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Institute of Electronics, Microelectronics and Nanotechnology

UMR CNRS 8520

Multi-Physics Characterization Platform

Pôle PCP

PCMP Plateforme de Caractérisation Multi-Physique

PCP

The Scanning Probe Microscopy service named **"Pole Champ Proche"** supplies premium tools, to observe and manipulate atoms, molecules or nanoscale objects on the micro to subnanometer scale, making these instruments essentials to Nanoscience and Nanotechnology. The PCP facility is organised into 2 domains depending on the measurement environment:

- AIR domain for microscopes operating in air ambient, liquid or controlled gas atmosphere
- UHV domain for microscopes operating under Ultra High Vacuum

With 8 instruments and 400m² of area in a ISO8-certified environment localized on the ground floor of IEMN, the facility hosts about 30 expert users. Part of the instruments are on free access and can be booked online. One day training for beginners is provided in request. The team is composed of 3 permanent engineers providing internal, external academic and industrial services in the framework of the RENATECH national network. Their mission concern also the development of new instruments and experimental techniques in collaboration with users, Start-up and SPM companies.

Head of PCP
D. Deresmes

- **Air domain SPM's** **I. 1-4**

→ Dominique Deresmes

ICON
DIMENSION
MULTIMODE
BIOSCOPE

- **UHV domain SPM's** **I. 5-8**

→ Dominique Deresmes → Maxime Berthe → Sylvie Godey

VTAFM
JT-SPM
LT-STM
NANOPROBE

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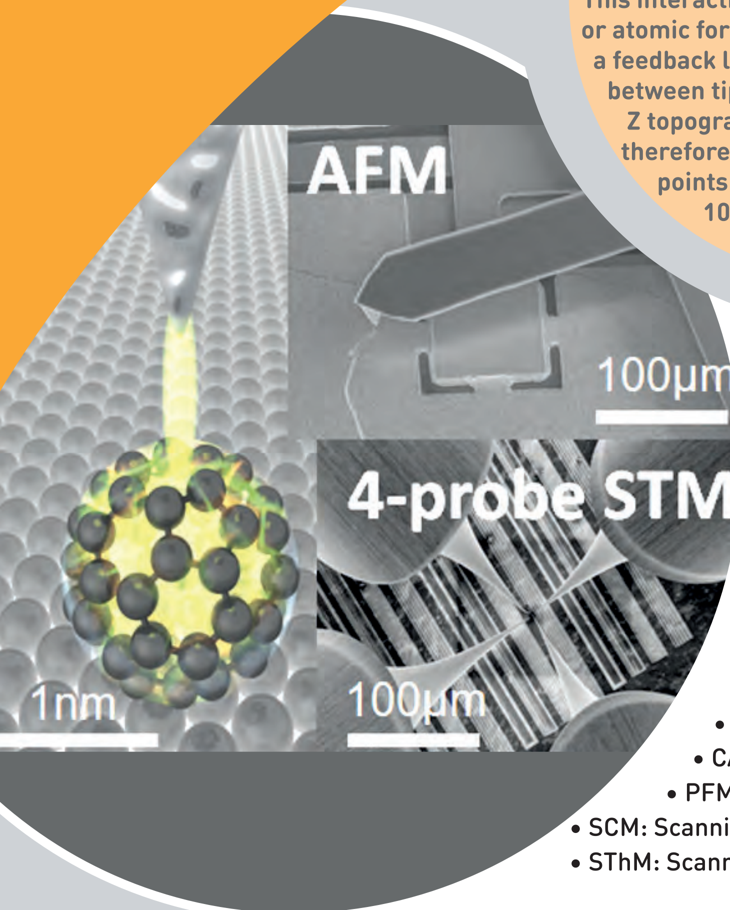
AIR DOMAIN SPM's

The Scanning Probe Microscopes use a recent technique (Nobel prize in Physics 1986) of microscopy where a probe (tip) interacts with the surface of the sample at a very short distance (Angstrom to 100nm). This interaction is based on tunneling current or atomic force that is kept constant thanks to a feedback loop which controls the distance between tip and surface with an actuator. Z topography (Angstrom to 10µm) can therefore be saved for each coordinate points (X,Y) ranging from 5nm to 100µm depending of the microscope model.



The probe interacts in contact (C) or non-contact (NC) mode and can work in static or dynamic mode. Various physical characteristics of the surface can be adressed through differents modes of measurement:

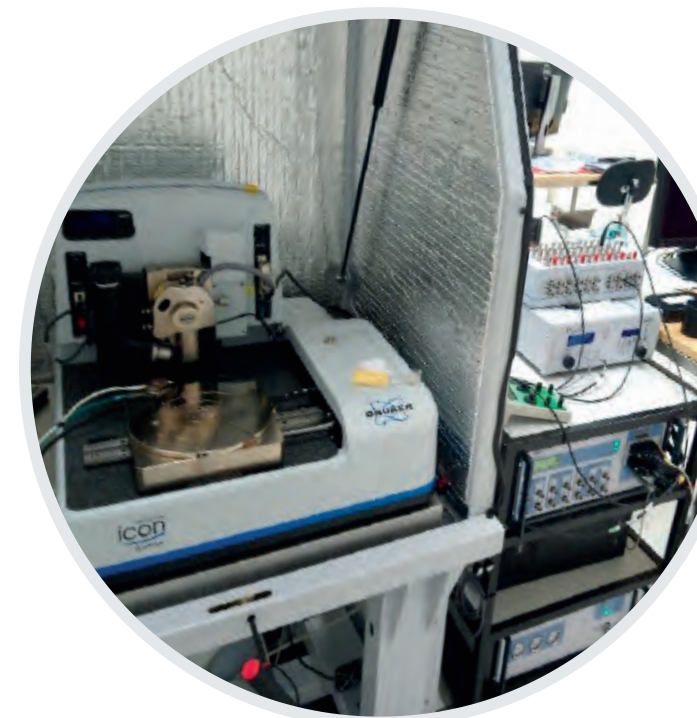
- STM: Scanning Tunneling Microscopy (NC),
- LDOS: Local Density of Electronic States (NC)
- AFM: Atomic Force Microscopy (C, NC), Force Spectroscopy (C)
- EFM: Electrostatic Force Microscopy (NC)
- MFM: Magnetic Force Microscopy (NC)
- KPFM: Kelvin Probe Force Microscopy (NC)
- CAFM: Conductive Atomic Force Microscopy (C)
- PFM: Piezoelectric Force Microscopy (C)
- SCM: Scanning Capacitance Microscopy (C)
- SThM: Scanning Thermal Microscopy (C)

→ APPLICATION EXAMPLES

- Topographic monitoring of technological processes and material growth: Molecular beam epitaxy, Etching, Film deposition, lithography
- Local characterization in contact mode of the physical properties of the material: Electrical conductivity by CAFM or thermal by SThM, Piezoelectric response by PFM, Measurement of adhesion force and mechanical property by force spectroscopy
- Local characterization in non-contact mode of the physical properties of the surface: Measurement of electrostatic and magnetic forces (EFM, MFM), measurement of charges , measurement of surface potential (KPFM), Density of states (STM)

→ ADVANTAGES & LIMITATIONS

(+) 3D nanometric topography measurement , sub nanometric roughness measurement
(+) Simultaneous local physical imaging and characterization
(−) Tip Convolution (−) Low scan speed



ICON

Dominique Deresmes

- **Sample dimension :** 5mm square to 20cm diameter
- **Scan range :** 10nm to 100µm (X and Y linearization feedback: close loop) - Max. Z range: 10µm
- **Resolution :** Lateral: nanometric - Vertical 30pm
- **Working Mode :** AFM Tapping, AFM Peakforce, EFM, KPFM, CAFM, PeakForce TUNA, PFM, SThM, Force spectroscopy
- **Environnement :** Ambient air, Nitrogen gas
- **Temperature :** -25°C to 250°C

→ APPLICATIONS

- PeakForce
- Thermal chuck for small sample

→ ADVANTAGES & LIMITATIONS

(+) Large sample, large coarse displacement of the chuck (2µm resolution)
(−) Acoustic and vibrational Noise sensitive

DIMENSION Bruker

Dominique Deresmes

- **Sample dimension :** 5mm square to 20cm diameter
- **Scan range :** 10nm to 100µm - Max. Z range: 6µm
- **Resolution :** Lateral: nanometric - Vertical 50pm
- **Working Mode :** AFM Tapping, EFM, KPFM, CAFM, PFM, SThM, Force spectroscopy, SCM
- **Environnement :** Ambient air, Nitrogen gas
- **Temperature :** Ambient

→ ADVANTAGES & LIMITATIONS

(+) Large sample, large coarse displacement of the chuck (2µm resolution)
(−) Acoustic and vibrational Noise sensitive

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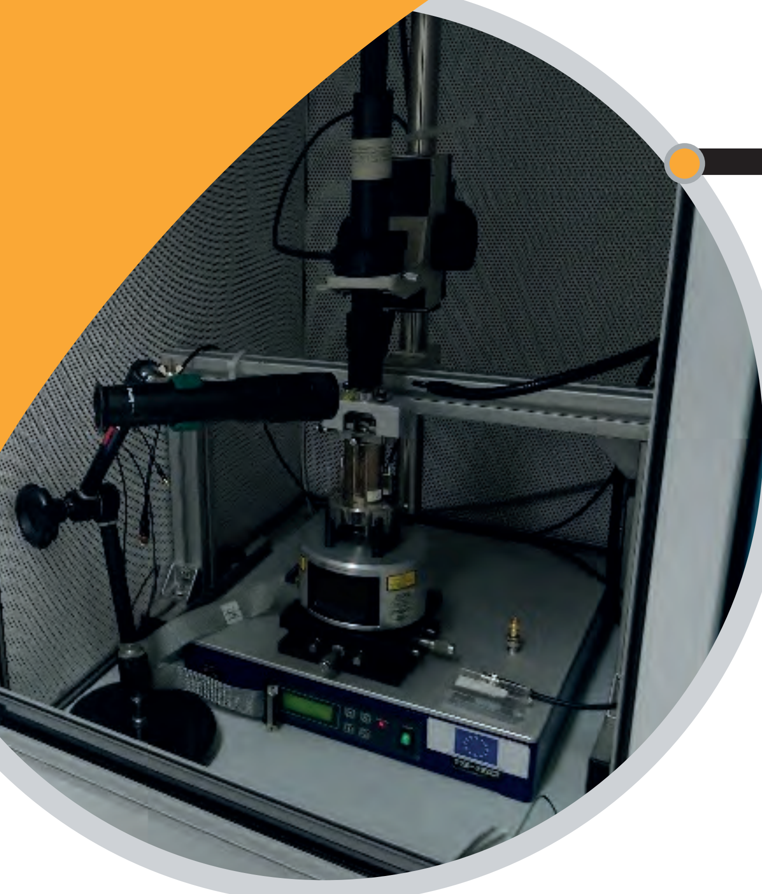
AIR DOMAIN SPM's



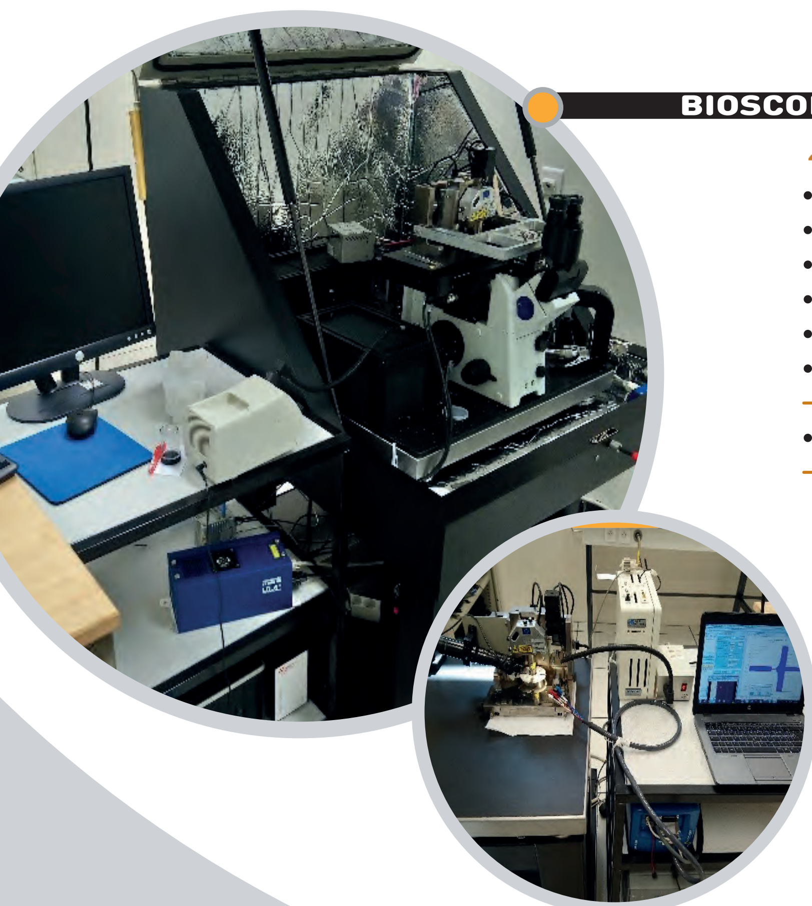
MULTIMODE Bruker

Dominique Deresmes

- **Sample dimension :** 5mm square to 15mm diameter
- **Scan range :** 10nm to 10 or 100µm (two scanners available) - Max. Z range: 2 or 5µm
- **Resolution :** Lateral: nanometric - Vertical 30pm
- **Working Mode :** AFM Tapping, EFM, KPFM, CAFM, PFM, Force spectroscopy
- **Environnement :** Ambient air, Nitrogen gas and Liquid
- **Temperature :** Ambient

→ ADVANTAGES & LIMITATIONS

+ Low noise imaging
- Small sample
- Limited coarse deplacement



BIOSCOPE Bruker

Dominique Deresmes

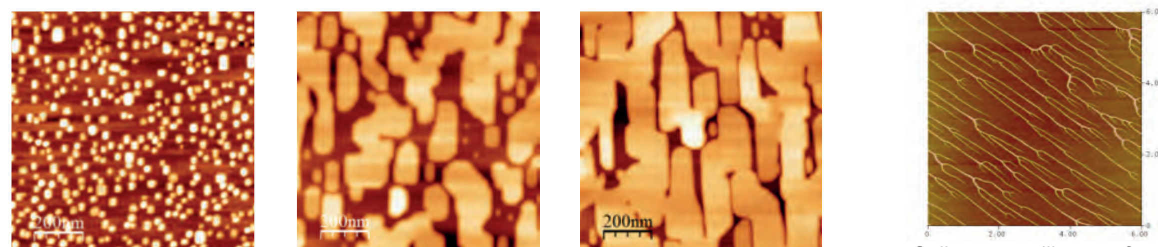
- **Sample dimension :** 5mm square to 5cm diameter
- **Scan range :** 10nm to 100µm - Max. Z range: 6µm
- **Resolution :** Lateral: nanometric - Vertical 80pm
- **Working Mode :** AFM Tapping
- **Environnement :** Ambient air and liquid
- **Temperature :** Ambient

→ APPLICATIONS

- In situ electrochemical growth monitoring

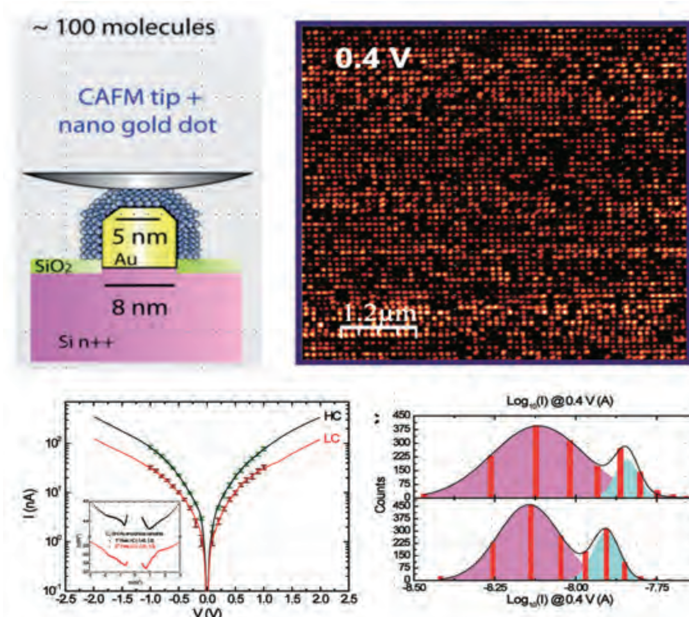
→ ADVANTAGES & LIMITATIONS

+ Tip Enhanced Raman Spectroscopy (TERS[1]) tip optical bench
- Acoustic and vibrational Noise sensitive

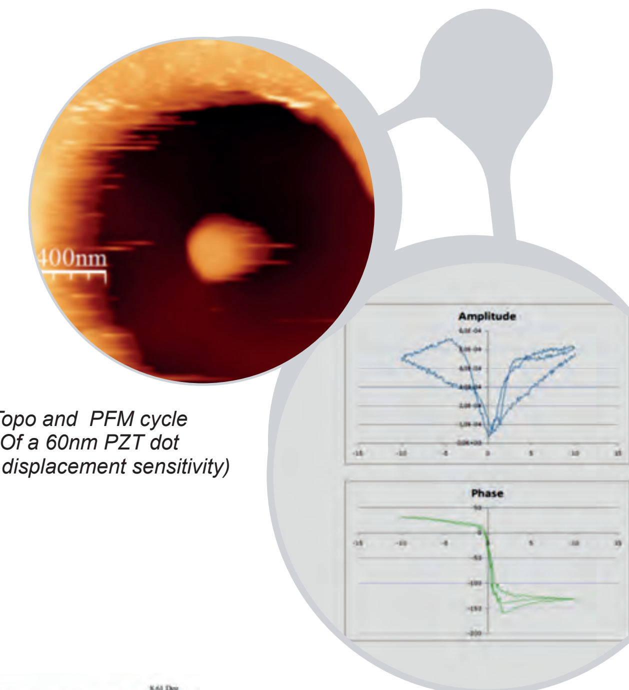


2D-3D growth GaSb/GaAs (AFM)

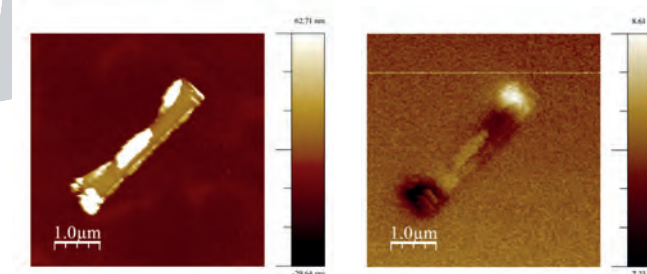
Collagen on silicon surface



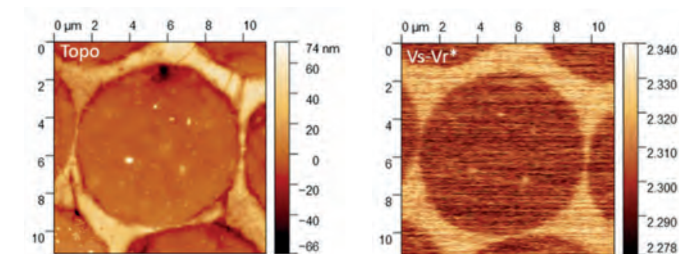
Conducting AFM statistics from a large array of sub-10 nm molecular junstions



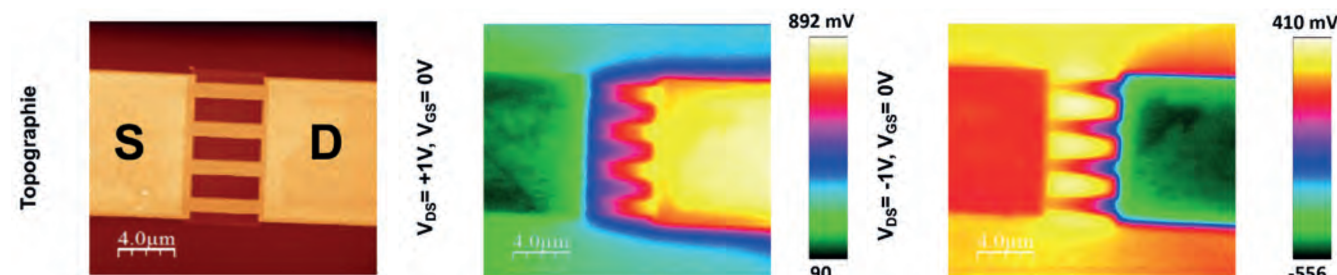
Topo and PFM cycle Of a 60nm PZT dot (1pm displacement sensitivity)



Topo and MFM image of ferromagnetic domain wall position in multiferroic heterostructures



Topography and thermal conductivity of carbon fiber in epoxy matrix (AFM-STHM)



Gas sensing transistor polarization (KPFM)

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UHV DOMAIN SPM's

VTAFM Omicron

Dominique Deresmes

- **Sample dimension :** 4x6mm to 15mm square
- **Scan range :** 10µm - Max. Z range: 2µm
- **Resolution :** Lateral: nanometric - Vertical 30pm
- **Working Mode :** AFM, EFM, KPFM, CAFM, PFM, STM
- **Environnement :** Ultra High Vacuum
- **Temperature :** 50K to 1000K

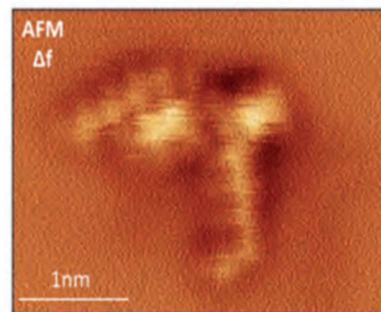
→ **APPLICATIONS**

- Laser beam deflection (allow contact modes)
- Preparation chamber for sample and Tip
- Sample heater
- Mass spectrometer
- Ion gun
- 3 metal evaporator

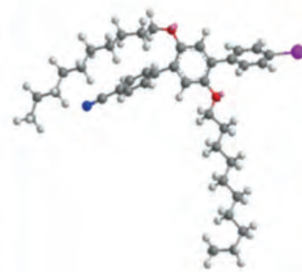
→ **ADVANTAGES & LIMITATIONS**

(+) Variable temperature operation

(−) Small sample



Δf image V_S=0mV

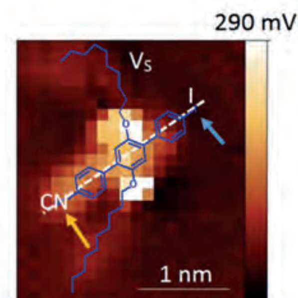


Model corresponding to nc-AFM image

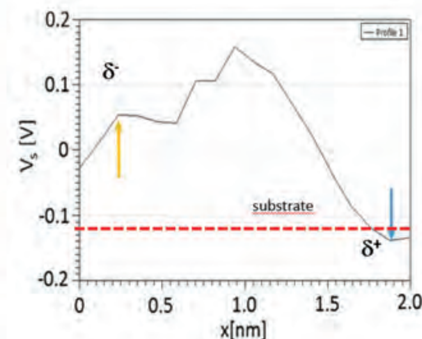
Sub-molecular resolution

KPFM Spectroscopy

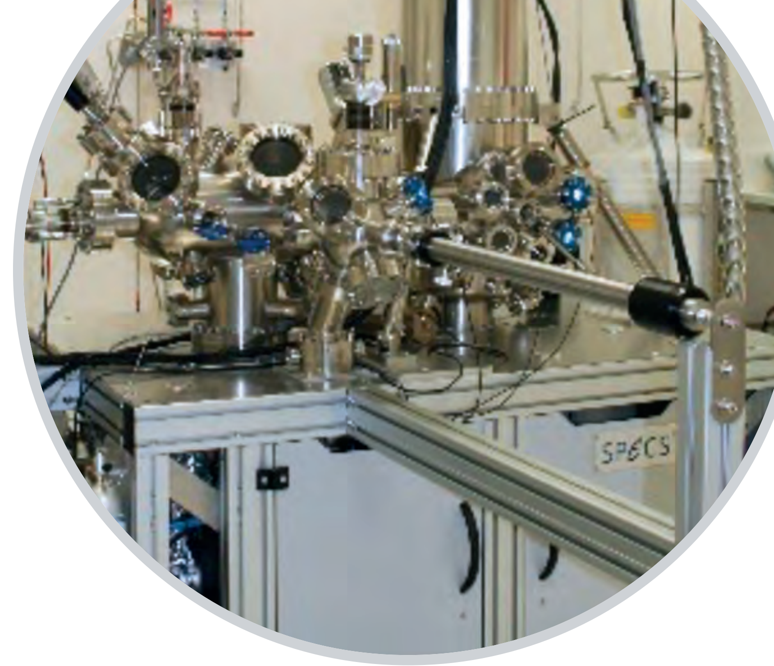
290 mV



-190



→ Identification of CN et I terminaison groups



JT-SPM SPECS

Sylvie Godey

- Low temperature Scanning Probe Microscope, 1.2 K minimum (Joule-Thomson stage)
- STM/AFM modes, nc-AFM, KPFM
- Length Extension Resonator (Kolibri sensor): f0= 1MHz K=540 kN/m Q≈100000 at 4K, - Nanonis controller
- XY Scan Range 300K/4K : ~22µm/~4µm, Z Scan Range 300K/4K : ~2.3µm/~0.42µm
- 3T maximum magnetic field perpendicular to sample surface
- Ar sputter gun for surface preparation, LEED-AES
- KENTAX evaporator, CO functionalisation

→ **APPLICATIONS**

- Structure and electrostatic properties of surfaces, adatoms, unique molecules or molecular assemblies, nanostructures, nano-objects
- Surface potential determination, single charge transfer detection

→ **ADVANTAGES & LIMITATIONS**

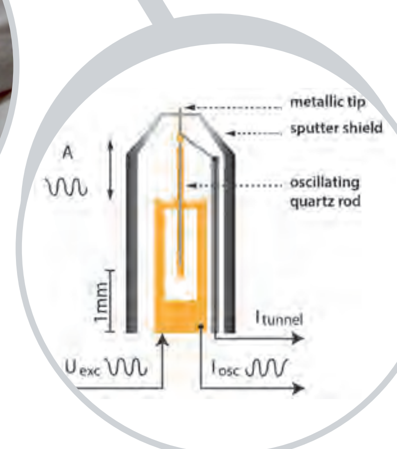
(+) AFM and STM simultaneous modes

(+) Submolecular resolution, tip functionalization

(+) constant height measurements

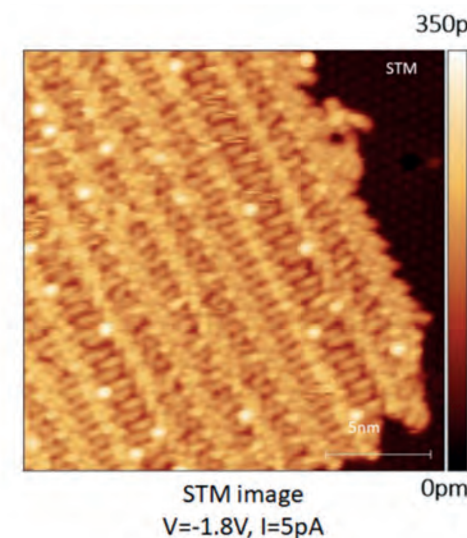
(−) need for a minimum density of objects of interest (of the order of one per 0.01 µm2) on about 1mm²

Kolibri Sensor



Self-assembled monolayers on Si:B

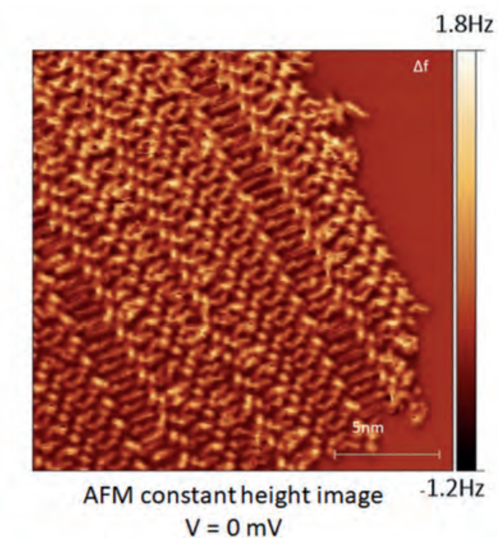
350pm



0pm

STM image
V=-1.8V, I=5pA

1.8Hz



-1.2Hz

AFM constant height image
V = 0 mV

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UHV DOMAIN SPM's

LT-STM Omicron

Maxime Berthe

- Surface imaging of conducting or semiconducting surfaces down to the atomic scale.
- Electrical testing on surfaces or nanostructures with atomic precision and ultra-low drift rate (<10pm/h).
- All modes of operation compatible with low temperature down to 4K.

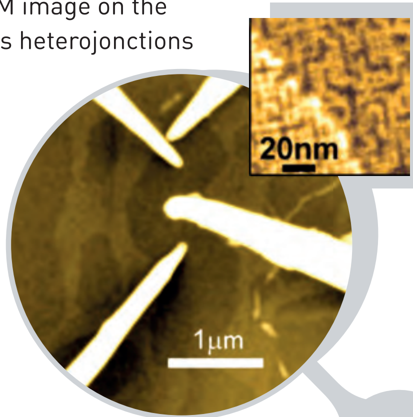
→ **APPLICATIONS**

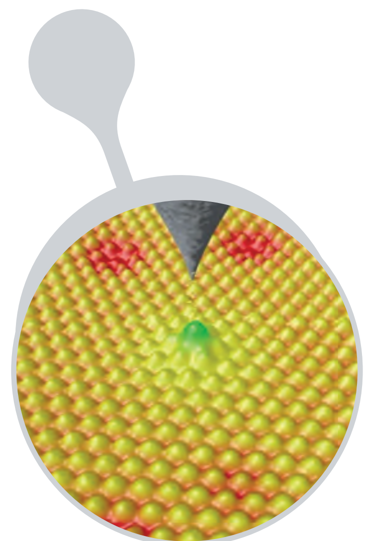
- Investigation of defects at the atomic scale in semiconductors and nanostructures by Scanning Tunneling microscopy (STM). Complementarity with TEM.
- Electronic properties of surfaces and nanostructures at the atomic scale by Scanning Tunneling Spectroscopy (STS). Complementarity with MBE, multiple-probe-STM, tunneling-induced light-emission spectroscopy.

→ **ADVANTAGES & LIMITATIONS**

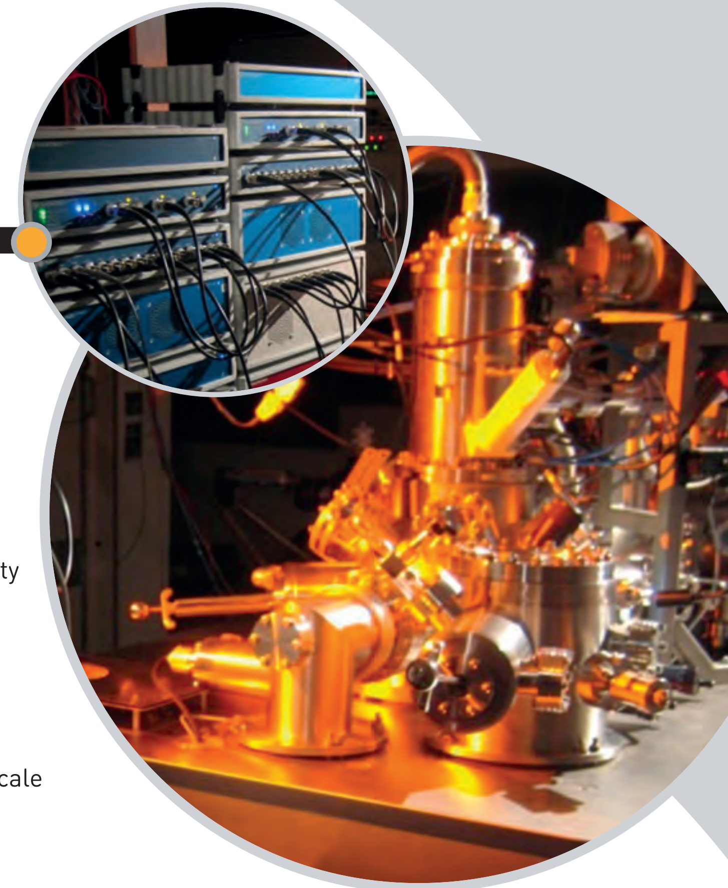
- Extreme resolution (better than 100pm)
- Electronic measurements (local electronic density of states)
- Limited aspect ratio : only flat surfaces
- Only conducting and semiconducting samples

SEM Image of a four-point-probe measurement on a single domain of colloidal nanocrystals heterojonctions. Inset : zoomed SEM image on the colloidal nanocrystals heterojonctions





3D representation of the reconstructed B-Si(111)-√3×√3 R 30°



NANOPROBE Omicron

Maxime Berthe

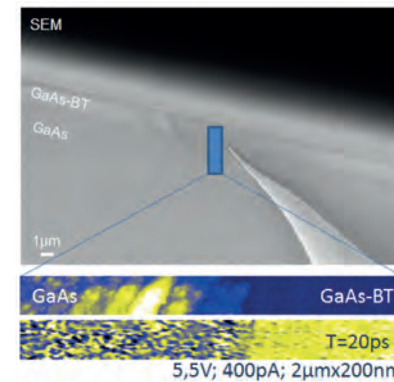
- Scanning Tunneling Microscopy (STM)
- Scanning Electron Microscopy (SEM)
- Nanoscale localization and manipulation
- Multiple-scale (100nm to 1mm) electronic transport measurements
- « fs-Laser-combined-multiple-probe-STM » for time-resolved (<1ps) nanoscale measurements .

→ **APPLICATIONS**

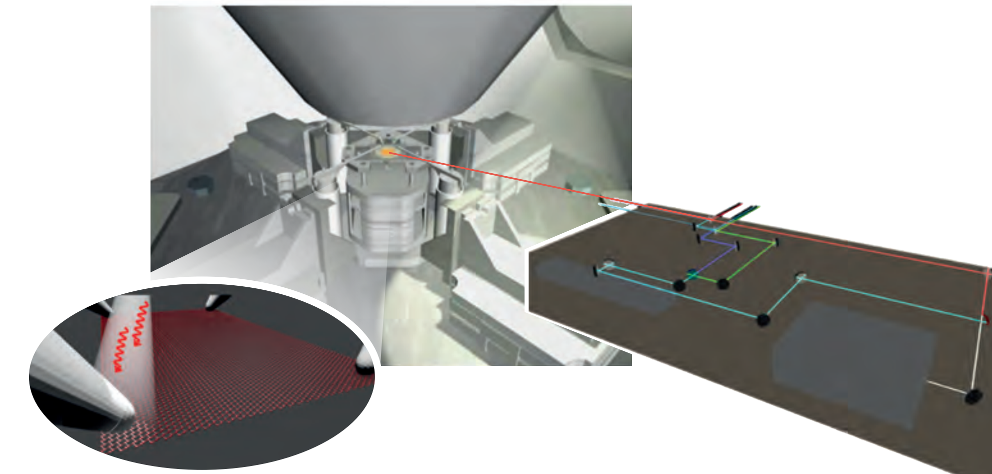
- Transport properties of surfaces and nanostructures. Complementarity with MBE, STM, tunneling-induced light-emission spectroscopy.
- Mapping of transport properties combined with STM. Complimentary with STM and electronics processing.

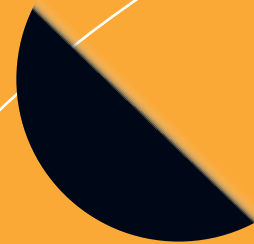
→ **ADVANTAGES & LIMITATIONS**

- Nanoscale imaging and manipulation with SEM monitoring
- Electronic transport measurements from nanometer to millimeter scale
- Limited STM resolution (nanometer) and stability



Top: SEM image of GaAs/LT-GaAs junction with one STM probe scanning accros the junction. Bottom: Simultaneous acquisition through STM probe of (i)Topographic STM image and (ii) Lock-in-demodulated ultrafast optical signal.





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