Performing probe experiments in the SEM

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Abstract

A four nanoprobe system has been installed inside a FEI XL30 F scanning electron microscope (SEM), and shown to be fully compatible with the normal functions of the SEM and also a Gatan cold stage (model C1003, -185 – 400 °C). With some selected examples of applications, we have shown that this nanoprobe system may be used effectively for gripping, moving and manipulating nanoobjects, e.g. carbon nanotubes, setting up electric contacts for electronic measurements, tailoring the structure of the nanoobject by cutting, etc. and even for making unexpected nanostructures, e.g. a nanohook. Applications in other areas have also been speculated, limitations or disadvantages of the current design of the probe system were discussed, and methods for possible improvement were suggested.

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1. Introduction

The ultimate goal of nanoscale science and technology is to create and manufacture things, e.g. computers, via a controllable fashion at the molecular or even atomic level (Feynman, 1960; Amato, 1999). The ability to measure and manipulate at the nanometer scale is crucial to the progress of nanoscale science and technology. Electron microscopes and scanning probe microscopes (SPM) made this possible and are essential tools in the development of nanotechnology.

While the scanning electron microscope (SEM) may be used most conveniently for observing complicated structures with nanometer resolution and large depth of field, it is basically a passive tool in that one can only observe objects but cannot do much to change it, although in few cases the damage on the object by the incident high energy electrons have been used to the advantage of the researcher, e.g. to drill a small hole or more generally to do electron beam lithography (Morgan et al., 1992). A probe system such as an SPM, on the other hand, may be used in many ways to manipulate and perceive objects actively (Wiesendanger, 1994). It is therefore highly desirable to integrate the probe system inside the SEM.

Four MM3A nanoprobe systems manufactured by Kleindiek company were recently delivered to our laboratory at Peking University. Because of the small dimension and high vacuum compatibility, these probes can be installed in the SEM thereby combines the advantages of the probe system and the electron microscope. With these probe systems in the SEM, we can not only observe the nanoobjects using the fine electron beam of the SEM, but also actively manipulate the nanoobjects, making contacts with them and to perceive and change them. In this article we will briefly describe our probe plus SEM system and illustrate, via several selected examples, the power of the system in the study of nanoobjects.

2. System description

Shown in Fig. 1 is an image showing a MM3A nanoprobe, together with a Chinese penny which has a diameter of 1.8 cm. The movement of the arm (onto which a sharp tungsten tip is mounted) is driven by two perpendicular rotation motors RM-1 and RM-2. The first motor RM-1 is to the left of Fig. 1, which controls the horizontal or ϕ angle rotation of the arm, while the second motor RM-2 is to the right of the first motor, and this motor controls the vertical or θ angle rotation using spherical coordinates. Together these two rotation motors enable the

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Fig. 1. A MM3A nanoprobe.

arm to move on a surface of a sphere with an accuracy of about 2.5 nm. The radial movement of the tip, which is mounted at the end of the arm, is controlled by an additional motor which drives the radial motion of the arm, and the finest step of this radial motion is 0.25 nm. The range of the motion of the rotation motor can be as large as more than 180°, while that of the radial motion is 12 mm. All three motors can be driven with varying speed, and no noticeable creeping or delayed motion was observed when changing from high speed to lower speed.

Fig. 2. (a) Four MM3A nanoprobe systems mounted on a probe cradle and (b) the nanoprobe cradle was installed inside the SEM with three MM3A nanoprobe systems approaching a specimen on a Gatan cold stage.

3. The nanoprobe systems in the SEM

Because of the small size and their high vacuum compatibility, the MM3A nanoprobe systems may be conveniently installed inside the SEM. Shown in Fig. 2a are four MM3A nanoprobe systems mounted on a probe cradle, and in Fig. 2b are three such systems installed inside the SEM (FEI XL30 FSEM). The probe cradle was so mounted inside the SEM that it was physically disconnected from the SEM sample stage. The sample stage shown in Fig. 2b is a Gatan cold stage (model C1300, temperature range from -185 to 400 °C). The probe cradle was mounted in the SEM via four screws, and may easily be uninstalled from the SEM. In principle the presence and operation of the MM3A nanoprobe systems inside the SEM do not interfere with the normal operation of the SEM.

Shown in Fig. 3a is a picture showing an external view of our SEM installed with four MM3A nanoprobe systems. The five small boxes near the SEM column to the right of the picture are the four nanocontrollers for the four MM3A nanoprobe systems inside the SEM and one master connector (on top of the four nanocontroller). Each controller can be used to define the movement of a MM3A nanoprobe, e.g. the speed and directions of motion of the probe, and the actual motion of the nanoprobe may be controlled either by this nanocontroller or the joy stick lying on the desk of the SEM. To the left of Fig. 3a is the computer screen of

Fig. 3. (a) An external view of the SEM installed with four MM3A nanoprobe systems. (b) SEM image showing the four nanoprobe systems operating inside the SEM.

Fig. 4. Two SEM images taken (a) before and (b) after a 1-h experiment.

the SEM showing the three nanoprobe installed as in Fig. 2b. Shown in Fig. 3b is another SEM image showing all four MM3A nanoprobe operating inside the SEM.

4. Stability of the system

To investigate the stability of the probe systems inside the SEM, we monitored the sample and the MM3A nanoprobe inside the SEM by recording one image every minute over a period of more than one hour. Shown in Fig. 4a is the SEM image recorded prior to the experiment, and in Fig. 4b is the SEM image recorded after the experiment. A quantitative analysis of the recorded SEM images shows that the motion of both the probes and the sample follows a same general trend. Shown in Fig. 5a are the x (parallel to the horizontal edge of Fig. 4, from left to the right) and y (vertically from top to the bottom) coordinates (in pixels) of the two marked points A and B of Fig. 4 as a function of time. It should be noted that direct measurements on a few SEM images like those shown in Fig. 4 would result in large errors. This is because one pixel in Fig. 4 corresponds to 4.6 nm and the precision with which the coordinates of points A and B were measured was around 2 pixels. Results shown in Fig. 5 which were obtained from many SEM images recorded over a long period (over 63 min) do provide a clear evidence for the general trend of the drift of the sample (point A, at which two nanotubes intersect) and the probe (point B, a feature at the probe tip) during the experiment. An average

Fig. 5. (a) Absolute drift of points A and B as a function of time and, (b) relative drift of the two points.

drift speed may be obtained from Fig. 5 by fitting each coordinate curve by a straight line, which gives the following drift velocities for the two points A and B: $V_x(A) = 2.9$ nm/min, $V_y(A) = 3.2$ nm/min, $V_x(B) = 2.9$ nm/min, and $V_y(B) = 4.5$ nm/min. A careful examination of the recorded images like that shown in Fig. 4 as well as Fig. 5a reveals, however, that the relative movement of the nanotip (point B) with respect to the sample (e.g. point A) is far less than the apparent movement of either the tip or the sample. Since the MM3A nanoprobe systems and the sample were mounted on separate bases, our measurement suggests that the real stability of our SEM sample stage and the MM3A nanoprobe systems is far better than what one would guess by comparing the absolute positions of Fig. 4a and b. Fig. 5b shows the relative movement of the two points A and B, i.e. $\Delta x = x(A) - x(B)$, $\Delta y = y(A) - y(B)$, between the tip and the sample, and direct fitting of the experimental data gives an average relative drift velocity of 0.02 and 1.25 nm/min, respectively, for the x and y directions. The apparent larger drift between Fig. 4a and b may result, among other effects, the instability of the high voltage and scanning coils of the SEM. Fig. 5 suggests that the relative drift of the probe

with respect to the sample is perhaps a better measure of the stability of the probe systems.

5. Results

MM3A nanoprobe may be used individually or in group to carry out more complicated tasks, e.g. to move nanoparticles, to set up electric contacts for current–voltage (I – V) measurement, and to inject chemical reagent to a nanoobject and to observe local chemical reactions. The MM3A nanoprobe controller may also be used to drive a nanogripper to perform nanomanipulations. In this section, we will present some of our preliminary results to illustrate some possible applications of the nanoprobe systems in nanotechnology.

Fig. 6 are two SEM images showing the procedure of gripping a nanoobject from a surface using two nanoprobe. A nanoprobe was first moved to be in contact with the nanoobject. The second nanoprobe was then used to grip the nanoobject together with the first nanoprobe. Since the two nanoprobe are controlled independently via two different nanocontrollers, it would be extremely difficult to move the nanoobject to a distance from the location where it was gripped. Fortunately as previously mentioned that the nanoprobe systems were mounted on a separate probe cradle, which was not connected to the SEM sample stage, the sample may therefore be moved independently of the nanoprobe systems. Instead of moving the nanoobjects using the nanoprobe, the sample stage or the substrate was firstly lowered to separate from the nanoprobe and the gripped nanoobjects. The substrate was then moved while monitored by the SEM to the desired location by the SEM sample stage, and moved up again so that the nanoobject may be released by the nanoprobe and dropped gently on the substrate surface without causing much damage to the nanoobject.

Carbon nanotubes are important building materials for nanotechnology. However, almost all synthetic methods result in bundles of carbon nanotubes rather than a well organized strand and it requires a complicated chemical procedure to separate individual carbon nanotubes that almost certainly would introduce damages of varying degree to the carbon nanotubes. Shown in Fig. 7a is a soot directly obtained from a chemical vapor deposition chamber (Che et al., 2003). By fixing one end of the soot, a carbon nanotube (in our case a multi-wall carbon nanotube) was drawn by using another nanoprobe. The nanoprobe may further be used to manipulate the carbon nanotube as shown in Fig. 8, e.g. to bend and to straight the carbon nanotube.

Accurate measurement of electronic transport properties of a single nanoobject, e.g. a carbon nanotube, remains one of the main challenges in nanoelectronics. Up to now only a handful of research groups around the world can perform such measurements. The principle obstacle that prevents such measurements is the establishment of proper electric

Fig. 6. Gripping nanoobjects on the surface using two nanoprobe.

contacts on the nanoobjects, e.g. carbon nanotubes. With the nanoprobe inside the SEM this can be done via a fairly straightforward fashion. Shown in Fig. 9 are two SEM images illustrating the procedure for setting up such

Fig. 7. Drawing a carbon nanotube from a bundle.

Fig. 8. Manipulation of a carbon nanotube by nanoprobe.

contacts on a carbon nanotube. Fig. 10 shows a different situation.

In Fig. 10a are shown two pre-patterned electrodes at the top and bottom of the image, and two carbon nanotubes connected to these electrodes. Unfortunately among the two carbon nanotubes only the one connecting both the top and bottom electrodes is desired, and the other one connecting only to the top electrode introduces additional current loop into the system and should be removed. Fig. 10b and c show that one can cut the undesired carbon nanotube using a nanoprobe. Electric contacts may then be set on the two pre-patterned electrodes as in Fig. 10d, and I – V measurements may then be carried out via the two conducting nanoprobe.

Apart from the conventional applications just described, the nanoprobe systems may also be used in certain unexpected situations. Fig. 11 shows the process of formation of a nanohook using the nanoprobe. The formation of the nanohook involves two conducting nanoprobe, which were kept in contact with each other as shown in Fig. 11a. By exerting a small force on the top nanoprobe, a nanohook automatically resulted when passing through the nanoprobe a large current and thereby providing more contacting area between the two nanoprobe and a reduced resistance between them. Fig. 11b and d show that the nanohook may be readily removed from the other nanoprobe and be used to manipulate the nanoprobe or other nanoobjects.

Fig. 9. The setting up of two electric contacts at a nanotube by using two nanoprobe so that I – V characteristics may be measured on the selected carbon nanotube.

Fig. 10. SEM images showing (a) two nanotubes connecting to the electrodes, (b) and (c) the cutting of one of the two nanotubes shown in (a) using a nanoprobe and (d) the establishment of two electric contacts at the two electrodes by two nanoprobe tips.

The MM3A nanoprobe controller may also be used to drive other tools such as a nanogripper as shown in Fig. 12. The four SEM images shown in Fig. 12 illustrate the open and close of the nanogripper, and the gripping of

a nanoobject by the gripper. Using this gripper one can conveniently lift an object from the substrate surface and move it directly to a different location. The finest step of movement of the current gripper is, however, of the order

Fig. 11. Four SEM images showing the formation of a nanohook.

Fig. 12. Four SEM images showing the movement of a nanogripper driven by a MM3A nanoprobe system.

of 0.1 μm, and the gripper jaws are 5 μm × 30 μm in dimension which may therefore not be used for manipulating true nanoobjects.

6. Discussions and conclusions

The chopstick is one of the greatest inventions by the ancient Chinese. From a practical point of view, the chopsticks enable people to eat food without using directly their fingers. Scientifically speaking the far reaching influence of the chopsticks is that the chopsticks extends human's fingers to reach otherwise unreachable or to work in dangerous environments, e.g. in boiling oil, and to perform otherwise impossible tasks.

The advancement of nanoscale science and technology demands that we extend the reach of human hands to the nanoworld, and being able to manipulate nanoobjects at the molecular level and ultimately to manufacture things via the bottom up approach. With the MM3A nanoprobe system we are now able to move a fine tip of a probe of tens nanometers with a subnanometer precision, to set up contacts with nanoobjects and to perceive them, which is a big step forward in nanotechnology.

To perceive a nanoobject one would need, in addition to an accurate probe driver, a feedback system that would tell us how much force the nanoobject is exerting on the approaching nanoprobe and to what extent the wavefunction of the nanoprobe and the nanoobject has overlapped via

e.g. monitoring the current passing through the nanoprobe. A simple implementation of such a feedback system to the existing one is to introduce a switch to the electric circuit of the MM3A nanoprobe controller. The switch would normally be in the on position, but once the current in the circuit exceeds a predefined threshold value the switch would stop the motion of the nanoprobe, thus enabling one to approach a nanoobject with relatively high speed without worrying too much about damaging the object. Once the nanoprobe is stopped by the switch, one can further approach the object via finer step of motion, e.g. the smallest step of 0.25 nm. A more sophisticated feedback system is that employed in the SPM, which may be used either in the STM mode by monitoring the current in the circuit or in the AFM mode by monitoring the force felt by the AFM tip.

While STM is currently the tool of the highest spatial resolution and capable of probing the local electronic structure of the sample, e.g. local density of state, it requires a conducting substrate to support the sample. In principle the electronic states of the sample are coupled with that of the supporting conducting substrate, and what was measured using a STM tip is therefore not necessary the property of the sample. And this is especially so for delocalized electronic states. AFM may be used for atomic resolution imaging of the surface structure of the sample in some favorable cases, but it cannot be used for measuring local electronic states of the object. Shown in Fig. 13 is a possible use of the nanoprobe to assist STM imaging of a nanoobject on an insulating substrate. A conducting

Fig. 13. A schematic diagram showing an arrangement for STM imaging of a nanoobject on the surface of an insulating substrate.

nanoprobe is first used to set up electric contact with the nanoobject lying on the surface of an insulating substrate. A STM probe may then be used, perhaps in constant height mode, to image the electronic structure of the conducting or semiconducting object, and to measure the electronic transport properties of the object.

Ideally one would also need a smart system to coordinate the movements of different nanoprobe, like human fingers. In principle using the current nanoprobe one could perform certain coordinated movements of different nanoprobe, but it is more like gripping a tiny object using two fingers of different people that requires a lot of practice and skill. The accuracy with which one can manipulate nanoobjects using the MM3A nanoprobe system is currently limited mainly by the finite size of the nanoprobe tip, and typically the tip is larger than 10 nm. The advancement of the nanotechnology now allows us to assemble a finer nanotube

of about one or two nanometers onto the tip of a nanoprobe. We expect by using such a fine nanotip the manipulation accuracy of the MM3A nanoprobe system may be further improved and converge ultimately to what one would achieve using a SPM system but with a much slower speed.

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