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QPlus: atomic force microscopy on single-crystal insulators with small oscillation amplitudes at 5 K

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Abstract

Based on a proven low temperature scanning tunneling microscope (STM) platform, we have integrated a QPlus sensor, which employs a quartz tuning fork for force detection in non-contact atomic force microscopy (AFM). For combined STM operation, this sensor has key advantages over conventional sensors. For quantitative force spectroscopy on insulating thin films or semiconductors, decoupling of the tunneling current and the piezo-electrically induced AFM signal is important. In addition, extremely low signals require the first amplification stage to be very close to the sensor, i.e. to be compatible with low temperatures. We present atomic resolution imaging on single-crystal NaCl(100) with oscillation amplitudes below 100 pm (peak-to-peak) and operation at higher flexural modes in constant frequency shift (df) imaging feedback. We also present atomic resolution measurements on MgO(100) and Au(111), and first evaluation measurements of the QPlus sensor in Kelvin probe microscopy on Si(111) 7 × 7.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Over many years, low temperature scanning tunneling microscopy (STM) has been established as an advanced imaging and spectroscopy tool in various scientific fields. A large number of self-built as well as commercial instruments are working on a high performance level with a z-stability in the picometer range, and they allow for applications such as molecule spectroscopy (including inelastic tunneling spectroscopy, IETS) [1], atom and molecule manipulation [2–6], spin-polarized STM/scanning tunneling spectroscopy (STS) [7], and many others. One reason for the widespread use of this technique is the relative technical simplicity of a scanning tunneling microscope, even under the stringent experimental conditions when working at liquid helium temperatures.

However, in the field of atomic force microscopy (AFM) the situation is different. A comparatively limited number of self-built or commercial low temperature instruments are successfully working on a high performance level [8, 9], which basically results from the technical complexity of optical beam deflection or interferometric AFM detection.

Nevertheless, applications are driving the development of an easy to handle, reliable and high performance AFM technique. A prominent example can be found within the PicoInside project [10]: a molecule based computing circuit requires a single molecule to be locally manipulated and electronically decoupled from the underlying substrate. Current approaches use wide band gap semiconductors for decoupling or thin insulation layers which would still allow for an STM based manipulation process. On the other hand, the use of an insulating substrate or thicker insulating layers would ensure perfect electronic decoupling of the molecule but require AFM based manipulation. The realization of a reproducible experimental route for this process represents a major milestone of the PicoInside project.

Aiming at a low level of technical complexity, purely electrical AFM sensors are favorable. Among a small number of approaches, the piezo-resistive (PR) detection scheme is one of the few that has proven atomic resolution but never been established as a reliable and routine AFM technology at a high performance level [11, 12]. One reason is that the first stage PR signal detection is highly integrated into the

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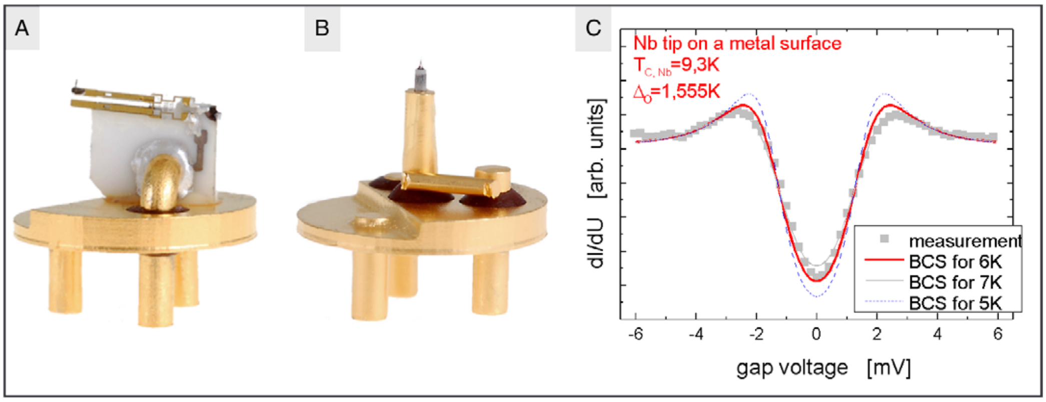

Figure 1. Pictures of a QPlus sensor (a) and an STM tip (b) mounted on the LT STM sensor carrier. In case of the QPlus sensor, tungsten tips are glued onto the oscillating prong of the tuning fork. The use of tungsten tips enables the QPlus sensor to be used simultaneously or alternatively in STM mode. Figure (c) shows the superconducting energy gap obtained for a niobium STM tip on a Au(111) surface at about 5 K.

cantilever itself, and thus requires a high level of cantilever process technology and basically depends on the sensor availability from commercial suppliers. In addition, the simultaneous use in high performance STM and STS modes is limited since cantilever probes need a metal coating for conductivity reasons. Also, electric power dissipation of the sensor has considerable impact when working at low temperatures.

An alternative (and also purely electrical) approach for non-contact AFM (NC-AFM) is based on a quartz tuning fork and uses the piezo-electric effect of quartz [13–17]. In general, tuning forks are mass products (e.g. used in wristwatches) and comparably cheap. Although the commercial availability of complete sensors is still very limited, they are accessible for the scientific community since probe tips can easily be mounted (glued) onto a tuning fork. In the late 1990s, Franz Giessibl presented a new approach that makes use of a quartz tuning fork, commonly called the 'QPlus sensor', which showed atomic resolution in NC-AFM images on Si(111) [18, 19]. One prong of the tuning fork is fixed while the scanning probe microscope (SPM) probe tip is mounted to the (free oscillating) second prong. It thus acts as a quartz lever transforming its oscillation (f_{res} typically 20–30 kHz, depending on tip mass) into an electrical signal as a result of the piezo-electric effect. The sensor can either be excited electrically by an AC voltage or mechanically by employing the scan piezo. The employed feedback signal is based on the frequency shift originating from tip–sample force interaction. However, for true force detection in non-contact AFM operation mode on conducting samples, the role of the tunneling current cross-talk needs careful consideration.

The basic motivation of the QPlus sensor is to improve AFM resolution for short range forces by the high spring constant of the sensor (approximately 1800 N m^{-1}, cantilever typically a few N m^{-1}), and small oscillation amplitudes in the range of 1 nm or below (cantilever typically 10 nm), which more precisely matches the range of the involved (chemical) forces. In addition, the high spring constant also helps to avoid jump-to-contact problems.

2. Experimental details

From a technical point of view, the integration of such a sensor is straightforward in terms of instrument design. Due to the high sensor spring constant and the close distance between the tip and the sample, the smallest external (mechanical) perturbations may have a strong impact since the forces involved are nearly three orders of magnitude larger and may easily induce image contrast changes by changing the tip's atomic formation, thus leading to instable imaging. This requires a stable SPM platform with a z-stability in the range of a few picometers. Following this demand, the QPlus technique was integrated into the STM platform of the 'Omicron LT STM' with a z-stability in the range of a few picometer.

A relevant design change is the implementation of three electrical contacts at the sensor carrier in conjunction with a fully UHV compatible transfer concept (figures 1(a) and (b)). Also here, well proven and existing technology from the 'Omicron VT SPM' has been employed, and two sensor carriers can alternatively be used (STM only, QPlus). The QPlus sensor can be operated in AFM feedback mode with simultaneous detection of a (dynamic) tunneling current or even in STM feedback mode. Using electrically connected tunneling tips has two advantages: (i) the amount of glue can be minimized for maximum sensor resonance frequency since the glue does not serve as electrical insulation, and (ii) the tunneling tip floating on U_{gap} potential allows for easy compensation of electrostatic forces originating for example from strong local charging on insulating single crystals such as MgO. Using insulating glue for tip mounting and separate wiring is an alternative approach for signal separation but is not required for the employed detection technique.

So far, QPlus sensors (figure 1) are not commercially available from sensor suppliers. We thus established a reliable manufacturing process for assembling sensors on dedicated LT STM carriers. After evaluation of Si cantilever tips from six different suppliers, the best image performance was achieved using conventionally wet-chemically etched tungsten tips, glued onto the tuning fork. This approach is very

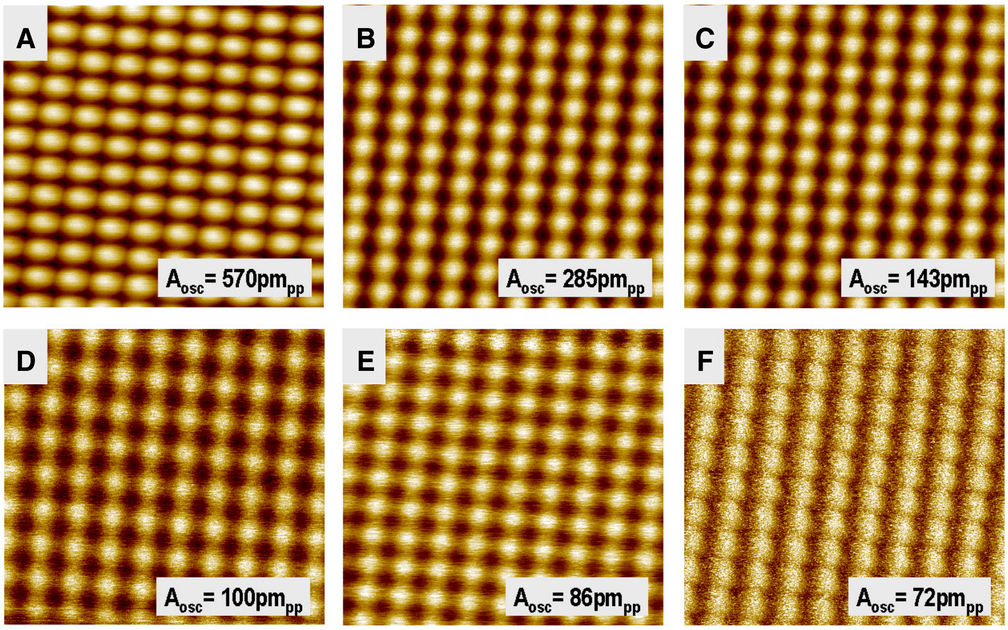

Figure 2. QPlus AFM on single-crystal NaCl(100) in df feedback at low oscillation amplitudes down to 72 pm_{pp}. Raw data, line-by-line background correction, no filtering. 4 nm × 4 nm, $T = 5$ K, $f_0 = 47\,874$ Hz, $Q = 90\,000$, $U_{\text{gap}} = +500$ mV ((a)–(c)) and $U_{\text{gap}} = -400$ mV ((d)–(f)); $df = -10.8$ Hz, -16.5 Hz, -22.3 Hz, -17.5 Hz, -19.3 Hz, -40.75 Hz ((a)–(f)).

simple, does not involve a sophisticated process technology, and basically gives access to employing virtually any tip material. In addition, the use of solid metal tips allows for highest performance in simultaneous or alternative STM/STS operation.

The major technical challenge in QPlus AFM is the pre-amplification technique. The detection circuit scheme guarantees distance control based on the pure vibrational (piezo-electric) AFM signal with clear separation from the tunneling current. Piezo-electrically generated signals are very small, and therefore the first stage detection circuit needs to be close to the sensor in order to ensure sufficient signal-to-noise (S/N) ratio. In addition, the design ensures continuous operation over the full temperature range up to room temperature without recalibration of the pre-amplifier. In fact, this required the development of a low temperature ($T < 5$ K) pre-amplifier which was fully UHV compatible and bakeable.

However, the use of a low temperature pre-amplification circuit requires careful consideration of the absolute temperature of the sample–tip system since the circuit generates heat and is thermally connected to the sensor by the signal lines. The sample temperature can be precisely measured by a thermally well coupled Si diode. With an active QPlus pre-amplifier, a slight temperature increase of the whole STM stage of about 0.5 K is observed, and the results prove that the pre-amplifier is thermally well coupled to the STM stage and that heat flow to the tip is minimal. In contrast, the temperature of the sensor tip is not directly accessible, since the wiring of a temperature sensor would change the thermal situation completely. Therefore, we utilized a superconducting niobium STM tip ($T_C \approx 9.3$ K) for modulation spectroscopy on a Au(111) surface with an active AFM pre-amplifier. The result of this measurement clearly shows the superconducting energy gap, which proves that the temperature of the tip must be below 9.3 K (figure 1(c)). The width of the superconducting gap is directly determined by the absolute temperature of the tunneling gap and thus represents a direct access to the sensor temperature. The measured width of $2\Delta \approx 2.5$ meV (modulation frequency $f_{\text{Mod}} = 1967$ Hz, modulating voltage $V_{\text{Mod}} = 150$ μV_{PP} (peak to peak)) corresponds well with the measurement by Pan *et al* in 1998 [20]. In addition, the measured data have been fitted corresponding to the BCS theory as described by Wiebe *et al* [21]. The curves from BCS theory at 5, 6, and 7 K are shown in figure 1(c). Best agreement was found for 6 K, and hence the temperature of the tip is increased by the pre-amplifier by less than 1.5 K.

The detection concept avoids electronic damping of the sensor (as known for IV-converter based concepts) to allow for Q-factors up to 100 000 at a temperature of 5 K, which results in increased force sensitivity. A large bandwidth of more than 100 kHz enables: (i) the use of sensors with higher resonance frequencies, potentially increasing the force sensitivity; (ii) higher harmonics AFM; or (iii) using a higher flexural mode of the QPlus sensor for frequency shift (df) feedback.

3. Results

Characterization measurements (figures 2, 3) prove that working at those high frequencies is possible with high resolution. Figure 2 shows a series of atomically resolved NaCl(100) NC-AFM images on subsequently decreasing the oscillation amplitude. Without any modifications of the control electronics (Matrix) it was easily possible to reduce the amplitude to 72 pm_{pp}. These measurements where done at a

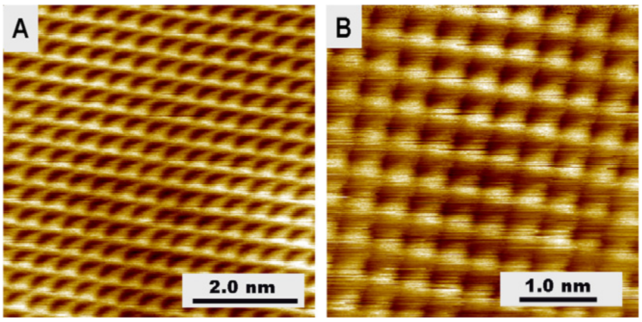

Figure 3. QPlus AFM on NaCl(100) using a flexural mode at $T = 5$ K. Sensor operated at a frequency of $f_0 = 318\,534$ Hz for df feedback. Base frequency is 47 kHz. $U_{\mathrm{gap}} = 450$ mV, $Q = 9300$. (a) $df = -2.37$ Hz, $A_{\mathrm{OSC}} = 1.5$ $\mathrm{nm_{pp}}$. (b) $df = -4.45$ Hz, $A_{\mathrm{OSC}} = 0.5$ $\mathrm{nm_{pp}}$. Raw data, line-by-line background correction, no filtering.

temperature of 5 K with a special QPlus sensor oscillating at a frequency of 47 kHz.

In addition to the measurements with the 47 kHz sensor, the same sensor has been operated at a flexural mode at a frequency of 318 534 Hz. The results are shown in figure 3. Even at these high frequencies imaging with atomic resolution is possible using oscillation amplitudes of 1.5 and 0.5 $\mathrm{nm_{pp}}$ (figures 3(a) and (b), respectively).

A NaCl single crystals have been selected to fundamentally prove the AFM performance due to the fact that preparation is very simple by just cleaving in UHV and that possible impact on the feedback loop from I_{T} cross-talk is excluded fundamentally. Figure 4 reveals a defect on an atomically resolved NaCl(100) single-crystal surface at a temperature of 5 K. The measurements show a typical corrugation of approximately 10 pm and prove that resolution on insulating samples is competitive with the best cantilever based AFM results. Measurements with comparable quality have been performed at 77 K and room temperature to prove the performance of the detection circuit at elevated temperatures.

A further example is shown in figure 4(c). MgO(001) single crystals are difficult to image with atomic resolution. The cleaving process leads to a high defect density and strong surface charges, which results in strong long range electrostatic forces. Usually vacuum annealing is required to reduce this strong charging and achieve atomic resolution with conventional cantilevers. Nevertheless, the performance shown has been achieved with the QPlus sensor without any annealing and can be explained by the fact that the QPlus sensor's small amplitudes predominantly sense chemical short range forces for atomic contrast in comparison to comparatively soft cantilevers with higher oscillation amplitudes.

Characterization measurements on single-crystal insulators such as NaCl and MgO have demonstrated that the set-up of the detection concept is suited for pure NC-AFM imaging. It also enables atomic resolution in NC-AFM mode on conducting samples on a high performance level. Examples are given in figure 5. The images show the atomically resolved Au(111) surface. Beside the atomic resolution, figure 5(a) reveals an adsorbate and the herringbone reconstruction of Au(111) with atomic corrugations of less than 3 $\mathrm{pm_{pp}}$. Figure 5(b) shows atomic resolution and the herringbone structure in the topography, damping, and df images.

Another interesting question is the use of a QPlus sensor for Kelvin probe force microscopy (KPM), a scanning probe technique capable of mapping the local surface potential or work function on various surfaces with high spatial resolution [22–25]. A way to increase the spatial resolution is to get as close as possible to the sample. We performed KPM measurements with a QPlus sensor on a Si(111) surface at 77 K. Figure 6(c) demonstrates atomic resolved Si(111) 7×7 in NC-AFM mode. The contact potential map (figure 6(a)) reveals contact potential differences (CPDs) between the clean areas with 7 × 7 reconstruction and the precipitate regions indicated by arrows. In addition, the contact potential map shows the Kelvin probe signal with atomic contrast. The recorded $\Delta f(V)$ curve (figure 6(b)) at a position in the precipitate region shows a CPD signal which agrees with the values measured in figure 6(a). These are the first results with this set-up which principally demonstrate the capability of the QPlus sensor for measurements of the contact potential difference. The theoretical explanation of the atomic contrast shown in figure 6(a) is still under discussion.

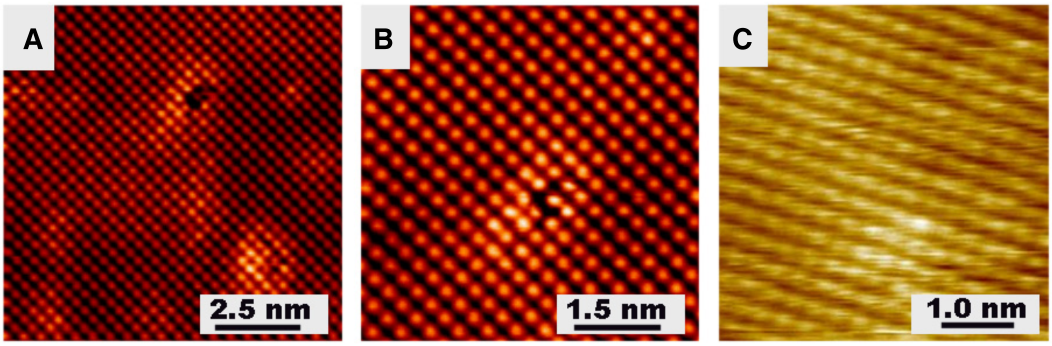

Figure 4. Atomic resolution on single-crystal NaCl(100) ((a), (b)) and MgO(001) (c) using QPlus force detection at $T < 5.5$ K. No FFT filtering applied, raw data and plane correction. (a) and (b) Measurement in df feedback on NaCl(100); imaging parameters: $df = -2$ Hz, $A_{\mathrm{osc}} \approx 1.5$ $\mathrm{nm_{pp}}$, $f_{\mathrm{res}} \approx 25$ kHz, $Q = 36\,671$. (c) QPlus AFM on MgO(001), corrugation ≈15 pm; imaging parameters: $f = 25$ kHz, $A_{\mathrm{OSC}} = 4$ $\mathrm{nm_{pp}}$, $U_{\mathrm{bias}} = -10$ V.

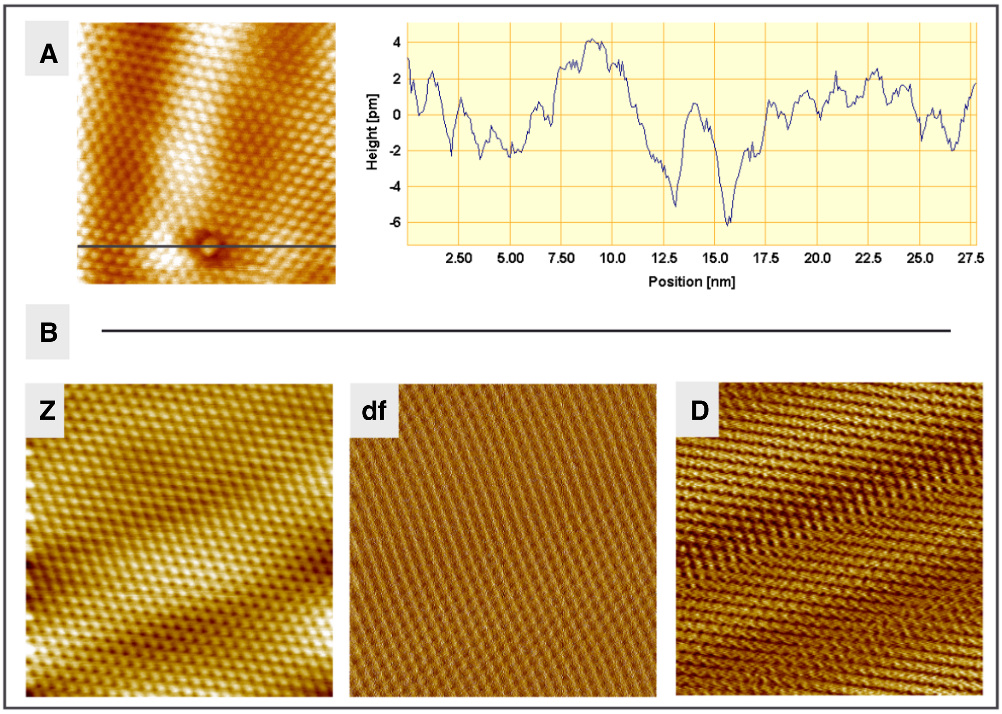

Figure 5. QPlus AFM on a single Au(111) crystal, $A_{\mathrm{OSC}} = 714$ pm$_{\mathrm{pp}}$ and $Q = 90\,000$ at $T = 5$ K. (a) Topography image showing an adsorbate on a Au(111) surface. Imaging parameters: $U_{\mathrm{gap}} = -500$ mV, $f_0 = 47\,873$ Hz, $df = -52.5$ Hz, (b) topography, damping, and df images. Imaging parameters: $U_{\mathrm{gap}} = -1$ V, $f_0 = 47\,870$ Hz, $df = -16.5$ Hz.

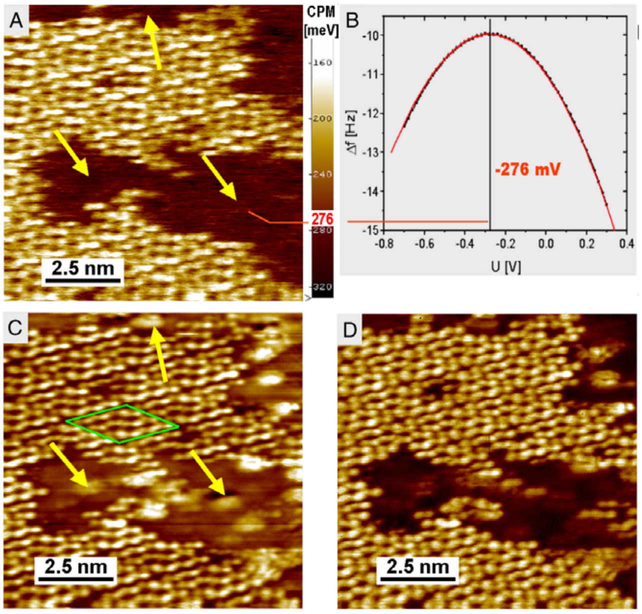

Figure 6. Kelvin probe microscopy on a Si(111) surface. (a) Kelvin signal (CPD), (b) $\Delta f(V)$ measurement at indicated position, (c) topography image, and (d) damping signal. The $\Delta f(V)$ value measured in (b) agrees with the scaling in image (a). The modulation frequency used was 500 Hz, the modulation amplitude 1 V$_{\mathrm{pp}}$, the oscillation amplitude ~2 nm, and measurements were made at $T = 77$ K.

4. Conclusions

In summary, a low temperature QPlus AFM detection technique has been developed successfully on a well-proven experimental platform without compromising the original STM/STS performance. In particular, the combination of both QPlus AFM and STM modes on an extremely high performance level has great potential for various applications, including AFM based molecule manipulation on insulating surfaces, representing a key application of the PicoInside project.

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