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DESIGN NOTE

In situ nanomanipulation system for electrical measurements in SEM

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Abstract
A versatile *in situ* measuring system in a SEM with four independently moveable tips was developed. The system allows manipulation as well as electrical contacting of objects on the micro- and nanometer scale. The SEM provides a high vacuum (HV) chamber, but also a variable pressure (VP) mode which allows imaging of conducting or nonconducting objects and surfaces. In this work, we show the experimental setup and capabilities of this system while measuring a platinum surface.

Keywords: SEM, nanomanipulation, nanoprobing, nanoprober, SEM-workbench

1. Introduction

With ongoing scientific effort in the field of synthesizing, ordering and structuring of materials in the nanometer regime as shown with nanoparticles, instruments for imaging such as the scanning tunneling microscope (STM) or atomic force microscope (AFM) cannot be thought away anymore. Such scanning probe microscopy (SPM) methods are based on a slow process of repetitive line scanning, which does not allow imaging in real time. Anyhow, it is possible to image objects also with scanning electron microscopy (SEM) with nanometer resolution by means of a fast electron beam which scans the surface, providing cycle times up to several milliseconds.

But imaging is just one point in characterizing samples. Besides structure and morphology, physical properties like electrical properties are vital for a comprehensive analysis on the nanometer scale. In order to measure such properties, STM and AFM or electrical force microscopy (EFM) are applicable. However, as a disadvantage imaging and measuring in parallel is not possible. Additionally, electrical two-point measurements can only be done through the sample and not in the lateral direction. In SEM the electrical connection with samples can be implemented using *ex situ* prepared electrode structures via lithographic techniques, such as photolithography [1, 2], extreme UV-lithography (EUV) [3, 4] or e-beam lithography [5, 6]. These methods are undoubtedly powerful, but disadvantageous is their complex multi-step preparation. *In situ* measurements can be done either by decomposing a gaseous precursor forming metallic connections between sample and macroscopic electrodes [7] or using moveable probing tips of nanorobotic systems [8–12]. In this field several approaches have been developed using up to eight tips prepared from tungsten wires, which has its own challenges due to oxidizing and handling [13]. Although applicable for STM purposes, it becomes difficult to reach metallic contacts directly. Even in the case of tips with diameters of several 10 nm, etched tungsten reaches its limit. A second disadvantage of etched tips made from metal wires is their unknown spring constant. This may be an issue in measurements where forces applied to a sample are important. Bringing electrodes into proper mechanical contact is important for a sufficient electrical contact, but bears the risk of modifying or even destroying the sample.

Depending on the purpose of the particular experiment (resistance, potential, transistor characteristics, etc), the device under test (DUT) can be contacted by two to four electrodes. Two-point measurements lead to $I(U)$ curves with series resistances of the measuring setup and contact resistances to

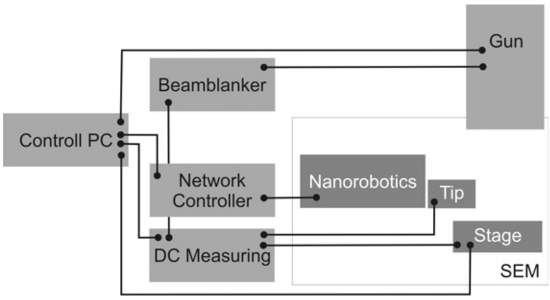

Figure 1. Schematic overview of the major components and their connections. The DC measuring system contains four source measuring units (SMUs).

the sample. Four-point measurements have the advantage of eliminating the contact resistance of the two electrodes, i.e. the mentioned oxide layers on tungsten, by measuring the voltage drop between two additional electrodes on the investigated area of the sample [9]. Nevertheless, the experimental effort to apply four electrodes is higher.

In this work, we introduce a novel and highly flexible nanorobotics system based on up to four steerable metal covered AFM tips representing nanometer sized electrodes.

2. Experimental setup

2.1. Overview

A high resolution scanning electron microscope (LEO/ZEISS Supra 35 VP, Germany) was expanded by a nanorobotics system (Klocke Nanotechnik [14], Germany), including four absolute positioning manipulators, a sample stage for e-beam lithography, accessories like tip approach sensing systems, different current or force sensitive probe tips and automation software including remote control and automation of SEM features (see figure 1) [14].

For two, three or four probe current measurements source measuring units (SMUs) can be included and controlled to record electrical data. The nanorobotics sample stage can be accessed by Raith Elphy software for high resolution e-beam lithography.

This basic system can be expanded, e.g. by different contact probes, microgrippers, a 3D-nanofinger for in-SEM dimensional measurements, *in situ* STM or AFM heads, pattern recognition for identifying nanometer sized objects in the SEM image and many more sub-systems [14].

2.2. SEM

The electron microscope is a commercially available SEM. The Gemini column is equipped with a field emission (FE) electron gun. The detection can be done using either the secondary electron (SE) detectors for high vacuum (HV) or variable pressure (VP) mode. While the SE detectors are used for lower resolutions, an SE in-lens detector can be used for resolutions down to 1 nm. Finally, the optional four-quadrant detector is used for detection of backscattered secondary electrons (BSE) and can be moved under the column. The energy dispersive electron x-ray spectroscope (EDX, Oxford, INCA Energy 200 with SiLi crystal, 133 eV, 10 mm^2) allows the determination of the elemental composition of a sample.

Figure 2. Photo of SEM chamber with four nanomanipulators installed around the cone of the electron gun in the center.

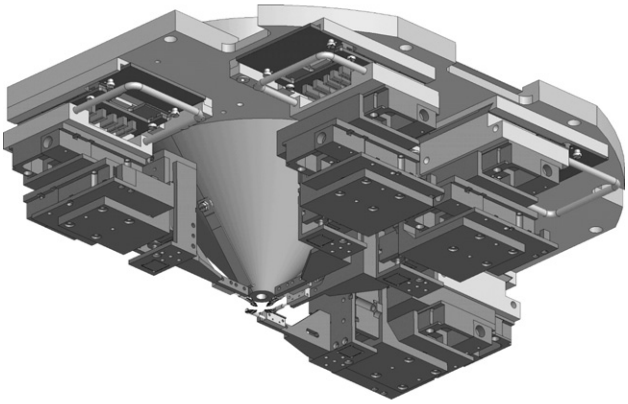

Figure 3. CAD image of the major parts of the nanorobotics system. Four manipulators are arranged in a half circle around the gun outlet of the SEM. The manipulators are docked in the adapter plate on top of the SEM.

Besides the usual HV mode, the SEM can be driven in VP mode with gas pressures from 33 to 130 Pa. Thus, also nonconductive samples, e.g. electrode structures on quartz, can be investigated without any additional surface coating with carbon or platinum, in order to prevent charging of the samples induced by the electron beam. In the VP mode surface charges are removed by gas molecules. In both modes the chamber is evacuated by a dry rotary pump and in HV mode additionally by a turbo pump.

The samples were placed on a five-axis stage that can be moved freely in x-, y- and z-directions with a precision of approximately 1 nm (combined with beam shift) and rotated as well as tilted with a precision of 0.1°.

2.3. Nanomanipulators used for nanoprobing

Up to four manipulators can be fixed and removed easily at the top of the chamber around the central electron gun outlet by docking stations (see figures 2 and 3). The cable system remains permanently in the chamber. This setup shows several advantages. First, the movement of the sample is fully decoupled from movements of the manipulator. Thus, the investigation area on the sample can be changed without rearrangement of the probing tips. Second, this configuration is independent of the type of sample. Even samples with huge lateral dimensions like wafers can be investigated at each area of interest. No space is lost for manipulator assembly on the

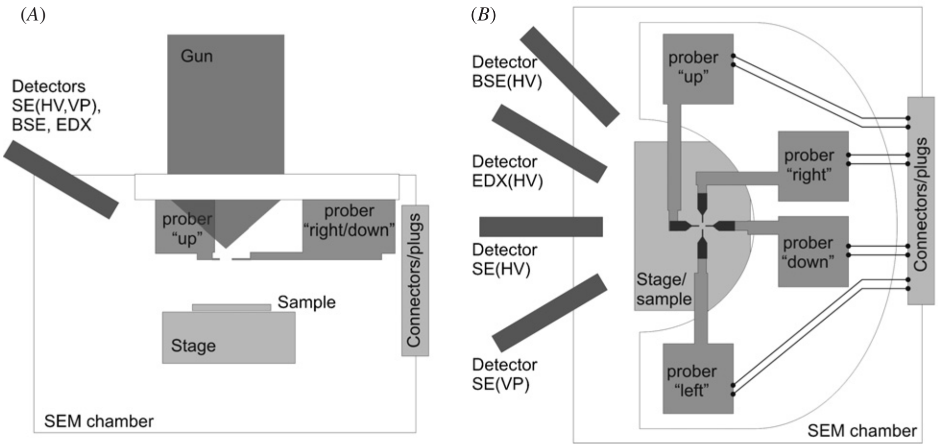

Figure 4. Sketch of the experimental setup for the nanomanipulation and electrical measurements with four probers. (*A*) Side view (prober 'left' not shown). (*B*) Top view from chamber top (gun outlet not shown). The chamber can be divided into the left part (detector side) and right part (prober installations side). Each prober is plugged individually into the adaptor plate at the chamber top and is connected via its own set of cables for controlling. Shielded cables for electrical measurements are used to connect the measuring tips with the electrical cable glands of the SEM chamber.

sample stage. In parking positions, the end-effectors fixed at these manipulators stay above the bottom line of the electron gun and thus do not restrict the free movement of the sample.

All four end-effectors can be moved automatically into the working position within a few seconds.

Each manipulator comprises three independent axes which can be moved with sub-nanometer precision and a repeatability of better than 50 nm. The manipulators move predictably along Cartesian axes, in contrast to tilting devices, where circular and linear movements are mixed. Different from electric motors coupled with piezo crystals, these manipulators have a resolution of a single nanometer over the whole stroke. Steps can be executed or inhibited at any time to guarantee a controlled movement without unwanted tip crashes. These features allow for a closed loop tip approach with 1 nm resolution over several millimeter strokes, without overshooting causing tip damage. The closed loop operation enables nearly drift-free positioning, for example to stay on contact points during probing. All movements can be stored by a sequencer for automation purposes. Real time movements can be executed by keypads for each manipulator. The step size can be changed dynamically by mouse click.

Every manipulator is equipped with a tool holder to incorporate exchangeable end-effectors. A shielded cable is included at the tool holder for connecting electrical probe tips with external measurement units. This system can also be equipped with further modules such as, e.g., microgrippers, (force-) sensors, complete SPM-heads or three-dimensional metrology.

For nanoprobing with probe tips the configuration shown in figure 4 was established. It shows the division of the SEM chamber into the left part containing all electron detectors (except the in-lens detector mounted in the column itself) and the right part, where all manipulators are placed. Because no further parts of the manipulators are in between sample and detectors except the probing tips, the influence of the manipulation system on the emitted electrons is negligible.

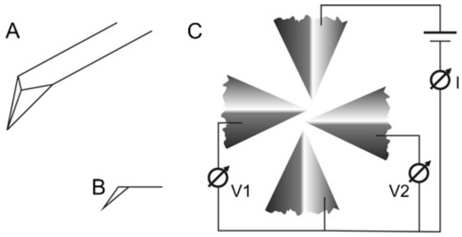

Figure 5. (*A*) Spatial and (*B*) side view of an AFM tip with the elongated tip in the front. (*C*) The arrangement in a four-point probe measurement (for the SEM image, see figure 9(*A*)).

2.4. Controlling system

Each manipulator is controlled by an external controller, which is connected via Ethernet with the control PC. The software contains the referencing procedures as well as simple and advanced control macros. Every axis can be accessed individually and driven either step-wise or with defined length values. Macro procedures also provide an automatic coarse approach to the surface with height control via an interface to the SEM software.

Because the scanning electron beam induces charge on the sample surface, it must be switched off during measurement of the electrical current between the tips. The internal beam blank of the SEM software places the beam in a corner of the scanning field and is not applicable for such measurements. Turning off the acceleration voltage takes several seconds and thus is also not feasible. A real termination of the beam is possible using a beam blanker from the lithography system (Raith, Dortmund, Germany) because this immediately deflects the beam and removes it from the column. Coupled via a TTL line with the measuring parameter analyzer, the beam is fully blanked during any electrical measuring procedure.

2.5. Tip production and characterization

For our measurements we used homemade metalized AFM tips with a force constant of approximately 40 N m^{-1} and a

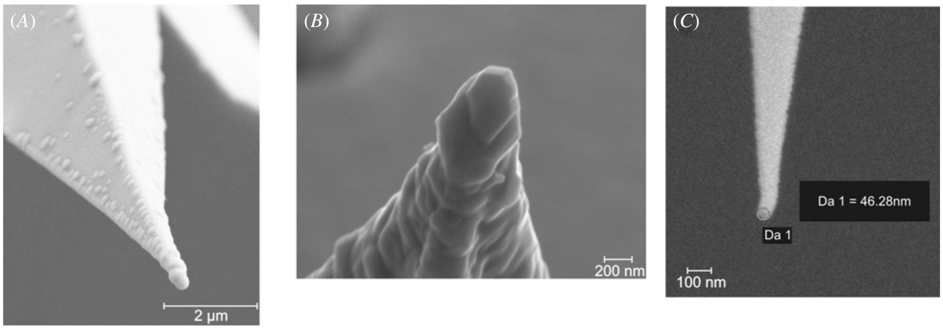

Figure 6. (*A*)–(*C*) Metal coated tips. Tip diameters can be varied down to approximately 46 nm.

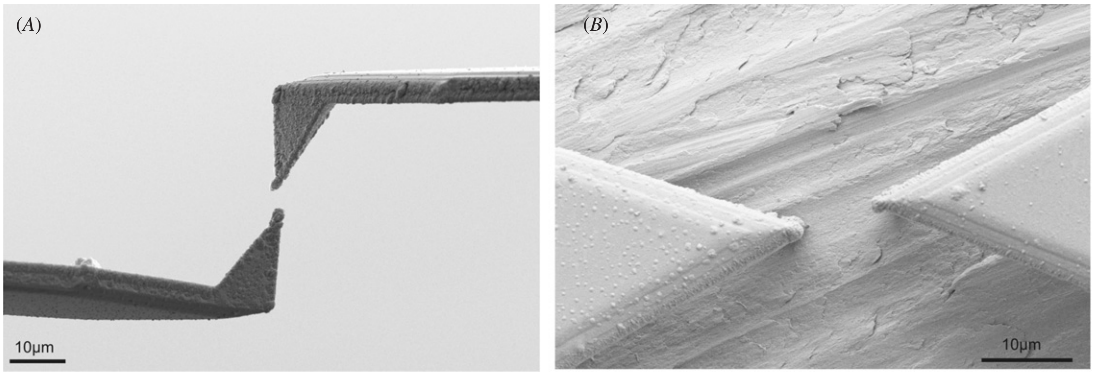

Figure 7. (*A*) Tilted tips before direct contact between them. (*B*) Two tips on a freshly cleaved platinum wire.

special geometry. Usually AFM tips are constructed in such a way that the tip is somewhere beyond the end of the cantilever and thus not visible from the top. For our purpose we used special elongated tips in the front part of the cantilever (see figure 5).

Such tips were coated either in a sputtering process with noble metals (e.g. gold or palladium) or by using electron beam evaporation of a platinum–iridium alloy. The thickness of this coating was as low as 30 nm which led to tip diameters of approximately 46 nm. Due to the tip curvature, the effective contact area can be even smaller. Such metalized tips with different tip diameters are shown in figure 6.

2.6. *Tip positioning*

After installation and referencing of a manipulator, it can be automatically moved into a working position which is visible in SEM. Further movements can be done manually via the keypads to pre-position the tips in the area of interest. Using the coarse approach macro the tips are brought into a close horizontal position above the sample surface. Close approach to the surface is done manually. While lowering the tips the shadow of the electron beam becomes visible before making mechanical contact. Further movement downward leads to a lateral movement on the sample surface. Due to the fact that AFM tips were slightly flexible, this horizontal movement is sprung. The applied force depends on the spring constant of the cantilever and is in the range of about several nN.

For comparison, table 1 summarizes the characteristic features of the nanorobotics system introduced here together with those of other systems.

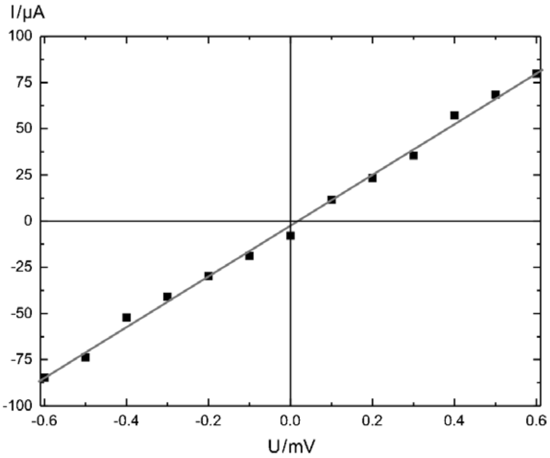

Figure 8. Sample $I(U)$ curve of a short circuit. The resistance was determined from linear fit ($r = 0.999$) to 8 Ω.

2.7. *Measurements*

All electrical measurements were carried out with a 4156C parameter analyzer (Agilent). The recorded $I(U)$ curves were limited to a maximum current to avoid high electric fields on the small conducting areas between tips and sample.

While recording the electrical current, the electron beam was blanked (switched off) using the beam blanker of the built-in lithography system to avoid additional current from incident primary electrons. Otherwise, a saw tooth like overlay with currents up to 100 pA appears in the $I(U)$ curves. Additionally, the contacted samples and tips were

Table 1. Comparison of nanorobotics systems.

	Comparison of Klocke Nanotechnik Nanorobotics manipulators with other manipulators	High performance nanorobotics	Low-cost nanorobotics	Tilting device[a]	Hybrid drives[b]
1	Cartesian movement of independent axes (important for easy operation)	Yes	Yes	No	Yes
2	Absolute positioning vertical axis (in the ‘blind’ direction of a SEM), repeatability better than 60 nm	Yes	Yes	No	No
3	Optional position sensors also for *xy*-axes, repeatability better than 60 nm	Yes	No	No	No
4	Small design (to fit in any chamber)	Yes	Yes	Yes	No
5	Many options to fix tools easily	Yes	Yes	No	Yes
6	Robust (crash resistant)	Yes	Yes	No	Yes
7	Moveable (by hand or by collisions) without losing the position information	Yes	No	No	No
8	Pure piezo drive from nm to cm stroke and not an electro motor coupled with a piezo	Yes	Yes	Yes	No
9	Stroke selectable in a range between 5 and 50 mm	Yes	No	No	Yes
10	Modular design to choose size and stroke for each axis	Yes	No	No	No
11	Stationary assembly designed ⇒ sample can move independently of manipulator. If necessary also moveable with sample stage	Yes	Yes	Yes	No
12	‘Micro-Jackhammer’ mode (with, e.g., 50 G acceleration) to process material	Yes	Yes	Yes	No
13	Secure approach of the sensor/actuator by different probe techniques	Yes	No	No	No
14	Force feedback option including electronics, FF-joystick, diagram software and automation	Yes	Yes	No	No
15	A compatible series of different microgrippers	Yes	Yes	No	No
16	Upgrade to form wafer probing systems including diagram software available	Yes	Yes	No	Yes
17	Option as micro tensile machine available	Yes	Yes	No	No
18	Upgrade to form the first real dimensional SEM/FIB with nm precision available	Yes	No	No	No
19	Option of vision system for pattern recognition as integrated software package available	Yes	Yes	No	No
20	Plenty of expendable items available: small sensors and actuators including adapters	Yes	No	No	No
21	Compatible absolute positioning ultra high precision sample stages available	Yes	Yes	No	No
22	Software on three levels: DLL, manual control (keypad, joystick) and automation by macros & process control sequencer	Yes	No	No	No
	Total Yes	22	14	4	5
	‘Performance factor’:	>4/1			
	‘Price factor for comparable items’:	≪4/1			

High performance and low-cost nanorobotics are from Klocke Nanotechnik.

[a] E.g., the older ‘Nanomanipulator’ from Klocke Nanotechnik or the MM3 from Kleindiek Nanotechnik.

[b] E.g., from Zyvex, USA or from Kammrath & Weiss, Germany.

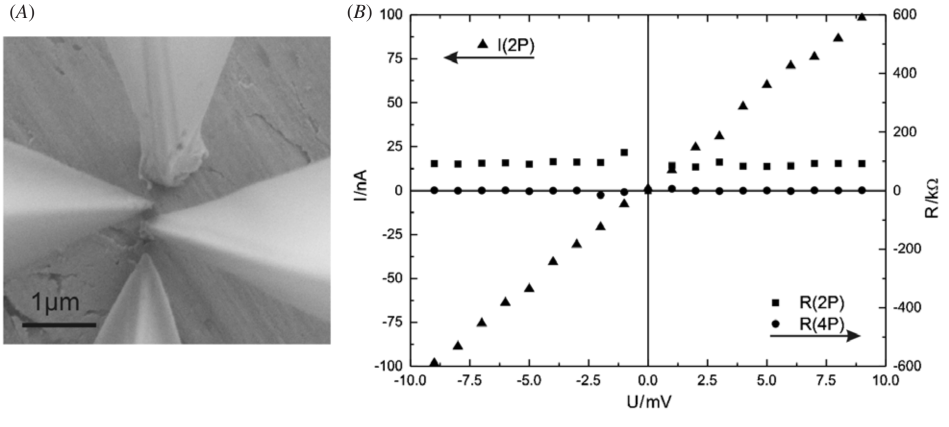

Figure 9. (*A*) SEM image and (*B*) $I(U)$ curve (left *y*-axis) of four-point probe measurement on a freshly cleaned Pt wire. The calculated resistance of the two- and four-point probe measurements is shown on the right *y*-axis of the diagram (*B*).

grounded to eliminate any charging caused by electron beam scanning.

To test this setup, the first experiments were done on short circuits. In these tests, the conductivity of the tips themselves was investigated. For this purpose a two-wiring setup was installed to determine the overall resistance of all plugs, cables and finally the two tips. The measuring tips were tilted toward each other at 90° to control the direct contact between them (see figure 7(*A*)). To prove the contact on a solid support, two tips were also brought into contact on a freshly cleaned platinum wire (see figure 7(*B*)).

3. Experimental results

The measured $I(U)$ curve reflects ohmic behavior with an overall resistance of the whole setup of about 8 Ω (see figure 8). This very low resistance shows impressively not only the metallic conductivity of the coated tips, but also the capability of measuring samples with a very low series resistance. Using this setup, nanometer-sized objects (i.e. metal nanoparticles) become measurable which will be shown in further experiments on nanoparticle arrangements [15].

To demonstrate the four-point probe measurement capability as shown in figure 5(*C*), the platinum wire of figure 7 is measured with four probe tips. These tips were coated with a hard metal alloy to increase the lifetime. In all measurements these tips showed an increased resistance compared to the platinum tips, reflecting the contact resistance typical for heterocontacts.

In a four-probe measurement four tips were brought into contact on a Pt wire within a maximum distance of 950 nm (upper and lower tips in figure 9(*A*)). These tips were used to measure the $I(U)$ curve representing two electrodes in a two-point probe measurement. The $I(U)$ curve is shown in figure 9(*B*). The resistance was calculated to be 100 kΩ. When, two additional tips were placed in between, with a distance of 200 nm, this setup allows a four-point probe measurement, where the two additional tips were applied to measure the voltage drop. Figure 9(*B*) shows the calculated resistances derived from respective $I(U)$ curves, for the two-point probe and for the four-point probe measurements, respectively. It becomes evident that the additional contact resistance of the two-probe setup almost vanishes giving an average resistance of 17 Ω for the platinum wire.

4. Conclusions

We presented a versatile nanorobotics system capable of electrical *in situ* measurements in SEM. The basic setup is applicable for up to four probes. Metal-covered electrodes with tip diameters below 50 nm were shown. Short circuit measurements of two metal-covered tips on a metal support resulted in good conductivity with ohmic behavior. Four-point probe measurements on a sub micrometer length scale help to suppress contact resistances of metal heterocontacts. This setup enables the direct visualization of surfaces as well as the measurement of structures with dimensions in the range of a few tens of nanometers.

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