Ρ С Ρ

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 M. Berthe

• Air domain SPM's

→ Dominique Deresmes ICON DIMENSION MULTIMODE BIOSCOPE

• UHV domain SPM's

 \rightarrow Dominique Deresmes \rightarrow Maxime Berthe \rightarrow Sylvie Godey

VTAFM JT-SPM LT-STM NANOPROBE

I. 5-8

PCP **AIR DOMAIN SPM's**

AFM

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

100um

• 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



- PeakForce
- Thermal chuck for small sample → ADVANTAGES & LIMITATIONS

- Acoustic and vibrational Noise sensitive

💂 Louis Thomas

- 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

ICON

💂 Louis Thomas

- 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

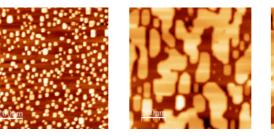
→ APPLICATIONS

- Large sample, large coarse displacement of the chuck
- (2µm resolution)





PCP **AIR DOMAIN SPM's**



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

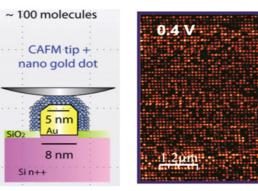
MULTIMODE Bruker

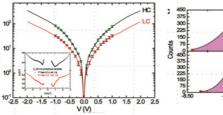
Louis Thomas

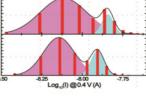
- Sample dimension : 5mm square to 15mm diameter
- Scan range : 10nm to 10 or 100µm (two scanners available) -Max. Z range: 2 or 5µm
- Resolution : Lateral: nanometric Vertical 30pm
- Working Mode : AFM Tapping, EFM, KPFM, CAFM, PFM, Force spectroscopy
- Environnement : Ambient air, Nitrogen gas and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

→ ADVANTAGES & LIMITATIONS

- Low noise imaging
- Small sample
- Limited coarse deplacement

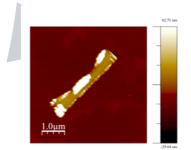


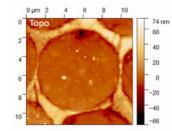




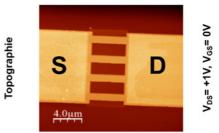
Log_(I) @0.4 V (A)

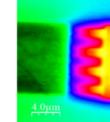
Conducting AFM statistics from a large array of sub-10 nm molecular junstions





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





BIOSCOPE Bruker

📕 Louis Thomas

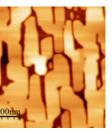
- Sample dimension : 5mm square to 5cm diameter
- Scan range : 10nm to 100µm Max. Z range: 6µm
- Resolution : Lateral: nanometric Vertical 80pm
- Working Mode : AFM Tapping
- Environnement : Ambient air and liquid
- Temperature : Ambient
- → APPLICATIONS
- In situ electrochemical growth monitoring

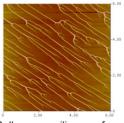
→ ADVANTAGES & LIMITATIONS

- O Tip Enhanced Raman Spectroscopy (TERS) tip optical bench
- Acoustic and vibrational Noise sensitive

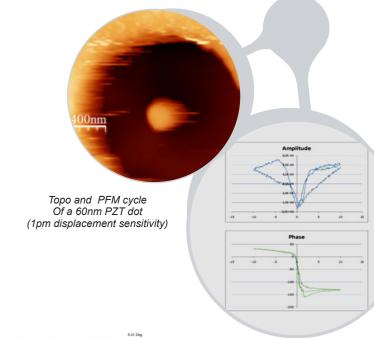
1.3

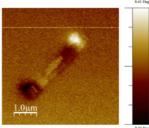
IEMN / PCMP



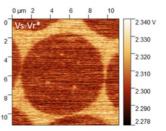


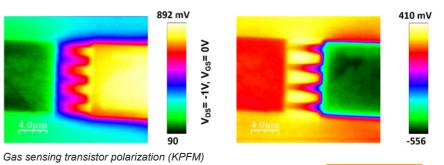
Collagen on silicon surface





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





IEMN / PCMP

1.4

VTAFM Omicron

Sylvie Godey

- 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



→ APPLICATIONS

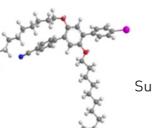
- Structure and electrostatic properties of surfaces, adatoms, unique molecules or molecular assemblies, nanostructures, nano-objects
- Surface potential determination, single charge transfer detection

→ ADVANTAGES & LIMITATIONS

- AFM and STM simultaneous modes
- OSubmolecular resolution, tip functionalization
- constant height measurements
- need for a minimum density of objects of interest (of the order of one per 0.01 μ m2) on about 1mm²

FM

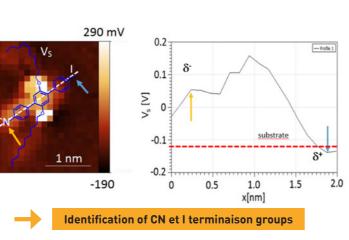
∆f image V_S=0mV



Sub-molecular resolution

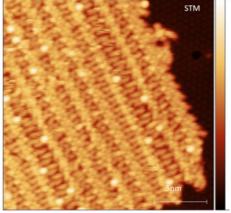
Model corresponding to nc-AFM image





Self-assembled monolayers on Si:B

350pm

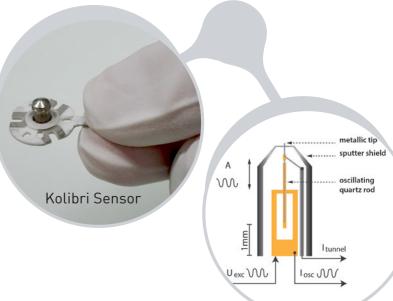


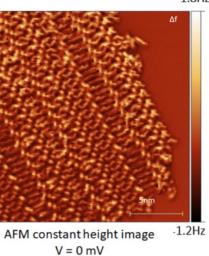
0pm STM image V=-1.8V, I=5pA

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





1.8Hz

PCP 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, tunnelinginduced 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

NANOPROBE Omicr

a 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 .

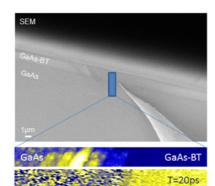
\rightarrow APPLICATIONS

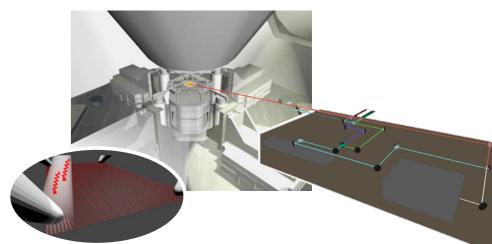
• Transport properties of surfaces and nanostructures. Complementarity with MBE, STM, tunneling-induced light-emission spectroscopy.

• Mapping of transport properties combined with STM. Complimentary with STM and electronics processing.

→ ADVANTAGES & LIMITATIONS

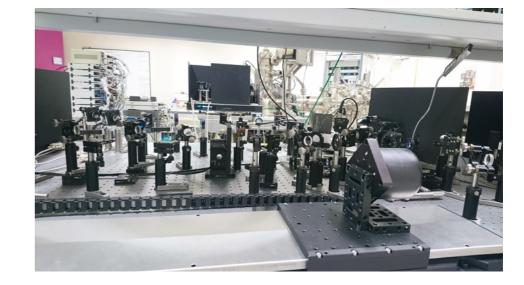
- Nanoscale imaging and manipulation with SEM monitoring
- S Electronic transport measurements from nanometer to millimeter scale
- Limited STM resolution (nanometer) and stability





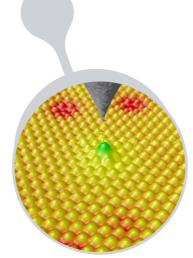
Top: SEM image of GaAs/LT-GaAs junction with one STM probe scanning accros the junction. Bottom: Simultaneous acquisition through STM probe of (i)Topographic STM image and (ii) Lockin-demodulated ultrafast optical signal.

.5V: 400pA: 2umx200nm

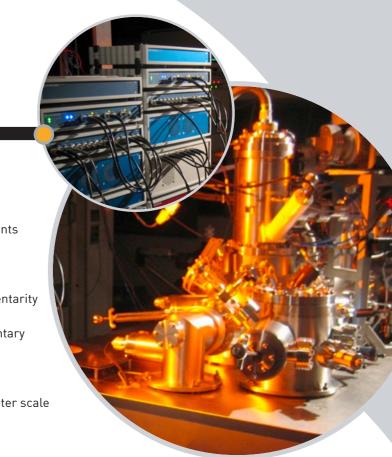


SEM Image of a four-point-probe measurement on a single domain of colloidal nanocrystals heterojonctions. Inset : zoomed SEM image on the colloidal nanocrystals heterojonctions

20nm 1µm



3D representation of the reconstructed B-Si(111)-V3×V3 R 30°





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