Cell based ultra high vacuum scanning probe microscope with an external scanning unit having gas or liquid processing capabilities

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Abstract. A scanning probe microscope (SPM), providing an ultra high vacuum (UHV), gas or liquid environment, is presented. It is intended for nanoscale processing and surface research, such as electron controlled chemical lithography (ECCL). The SPM device is mounted on the preparation chamber of a molecular beam epitaxy (MBE) UHV system. Tips and samples can be transferred under vacuum, to and from a small-volume UHV compatible SPM cell which has electrical feedthroughs and gas/liquid inlets and is closable from the MBE system. The air-side of the SPM cell is deformable with three \((x, y, z)\) external piezoelectric actuators, controlling the tip-sample distance, and the \((x, y)\) scanning. Piezoelectric actuators, capacitive displacement sensors and coarse-approach unit are all in a single scanning-unit in air, which is removed for the vacuum system bakeout procedure. Characterization measurements are presented of imaging capabilities in atomic resolution.

1. Introduction

Since the introduction of the scanning tunneling microscope (STM) \([1]\) and the atomic force microscope (AFM) \([2]\), named scanning probe microscopes (SPM) in general, great and exciting advancement has been made in imaging and manipulation at the molecular level. It is now a well established approach to use the STM to maneuver individual atoms and molecules on a surface \([3]\) and more recently inelastic tunneling from the STM tip has been shown viable to selectively induce vibrational excitation and/or dissociation of molecules \([4, 5, 6]\).

For example, by dosing the target molecule with tunneling electrons in the energy range from 2 to 5 eV, controlled dissociation of C-H bonds in benzene molecules has been induced \([7]\). The controlled step-by-step dissociation of single iodobenzene molecules has been demonstrated on a Cu(111) surface by using 1.5 eV tunneling electrons \([8]\). Individual hydrogen bond breaking on a Si(100) surface has also been used to pattern the surface with atomic resolution \([9]\) and electrons from the STM tip have been used to induce C-Cl bond cleavage in chlorobenzenes with subsequent attachment of the remaining organic radical to the surface \([10, 11]\).

These examples clearly demonstrate the potential that the STM has, to allow tailored synthesis of molecules on surfaces, which is an important step towards molecular engineering. The adoption of STM technology together with the technique of scanning a tuned beam of low energy electrons across a prepared surface to form arrays of functionalised chemical groups with
specific chemical and/or physical properties, provide the basis of the next generation of electron controlled chemical lithography (ECCL).

Here, a construction of a SPM system intended for ECCL is presented. It is an ultra high vacuum (UHV) SPM, based on the deformable-cell-SPM design [12] where inlets/outlets for gases and/or liquids have been added to the tip-sample zone. With this design, molecules or growth media may be introduced to the SPM-cell either in solid phase on the sample or separately in liquid or gas phase. Furthermore, the horizontal $x,y$ displacements are monitored with capacitance displacement sensors. Basic tests performed in order to verify the design are finally presented.

2. Construction

An overview of the device is shown in Fig. 1. The SPM is mounted on a small standard UHV chamber (1) which is mounted on the UHV sample preparation chamber of a molecular beam epitaxy (MBE) system through a gate valve (2). A sample (3) and a tip (4) are mounted on modified standard Omicron sample plates (5), shown in Fig. 2 (left), that are transferred under UHV from the MBE preparation chamber to the SPM head (6) with a sample plate holder (7) mounted on an already built in preparation chamber transfer mechanism. There, two sample plates are slided into the head via a standard wobble stick. The sample plates are in a parallel configuration facing each other on the SPM head. One sample plate is fixed to the head and the other sample plate fixed to the head via two small flexible metal sheet strips (8), also visible in Fig. 2 (center).

![Figure 1](image)

**Figure 1.** A scheme of the scanning probe microscope (SPM). (1) Small standard UHV chamber, (2) gate valve, (3) sample, (4) tip, (5) modified standard Omicron sample plates, (6) the SPM head, (7) the sample plate holder on the MBE system transfer mechanism, (8) small flexible metal strips, (9) heavy duty linear feedthrough, (10) the SPM-cell connection part, (11) electrical feedthroughs, (12) gas/liquid inlets, (13) internal valves, (14) bellows deformable part, (15) a stiff stainless steel plate, (16) three small tips, (17) the scanning unit, (18) $x,y$ piezoelectric stack actuators, (19) $x,y$ displacement sensors, (20) high torque stepper motor, (21) worm gear, (22) power screw, (23) nut, (24) the $z$ piezoelectric stack actuator, (25) Helicoflex Delta-seal.

The SPM head is transferred with a heavy duty linear feedthrough (9) down to a small volume SPM cell and closes it. The SPM cell is sealed from the UHV chamber with a Helicoflex Delta-seal [13]. The SPM cell connection part (10) comprises two electrical feedthroughs (11) and two gas/liquid inlets (12) that connect to the SPM head. Valves (13) for the gas/liquid
inlet are built into the cell to minimize the cell volume, shown in Fig. 2 (right). The cell has a deformable part (14), a small piece of metal flexible hose (bellows) is silver brazed between the cell connection part and a stiff stainless steel plate (15). When the SPM head goes into the SPM cell, one sample plate, connected to the head via the flexible metal strips, touches the stiff plate of the deformable part and is constrained to move with the plate via three small tips (16) that penetrate into small holes on the movable plate. Electrical connections on the SPM head come into contact with the electrical feedthroughs on the SPM cell connection part when in closed-cell-mode. The sample plate, mounted via the metal strips, is electrically connected through the strips which is electrically insulated from the environment. All electrical insulation material is made of Macor.

A scanning unit (17) is mounted on the air side of the cell unit, which comprises $x, y, z$ piezoelectric stack actuators (18), $x, y$ displacement sensors (19) and a $z$ coarse approach part. The piezoelectric actuators touch the periphery of the plate of the deformable part of the cell in mutually orthogonal directions and provide displacement and scanning functions as described elsewhere [12].

The coarse approach part is implemented with a high torque stepper motor (20) mounted on a worm gear (21). A power screw (22) is mounted on the worm gear which provides a linear motion to the nut (23) on the screw when it is rotated. The $z$ piezoelectric actuator (24) rests on the nut. The coarse approach is designed to apply a necessary force to deform the SPM cell. The power screw pitch is 2 mm, the worm gear ratio is 1:100 and one step from the stepper motor is $1.8^\circ$. The result is a theoretical displacement resolution of 100 nm for each coarse approach step.

3. Characterization
A pressure of $3 \times 10^{-9}$ mbar has been acquired after the MBE system bakeout, verifying the SPM system UHV capabilities. When the SPM cell is under vacuum it contracts about 0.5 to 1.0 mm due to the pressure difference. That does not however affect its functionality, since it still remains deformable enough for approach procedure and scanning operations.
A scanning tunneling measurement was conducted in order to evaluate the device’s performance. An etched wolfram tip was used on a highly oriented pyrolytic graphite (HOPG) sample. Fig. 3 shows a constant tunneling current scan measurement in atomic resolution which verifies that the SPM system is functioning properly, although distortions in the image are apparent due to drift and hysteresis in the piezoelectric actuators and due to lack of orthogonality between the scanning directions.

4. Summary
We have presented preliminary results from an ongoing work on the design and construction of an SPM for electron controlled chemical lithography. Future work comprises enhanced control systems and further tests under different conditions.

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