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LT NANOPROBE Atomically Precise Electrical Probing



- Transport measurements with atomic scale precision
- Four independent atomic resolution SPMs for probe approach and imaging
- Extremely low thermal drift at T < 5K</p>
- Simultaneous SEM imaging at low temperatures for rapid tip navigation
- STM spectroscopy and atom manipulation
- **QPlus nc-AFM**

Ultimate Nanoprobing at Low Temperatures



The Low Temperature NANOPROBE defines a new class of analytical instrumentation that merges SEM-navigated local transport measurements with atomic scale precision, high performance STM imaging, spectroscopy, and manipulation at LHe temperatures.

A major challenge in the development of novel devices in molecular and atomic scale electronics is their interconnection with larger scaled electrical circuits to control and characterize their functional properties. Local electrical probing by multiple probes with atomic precision can significantly improve efficiency in analysing electrical properties of individual structures without the need for full electrical integration. The LT NANOPROBE employs four independent, ultimate stability SPM's with precision in the picometer-range as individual probes for electrical transport measurements, and uses a scanning electron microscope for efficient and fast navigation of the probes.

The excellent stability allows for atomic resolution in STM and nc-AFM (QPlus) and expands applications towards the creation of atomically precise structures as well as their direct analysis by electrical transport measurements and SPM-based spectroscopy methods. The system - including simultaneous SPM and SEM operation - is operated near thermal equilibrium at T< 5K and has been optimised towards extremely low thermal drift as the most important ingredient to allow for enough measurement time on atomic structures, a precision regime that is virtually inaccessible at room temperature.

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Manipulation of a 4-Acetylbiphenyl (ABP) molecule [1,2] on Au(111), driven by inelastic tunneling effect. Data obtained during the TU Dresden team training session for the nanocar race at the PicoLab in Toulouse. Data by courtesy of F. Moresco, TU Dresden, Germany. References:

[1] F. Moresco et al., ACS Nano, 7, 191 (2013); [2] F. Moresco et al., ACS Nano, 9, 8394 (2015)

Inside the LT NANOPROBE Microscope stage design and optical access

Four SPMs

The microscope stage carries four individual SPM modules with independent and guided 3D coarse positioning of XYZ = 5 x 5 x 3mm. The sample can be independently positioned by XY = 4 x 4mm. Our patented piezo-electric inertia drives provide highly reliable and efficient navigation with step sizes from a few tens of nm's up to several hundred nm. Fine positioning and atomic resolution STM imaging is achieved by shared stack scanners with a range of $1.5\mu m \times 1.5\mu m$ at 5K. For ultimate STM performance the microscope stage employs an effective eddy current-damped spring suspension.

Sample & tip exchange

A fast and secure tip and sample exchange is crucial for ease of use and high throughput. Samples and spring loaded tip carriers can easily be exchanged by wobble stick.

Thermal shielding

An efficient thermal shield compartment allows for temperatures well below 5 K, extremely low thermal drift and thermal equilibrium of sample and probes. In addition, high resolution SEM navigation requires a small SEM working distance and thus makes a dedicated STM concept indispensable. A sophisticated shared stack scanner allows for a very compact and flat design, while ensuring highly linear, orthogonal and stable STM scanning characteristics.

Magnetic Fields

A compact superconducting coil is mounted beneath the sample plate and provides a vertical magnetic field of 20 mT.





LT NANOPROBE SEM Imaging and Tip Navigation at T< 5K

For the navigation of four independent STM probes, simultaneous SEM imaging is indispensable as it bridges dimensions from the mm-scale down to the nm-scale. The SEM enables a large field of view for probe coarse positioning as well as fine positioning and rapid localisation of nanometersized structures.





Sequence of SEM images taken at a sample temperature of T = 4.5 K. Probe navigation and positioning at the mm scale and high resolution imaging on Au islands on carbon with

The UHV Gemini column is the ultimate tool for that purpose. It offers unsurpassed resolution under true UHV conditions and at low temperatures.

In combination with the LT NANO-PROBE, the in-lens Secondary

Electron Detector (SED) represents a key advantage. Only one small access port is needed in the thermal shield compartment of the microscope stage (at T <5 K). Thus thermal impact is minimised, while still offering a suitable signal for high resolution imaging.

Alternatively, other UHV SEM columns or optical microscopes can cover the lower resolution range if sample structures do not require ultimate resolution.

typical size of 10-100nm. The SEM resolution with UHV Gemini column is specified to < 20nm using a 20/80 criteria (insert). Sub 10nm structures are visible.

20/80% Line profile (acc. to ASTM Standard)

Distance [nm]

809

ntensity [a.u.]

UHV Gemini

column SEM

and the sea

dx <20r

Error ± 0.6nm (8.6%)



ZnO nanowires with Au contacts characterised in the as-grown real device configuration using a local multi-probe method. Data courtesy: Steve P. Wilks et. al. / Nanotechnology 25 (2014) 425706

LT NANOPROBE STM for Probe Approach

STM is the key to advancing probing technology into the sub-nanometer scale. It ensures extremely accurate probe positioning and STM-based safe tip approach of fragile probe tips having diameters in the range of a few ten's of nanometers.

SEM ensures fast and efficient navigation of STM probes from the millimeter- to the nanometer-range. However, the STM tip shadows SEM imaging of structures smaller than the tip apex (please refer to page 10). Subsequently, STM is employed for atomically precise positioning of the tip and its point of contact.

Tip re-positioning accuracy is important as the probes are approached and positioned sequentially. During the approach process of an individual probe, the other probes need to be retracted to a safety distance of a few 100nm. When the positioning process is finished, all four probes are approached to contact. The outstanding precision of the LT Nanoprobe allows to land on the same atom again.

Electrical Transport Measurements

During STM approach the distance control is based on tunnelling current feedback and therefore requires a dedicated low noise I/V converter. When the tunnelling contact is established, the individual probe-sample distance is well controlled in the nm range.

To establish electrical contact and to control its resistance, the STM feedback is de-activated and the probe is manually approached by setting a piezo scanner z-offset. The individual contact properties are analysed by an IV measurement between the tip and the grounded sample.

Transport measurements in various configurations such as four-point transport or three terminal measurements with one tip acting as (tunnelling) gate, require the I/V converter to be taken out of the signal line. Thus, a pA STM compatible and TTL trigger controlled switching technology is used to route signals of the four probes to external BNC connectors. Using LabVIEW, experimental workflows can be integrated with third party measurement electronics.



Sequence of STM images on Si(111) over 10 minutes with 200nm tip retraction in-between. Absolute lateral thermal drift X/Y = 33/30 pm in 10 minutes. Virtually identical line profiles show re-positioning accuracy at the atomic scale (blue and black curves show backward and forward scan, respectively).



Schematic showing the function principle of signal re-routing from the probe tip either to the I/V converter for STM feedback to the MATRIX control system or to a BNC connector to connect external electronics.

LT NANOPROBE SPM Performance

Each STM module is designed to achieve atomic resolution on metal surfaces with pm stability, enabled by the high intrinsic stability of the STM stage and efficient decoupling from external vibrations by a spring suspension and eddy current damping. During the tip navigation phase, the damping stage can be locked for optimal SEM resolution while still achieving good atomic resolution.





pm (peak-to-peak). (10 nm x 10 nm, U__ = 150 mV, I_= 40 nA)

Atom manipulation

The LT NANOPROBE stability virtually matches state-of-the art performance of well-established single tip low temperature STMs and therefore enables extended experiments such as atom and molecule manipulation and tunnelling spectroscopy.



QPlus AFM

A suitable AFM technology is required for tip approach on structures on insulating substrates or electronically decoupling layers. The QPlus AFM detection principle is purely electrical and thus compact enough to be integrated into each probe module, using 3 electrical tip contacts. The concept of the QPlus AFM with a tip glued to the oscillation prong allows both to mount solid metal tips for transport measurements or to use alternative tip types.



LT NANOPROBE Low thermal drift

The thermal stability and resulting thermal drift of the whole microscope stage - four probe modules relative to the sample - is crucial for long-term measurements on nm-sized structures. It essentially defines the time window for sequential positioning of the four probes and the measurement time, during which probes can be held in electrical contact at atomic scale dimensions. The excellent drift performance in the few Å /h regime also enables single or multi-tip tunnelling spectroscopy experiments.



time approx. 2hrs and resulting lateral drift of 1.3Å /h.

Cooling Down to T < 5K

The use of a high-resolution SEM column for tip navigation from above implies an unconventional cryostat concept. A specifically designed bath cryostat with LN, and LHe reservoirs allows

for a measurement time of > 45hours at T < 5 K and cools the whole microscope stage from below. LN₂ and LHe double shields minimise the thermal impact on the stage. Doors to exchange tips or samples are operated by wobble stick.

As the UHV Gemini column uses an in-lens secondary electron detector, the thermal impact during SEM imaging is minimised and the microscope stage maintains the minimal temperature of T < 5K during SEM operation.



Two-probe STM experiments at the atomic level



The unprecedented stability and drift performance of the LT NANOPROBE opens the route towards multi-probe transport measurements of planar atomic or molecular nano-scale systems. So far the stability of single atom contacts and probe navigation with real atomic precision has only been accessible by single probe SPM experiments. Parallel STM imaging of probes is crucial to control the individual points of contact and to identify the minimal distance between probes by inter-probe tunnelling.

(A) Two-probe STM measurement on a dangling bond dimer wire supported on hydrogenated Ge (001).

Tip 2 is in electrical contact ($\Delta z = -0.45$ nm, R $\approx 25M\Omega$) while Tip 1 is in tunnelling regime (U_{Gan} = -0.5V, I_T = 20pA) and collects I/V spectroscopy data. Probe to probe distance reaching 30 nm. Insert I/V curves at 34.5 nm and 48 nm, respectively. Data by courtesy of: M. Kolmer¹, P. Olszowski¹, R. Zuzak¹, S. Godlewski¹, Ch. Joachim^{2,3}, M. Szymonski¹ (1) NANOSAM, Faculty of Physics, Krakow, Poland (2) Nanoscience Group CEMES/CNRS, Toulouse, France (3) MANA Tsukuba, Japan



(A) SEM image showing two relatively large STM probes in close proximity. The respective points of surface contacts are identified by matching large scan STM images of both probes. (B) STM image by Tip C. Tip B appears as a wall as tunnelling between the two probes occurs in close proximity. Data by courtesy of J. Yang, D. Sordes, M. Kolmer, D. Martrou, and Ch. Joachim, Eur. Phys. J. Appl. Phys. (2016) 73.

The Nanocar Race -An international scientific experiment

Nanocars have competed for the first time ever during an international molecule-car race in April 2017 in Toulouse (France). The vehicles, which consist of a few hundred atoms, were powered by electrical pulses during the 36 hours of the race, in which they had to navigate a racecourse made of gold atoms, measuring a maximum of 100 nanometers in length.

Four out of six nanocars have

the LT NANOPROBE. The race,

simultaneously been driven with

which was organized by the CNRS,

is first and foremost a scientific and technological challenge.



Principle 3D graphic showing two of four SPM probes moving the nanocars within the LT NANOPROBE.

> The race teams in the remote control room

Nanocar Race - the official results*

Position	1st	1st	3rd
Vehicle	/	57N	
University/ Country	Uni Graz / Rice Univ. Austria / USA	Univ. Basel Switzerland	Ohio Univers USA
Team Leaders	Leonhard Grill & James Tour	Remy Pawlack	Saw-Wai Hla Eric Masson
Surface	Ag(111)	Au(111) (shared)	Au(111)
Propulsion mechanism	Dipolar	Inelastic	Dipolar
Driving distance	150nm 1.5 hours	133nm 6 hours	43 nm 29 hours
Incidents	-	-	-



The LT NANOPROBE system located at the CNRS Centre d'Elaboration de Matériaux et d'Etudes Structurales (CEMES) in Toulouse.



The LT NANOPROBE UHV system concept



LT NANOPROBE Customer solutions



ports for sample sputtering, thin film evaporation, and a sample manipulator for heating up to 900°C.



is designed for direct connection to a transfer line and is equipped with a horizontal XP type preparation ber, and a separate manipulator for Lcooling and heating. An Orsay Vortex SEM

Inside the LT NANOPROBE Transfer through the system



LT NANOPROBE transfer

The possibilities for smart transfer of tips and samples throughout the complete UHV chamber and into the LT NANOPROBE stage are fundamental for comfortable routines and processes.

The LT NANOPROBE offers two different fast entry locks (FEL), located opposite to each other on both sides of the system. This positioning allows for easy provision of tips and samples. Two wobble sticks - both right angled to the transfer rods of the FEL - are taking over the 'inner tasks' like adjustment, handling, positioning or 'parking' of tips and samples within the 28-position-carousel.

Various lock valves assure for short pump-down times as well as stable vacuum conditions. The pumping is realized via efficient turboand ion-pumps.

The manipulator in the preparation chamber allows for tip and sample preparation, sputtering and depositioning.



Top view of the LT NANOPROBE UHV System

How to contact us



Technical Data

Probe module coarse positioning: Sample stage coarse positioning:

XYZ = 5 mm x 5 mm x 3 mm $XY = 4mm \times 4mm$ piezo-electric inertia drives

STM fine positioning / scan range: STM resolution: STM preamplification:

STM preamplifiers switch:

QPlus AFM resolution:

SEM resolution:

Probe / tip exchange:

Sample and tip storage: Sample and tip transfer: Sample exchange: Sample Size: Sample temp. measurement: XYZ = 1.5 μm x 1.5 μm x 0.3 μm @ LHe Atomic resolution on Au(111), Z-stability better 5pm @ LHe 3nA / 330nA ranges lowest STM imaging current < 2 pA Signal re-routing from STM pre-amplifier to external BNC connector (TTL controlled) Atomic resolution on NaCl(100) or Si(111)

30nm @ LHe 17mm working distance, in lens SED

In-vacuum spring loaded tip carrier exchange by wobble-stick 28 storage positions inside analysis chamber Fast entry lock with 5 transfer positions In-vacuum wobble-stick transfer 10mm x 10mm Si diode sensor at sample stage

Base temperatures: LHe and LN, Holding time: Initial cool down time (300K to LHe): < 9h Cool down time (77K to LHe): Max. bakeout temperature: Base pressure:

Control electronics:

- T < 5K, 77K, 300K > 45h

Two-probe STM measurement on a dangling bond dimer wire

supported on hydrogenated Ge (001). (See page 10)

- < 2h
- 150°C
- < 3 x 10⁻¹⁰ mbar (@ 300 K)
- Matrix SPM control system for 4 SPM's
- QPlus AFM extension

How to contact us:

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