

Development and performance of the nanoworkbench: A four tip STM for conductivity measurements down to submicrometer scales

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A multiple-tip ultrahigh vacuum (UHV) scanning tunneling microscope (MTSTM) with a scanning electron microscope (SEM) for imaging and molecular-beam epitaxy growth capabilities has been developed. This instrument (nanoworkbench) is used to perform four-point probe conductivity measurements at μm spatial dimension. The system is composed of four chambers, the multiple-tip STM/SEM chamber, a surface analysis and preparation chamber, a molecular-beam epitaxy chamber, and a load-lock chamber for fast transfer of samples and probes. The four chambers are interconnected by a unique transfer system based on a sample box with integrated heating and temperature-measuring capabilities. We demonstrate the operation and the performance of the nanoworkbench with STM imaging on graphite and with four-point-probe conductivity measurements on a silicon-on-insulator (SOI) crystal. The creation of a local FET, whose dimension and localization are, respectively, determined by the spacing between the probes and their position on the SOI surface, is demonstrated. © 2005 American Institute of Physics. [DOI: 10.1063/1.1878213]

I. INTRODUCTION

In order to measure the electrical conductivity of surfaces and the influence of adsorbates on conductivity, it is desirable to perform local four-point conductivity measurements. The typical in-line setup with four probes involves driving a current between the two outer probes while measuring the resulting voltage drop between the inner two probes using a high-impedance voltmeter ($>10\text{ G}\Omega$ in our case) so that very little current is drawn. This yields a current-voltage (I - V) curve dependent on the separation d between the probes. It has a strong advantage over the two-probe measurements since it eliminates measurement error due to probe resistance and the contact resistance between the substrate and the probes. The four-terminal configuration also presents the advantage of being more versatile, provided that the probes can be controlled independently, allowing not only in-line measurements but also van der Pauw configuration electrical conductivity measurements in which the probes are placed in a square or rectangular configuration.^{1,2}

Four-point-probe instruments are commercially available but the spacing between the probes is usually fixed and too large to be able to probe structures at micrometer or submi-

cometer levels. For this reason, it is desirable to implement instruments with smaller probe-to-probe distances.

Recently, the development of multiple-probe instruments for conductivity measurements has been reported.³ Dual probe configuration measurements have been performed⁴⁻⁷ as well as four-point probe measurements.⁸⁻¹⁰ One reported instrument utilized four parallel insulating cantilevers, which cannot be driven independently, fabricated by a conventional silicon processing technique.^{11,12} Another approach is based on four independently-driven probes for conductivity measurements in ultrahigh vacuum.^{13,14}

We report the development and operation of the Nanoworkbench (NWB), a multiple-chamber ultrahigh vacuum system based on a multiple-probe STM/SEM instrument designed to perform four-point-electrical measurements. Each of the four probes of the multiple-tip system can be operated independently as a STM with nanometer-scale lateral resolution and atomic-scale vertical resolution. This specificity greatly increases the potential of the instrument for conductivity measurement at submicrometer levels by allowing precise positioning of the probes and characterization of the surface or object of interest. The NWB also features a surface analysis and preparation chamber equipped with standard surface science tools as well as a molecular-beam epitaxy system.

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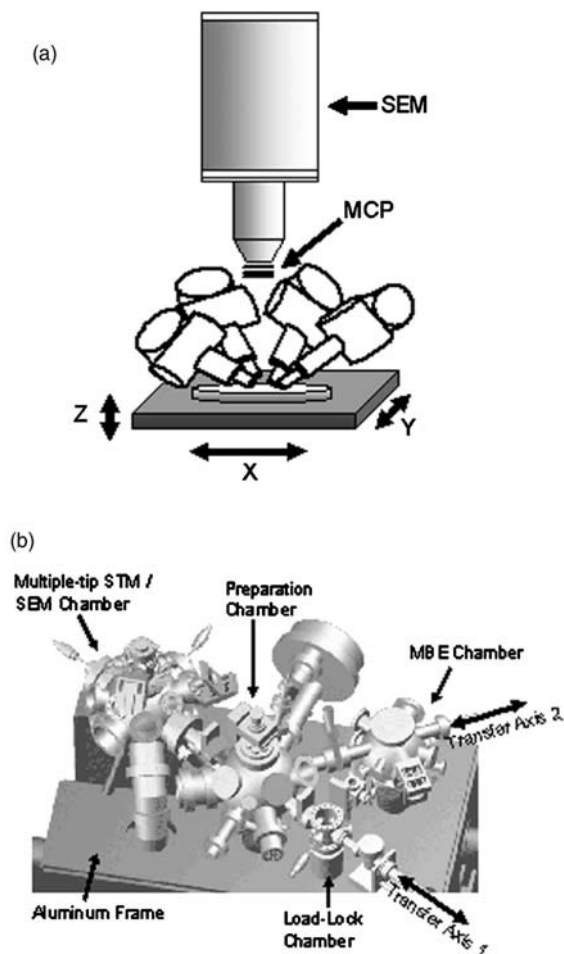


FIG. 1. (a) Schematic image of the Nanoworkbench; (b) overview of the Nanoworkbench showing a multiple-tip STM/SEM chamber (left), a surface analysis and preparation chamber in the center featuring standard surface science tools and a molecular-beam epitaxy chamber (MBE) on the right equipped with Ge, Si, and C evaporation sources. Additionally, a load-lock chamber allows for fast and easy transfer of the samples and probes following two transfer axes. Axis 1: load-lock/preparation chamber/STM-SEM chamber and axis 2: MBE chamber/preparation chamber.

II. OVERVIEW OF THE SYSTEM

A schematic image of the basic structure of the Nanoworkbench (NWB) is shown in Fig. 1(a). Four STM nanomanipulators, oriented at 45° from the normal, and at 90° to each other, electrically probe a small region on the surface of a material of interest. They are guided by an SEM which images the surface and the tips. The sample is mounted on a positioning platform with X, Y, and Z motion. Attached to the NWB are: (1) a chemical preparation and characterization chamber; (2) a molecular-beam epitaxy chamber (MBE); and (3) a fast-entry load lock chamber [see Fig. 1(b)]. Samples and probes may be transferred from these chambers to the NWB. The system was designed using AutoCAD 14 software (Autodesk, Inc.) in combination with ACIS 3D Open Viewer (Spatial Technology, Inc.). The entire system is mounted on an aluminum frame box designed and built as a highly rigid structure.

Each chamber is equipped with its own pumping system: a 500 L/s ion pump (Leybold), a 500 L/s magnetic turbo-

molecular pump (Leybold, Pfeiffer, and Shimadzu), and a titanium sublimation pump (Thermionics—model SB-1020). A common roughing line is used for the three experimental chambers. The pumps can be isolated from each other via a system of valves and gate valves. Pressure measurement in the system is achieved through cold-cathode gauges (MKS—model 421 cold cathode sensor), and pressure in the low 10^{-10} mbar range is routinely achieved for each chamber after bakeout. In addition, the load-lock chamber is pumped separately by a mechanical pump coupled with a 150 L/s Leybold turbomolecular pump. A pressure of 5×10^{-8} mbar is achieved within 1 1/2 hour and an ultimate pressure of 6×10^{-9} mbar is readily achieved in the load-lock chamber without bakeout after 24 h.

The surface analysis and preparation chamber [see Fig. 1(b)] is equipped with standard surface science tools, including a Perkin-Elmer Cylindrical Mirror Analyzer for Auger electron spectroscopy (AES model 10-155), a hemispherical Leybold-Heraeus EA-10 electron analyzer combined with a dual Mg/Al anode x-ray source for x-ray photoelectron spectroscopy (XPS), a UTI-100C quadrupole mass spectrometer (QMS), a homemade reverse view low-energy electron diffraction analyzer (LEED), an ion sputter gun and a gas dosing system. An XYZ manipulator with heating and cooling capability is used as a receiving stage for the sample box. The MBE chamber [see Fig. 1(b)] is used for thin film deposition, doping or growth of quantum dots. It contains three 3 kW solid-source electron-beam evaporators (MDC—evap3000) and one 100 W wire source (MDC—evap100). A quartz crystal microbalance (QCM—Sycon model STM-100) is used to monitor thin film growth. Each evaporation source can be isolated via a shutter mounted on a linear feedthrough. Finally, the STM/SEM chamber is equipped with four nanomanipulators (Kleindiek Nanotechnik—model MM3 and MM3a) holding etched PtIr probes (Fig. 1). The addition of an UHV-SEM (FEI—model 2LE-EVA) based on a Schottky emitter source allows fast and precise approach and positioning of the probes. The STM/SEM chamber also features a UTI-100C quadrupole mass spectrometer, a gas handling system as well as a probe-garage holding up to 10 tips and a sample box carousel (three sample spots).

III. TRANSFER SYSTEM AND SAMPLE BOX

A. Sample box and receiving stages

For the preparation of clean Si(100)- 2×1 surfaces,^{15,16} it is important to have precise control over the temperature of the sample in each UHV chamber. Moreover, it is desirable to have a system which can accommodate samples with various sizes and requirements. For this purpose a special transferable sample box with integrated heating and temperature measuring capabilities has been developed. As shown in Fig. 2 the sample box is made of OFHC copper with highly polished outer walls. Four UHV-compatible male banana plugs (Transfer Engineering and Manufacturing—model BPBPM) are tightly connected to the sample box and isolated from it with ceramic spacers. The outer plugs are made one of chromel and the other one of alumel. They are used for K-type thermocouple connections. The inner plugs are used

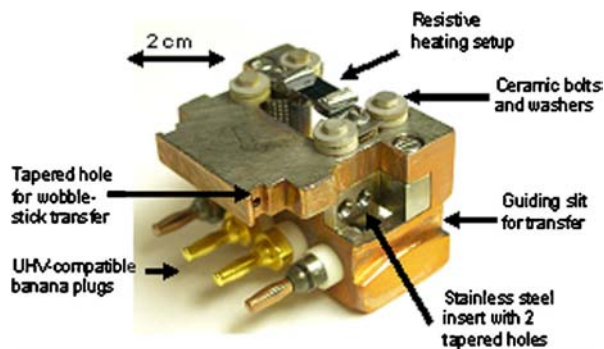


FIG. 2. (Color online). Sample box equipped with four UHV-compatible banana plugs (models BPBPM) for heating and temperature measurements. Stainless steel inserts are used to tightly grab the sample box for transfer from one chamber to the next. A resistive heating device is mounted on this sample box.

as electrical heating connections and are made of BeCu, 304 stainless steel, and gold-coated for enhanced electrical performance and reliability. The sample box is equipped with stainless steel inserts on each side. These inserts with tapered holes match tapered pins on the transfer manipulator and are used to grab the sample box for UHV transfer.

The sample box can be equipped with either resistive or radiative heating devices. Figure 2 shows the resistive heating setup with a $10\text{ mm} \times 5\text{ mm} \times 0.5\text{ mm}$ Si(100) crystal. The crystal is resistively heated using noninteracting Ta contacts following a method described by Yates.¹⁷ Alternatively, the sample box can be equipped with a radiative heater (Tetra—Boraelectric—not shown here).

Each UHV chamber is equipped with a receiving stage such as the one shown in Fig. 3. The receiving stage is also made of OFHC copper—with highly polished inner walls closely matching the outer walls of the sample box, and is

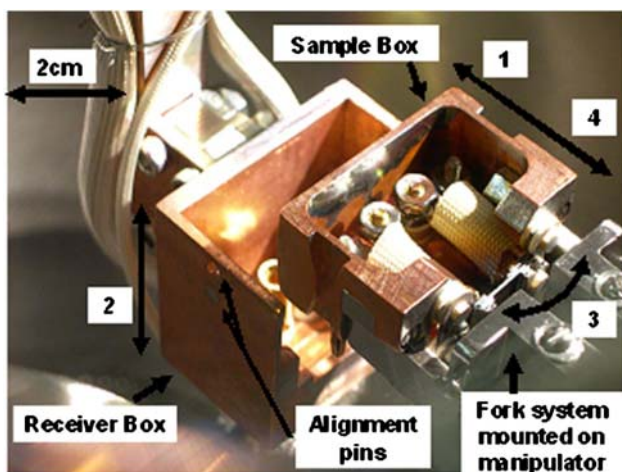


FIG. 3. (Color online). Image of the sample box and receiver box in the preparation chamber. The transfer sequence is divided into 4 steps: (1) The sample box—held by the fork system of the linear/rotary manipulator approaches the XYZ receiver; (2) once the sample box is positioned above the receiving box, the receiver (controlled by an XYZ manipulator) is moved up, so that the receiving box tightly houses the sample box; (3) once the connection is tight, the fork system is opened; and (4) the linear manipulator is moved away from the sample box. The sample box and receiving box are both made of highly polished OFHC copper.

equipped with a similar set of female banana plugs. The receiving stage features a set of 2 mm diameter stainless steel pins on each side to ensure rigorous alignment of the sample box during transfer. In addition, each receiving stage features a liquid nitrogen reservoir for cooling the sample.

B. Transfer system

The transfer system is articulated around two main axes: axis 1: load-lock chamber—preparation chamber—STM/SEM chamber, and axis 2: MBE chamber—preparation chamber, as can be seen in Fig. 1(a). Linear/rotary manipulators are used to transfer the sample throughout the system. In addition, two wobble sticks are mounted on the STM/SEM chamber to allow for easy transfer of the sample box to the STM stage and sample carousel as well as *in situ* tip exchange. We use custom-made magnetically actuated wobble-stick manipulators (Ferrovac) which are not affected by the pressure difference between atmospheric pressure and ultrahigh vacuum, allowing the shafts of the wobble stick to be guided in a smooth and controlled way. Both linear/rotary manipulators and wobble sticks are equipped with a stainless steel fork system (pincer/grabber) featuring jaws with two stainless steel tapered pins at the end. These pins match the stainless steel inserts with tapered holes (see Fig. 2) in the sample box and ensure enhanced stability during transfer of the sample box.

The typical transfer sequence is divided into four steps (see Fig. 3):

- (1) The linear transfer manipulator holding the sample box approaches the XYZ manipulator.
- (2) Once the sample box is precisely positioned above the receiving stage, the XYZ manipulator is translated up, so that the receiving stage houses the sample box.
- (3) Once the connection between the sample box and the receiving stage is tight, the fork system (pincer) at the end of the transfer manipulator is opened, and
- (4) the linear manipulator is moved away from the sample box.

IV. MULTIPLE-TIP STM/SEM CHAMBER

A. Overview: nanomanipulators and XYZ table

The STM/SEM chamber is directly connected to the preparation chamber [see Fig. 1(b)]. Its core is a multiple-tip scanning tunneling microscope (MT-STM) combined with a scanning electron microscope (SEM).

The MT-STM assembly is shown in Fig. 4 (4a: side view and 4b: top view). It comprises four nanomanipulators models MM3 and MM3a (Kleindiek Nanotechnik) mounted on a stainless steel ring at 90° from each other in the horizontal plane [see Fig. 4(b)] and inclined at 45° from the sample plane [see Fig. 4(a)]. This design ensures a large opening from the top to accommodate the SEM and its detector. Electronic components such as the preamplifiers for STM feedback control and imaging are also mounted on the STM stage mounting ring [Fig. 4(a)] and are connected to the outside *via* electrical feedthroughs.

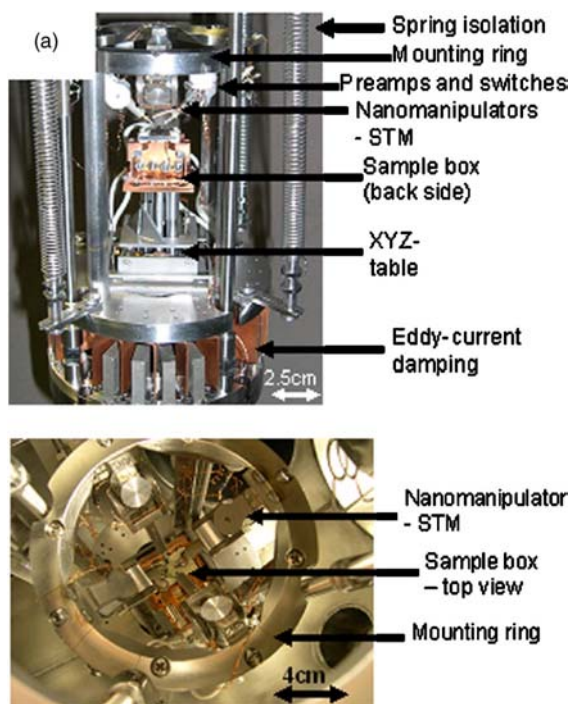


FIG. 4. (Color online). (a) Overview of the STM stage in the STM/SEM chamber. The STM stage features four nanomanipulators that can be used as STMs (models MM3 and MM3a—Kleindiek Nanotechnik) mounted on a ring at 45° from the sample plane, a sample stage mounted on an XYZ table, as well as spring isolation and an eddy-current damping system; (b) top view (SEM point of view) showing the four nanomanipulators mounted at 90° from each other in the sample plane (horizontal plane). The large solid angle allows enough space to accommodate the SEM gun.

Both the STM nanomanipulators and the sample stage must be capable of coarse and fine motion. The design of the Nanomotor[®]¹⁸ achieves both through its unique design. For fast large travel, the Nanomotor[®] can be driven in the coarse step mode which exploits the well known stick-slip mechanism.¹⁹ At the same time, positioning in the nanometer range is possible in the fine step mode, which utilizes the piezoelectric effect in the conventional way.²⁰

The sample table (Kleindiek Nanotechnik, model LT6820XYZ) has three perpendicular degrees of freedom and is equipped with three nanomotors. The table can travel 20 mm in the X and Y directions and 15 mm in the Z direction. The integrated optical encoding system allows positioning of the sample with 100 nm repeatability. The stiffness of the table was enhanced by mounting additional vertical aluminum columns.

The nanomanipulators have been developed in collaboration with Kleindiek Nanotechnik for STM applications. As shown in Fig. 5 the nanomanipulators have two rotational axes (X, Y) and one linear motion (Z). Two slightly different models are being used: the MM3 model with a length of 80 mm and the MM3a model with an overall length of 65 mm.

From the standpoint of control electronics, the simultaneous operation of four STM devices or the performance of four point probe conductivity measurements on a small scale is a challenge by itself. The SPM electronics for the 4-tip STM is provided by Kleindiek Nanotechnik (Nanomotor[®]

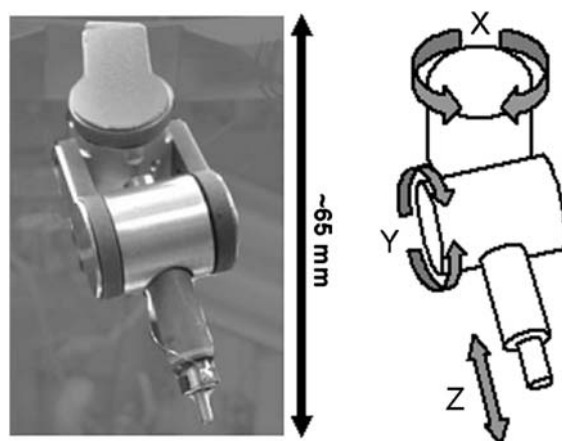


FIG. 5. Left: Image of the MM3a nanomanipulator; right: schematic showing both rotational axes (X, Y) and the linear motion (Z) of the nanomanipulator.

STM Control and Image Acquisition Electronics/Software). The electronics is controlled via a computer through an RS-232 serial interface. Each SPM electronics system is controlled by a dedicated PC to guarantee simultaneous independent operation of all four nanomanipulators. An analog feedback loop is used in constant-current mode. To reduce coupling of ambient electromagnetic noise the first amplification stage of the tunneling current is located inside the UHV system close to the nanomanipulators using a low-noise fast operational amplifier OPA111 (Burr-Brown). The gain of the OPA111 is set with a 100 M Ω resistor. With this setup the range of voltage/current is -2.5 to $+2.5$ V (restricted by SPM electronics)/ -100 to $+100$ nA (restricted by the specification of OPA111 in combination with a 100 M Ω resistor). To enable four-point conductivity measurements, direct electrical access to the probes is also desirable. Low dissipation electromagnetic relays (Teledyne, type 732-12) are mounted close to the OPA111, as shown in Fig. 6.

B. UHV-SEM+MCP detection

The SEM column (FEI-model 2LE EVA) is based on a Schottky emitter source with a two-lens electron technology and an electronically variable aperture. Mounted on top of the chamber, it is mainly used to locate features on the surface and to guide the STM tips near objects of interest.

Conventional scanning electron microscopes (SEM) operate at pressures near 10^{-6} mbar. Therefore typical SEM detection systems are based on scintillation amplifiers (a summary of SEM detectors can be found in Ref. 21). In these systems secondary electrons are converted into light and subsequently the photons are converted back into an electrical signal, which limits the sensitivity and signal-to-noise ratio (typical gains are in the range of 10^4).

The UHV environment of the nanoworkbench system permits the use of a high efficiency multichannel plate (MCP) system, similar to those used in UHV instruments for ion and electron spectroscopy. A typical MCP consists of an annular array of tiny glass tubes (12–23 μm diameter and 0.5–1 mm long) fused together to form an array of thousands of independent electron multipliers. An accelerating

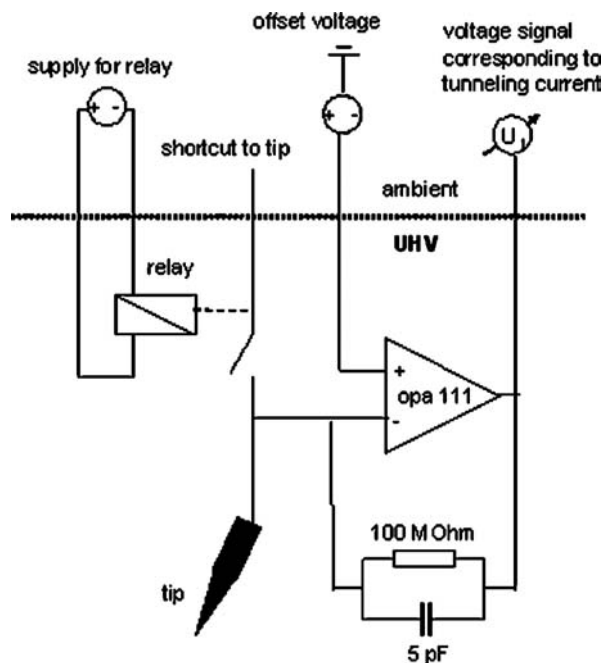


FIG. 6. Schematic of preamplifier and relay electronics close to tip. The parts shown below the dashed line are located inside the UHV chamber.

potential is applied to a metal film deposited across the ends of the tubes, and when an electron enters a tube it produces additional secondary electrons as a result of striking a special highly emissive coating on the inside of the tube. Two MCPs stacked together are used to create a gain approaching 10^7 .

Figure 7 shows a schematic diagram of the ultrahigh efficient secondary electron detection, the electron beam deflection and image recording system used in the nanoworkbench.

We use an ultrathin (2.5 mm maximum) annular MCP assembly with a center hole (Hamamatsu F6589), which is directly mounted below the SEM electron column. The shielded center hole allows undisturbed travel of the primary electron beam to the sample surface. The detector position above the specimen surface is ideal for collecting a large percentage of secondary and backscattered electrons emitted

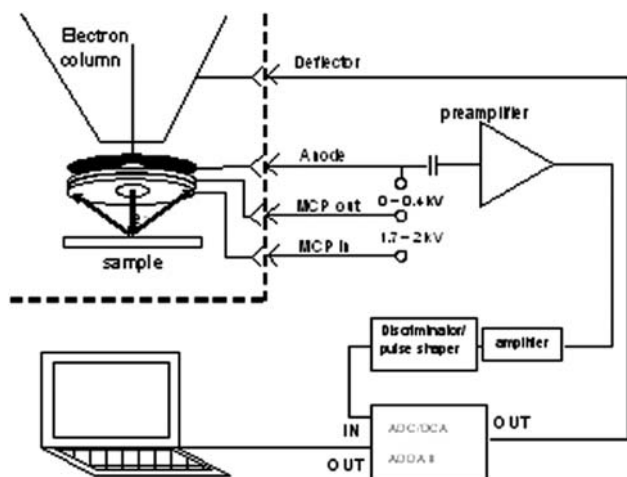


FIG. 7. Schematic of the SEM-MCP detection system.

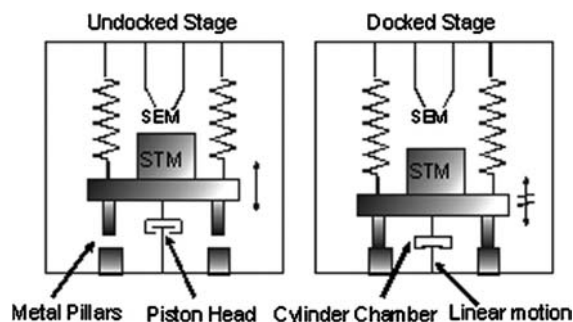


FIG. 8. Schematic view of the docking system. The STM unit is connected to the docking system without any mechanical contacts to the frame, (left) undocked and (right) docked. The undocked position is used for STM and conductivity measurements applications while the docked position is used for SEM imaging to keep the working distance constant.

from the specimen surface. Because of the large solid angle detection efficiency of the MCP, shadowing due to the nanomanipulators and the topography of the specimen is minimized; even deep holes and vias can be seen with greater efficiency than with side mounted secondary electron detectors. In particular, in the area of critical dimensions, the MCP can provide uniformly illuminated shadow-free images from which highly accurate measurements can be obtained. The front surface of the detector array may be biased positively or negatively to attract or repel secondary electrons from the sample. In this way a secondary electron image or a back-scattered electron image may be selected easily.

The electronic amplification and pulse shaping of the MCP output signal is performed by a combination of a commercially available preamplifier (ORTEC Fast Preamplifier model 9301), and an amplifier (ORTEC Amplifier Discriminator model 9302) along with a home-built pulse shaper. The resulting pulse sequences (up to 20 MHz frequency) are digitized and synchronized with the deflection controller using a commercially available, fully programmable scan system (ADDA II, Soft Imaging System). The ADDA II system is connected to a PC for control of scan parameters and image recording.

Under experimental conditions, in four-point probe configuration, the SEM resolution is ~ 100 nm. Large working distances imposed by the design of the STM stage limit the SEM resolution of the instrument.

C. Docking stage

The STM unit is decoupled from the electron column mounted on top of the STM/SEM chamber via the spring isolation system and the eddy-current damping unit (see Sec. IV D). As a result, the distance between the sample (which floats during STM imaging) and the electron gun aperture is not constant. Such variation in the working distance makes it difficult to focus the SEM, especially at high magnifications ($\geq 10^3$).

A docking system has been developed to address this problem. Figure 8 shows the design of the docking mechanism. It consists of three pairs of metal pillars and a piston-like linear actuator. One metal pillar is mounted on the STM unit, whereas the second pillar in a given pair is mounted on

the frame under the STM unit. The mating faces are mirror finished to ensure excellent mechanical contact. The actuator is composed of a piston head and a cylindrical chamber without any contact with one another. These two elements are connected to the linear motion feedthrough and the STM unit, respectively. When the piston head of the actuator is pulled down (using the linear motion feedthrough) it makes contact with the cylindrical chamber to transmit the linear motion and, consequently, pulls down the STM unit until both pillars are in contact. A spring between the linear motion feedthrough and the actuator regulates the linear force to give a consistent pulling force. When the STM unit is undocked, the piston head does not have any contact with the cylindrical chamber, and the STM unit is floating, completely decoupled from the rest of the system [Fig. 8(left)]. This position is used for STM operation and conductivity measurements. For high-resolution SEM imaging, it is preferable to dock the stage [Fig. 8(right)].

D. Vibration isolation

Vibration isolation is especially challenging since the NWB is a large system with UHV requirements. Nonetheless, vibration isolation is crucial not only for STM operation but also for conductivity measurements since one would like to achieve stable contact of the probes with the substrate over a long period of time.

As a first step towards vibration isolation, the main mechanical pump is isolated by coupling the rough vacuum line to a heavy concrete block. In addition, each turbomolecular pump is connected to the rough vacuum line via flexible tubing to minimize vibration transfer. During STM operation and conductivity measurements, all turbomolecular pumps are turned off. Additional isolation of the entire NWB from the building is achieved through a six-point commercial active vibration system (HWLbioanalytic SYSTEMS, MOD-4). Each of the six elements senses vibrations along both horizontal and vertical directions, and a closed-loop feedback system compensates these disturbances. The isolation elements are active within the range of 0.75–35 Hz and passive above that. Additional vibration isolation elements are mounted inside the STM/SEM chamber.

The STM assembly is suspended from the chamber with custom UHV-compatible springs, fabricated using Inconel X750 wire, 0.041 in. diameter (Gibbs Wire and Steel Co., Inc.). The springs were electropolished to reduce their porosity and limit their outgassing in vacuum by immersion in a 3:1 mixture bath of phosphoric acid and sulfuric acid,²² and a 9 V potential with respect to an array of graphite electrodes was applied for 3 min. The measured spring constant is 110.3 kg s⁻² for each spring used.

To further dampen vibrations in the STM stage, an eddy-current system was employed, consisting of a circular array of samarium–cobalt magnets (Magnetic Component Engineering, Inc., model S2669, 1T) with interposed conducting copper blades. The copper blades are mounted on the STM unit and the magnets are mechanically clamped on the frame [Fig. 4(a)]. The samarium–cobalt magnets have good corrosion resistance and high temperature stability. The Curie

temperature is about 800 °C and the maximum working temperature is 320 °C, well above the requirement of our system for bakeout.

V. PERFORMANCE

The nanoworkbench is capable of performing measurements in a variety of operational modes, some of which are illustrated below.

A. STM

The possibility of performing STM measurements distinguishes the nanoworkbench from other four probe setups. STM operation also expands the imaging capabilities of the NWB.

1. Sample preparation

A highly oriented pyrolytic graphite (HOPG) sample of 10 mm × 10 mm × 2 mm (SPI—model 440HP-AB) is used for testing the STM capability of the nanomanipulators. The usual approach to prepare the HOPG sample for STM is to take a piece of tape, press it onto the flat surface and pull it off. The tape takes with it a thin layer of HOPG, leaving a freshly cleaved HOPG surface.

2. STM operation and STM imaging

The specifications of the nanoworkbench STM probes differ in several important respects from typical single-probe STM instruments. First, as mentioned earlier, the manipulators are mounted at 45° from the surface plane. Therefore, the Z motion of the manipulators is established with a similar angle and is not perpendicular to the sample surface as is customary for STM systems. The SPM software incorporates a tilt correction feature to compensate for the large angle. Improved STM operation was achieved by bending the tips ~25° downwards (in direction of sample) at a distance of 1 mm before the end of the tip. Second, the nanomanipulators themselves are less compact and rigid than most STMs. Therefore the lowest resonance frequency of the nanomanipulators is close to 300 Hz. Despite the described procedures for vibration isolation and the increase of the overall stiffness built into the STM stage, slow scanning (~1 Hz) was required to acquire satisfactory STM images.

Using custom-made PtIr etched probes (Custom Probes Unlimited) we were able to reproducibly image atomic steps (see Fig. 9) and small features with a lateral width as small as 4 nm on a cleaved HOPG surface. Similar images were acquired with all four nanomanipulators.

B. Four-point probe

As explained above four terminal measurements have several advantages over two-terminal measurements. The nanoworkbench allows *in situ* positioning of four nanoscale contacts, allowing four probe measurements to be obtained.

1. Setup and strategy

To perform the measurements the precise placement of the tips is crucial. The following procedure is employed for positioning the four probes:

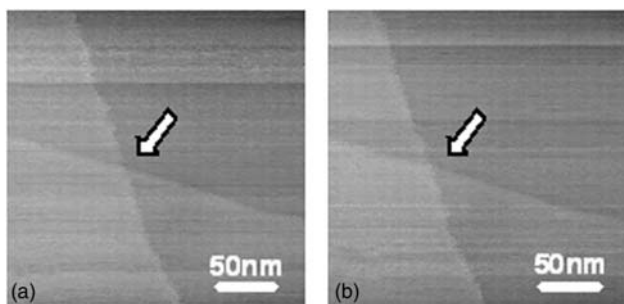


FIG. 9. Reproducible STM imaging of atomic steps on HOPG acquired with MM3a nanomanipulator. The images were acquired consecutively. Experimental conditions for both images: 1×10^{-8} mbar; $U_{\text{bias}}=200$ mV, $I_{\text{setpoint}}=0.43$ nA, image size 271×271 nm, acquisition time per image was 260 s. The bar scale represents 50 nm.

- (1) The tips are brought into close proximity to the sample surface, using the UHV-SEM as a guide. The “electron shadow” of the probes in the SEM images can be used to estimate the distance between the tip and the sample.
- (2) When the distance between the tip and the sample is small enough the automatic STM Z coarse approach with the feedback loop control is activated.
- (3) Steps 1 and 2 are repeated until all four tips are in tunneling contact with the surface. The spacing between the probes can be measured to an accuracy of ± 100 nm from the SEM image. Fine positioning of the tips in tunneling contact is possible.
- (4) Gentle contact is achieved between the tips and the sample surface. By changing the tunneling parameters (increase of current setpoint, decrease of bias voltage) the tunneling gap is minimized. By setting the gain of the feedback loop (SPM software) to zero, the feedback control is switched off. To prevent damage and/or interference of the operational amplifier (OPA111) all connections to the preamplifier are disabled via a switchbox. By switching an electromagnetic relay (see Fig. 6), an electrical shortcut to the tip is established and the resistance between tip and sample can be monitored. With all four tips in electrical contact with the sample, four-probe measurements can be performed. A Keithley 6487 picoammeter/voltage source is used as a current source between the outer probes while the voltage drop between the inner probes is measured with an Agilent 34401A 6 $\frac{1}{2}$ digit multimeter (input impedance >10 G Ω).

2. Sample preparation

Four probe experiments were performed on silicon-on-insulator (SOI) samples (Ultrasil, Inc.), which consist of an undoped Si(100) layer with a thickness of $3.5 \mu\text{m}$, separated from a highly doped Si(100) substrate called a “handle” ($410 \mu\text{m}$ thick) by a buried oxide layer (500 nm thick). The handle is highly doped (resistivity of $1-10 \Omega \text{ cm}$) allowing the insulating top layer to be flash cleaned by resistive heating of the handle [see schematic of the SOI sample in Fig. 10(b)]. The dimensions of the crystal are $10 \times 5 \times 0.41 \text{ mm}^3$. Prior to introduction in vacuum the crystal was

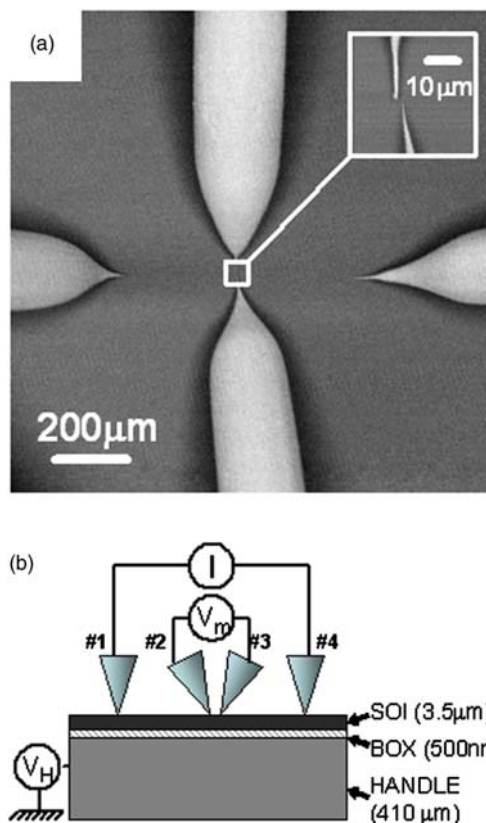


FIG. 10. (Color online). (a) SEM image showing the in-line configuration with the inner tips at a distance of $2.5 \mu\text{m}$. The inset in the upper right corner is a magnified view of the marked area. The scale bar represents $200 \mu\text{m}$ for the overview image and $10 \mu\text{m}$ for the inset; (b) schematic of the experimental setup.

chemically cleaned *ex situ* using the following recipe: (1) 10 min acetone; (2) 10 min ($\text{H}_2\text{O}_2:\text{H}_2\text{SO}_4$ 1:2) at 130°C ;²³ (3) Standard Clean-1 at 65°C for 5 min ($\text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{NH}_4\text{OH}$ 5:1:1); (4) Standard Clean-2 at 65°C for 10 min ($\text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{HCl}$ 6:1:1);^{24,25} and (5) oxide etch for 15 s using ($\text{HF}:\text{H}_2\text{O}$ 2:100). This treatment is known to produce a hydrogen-terminated Si(100) surface free of organic contaminants.²⁶ The crystal was subsequently annealed in vacuum for 20 min at 870 K , followed by 20 min at 1170 K and then by a 15 s flash at 1470 K . Following this treatment the Auger spectrum reveals an intense Si peak at 92 eV and only tiny signals from carbon (272 eV) and oxygen (502 eV) (not shown). The C/Si peak-to-peak ratio of the clean sample was found to be less than 0.002, which corresponds to a C atomic fraction of 0.005 in the depth of Auger sampling.

3. Results

A typical experimental configuration is shown in Fig. 10(b). In a first step, all four tips were brought in contact with the clean Si(100) crystal, according to the positioning procedure described above. The four probes are arranged in a linear fashion as shown in the SEM image Fig. 10(a). The spacing d_1 between the inner probes and d_2 between the outer probes was determined by SEM. Throughout the experiment d_1 and d_2 were maintained constant respectively at

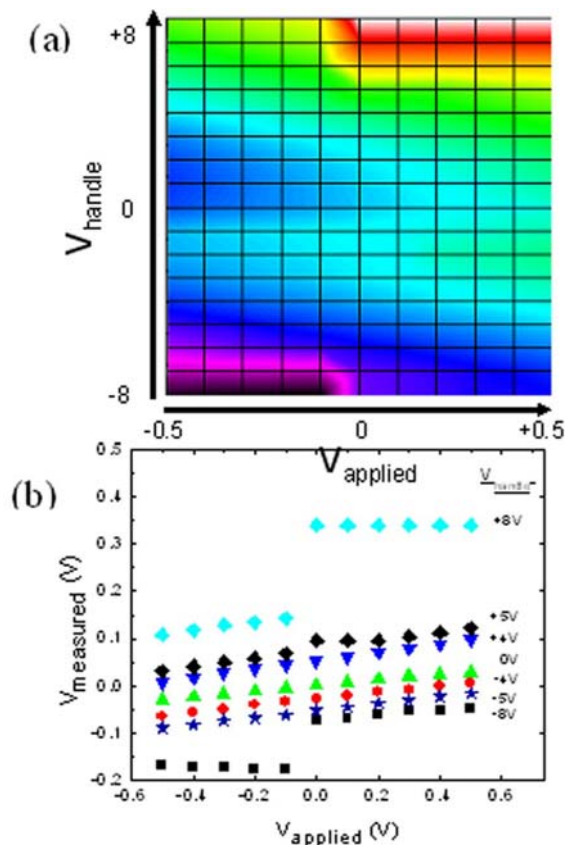


FIG. 11. (Color online). Four-point probe measurement on SOI sample: (a) map of the voltage drop measured between the inner probes (V_{measured}) as a function of the voltage applied between the outer probes (V_{applied}) and the bias voltage on the handle (V_{handle}) and (b) cross section of (a) for various values of V_{handle} .

2.0 and 10 μm . A voltage (V_{applied}) was applied between the outer tip No. 1 and tip No. 4, and the resulting voltage drop between tip No. 2 and tip No. 3 (V_{measured}) was measured for different values of the handle voltage V_{handle} .

Figures 11(a) and 11(b) summarize the resulting characteristics of the “device” formed by the SOI sample, the handle and the four probes. Figure 11(a) shows an intensity map of V_{measured} as a function of V_{handle} and V_{applied} . The characteristics are linear for low values of V_{handle} , and become highly nonlinear at sufficiently large V_{handle} . Figure 11(b) shows a “cross section” of Fig. 11(a) for seven values of V_{handle} : -8 , -5 , -4 , 0 , $+4$, $+5$, and $+8$ V. The handle voltage causes a local depletion region at the “source” and “drain” which are the outer tips No. 1 and No. 4, thus reducing the measured value of the voltage. This transition from ohmic behavior to a highly nonlinear device characteristic is typical of a field effect transistor (FET). The difference is that this FET is

“roaming” because the source and drain leads are STM probes that can touch down at different locations on the sample. A roaming FET may prove useful in probing the transfer characteristics of a variety of nanoscale devices such as quantum dot-based single electron transistors.

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