *Rev. Sci. Instr.*, **2002**, 73, 1198-1203.

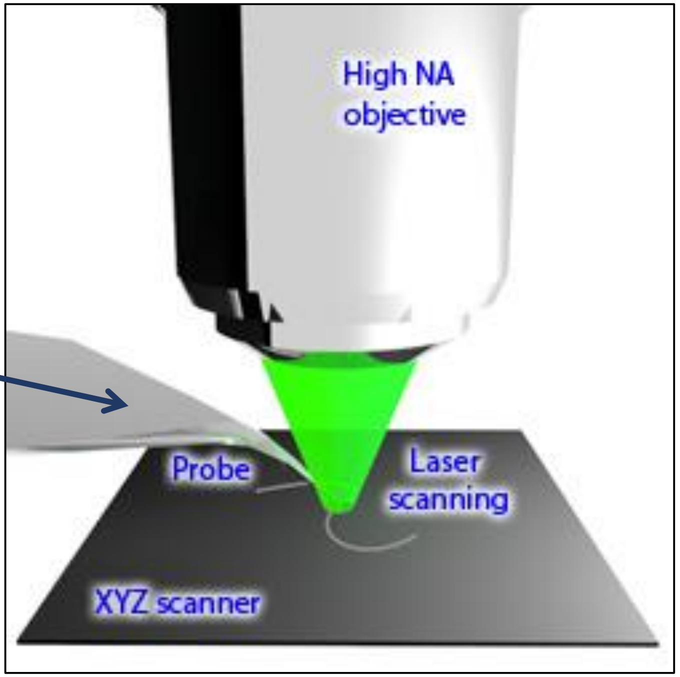
Instrumental configurations

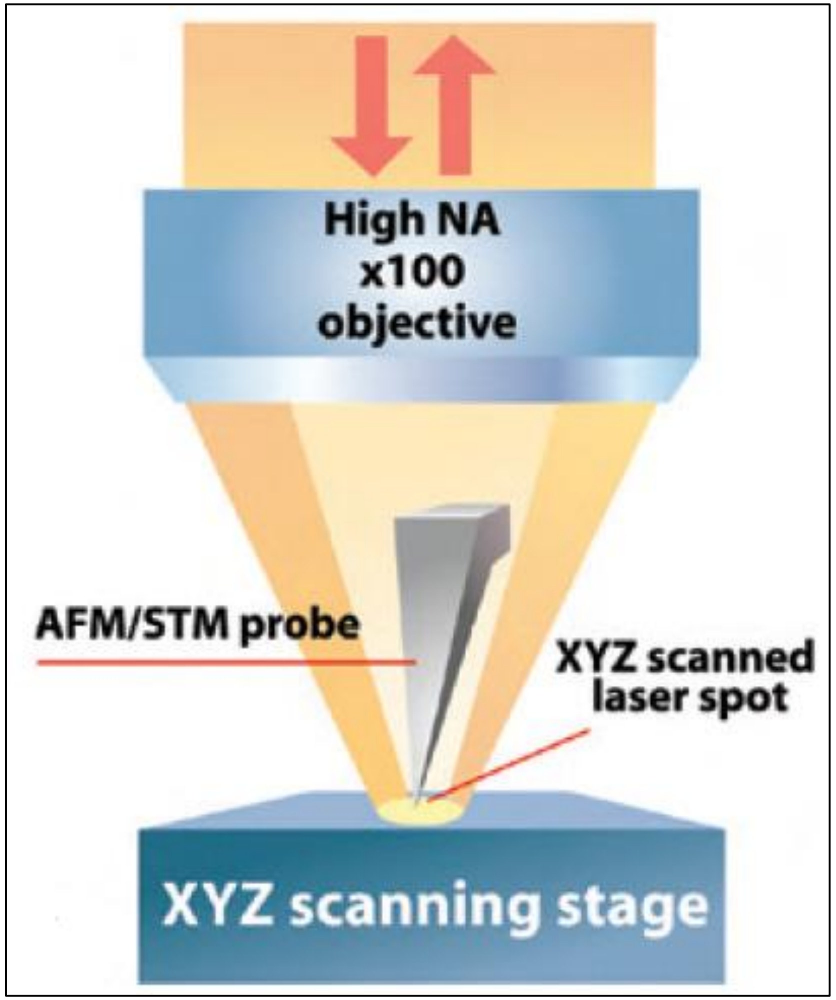
1. Upright configuration

- ✓ Opaque samples can be studied;
- ✓ High-resolution optics is positioned from the same side of the sample as the cantilever;
- ✓ "Elephant trunk-shaped" probes allow focusing of the laser light directly at the apex of the AFM tip.

Park SYSTEMS

HORIBA JOBIN YVON



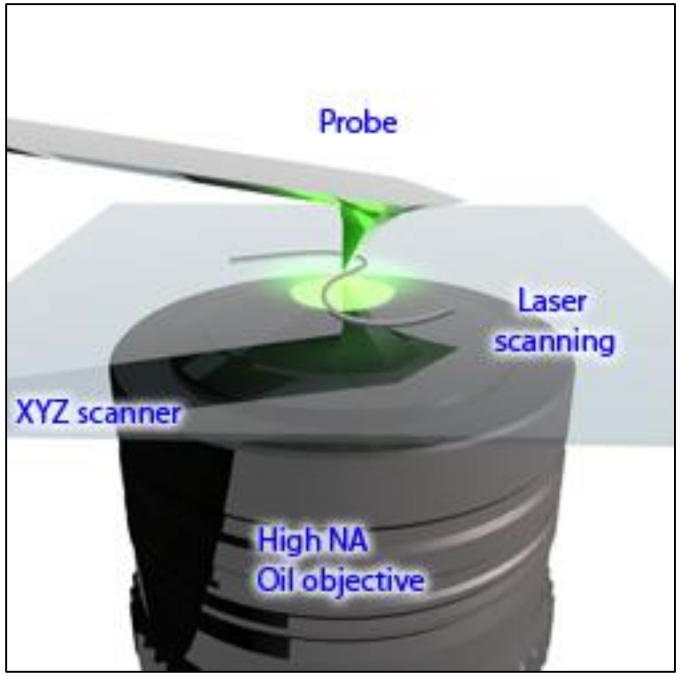


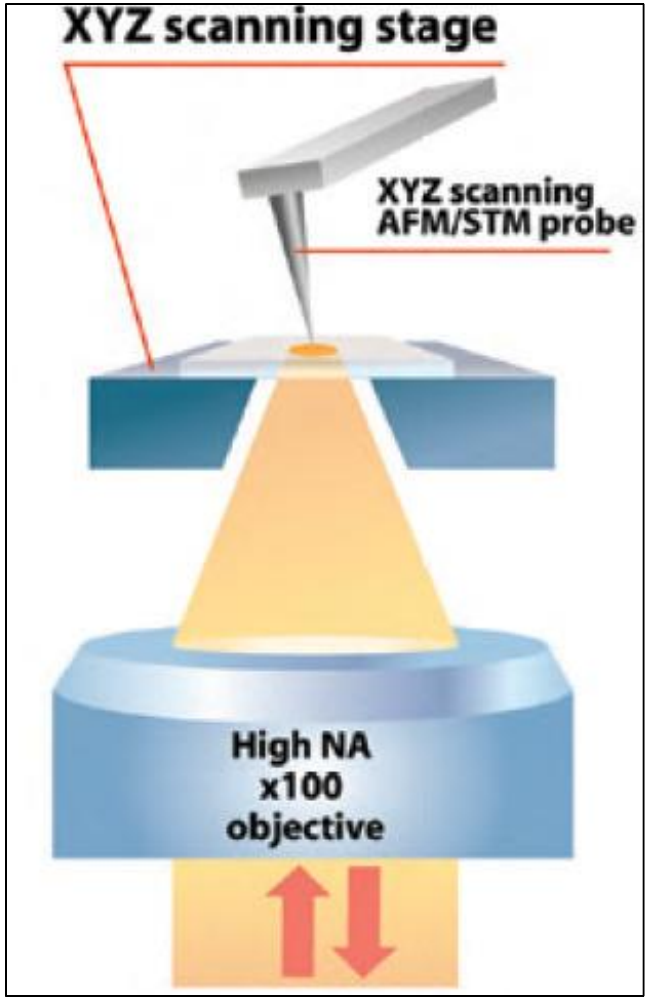
Instrumental configurations

2. Inverted configuration

- ✓ Opaque samples cannot be studied;
- ✓ Has the advantage of having the cantilever and laser spot coming from different sides of the sample;
- ✓ Allows the use of oil immersion optics, achieving the highest spatial resolution;

NANONICS IMAGING Ltd.





55 Cs $[Xe]6s^1$	56 Ba $[Xe]6s^2$	57-71 Lanthanides	72 Hf $[Xe]4f^{14}5d^2 6s^2$	73 Ta $[Xe]4f^{14}5d^3 6s^2$	74 W $[Xe]4f^{14}5d^4 6s^2$	75 Re $[Xe]4f^{14}5d^5 6s^2$	76 Os $[Xe]4f^{14}5d^6 6s^2$	77 Ir $[Xe]4f^{14}5d^7 6s^2$	78 Pt $[Xe]4f^{14}5d^9 6s^1$	79 Au $[Xe]4f^{14}5d^{10} 6s^1$	80 Hg $[Xe]4f^{14}5d^{10} 6s^2$	81 Tl $[Xe]4f^{14}5d^{10}6s^2p^1$	82 Pb $[Xe]4f^{14}5d^{10}6s^2p^2$	83 Bi $[Xe]4f^{14}5d^{10}6s^2p^3$	84 Po $[Xe]4f^{14}5d^{10}6s^2p^4$	85 At $[Xe]4f^{14}5d^{10}6s^2p^5$	86 Rn $[Xe]4f^{14}5d^{10}6s^2p^6$
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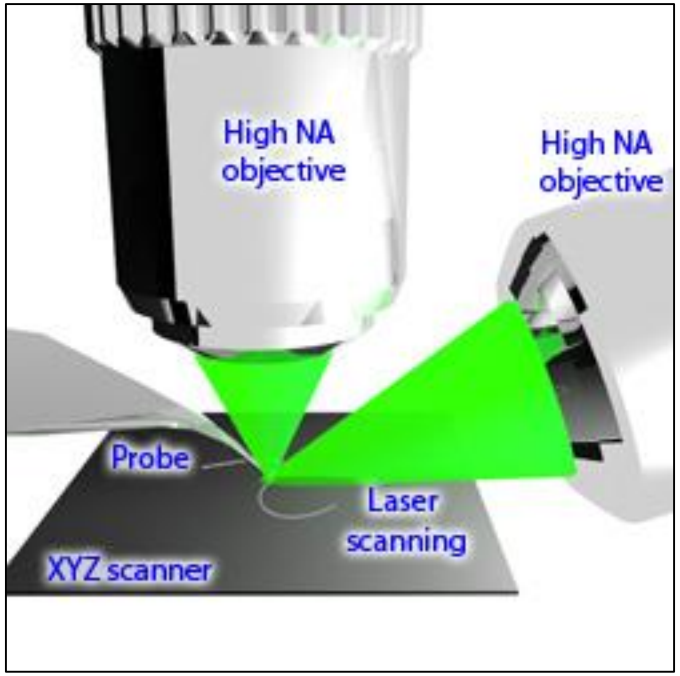
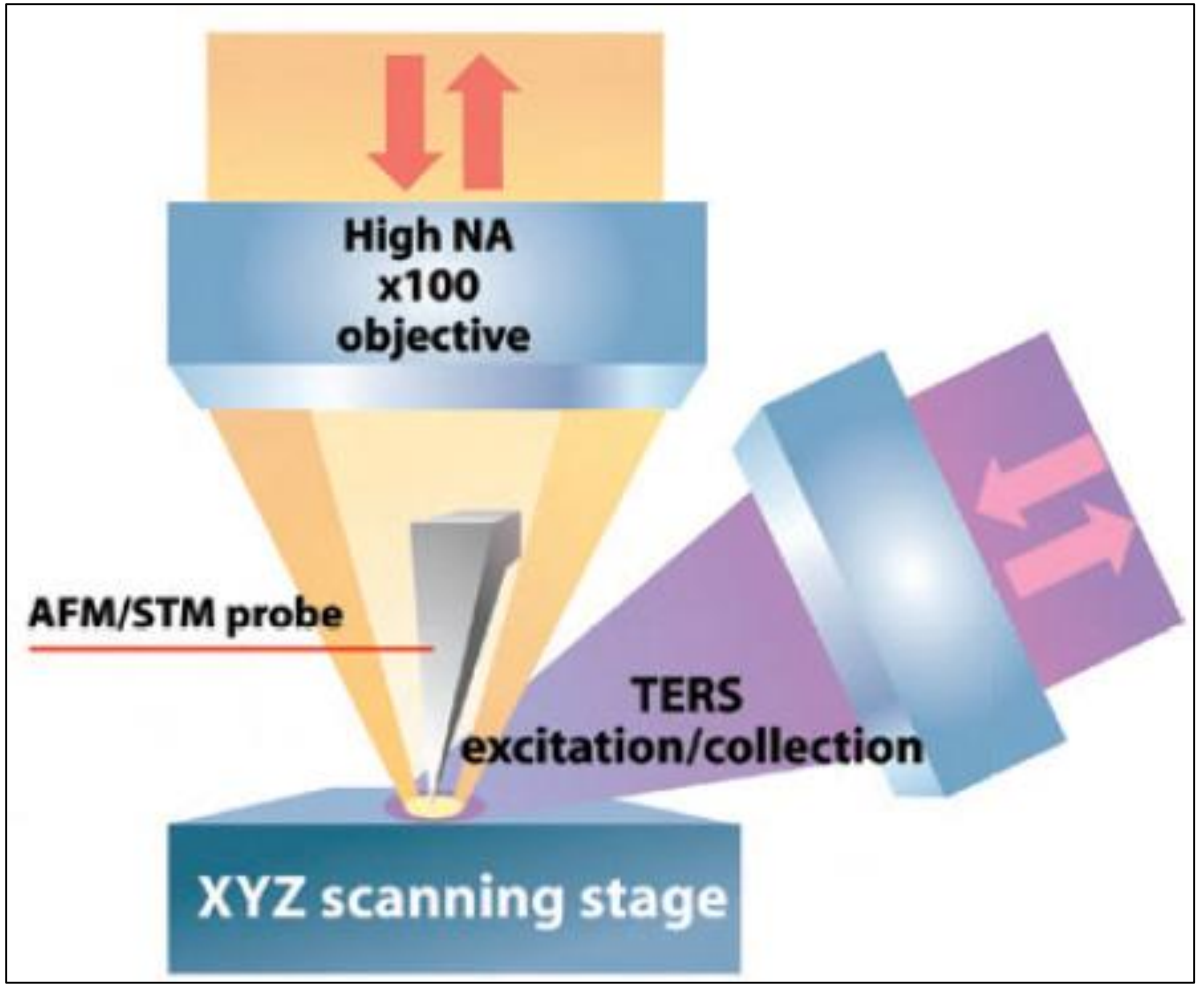
http://http://www.nanonics.co.il/

Tip-enhanced Raman Scattering (TERS)

- ✓ Side optical access is realized by an additional objective lens to focus the laser light from the side of the AFM probe;
- ✓ Nanometer sized metallic tips are used, since large field enhancement occurs for elongated structures with high curvature.

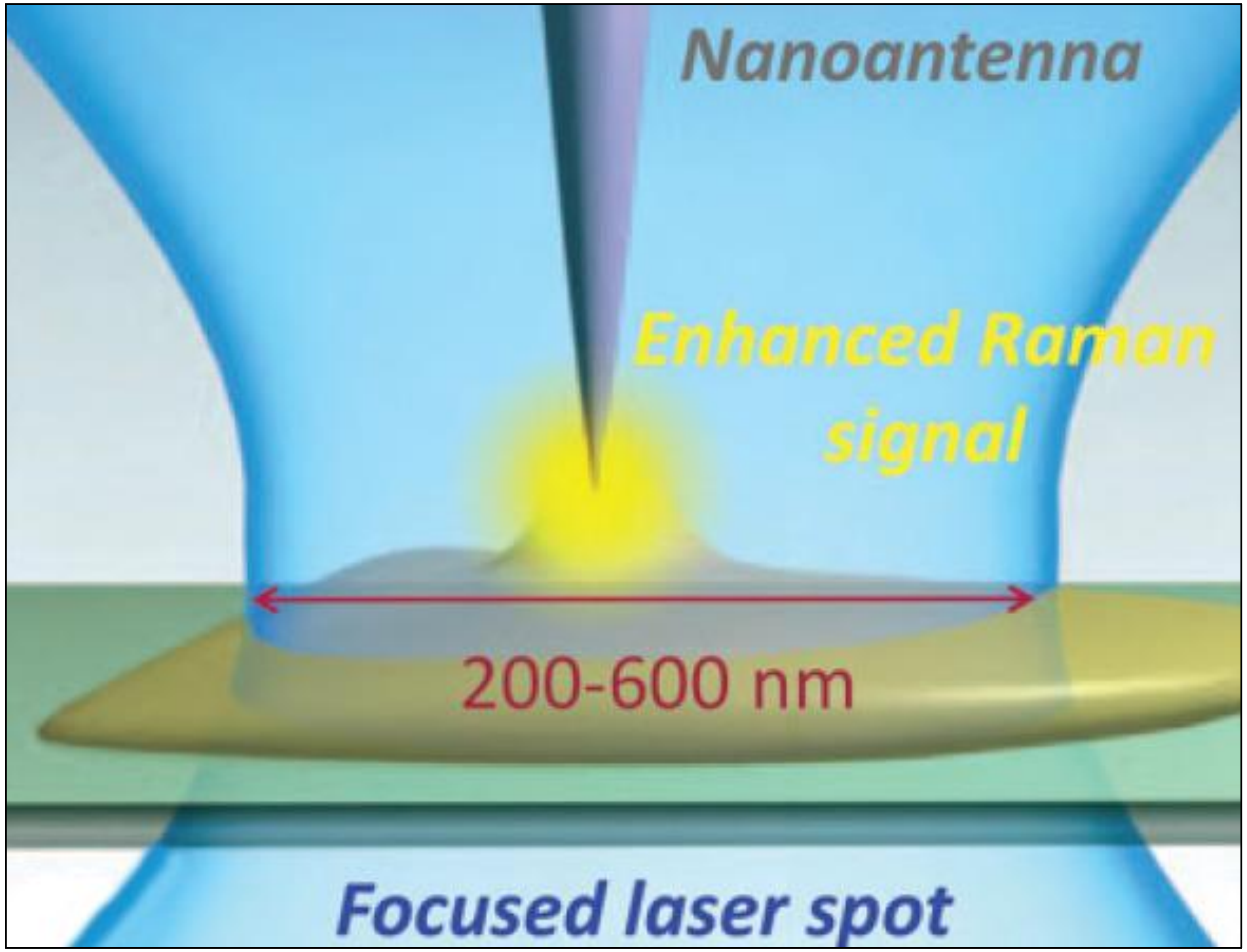
HORIBA JOBIN YVON





http://www.horiba.com/scientific/

Tip-enhanced Raman Scattering (TERS)



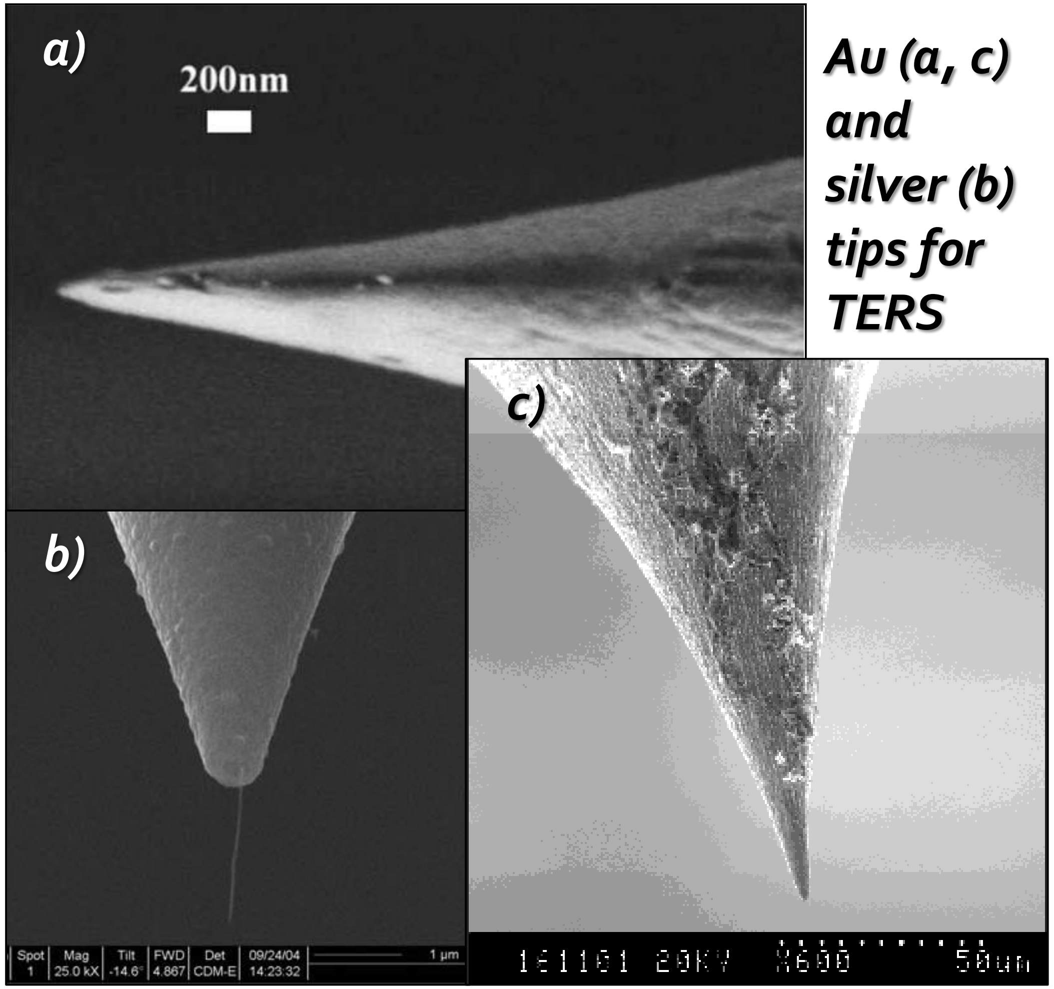
Mechanism for TERS enhancement

1. **Electromagnetic effect**: On the surface of metal nanostructures surface plasmons are excited by light. The involved electromagnetic fields can be strongly enhanced in the presence of surface plasmons if certain resonance conditions are fulfilled;

2. **Chemical or charge transfer effect**: Corresponds to an amplification in the polarizability of the molecule due to a charge transfer between the metal and the adsorbed molecule.

E. Bailo et al., *Chem. Soc. Rev.*, **2008**, 37, 921 - 930

Fabricating TERS tips



Au (a, c) and silver (b) tips for TERS

- ✓ Made by electrochemical etching of a pure metal wire;
- ✓ Focused ion beam milling can be used to further sharpen the apex;
- ✓ Vapor deposition can be also used to metallize common AFM tips;
- ✓ Heating of the cantilever during CVD will distort it and make it unsuitable for scanning;
- ✓ The adhesion of the metal coating (especially for Au films) to the tip is fairly weak, and the coating may peel after several hours of scanning

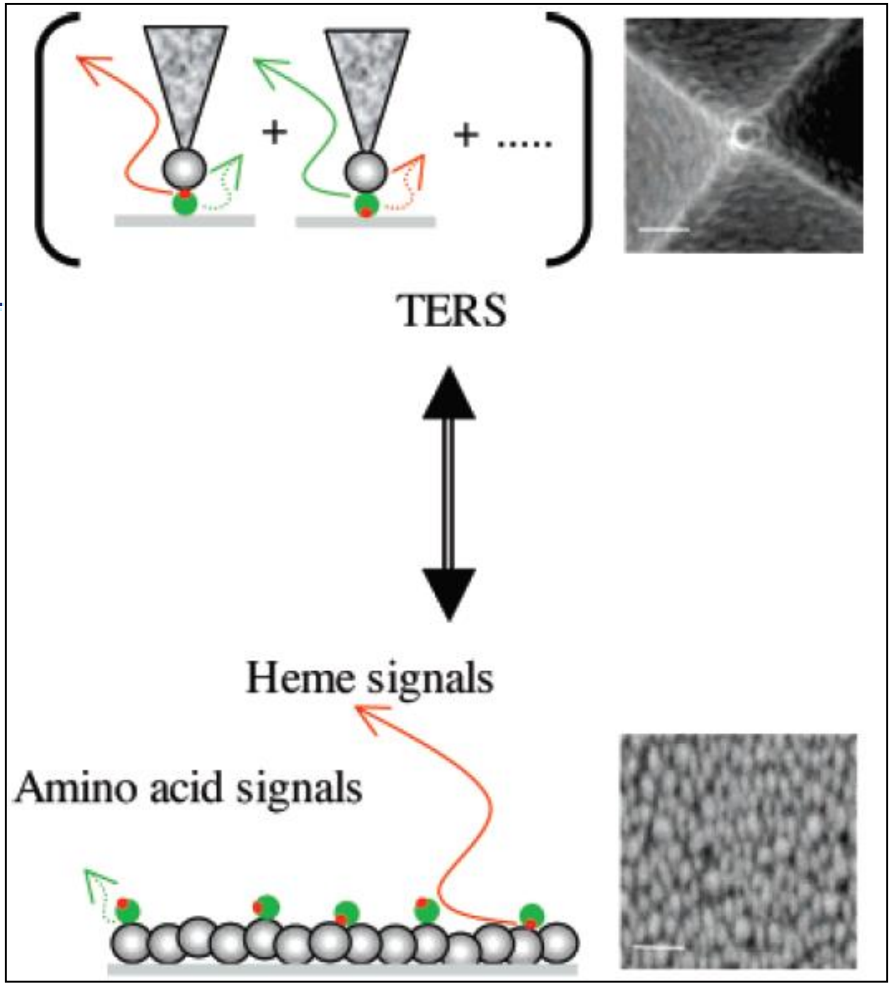
B. Yeo et al., *Chem. Phys. Lett.*, **2009**, 472, 1-13

Applications: biotechnology

J. Phys. Chem. C **2008,** *112,* 4867–4873

Tip-Enhanced Raman Spectroscopy Can See More: The Case of Cytochrome c

Boon-Siang Yeo, Stefanie Mädler, Thomas Schmid, Weihua Zhang, and Renato Zenobi*



- ✓ The heme protein, Cytochrome C has been studied using TERS;
- ✓ TERS detected both the heme and amino acid vibrational bands of Cytochrome C, using resonance excitation at 532 nm;
- ✓ With conventional Raman spectroscopy, only ensemble information is obtained, and is not possible to distinguish between the two signal classes.

55	56	57-71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
Cs	Ba		Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
$[Xe]6s^1$	$[Xe]6s^2$	Lanthanides	$[Xe]4f^{14}5d^2 6s^2$	$[Xe]4f^{14}5d^3 6s^2$	$[Xe]4f^{14}5d^4 6s^2$	$[Xe]4f^{14}5d^5 6s^2$	$[Xe]4f^{14}5d^6 6s^2$	$[Xe]4f^{14}5d^7 6s^2$	$[Xe]4f^{14}5d^9 6s^1$	$[Xe]4f^{14}5d^{10}6s^1$	$[Xe]4f^{14}5d^{10}6s^2$	$[Xe]4f^{14}5d^{10}6s^2p^1$	$[Xe]4f^{14}5d^{10}6s^2p^2$	$[Xe]4f^{14}5d^{10}6s^2p^3$	$[Xe]4f^{14}5d^{10}6s^2p^4$	$[Xe]4f^{14}5d^{10}6s^2p^5$	$[Xe]4f^{14}5d^{10}6s^2p^6$
87	88	89-103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118
Fr	Ra		Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn	Uut	Fl	Uup	Lv	Uus	Uuo
$[Rn]7s^1$	$[Rn]7s^2$	Actinides	$[Rn]5f^{14}6d^2 7s^2$*	$[Rn]5f^{14}6d^3 7s^2$*	$[Rn]5f^{14}6d^4 7s^2$*	$[Rn]5f^{14}6d^5 7s^2$*	$[Rn]5f^{14}6d^6 7s^2$*	$[Rn]5f^{14}6d^7 7s^2$*	$[Rn]5f^{14}6d^9 7s^1$*	$[Rn]5f^{14}6d^{10}7s^1$*	$[Rn]5f^{14}6d^{10}7s^2$*	$[Rn]5f^{14}6d^{10}7s^2 7p^1$*	$[Rn]5f^{14}6d^{10}7s^2 7p^2$*	$[Rn]5f^{14}6d^{10}7s^2 7p^3$*	$[Rn]5f^{14}6d^{10}7s^2 7p^4$*	$[Rn]5f^{14}6d^{10}7s^2 7p^5$*	$[Rn]5f^{14}6d^{10}7s^2 7p^6$*

B. Yeo et al., *J. Phys. Chem. C*, **2008**, 112, 4867-4837

Applications: catalysis

Top Catal (2013) 56:1088–1095
DOI 10.1007/s11244-013-0074-6

ORIGINAL PAPER

Far Field Combined AFM and Micro-Raman Imaging for Characterisation of Surface of Structured Catalysts: Example of Pd Doped CoO_x Catalysts on Precalcined Kanthal Steel

J. Łojewska · A. Knapik · A. Kołodziej · P. Jodłowski

- ✓ AFM–Raman system was used to study the surface heterogeneity of catalytic materials at various stages of their preparation;
- ✓ The catalysts chosen for the analyses were cobalt oxide with and without palladium dopant deposited on surface.

55 Cs	56 Ba	57-71 Lanthanides	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
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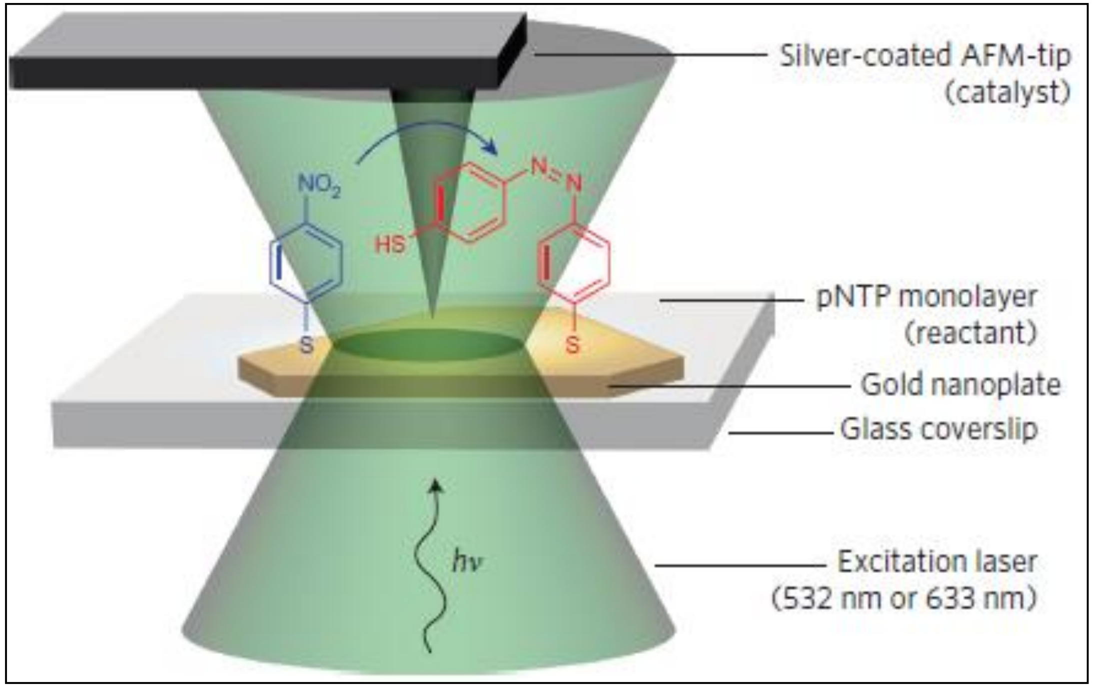
J. Łojewska et al., *Top. Catal.*, **2013**, 56, 1088–1095

Applications: catalysis

nature nanotechnology | LETTERS
PUBLISHED ONLINE: 19 AUGUST 2012 | DOI: 10.1038/NNANO.2012.131

Catalytic processes monitored at the nanoscale with tip-enhanced Raman spectroscopy

Evelien M. van Schrojenstein Lantman[1], Tanja Deckert-Gaudig[2], Arjan J. G. Mank[1,3], Volker Deckert[2,4]* and Bert M. Weckhuysen[1]*



- ✓ Time-resolved TERS is used to monitor photocatalytic reactions at the nanoscale;
- ✓ Authors used a silver coated atomic force microscope tip to both enhance the Raman signal and to act as the catalyst;
- ✓ Photocatalytic reduction process on a Self-Assebled Monolayer is induced at the apex of the tip with green laser light, while red laser light is used to monitor the transformation process during the reaction.

55 Cs [Xe]6s^1	56 Ba [Xe]6s^2	57-71 Lanthanides	72 Hf [Xe]4f^{14}5d^{2}6s^2	73 Ta [Xe]4f^{14}5d^{3}6s^2	74 W [Xe]4f^{14}5d^{4}6s^2	75 Re [Xe]4f^{14}5d^{5}6s^2	76 Os [Xe]4f^{14}5d^{6}6s^2	77 Ir [Xe]4f^{14}5d^{7}6s^2	78 Pt [Xe]4f^{14}5d^{9}6s^1	79 Au [Xe]4f^{14}5d^{10}6s^1	80 Hg [Xe]4f^{14}5d^{10}6s^2	81 Tl [Xe]4f^{14}5d^{10}6s^{2}p^1	82 Pb [Xe]4f^{14}5d^{10}6s^{2}p^2	83 Bi [Xe]4f^{14}5d^{10}6s^{2}p^3	84 Po [Xe]4f^{14}5d^{10}6s^{2}p^4	85 At [Xe]4f^{14}5d^{10}6s^{2}p^5	86 Rn [Xe]4f^{14}5d^{10}6s^{2}p^6
87 Fr [Rn]7s^1	88 Ra [Rn]7s^2	89-103 Actinides	104 Rf [Rn]5f^{14}6d^{2}7s^2*	105 Db [Rn]5f^{14}6d^{3}7s^2*	106 Sg [Rn]5f^{14}6d^{4}7s^2*	107 Bh [Rn]5f^{14}6d^{5}7s^2*	108 Hs [Rn]5f^{14}6d^{6}7s^2*	109 Mt [Rn]5f^{14}6d^{7}7s^2*	110 Ds [Rn]5f^{14}6d^{9}7s^1*	111 Rg [Rn]5f^{14}6d^{10}7s^1*	112 Cn [Rn]5f^{14}6d^{10}7s^2*	113 Uut [Rn]5f^{14}6d^{10}7s^{2}7p^1*	114 Fl [Rn]5f^{14}6d^{10}7s^{2}7p^2*	115 Uup [Rn]5f^{14}6d^{10}7s^{2}7p^3*	116 Lv [Rn]5f^{14}6d^{10}7s^{2}7p^4*	117 Uus [Rn]5f^{14}6d^{10}7s^{2}7p^5*	118 Uuo [Rn]5f^{14}6d^{10}7s^{2}7p^6*

E. M. van Schrojenstein Lantman et al., *Nature Tech.*, **2012**, 131, 1-4

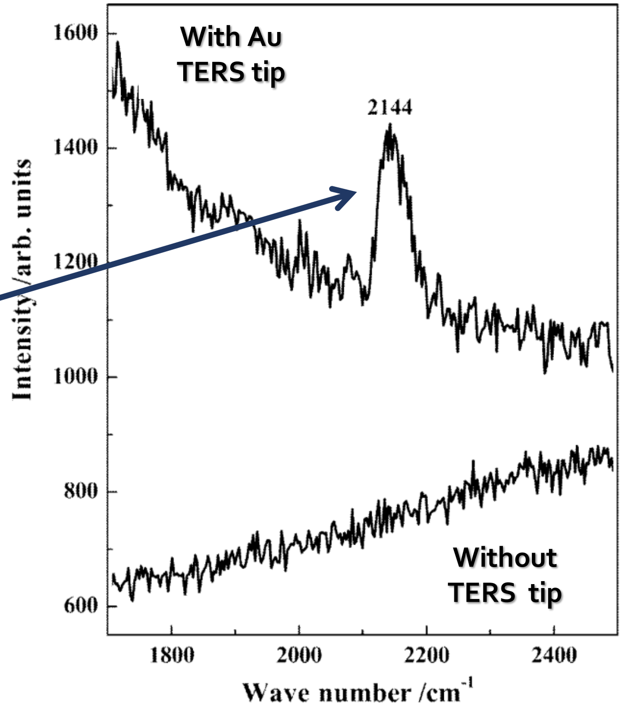
Applications: Self-Assembled Monolayers (SAM)

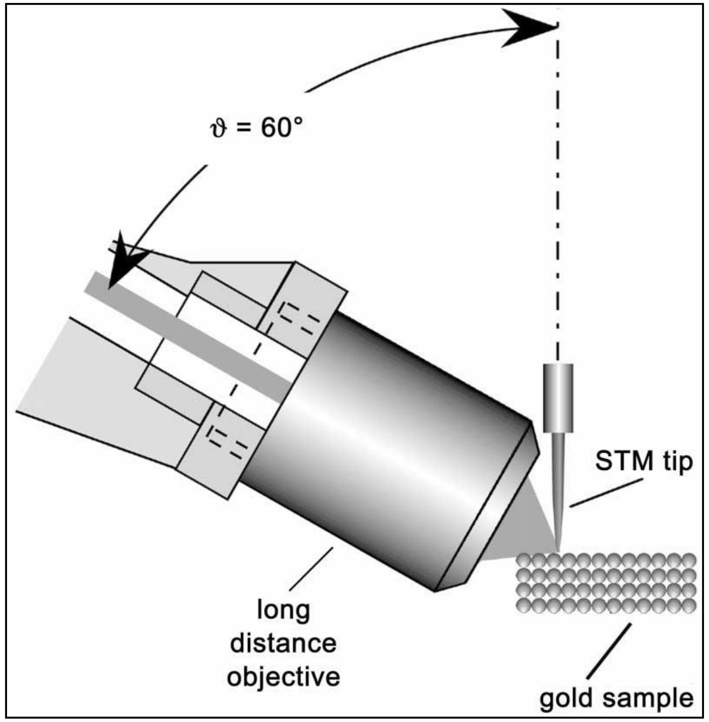
VOLUME 92, NUMBER 9 PHYSICAL REVIEW LETTERS week ending 5 MARCH 2004

Nanoscale Probing of Adsorbed Species by Tip-Enhanced Raman Spectroscopy

Bruno Pettinger,[1,*] Bin Ren,[1,2] Gennaro Picardi,[1] Rolf Schuster,[1] and Gerhard Ertl[1]

- ✓ The application of suitable Au tips allowed to record, for the first time, Raman spectra of an optically nonresonant species;
- ✓ The data analysis yields Raman enhancements of about $4 \cdot 10^5$ for CN^- signal at Au(111) with a Au tip





B. Pettinger et al., *Phys. Rev. Lett.*, **2004**, 92, 096101-1 - 096101-4

Applications: Self-Assembled Monolayers (SAM)

Research Article

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Tip enhanced Raman spectroscopy on azobenzene thiol self-assembled monolayers on Au(111)

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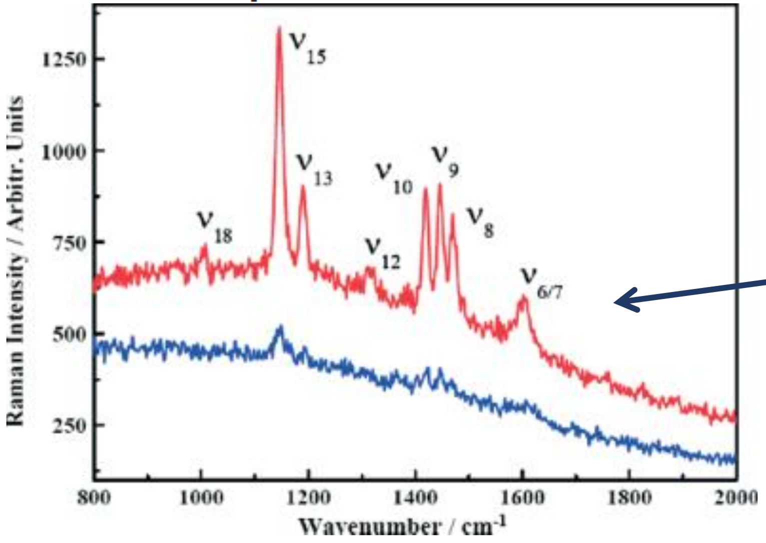
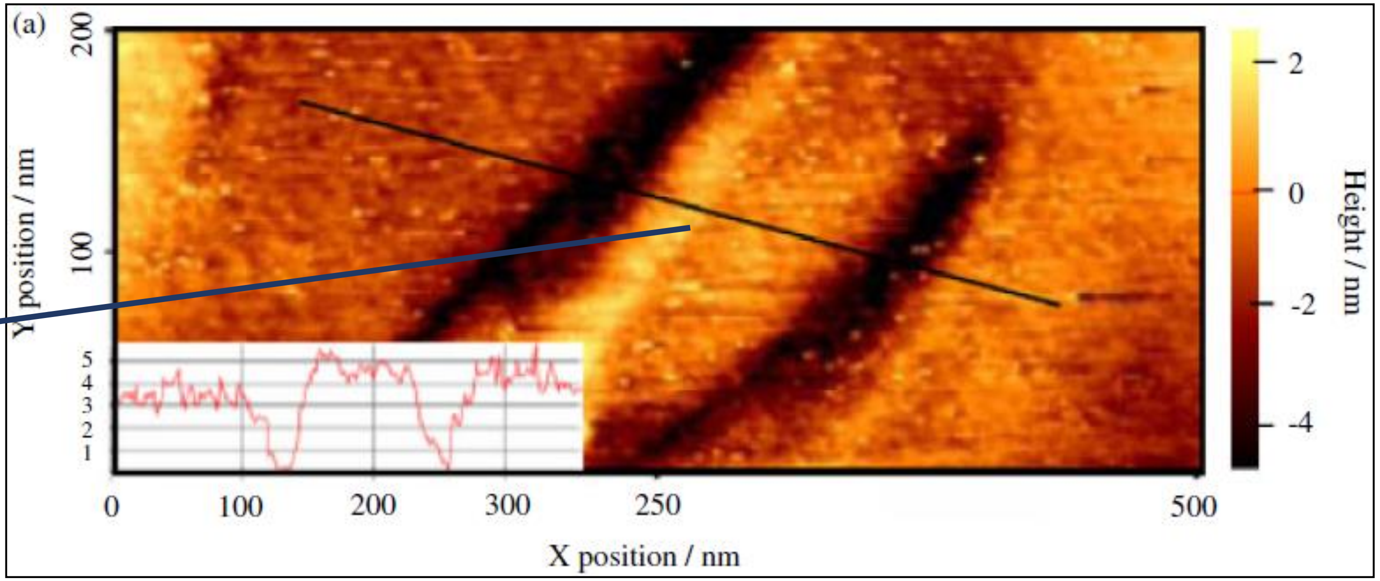
TERS spectrum (red) from a chemisorbed layer at the Au(111) surface.





55 Cs $[Xe]6s^1$	56 Ba $[Xe]6s^2$	57-71 Lanthanides	72 Hf $[Xe]4f^{14}5d^26s^2$	73 Ta $[Xe]4f^{14}5d^36s^2$	74 W $[Xe]4f^{14}5d^46s^2$	75 Re $[Xe]4f^{14}5d^56s^2$	76 Os $[Xe]4f^{14}5d^66s^2$	77 Ir $[Xe]4f^{14}5d^76s^2$	78 Pt $[Xe]4f^{14}5d^96s^1$	79 Au $[Xe]4f^{14}5d^{10}6s^1$	80 Hg $[Xe]4f^{14}5d^{10}6s^2$	81 Tl $[Xe]4f^{14}5d^{10}6s^2p^1$	82 Pb $[Xe]4f^{14}5d^{10}6s^2p^2$	83 Bi $[Xe]4f^{14}5d^{10}6s^2p^3$	84 Po $[Xe]4f^{14}5d^{10}6s^2p^4$	85 At $[Xe]4f^{14}5d^{10}6s^2p^5$	86 Rn $[Xe]4f^{14}5d^{10}6s^2p^6$
87 Fr $[Rn]7s^1$	88 Ra $[Rn]7s^2$	89-103 Actinides	104 Rf $[Rn]5f^{14}6d^27s^{2*}$	105 Db $[Rn]5f^{14}6d^37s^{2*}$	106 Sg $[Rn]5f^{14}6d^47s^{2*}$	107 Bh $[Rn]5f^{14}6d^57s^{2*}$	108 Hs $[Rn]5f^{14}6d^67s^{2*}$	109 Mt $[Rn]5f^{14}6d^77s^{2*}$	110 Ds $[Rn]5f^{14}6d^97s^{1*}$	111 Rg $[Rn]5f^{14}6d^{10}7s^{1*}$	112 Cn $[Rn]5f^{14}6d^{10}7s^{2*}$	113 Uut $[Rn]5f^{14}6d^{10}7s^27p^{1*}$	114 Fl $[Rn]5f^{14}6d^{10}7s^27p^{2*}$	115 Uup $[Rn]5f^{14}6d^{10}7s^27p^{3*}$	116 Lv $[Rn]5f^{14}6d^{10}7s^27p^{4*}$	117 Uus $[Rn]5f^{14}6d^{10}7s^27p^{5*}$	118 Uuo $[Rn]5f^{14}6d^{10}7s^27p^{6*}$

G. Picardi et al., *J. Raman Spec.*, **2009**, 40, 1407–1412

Conclusions

- ✓ **AFM-Raman allows imaging and chemical speciation simultaneously;**
- ✓ **Many experimental set-up have been developed;**
- ✓ **Nanometer sized metallic tips were found to be responsible of large field enhancement;**
- ✓ **Tip-enhanced Raman Scattering (TERS) is widely used in many research fields.**