VT XA Series - Variable Temperature UHV SPM

Benchmarking UHV STM
and AFM Technology

- 50 K – 500 K / 650 K
- True pA STM
- Improved dI/dV Spectroscopy
- Beam Deflection AFM
- QPlus AFM
- In-situ Evaporation

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and AFM Technology

VT Series: Major Product Releases:

1994 VT STM
1998 VT with Beam Deflection AFM
2003 sub -pA STM & improved spectroscopy
2008 VT with QPlus AFM

STM & AFM in UHV at Variable Temperatures

The proven Scienta Omicron eddy current damping allows for high resolution SPM even under non-ideal conditions.

After a period of rapid development, Scanning Probe Microscopy (SPM) has become a standard analysis technique in surface science.

The Scienta Omicron VT SPM is the established microscope in many research labs. It won the prominent R&D 100 award in 1996 and to the present day more than 500 instruments have been delivered and successfully installed around the world. The design concept and ultimate performance as well as the concept of easy and reliable operation guarantee the maximum output of results.

The 'VT series' has undergone a process of continuos improvement. In addition to major improvements such as sub-pA STM and QPlus AFM, a variety of unique customer requests have been realised and are now available as options to match today's research needs.

The volume of our customers' research results and publications is conclusive proof of the performance, quality, and versatility of the Variable Temperature SPM design.

VT XA Series - Variable Temperature UHV SPM

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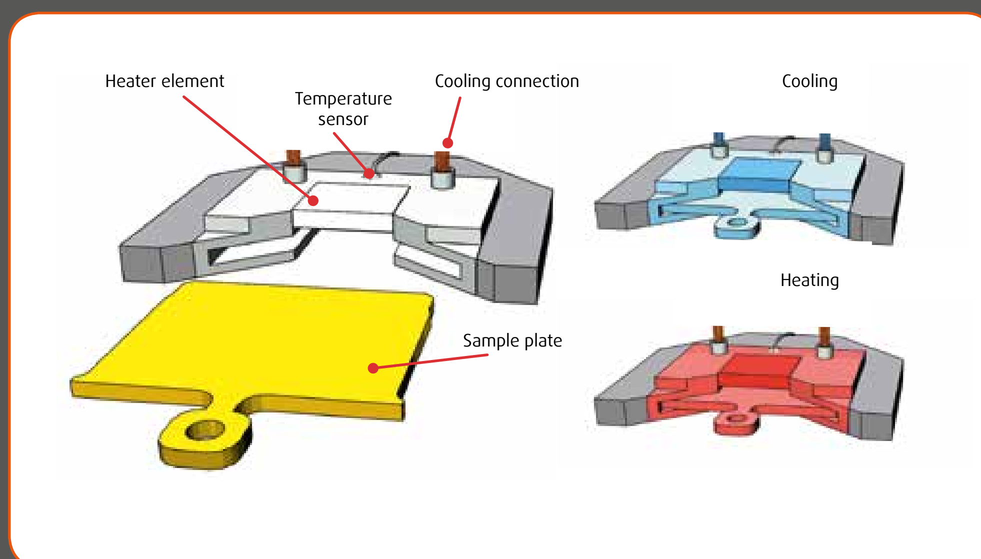
The VT XA Design

Heating and Cooling Concept for Temperatures from 50 K to 650 K

Cooling (down to 50 K)
The thermal connection (copper braid) between flow cryostat and sample stage is fixed and thermally isolated from the microscope stage.

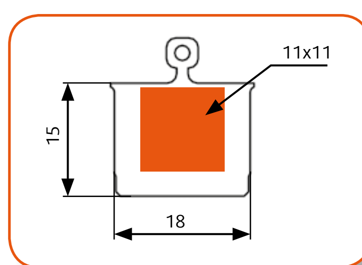
Heating (up to 650 K)
An embedded solid-state heater element in the sample stage allows indirect radiative heating of samples. The stage is equipped with a temperature sensor to measure the sample temperature. A temperature range between 50 K and 500 K is possible. Instruments without cooling option can heat the sample up to 650 K.



Sample Plates

The VT XA design forms a new series of VT instruments using standard Scienta Omicron samples plates. The pilosophy of the XA series is to provide maximum compatibility with many different surface science techniques, like MBE, RHEED and different kinds of electron spectroscopy.

Standard Scienta Omicron sample plates are available in Mo, Ta or stainless steel. Maximum sample
size is 11 mm x 11 mm.



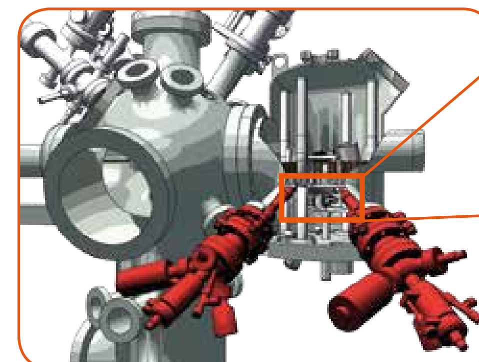
Dimensions of a standard sample plate [mm]

Standard sample plate

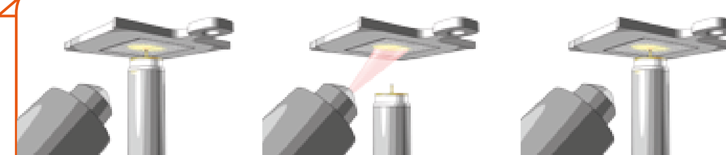
Direct-heating sample plate for sample preparation on the system manipulator

PEEM sample plate

In-Situ Growth

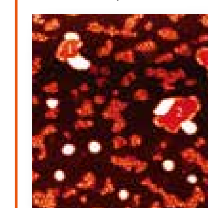


The VT series enables the mounting of up to two EFM evaporators on the VT-chamber for in-situ materials growth even during imaging.
The Scienta Omicron patented piezo inertia drive allows the same sample area to be relocated and imaged even after the sensor has been retracted during
evaporation.

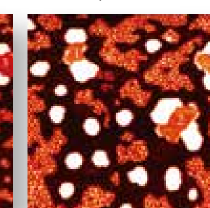


The VT STM incorporates a unique 3D tip positioning design ensuring completely independent movement in X, Y, and Z. The 3D positioning unit consequently allows precise tip coarse positioning in the sub-micrometer range. The guided positioning within a range of X/Y/Z = (10 x 10 x 10) mm allows tip coarse re-approach with an ultra-small lateral shift (typically < 50 nm) for several hundred coarse steps.

Before Evaporation



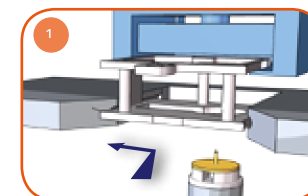
After Evaporation



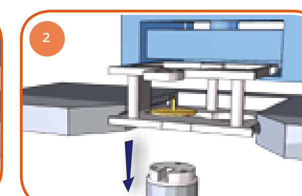
Growth of Pb on a Si(111) surface, image size: 80 nm x 80 nm, sample temperature 160 K, time interval: 16 min. The tip was completely retracted 10 mm between the two scans. An additional deposition of Pb was performed and the tip re-approached to the sample.

Data courtesy: M. Hupalo and M. C. Tringides, Dept. of Physics Iowa State Univ., Ames Lab. USDOE, USA, PHYSICAL REVIEW B, VOLUME 65

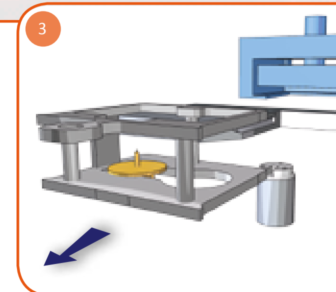
Easy and Safe Sensor Exchange



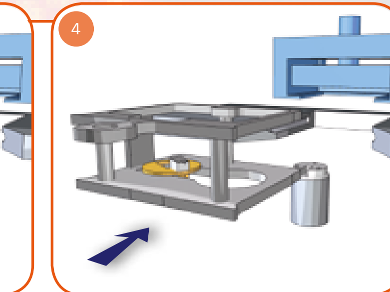
1) The transfer plate is inserted into the sample stage to pick up a sensor using the 3D tip coarse positioning.



2) The scanner is moved out of the ‘keyhole‘ of the transfer plate.



3) The sensor is removed from sample stage using a wobble stick (not shown).



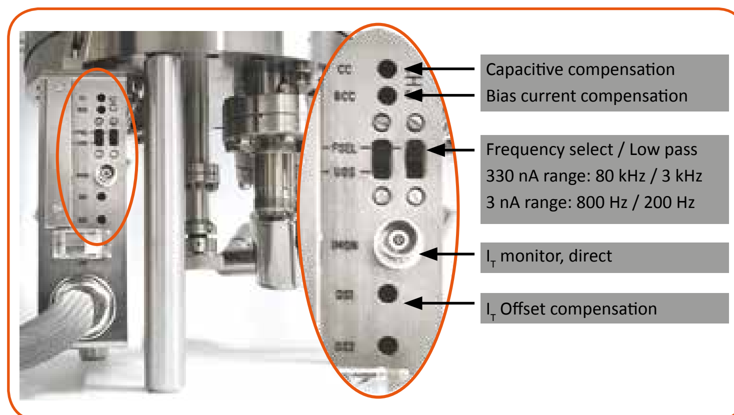
4) A different sensor is inserted into the sample stage.

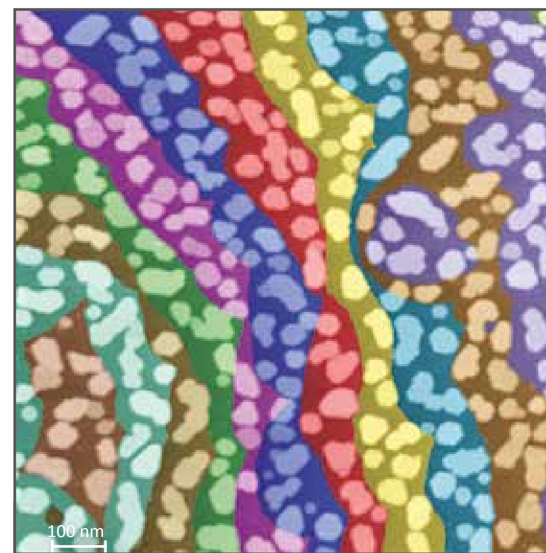
In-situ tip exchange is crucial for flexible UHV scanning probe microscopy. Up to 12 different sensors can be stored in the carousel of the VT chamber. The different sensors are exchanged by remote-control using Scienta Omicron’s patented piezo-inertia coarse positioning drives.

A sensor is transferred through the UHV system on a transfer plate with a ‘keyhole‘ cut-out. It is picked up by the scanner under user-driven remote-control, reducing risk of mechanical damage to a minimum.

True pA STM and Tunneling Spectroscopy

All instruments of the VT series are equipped with state-of-the-art STM and STM spectroscopy capabilities. It is possible to extend this with AFM functionality, and the VT instruments can be used either with Beam Deflection AFM, the 'QPlus' AFM, or with both types of AFM.





Growth of multilayer ice films and the formation of cubic ice imaged with STM at I_T = 0.4 pA. Ice films as much as 30 molecular layers thick can be imaged with scanning tunneling microscopy (STM) when negative sample biases -6 ±1 V and sub-picoamp tunneling currents are used. (K. Thürmer and N. C. Bartelt, PHYSICAL REVIEW B 77, 195425 (2008), Physical Review Letters 100,186101 (2008))

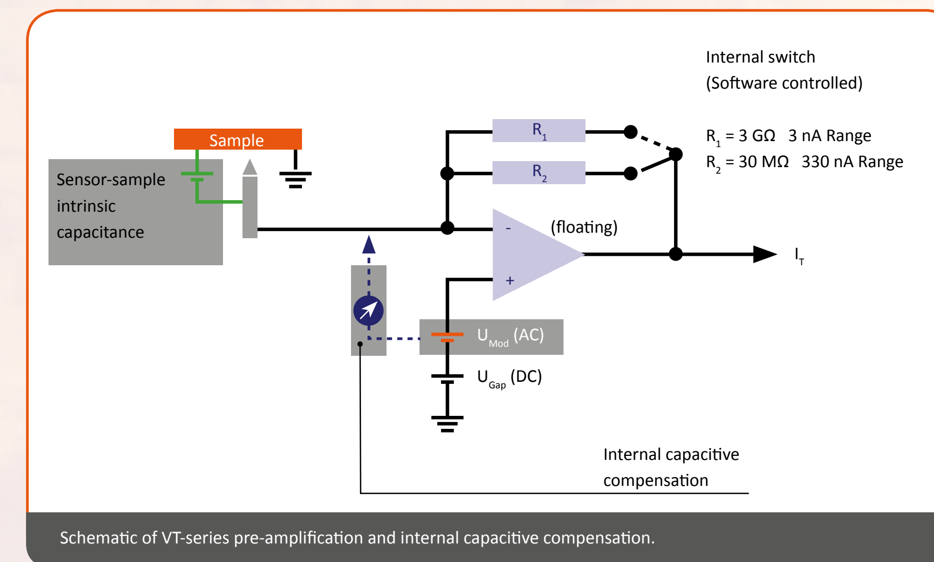
IVC H3 - Superior STM Pre-Amplification

The IVC H3 is the latest development of Scienta Omicron's in-vacuum pre-amplifier, designed to provide signal/noise ratios close to the theoretical minimum. The IVC H3 allows low current STM (<1 pA) and applications where higher currents (up to 330 nA) are required. Sophisticated experiments have shown that the IVC H3 is capable of stable STM operation at 50 fA. Two internal feedback resistors, switchable by the MATRIX software, allow a tunneling current range from 1 pA to 330 nA without interruption of the experiment, changing cables or changing the preamplifier.

Offset Compensation for *True* pA STM

Normally, intrinsic current and voltage offsets are mixed due to the I/V conversion by the operational amplifier. These intrinsic currents cause typical offsets of 1 pA. Normal I/V converters do not separate between these currents in an offset adjustment. The IVC H3, however, offers separate offset compensation, thus allowing *true* pA STM operation.

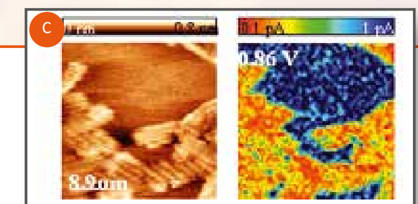
Capacitive Compensation via Preamplifier Technology

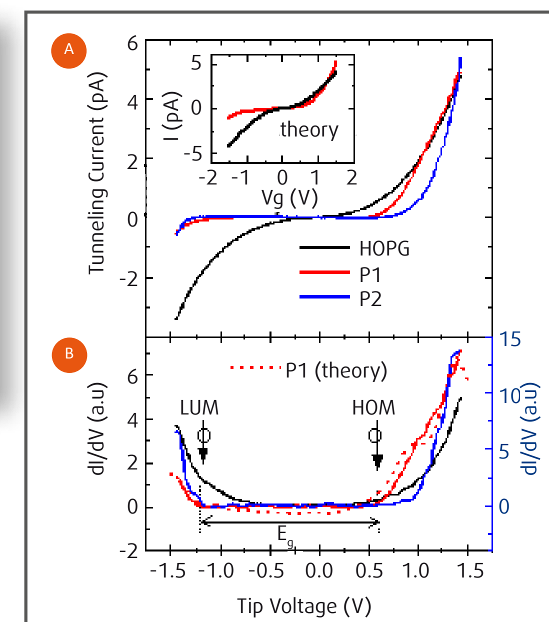


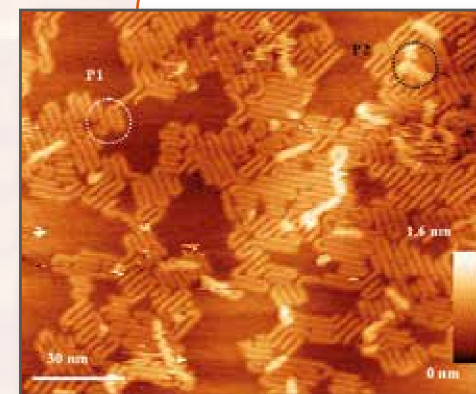
Schematic of VT-series pre-amplification and internal capacitive compensation.

Optimized for Modulation Spectroscopy

The intrinsic capacitance, both between tip and sample as well as in the microscope wiring, is typically in the range of a few picofarads. Modulation techniques that apply a modulated voltage to U_{gap} can thus cause parasitic currents up to several nA. The IVC H3 reduces these parasitic currents through its internal capacitive compensation, reducing acquisition time and improving results.







STM image (150 nm × 123 nm) of a poly (3-dodecylthiophene) P3DDT film on HOPG with sub-monolayer coverage obtained in the low-current mode. Set-point values I_T = 2.5 pA, U_{Gap} = 1.4 V. The white and black dotted circles highlight the first polymer layer and a partially nucleated second layer, respectively.

(A-B) Experimental and calculated STS spectra.
(A) Experimental I-V curves recorded during a CITS measurement. Over the bare substrate (HOPG, black), the first polymer monolayer (P1, red) and the second polymer layer (P2, blue).
(B) Conductance dI/dV spectra (solid lines).
(C) Topographic and current images acquired during a CITS measurement. Left: topographic image (50 nm × 50 nm) recorded with I_T = 3.9 pA, U_{Gap} = 1.2 V. Right: current image at 0.86 V.
Literature: Probing the Electronic Properties of Self-Organized Poly(3-dodecylthiophene) Monolayers by Two-Dimensional Scanning Tunnelling Spectroscopy Imaging at the Single Chain Scale; L. Scifo et al., Nano Lett. 6, 1711–1718 (2006)

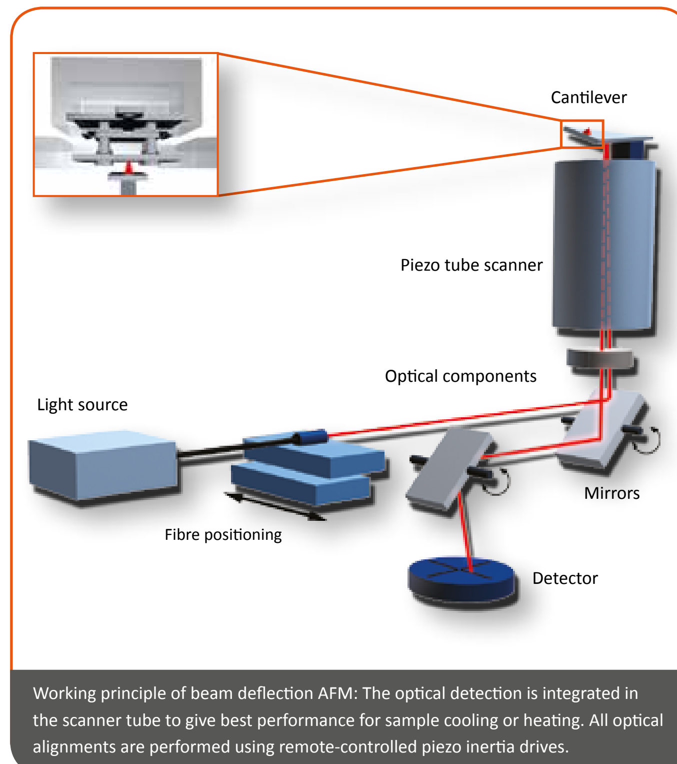
Beam Deflection AFM

The classical Beam Deflection AFM method uses a microscale cantilever with a sharp tip at its end. The cantilever is typically silicon or silicon nitride with a tip radius curvature on the order of several nanometers.

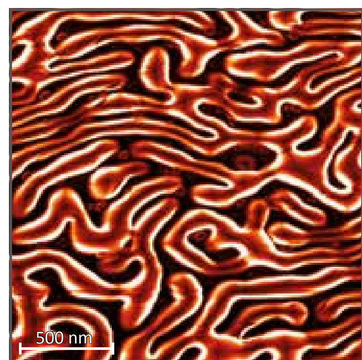
The force interaction between the tip and the sample surface leads to a deflection of the cantilever. In a Beam Deflection AFM the deflection is measured using a laser spot reflected from the top surface of the cantilever into an array of photodiodes. Depending on the application it is possible to operate in the static contact mode or in the dynamic non contact mode.
High resolution AFM, as well as Electrostatic Force Microscopy (EFM), Scanning Kelvin Probe Microscopy (SKPM), and Magnetic Force Microscopy (MFM), is possible.

There is a broad range of commercially available cantilevers with spring constants from 0.1 N/m up to 100 N/m. In dynamic modes, typical oscillation amplitudes are about 10 nm, and oscillation amplitudes down to 1 nm are possible with stiff cantilevers.

Cantilever mounted on an exchangeable tip carrier.



Working principle of beam deflection AFM: The optical detection is integrated in the scanner tube to give best performance for sample cooling or heating. All optical alignments are performed using remote-controlled piezo inertia drives.



Magnetic force Microscopy (MFM) image taken at a Co/Pt multilayer surface.

QPlus AFM

The 'QPlus' sensor is a new approach for non-contact-AFM. It is ideally suited for high resolution AFM with small oscillation amplitudes.

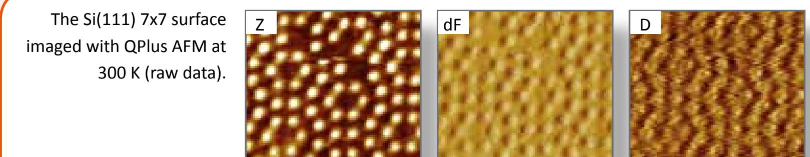
The QPlus sensor uses a modified quartz tuning fork. One prong of the tuning fork is fixed while the SPM probe tip is mounted to the (free oscillating) second prong. It acts as a quartz lever, transforming its oscillation into an electrical signal as a result of the piezo-electric effect. The sensor is excited mechanically by employing the scan piezo.

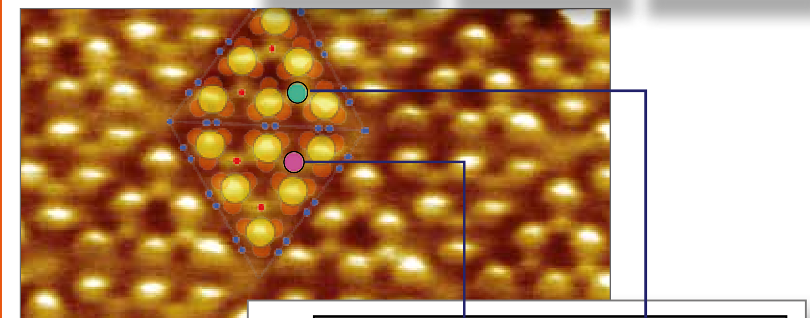
Due to the stiffness of the QPlus sensor (spring constant ~ 1800 N/m), it can be operated with smaller oscillation amplitudes compared to conventional cantilevers. With a unique signal detection concept, ultimate QPlus performance, low oscillation amplitudes, and high frequencies are possible, making the QPlus sensor ideally suited for high resolution AFM with small oscillation amplitudes. The tip of the QPlus sensor can be tungsten or any other suitable tip material and is easily mounted by gluing it onto the end of the tuning fork.

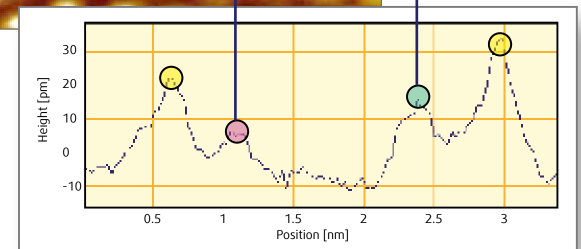
The sensor is also useful for easy AFM navigation of an STM tip and subsequent STM imaging and spectroscopy. Due to the stiffness and the use of solid metal tips, there are no negative impacts on the STM performance. The QPlus sensor is therefore ideal for inclusion in STM's, and also as a complementary sensor for beam deflection instruments.

QPlus sensor mounted on an exchangeable tip carrier.

The Si(111) 7x7 surface imaged with QPlus AFM at 300 K (raw data).



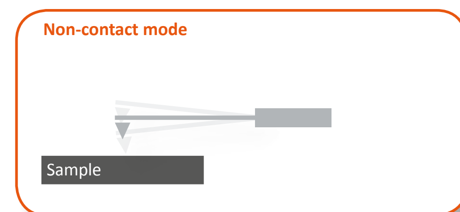




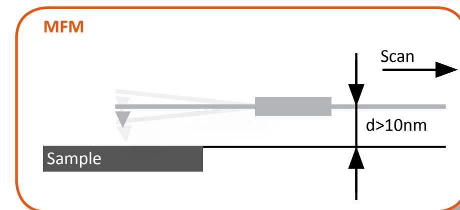
Imaging of rest atoms (raw data) of the Si(111) 7x7 surface with QPlus AFM at 50 K. Line profile showing the height difference of the rest atoms. Measurement: Scienta Omicron

AFM Modes and Forces

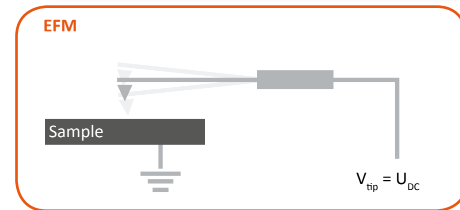
Non-Contact Modes



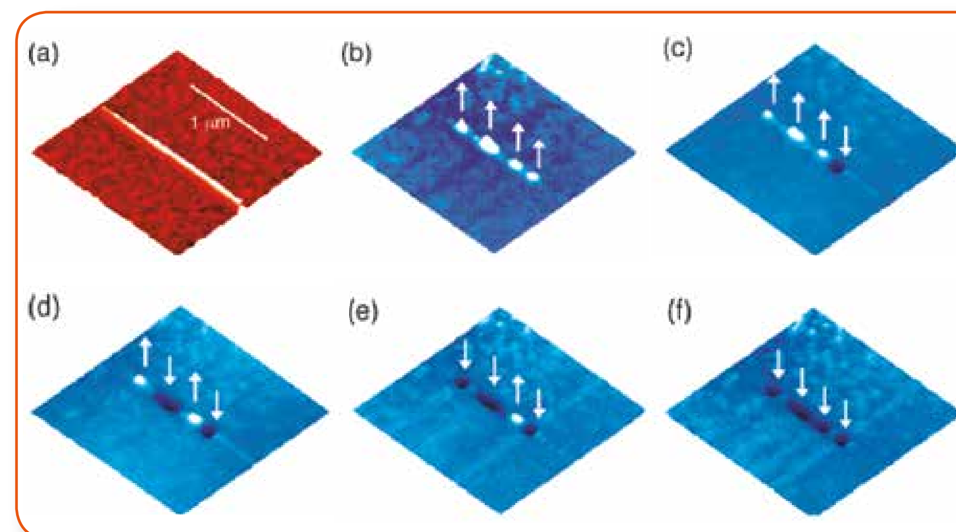
Non-Contact Operation Modes: The sensor is forced to oscillate or is excited to oscillate at its resonance frequency (eigenfrequency). There are two basic modes of operation; amplitude modulation (AM) and frequency modulation (FM). FM is the preferred mode in-vacuum, as the quality factors of the sensors (q-factors) are typically higher than in air.



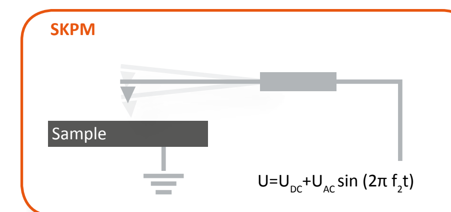
Magnetic Force Microscopy (MFM): Non-contact AFM can be used to measure magnetic forces. The magnetic dipole interaction is recorded at a constant tip-sample distance. The typical distance between tip and surface is >10 nm to exclude influences by van der Waals forces.



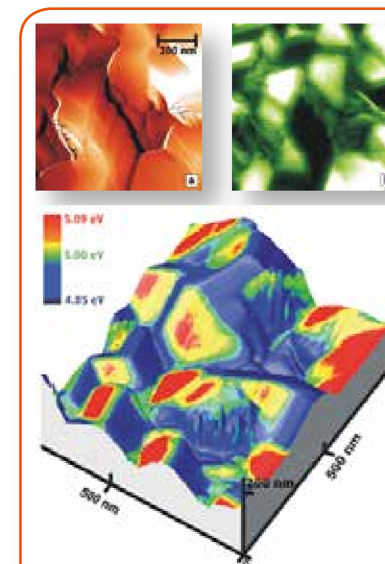
Electrostatic Force Microscopy (EFM): By applying a voltage between tip and sample, EFM can be used to study capacitance, surface potential, charge or dopant distribution, topography, and dielectric properties of metallic and insulating surfaces, and can be used to deposit and image localized charges on insulators.



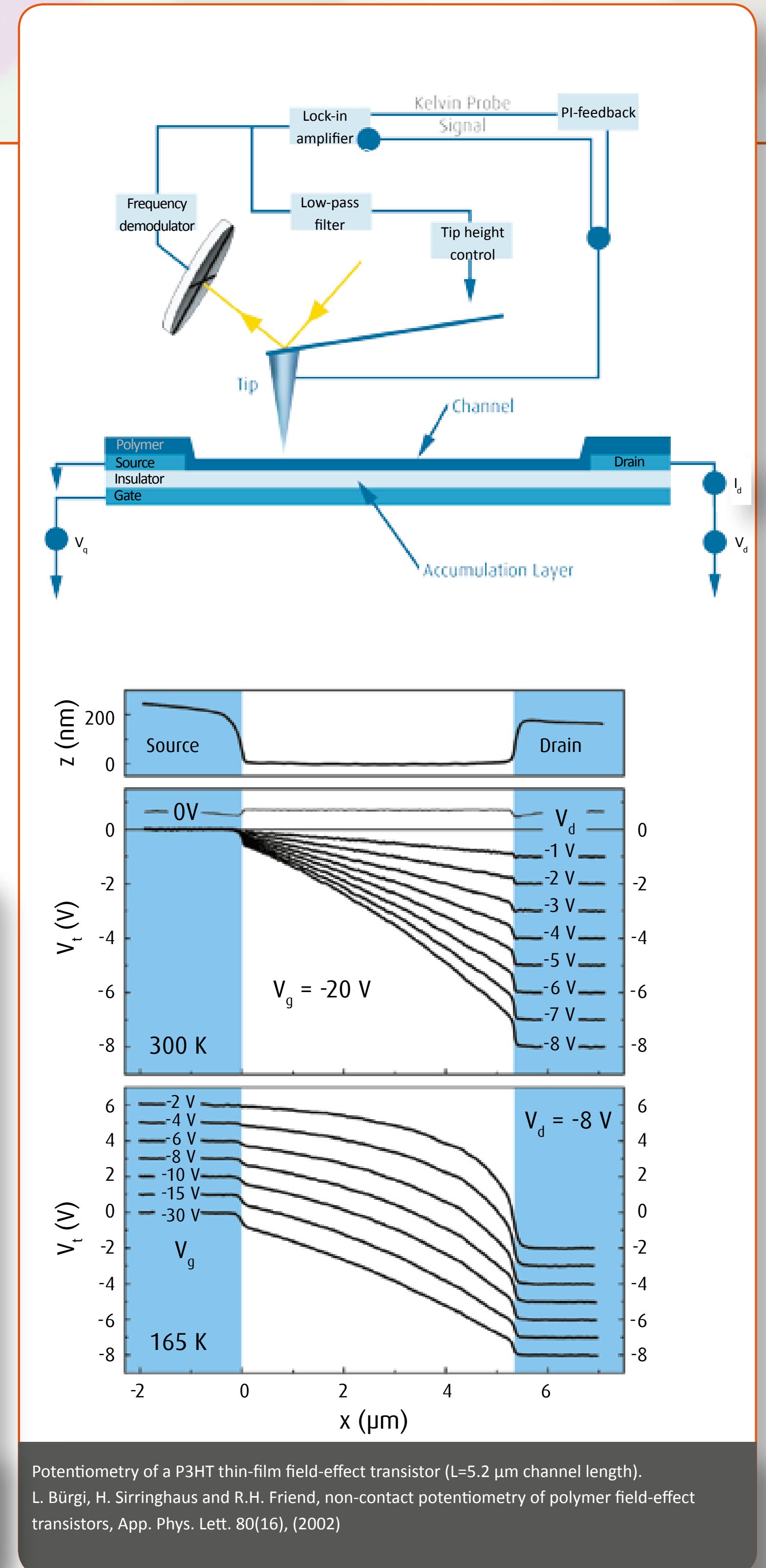
EFM: scanned probe investigations on the ferroelectric properties of individual single-crystalline barium titanate nanowires. Electrical polarization can be reproducibly induced and manipulated on these nanowires, thereby demonstrating that nanowires as small as 10 nm in diameter retain ferroelectricity. Scan range: 1800 nm x 1800 nm. Data courtesy: Hongkun Park, Harvard University, Cambridge, Massachusetts 02138, USA.



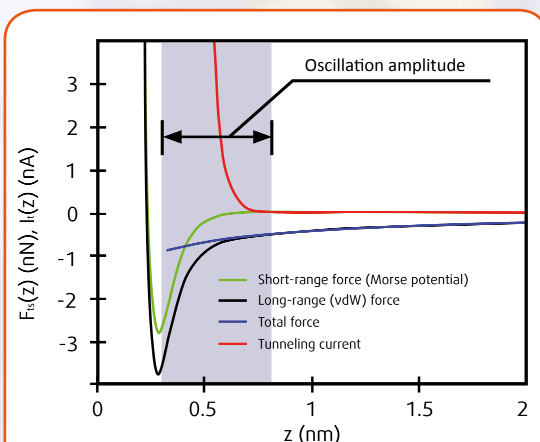
Scanning Kelvin Probe Microscopy (SKPM):
In SKPM an AC and a DC voltage are applied between tip and sample. The lock-in technique provides information on work function changes on a surface and correlates it with the simultaneously recorded topography information. Detection at frequency $2f_2$ can be used for Scanning Capacitance Microscopy (SCM).



Work function imaging of semiconductor surfaces using UHV Kelvin Probe Force Microscopy
Data courtesy of: S. Sadewasser, Th. Glatzel, M. Rusu, A. Jäger-Waldau, M. Ch. Lux-Steiner, Hahn-Meitner Inst., Berlin, Germany



Potentiometry of a P3HT thin-film field-effect transistor (L=5.2 µm channel length).
L. Bürgi, H. Sirringhaus and R.H. Friend, non-contact potentiometry of polymer field-effect transistors, App. Phys. Lett. 80(16), (2002)



Plot of tunnelling current and typical force potentials as a function of distance between surface and front sensor atom. For AFM, interaction involves many more tip atoms than for STM. Shaded area: small QPlus oscillation amplitudes in combination with specific df setpoints allow for tuning the sensitivity to various force ranges.

Non-Contact AFM with Small Oscillation Amplitudes using a QPlus Sensor [1]

Short range forces determine the atomic contrast in non-contact AFM mode. Conventional cantilevers with a typical spring constant of 10 N/m access this regime by employing comparably large oscaillation amplitudes (~10 nm) to avoid a 'jump-to-contact' of the tip.
Giessibl[1] et al. found empirically, that atomic resolution is possible when the product of the cantilever spring constant k and the amplitude A of the cantilever oscillation (i.e. the restoring force) is larger than about 100 nN.
However, the interpretation of the images is difficult, since long range forces contribute heavily to the frequency shift and the tip-sample force gradient can vary by orders of magnitude during a single oscillation of the tip.

In contrast, small oscillation amplitudes allow for straight-forward data interpretation since the frequency shift Δf is independent of the amplitude and proportional to the tip-sample force gradient.

Optimum resolution is achieved when working with sub-nm amplitudes close to the surface. This requires a spring constant k of larger than 1000 N/m.

[1] F. J. Giessibl et al., Appl. Surf. Sci. 140, 352 (1999)

Contact Mode

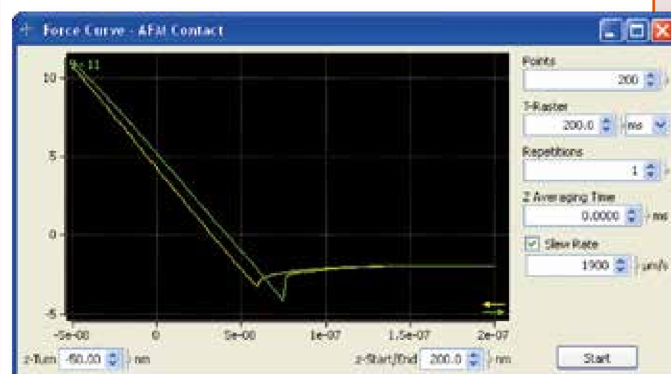
Contact mode

Contact Mode:

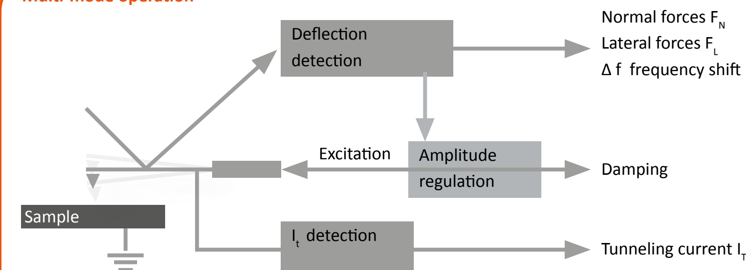
A tip mounted on a spring (cantilever) is brought into contact with the sample surface. Scanning the tip across the surface bends the cantilever, which is detected by the deflection of a light beam.
The measurement of force-distance curves gives information about the forces between the tip and the sample and can be used to measure surface properties such as adhesion, elasticity, nanohardness, etc.

Friction Measurements

Cantilever AFM also allows the measurement of lateral forces in addition to the detection of normal forces between tip and surface.



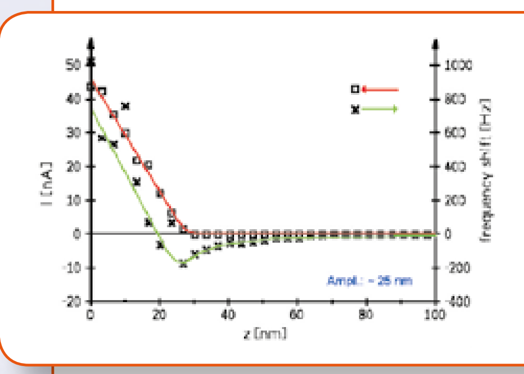
Multi-Mode



Multi-Mode Operation & Spectroscopy:

Multi-mode operation uses simultaneous data collection of several signals. Tunnelling current, normal force, lateral force, frequency shift, and damping signals are available simultaneously with the MATRIX SPM controller. Furthermore, external inputs can be selected in order to obtain useful multi-mode spectroscopy combinations.

The choice of feedback regulation is independent of the combination of different channels for data acquisition. A wealth of possibilities is opened up by the combination of AFM measurements with current detection. More physical properties of the sample, e.g. piezoelectricity or transport characteristics, can be investigated.



Simultaneous spectroscopic measurements of tunnelling current and frequency shift versus distance using a conductive cantilever. The onset of the tunnelling current signal coincides with the onset of repulsive force interactions.

Forces in Atomic Force Microscopy AFM

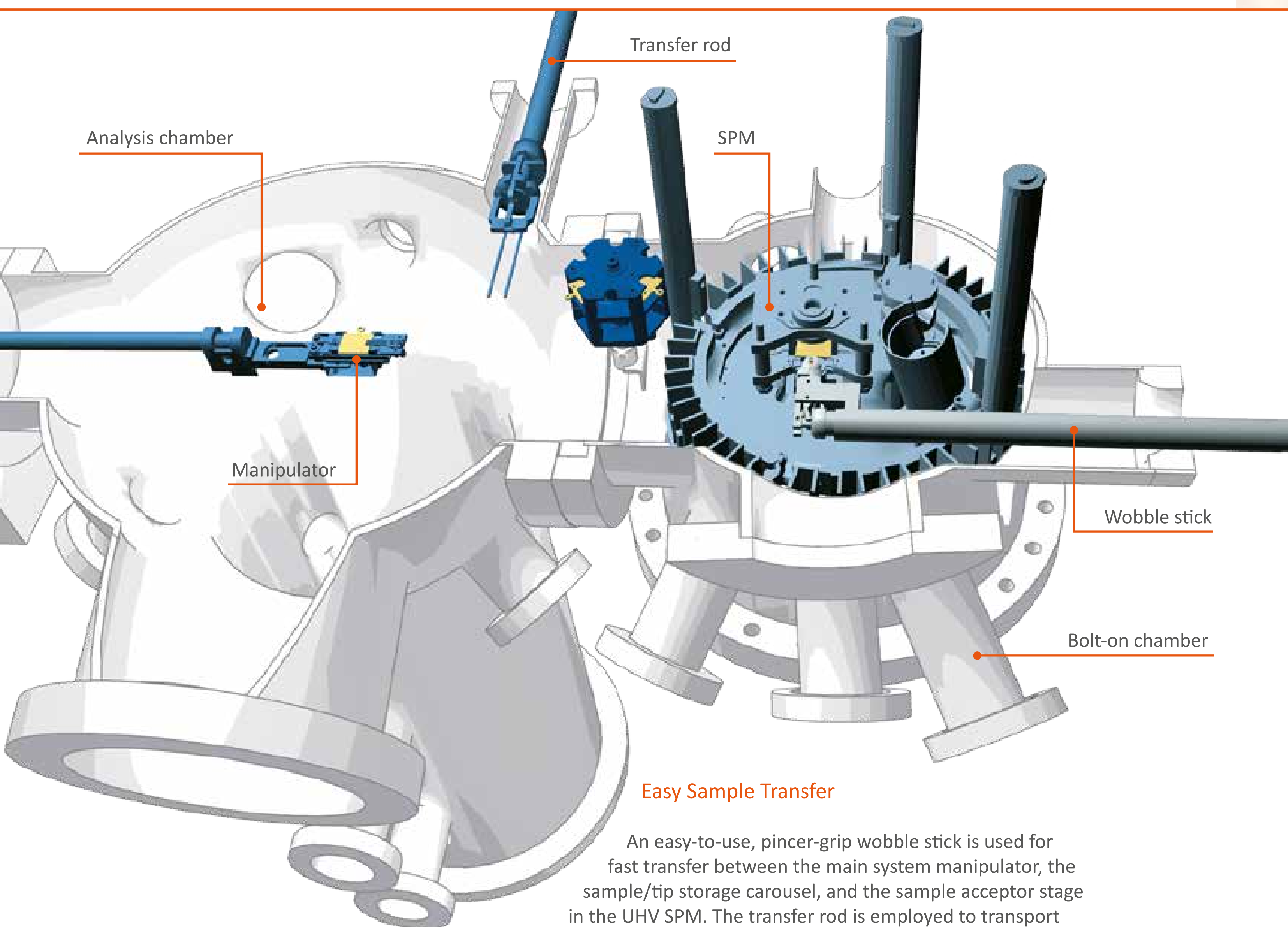
Electrostatic Forces (image forces, capacitance forces, work function inhomogenities, forces due to tip and surface charging) are always present. The electrostatic force is a long range force (several 100 nm) and can be attractive or repulsive. Electrostatic forces can be measured to characterize various surface properties (e.g. capacitance, surface potential and charge or dopant distribution).

Van der Waals Forces are always attractive. Although they are small by macroscopic standards, they significantly exceed the chemical forces and in many cases dominate tip-surface interaction. The van der Waals force is a long range force (up to 100 nm).

Chemical Forces are short range forces (fractions of a nm). They can be repulsive or attractive. Although chemical forces are not the strongest forces present between tip and surface, they are the most important as they can distinguish atomic identities, and are therefore responsible for atomic resolution in images.

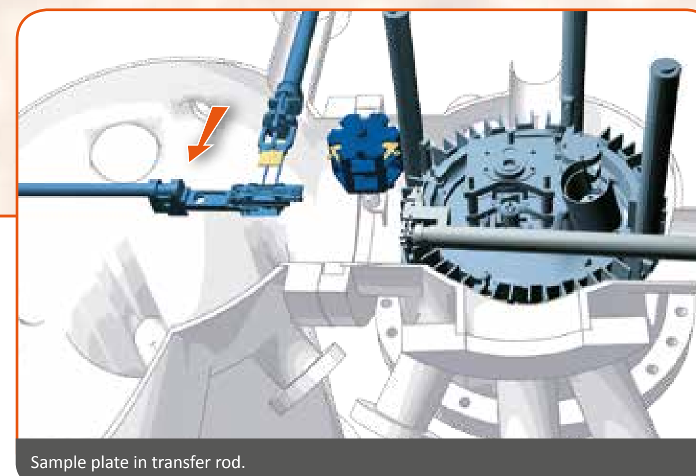
Magnetic Forces are important when both tip and sample have magnetic attributes.
The magnetic dipole interaction between the magnetic tip and the stray field of a magnetic sample is long-range (several 100 nm). Typical tip sample distances for MFM (Magnetic Force Microscopy) are above 10 nm.

Handling & Navigation

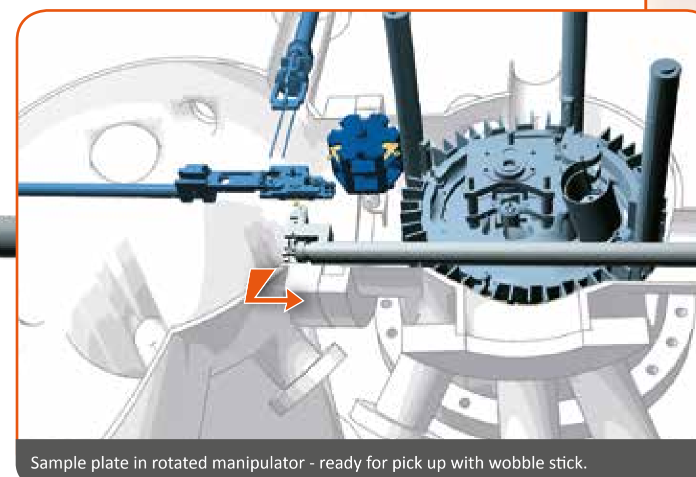


Easy Sample Transfer

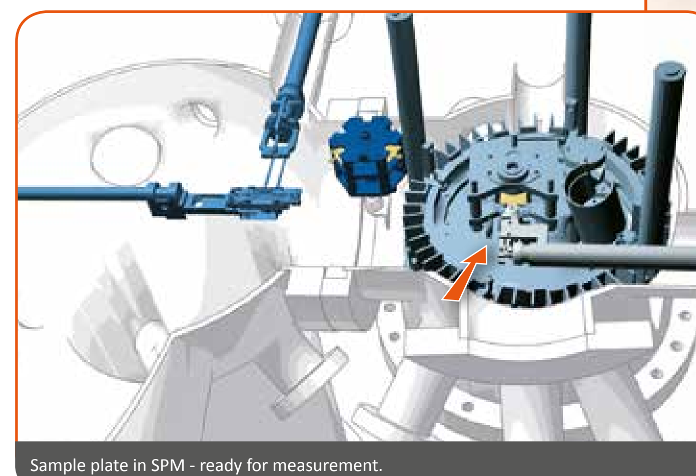
An easy-to-use, pincer-grip wobble stick is used for fast transfer between the main system manipulator, the sample/tip storage carousel, and the sample acceptor stage in the UHV SPM. The transfer rod is employed to transport samples and tips to and from the fast sample entry chamber or preparation chamber.
The bolt-on chamber houses a sample carousel for up to 12 samples/tips. Transfer of samples is quick and reliable using a wobble stick. Viewports on the bolt-on chamber allow optimal observation of the tip/sample coarse positioning using the external CCD camera.



Sample plate in transfer rod.



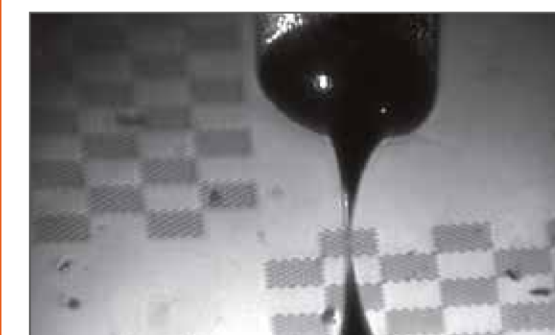
Sample plate in rotated manipulator - ready for pick up with wobble stick.



Sample plate in SPM - ready for measurement.

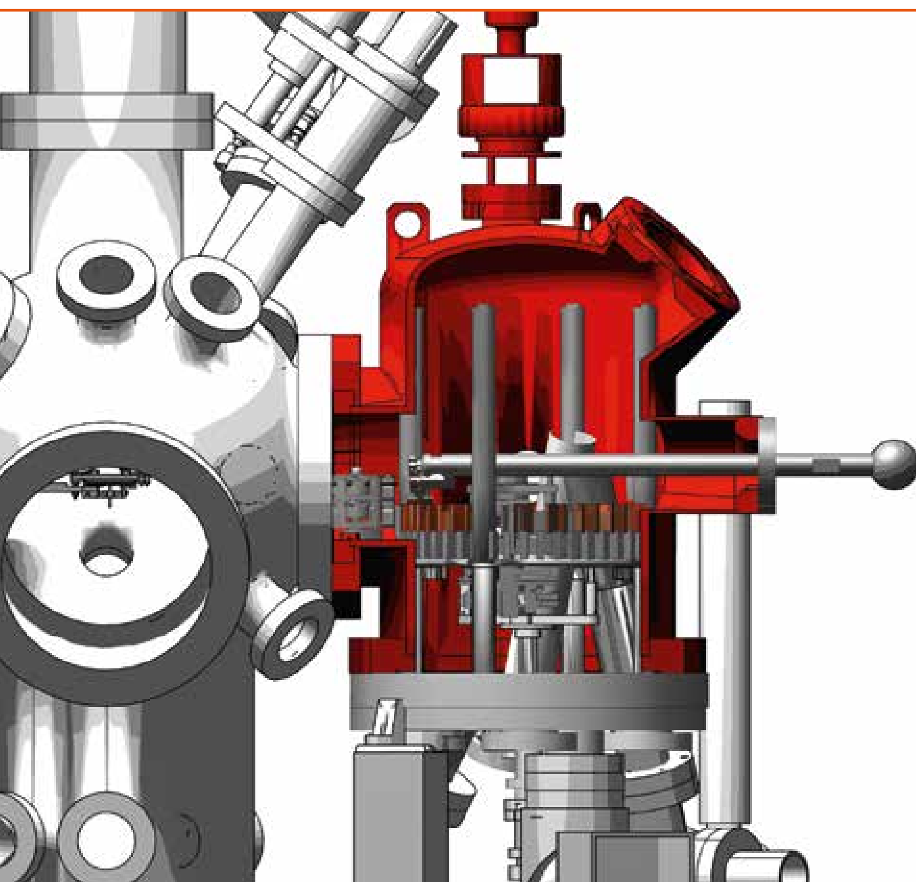
Optical Access

The VT STM can optionally be equipped with a long focal length optical microscope for sensor navigation (shown above on a Scienta Omicron system).
A dedicated set-up with a high-resolution CCD camera provides optical resolution below 10 µm.



The combination of the VT STM´s large scan range and (10 x 10 mm) orthogonal X/Y coarse positioning allows for locating and re-locating nanostructures by employing suitable navigation marks on the surface.

Integration & Bolt-on Concept

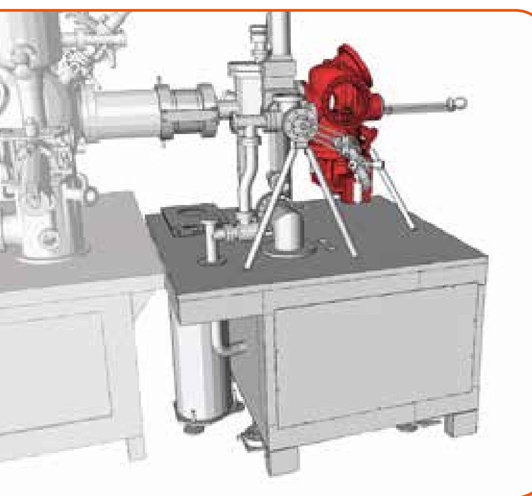


Easy UHV System Integration

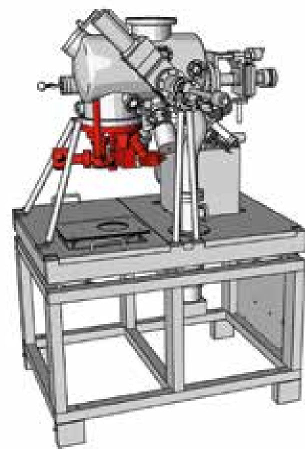
The VT instruments are housed in their own, dedicated UHV chambers which can either be mounted onto a standard Scienta Omicron system (MULTIPROBE™...) or bolted-on to an existing vacuum system. In this case the wobble stick enables the sample transfer from the main vacuum system to the VT instrument.

The VT bolt-on instrument concept allows easy access to the microscope (optical access, in-situ evaporation, sputtering, oxidation etc.) and avoids interference with other techniques.

Scienta Omicron's proven internal spring suspension system with eddy current damping ensures excellent vibration isolation. Additional external vibration damping on the UHV system is usually not required.



SPM Subsystem:
Instead of directly mounting the bolt-on SPM chamber to an existing system, Scienta Omicron also offers specifically designed small sub-systems. These operate as stand-alone systems and can be connected via a sample transfer to any existing system.

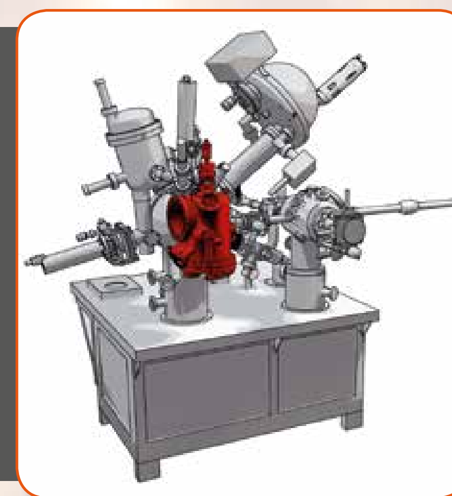


SPM Probe:
The SPM Probe is a compact and cost-effective complete UHV system solution for Scienta Omicron's UHV VT SPMs.
The rigid bench is ideally suited for high resolution SPM work. The UHV SPM is directly mounted to the experimental chamber. The experimental chamber can be easily equipped with auxiliary techniques for sample preparation and characterization.

SPM Probe (left) and two customized subsystems with VT SPM.

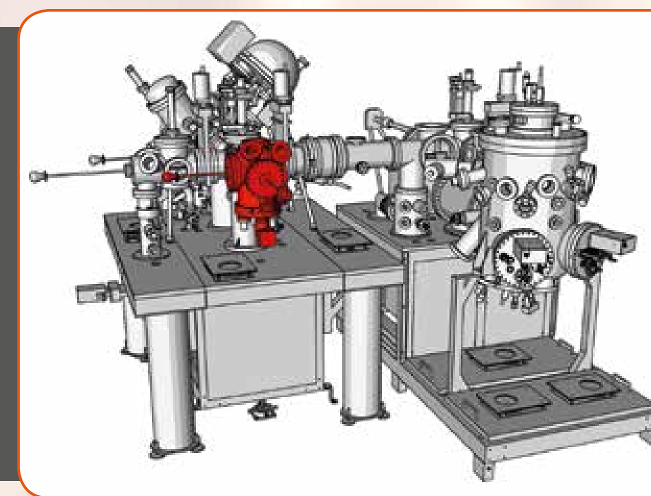
MULTIPROBE system with MBE and analysis chamber.

Multi-module cluster tool: combined SiGe-MBE, metal MBE, sputtering, and annealing for up to 4" wafers.

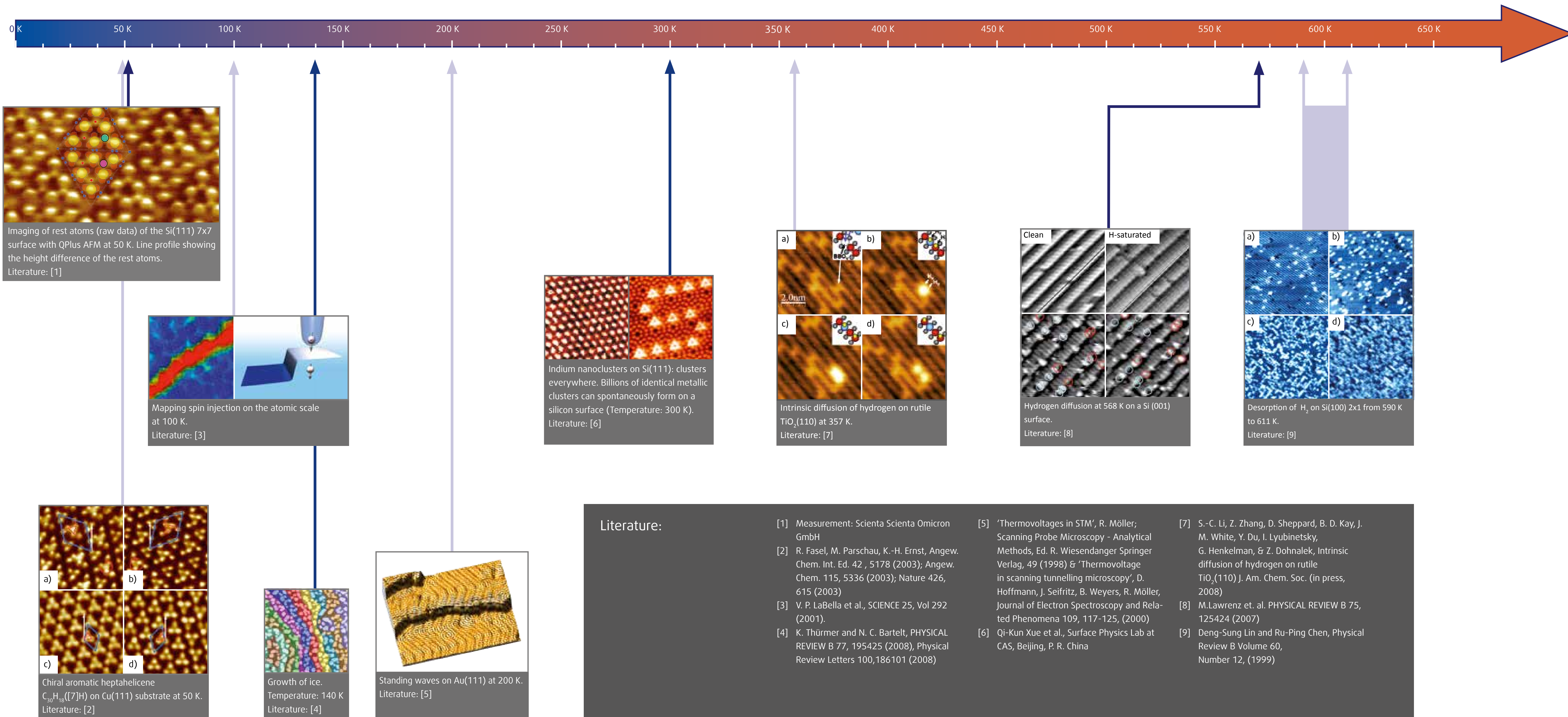


MULTIPROBE:
The standard MULTIPROBE systems demonstrate Scienta Omicron's successful concept of combining atomic reso-lution UHV SPM with a comprehensive range of complementary multi-technique surface analysis and sample preparation techniques.

SPM as part of a complex system:
State-of-the-art MBE growth and surface analysis in a single, integrated system. SPM and multi-technique analysis can be combined to virtually any kind of complex system.



Impressive Results at Various Temperatures



Imaging of rest atoms (raw data) of the Si(111) 7x7 surface with QPlus AFM at 50 K. Line profile showing the height difference of the rest atoms. Literature: [1]

Chiral aromatic heptahelicene $C_{30}H_{18}$([7]H) on Cu(111) substrate at 50 K. Literature: [2]

Mapping spin injection on the atomic scale at 100 K. Literature: [3]

Growth of ice. Temperature: 140 K Literature: [4]

Standing waves on Au(111) at 200 K. Literature: [5]

Indium nanoclusters on Si(111): clusters everywhere. Billions of identical metallic clusters can spontaneously form on a silicon surface (Temperature: 300 K). Literature: [6]

Intrinsic diffusion of hydrogen on rutile TiO_2(110) at 357 K. Literature: [7]

Hydrogen diffusion at 568 K on a Si (001) surface. Literature: [8]

Desorption of H_2 on Si(100) 2x1 from 590 K to 611 K. Literature: [9]

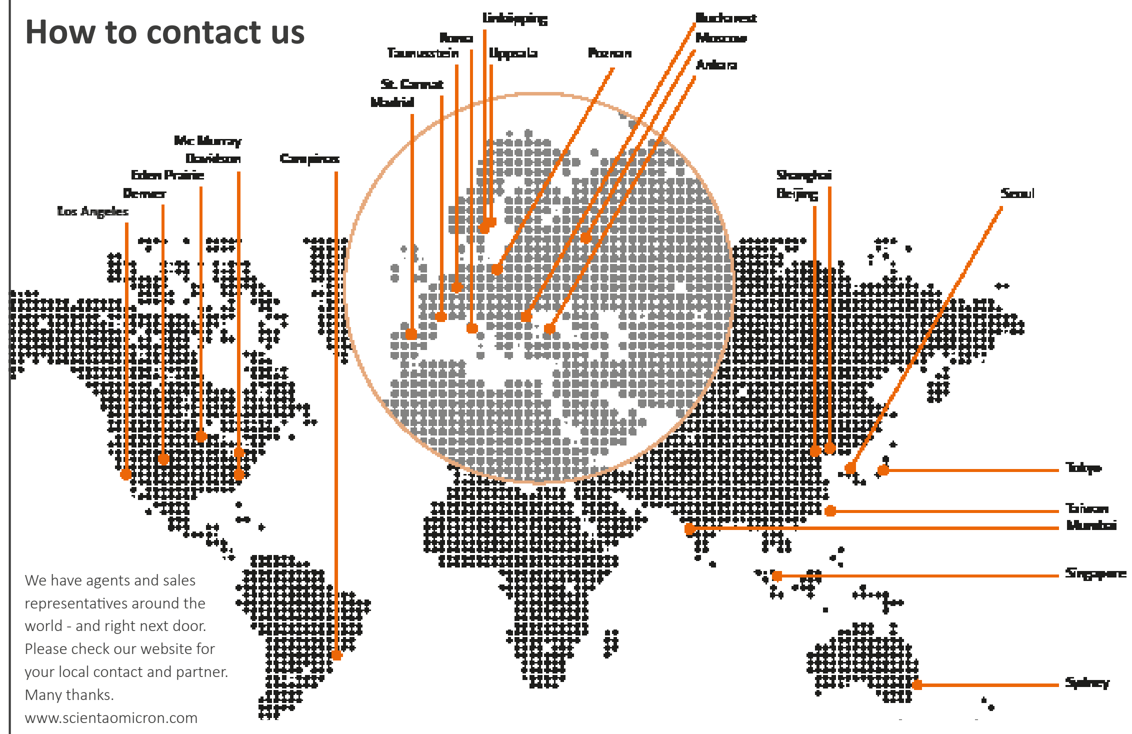
Literature:

[1] Measurement: Scienta Scienta Omicron GmbH
[2] R. Fasel, M. Parschau, K.-H. Ernst, Angew. Chem. Int. Ed. 42 , 5178 (2003); Angew. Chem. 115, 5336 (2003); Nature 426, 615 (2003)
[3] V. P. LaBella et al., SCIENCE 25, Vol 292 (2001).
[4] K. Thürmer and N. C. Bartelt, PHYSICAL REVIEW B 77, 195425 (2008), Physical Review Letters 100,186101 (2008)
[5] 'Thermovoltages in STM', R. Möller; Scanning Probe Microscopy - Analytical Methods, Ed. R. Wiesendanger Springer Verlag, 49 (1998) & 'Thermovoltage in scanning tunnelling microscopy', D. Hoffmann, J. Seifritz, B. Weyers, R. Möller, Journal of Electron Spectroscopy and Related Phenomena 109, 117-125, (2000)
[6] Qi-Kun Xue et al., Surface Physics Lab at CAS, Beijing, P. R. China
[7] S.-C. Li, Z. Zhang, D. Sheppard, B. D. Kay, J. M. White, Y. Du, I. Lyubinetsky, G. Henkelman, & Z. Dohnalek, Intrinsic diffusion of hydrogen on rutile TiO_2(110) J. Am. Chem. Soc. (in press, 2008)
[8] M.Lawrenz et. al. PHYSICAL REVIEW B 75, 125424 (2007)
[9] Deng-Sung Lin and Ru-Ping Chen, Physical Review B Volume 60, Number 12, (1999)

VT XA Series - Variable Temperature UHV SPM

Benchmarking UHV STM
and AFM Technology

How to contact us



We have agents and sales representatives around the world - and right next door. Please check our website for your local contact and partner. Many thanks.
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Technical Data

Scan (and offset) range X/Y/Z:
12 µm x 12 µm x 1.5 µm (VT STM XA)
10 µm x 10 µm x 1.5 µm (VT AFM XA)

Coarse movement X/Y/Z:
10 mm x 10 mm x 10 mm
Step size: 40 nm – 500 nm

Tip/cantilever exchange:
Remote controlled tip/cantilever exchange

Z – resolution:
< 0.01 nm

Preamplifier:
In-vacuum first stage I/V converter with software-controlled gain switch, separate compensation for I_t offset and voltage offset, capacitive compensation for modulation spectroscopy, maximum bandwidth 80 kHz

Tunnelling current:
< 1pA – 330 nA,
1 pA (200 Hz bandwidth)
3 pA (800 Hz bandwidth)
Noise on tunnelling current:
0.1 pArms (200Hz), 0.2 pArms (800 Hz)

Capacitive compensation:
> 200 (50 Hz – 10 kHz, 30 MΩ, 80 kHz)
> 100 (10 Hz – 6 kHz, 3 GΩ, 800 Hz)

Vibration isolation:
Internal eddy current damping

Gap voltage:
± 5 mV to ± 10 V; applied to tip/cantilever, sample grounded

Maximum bakeout temperature:
170° C (VT STM XA)
160° C (VT AFM XA)

Vacuum achievable:
10^{-11} mbar range

Variable temp. operation in microscope stage:
With LHe flow cryostat: 50 K - 500 K / 650 K*
With LN_2 bath cryostat: 100 K - 500 K / 650 K*
*heating version only
Max. heating power allowed in stage: 7.5 W
Max. heating current allowed in stage: 0.5 A

Flow cryostat (LHe, LN_2):
Resolution (with temperature controller) < 0.02 K,
temperature drift < 0.1 K/hr,
LHe consumption typically 1 l/hr.
Initial cool-down time < 0.5 hr.
Integrated counter - heating element for temperature control.

LN_2 bath cryostat:
Initial cool-down time < 1 hr (for equilibrium), < 0.5 hr. after refill.
Capacity 1.6 l; duration 8 hr. typical
Stability (at sample) < 1 K/hr.