APPLICATION NOTE

Nanomanipulation in the SEM: Theory and Practice

Technological progress is moving toward an increase of complexity, power and integration, leading to the miniaturization of single device elements down to the nanoscale. New tools allowing engineers to interact with sub-micron scale objects are therefore needed to improve the control over the characterization and test of these devices.

Unfortunately, handling and sensing single nanoparticles can be very cumbersome to novices with no expert support. In fact, because of their size, nano-objects have to be observed under vacuum conditions in a scanning electron microscope (SEM). In this environment, forces acting between objects are fundamentally different from those at macroscale: the gravity plays a negligible role, while van der Waals and electrostatic forces take precedence.

This application note intends to provide the reader a basic understanding of the theory behind the forces acting on objects at nanoscale, and to propose a few tips and tricks to increase the efficiency in manipulating such objects in the SEM. The experimental section illustrates the theory with an example of nanomanipulators operated to pick up nanowires from a native substrate and to transfer them to another substrate.

FORCES ACTING AT NANOSCALE

The two most prominent forces acting between particles with radius R = [5, 1000] nm are the van der Waals forces (F_{vdW} = [0.8, 20] nN) and the electrostatic forces (F_{el} = [0.8, 10’000] nN). The gravity and electromagnetic forces are negligible for particles of that size.

Van der Waals forces are acting between molecules and surfaces separated by short distances (typ. between $1/R^6$ and $1/R^{12}$) and cause the adhesion of the objects. They are commonly used to pick and place particles with sharp probe tips.

Electrostatic forces are acting over long range (typ. $1/R^2$) and, depending if the objects are charged positively or negatively, they can be attractive or repulsive. Inside an SEM, dielectric particles sitting on a conductive substrate are charged with the electron beam irradiation. The charging depends on the ratio between the particle size and the electron beam interaction volume [Figure 2], volume which varies in function of the electrons energy and the particle material (typ. between 20 nm to 5 um). By playing with these parameters, the operator

In collaboration with:

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http://www.laboratorionest.it

Imina Technologies products in use:

– miBot™ BT-14 nanomanipulator
– Nanoprobing SEM Platform kit

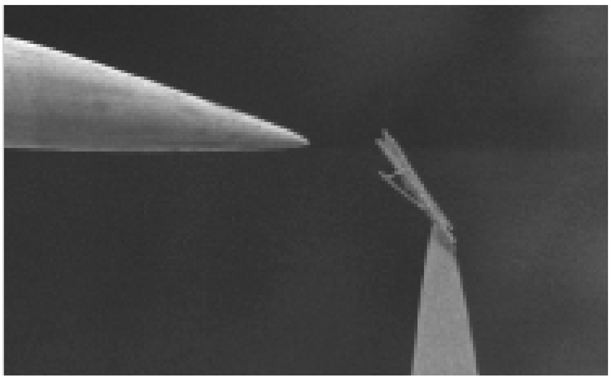

Figure 1. Manipulation of nanowires in situ in an SEM with two probe tips moved by high precision robots.

has thus some possibilities to adjust the electrostatic forces acting on the particles to manipulate, and possibly make the operation easier.

Playing with electrostatic forces to pick and place nanoparticles

To pick up a particle with a probe tip, the electrostatic forces acting on the particle must overcome the van der Waals forces which retain it on the substrate. In the case the size and shape of the particle would allow it, the electrostatic forces that would pull it on the tip can be generated with the SEM electron beam [Figure 3]. The particle must be small enough to get charged positively, and the probe tip must be metallic and non-grounded to get charged negatively. A particle will stick on the probe tip due to both the van der Waals and the electrostatic forces, as long as the tip is charged.

To drop off a particle from a probe tip, the opposite effect used for the pick-up is sought. The larger is the particle surface exposed to the substrate, the stronger are the van der Waals forces to tear it off the tip. And to amplify this effect, leaking away the charges from the tip will release the electrostatic forces. This can either be done by moving away the electron beam irradiation from the tip, or by grounding the tip.

Use case: manipulation of nanowires for transport characterization

At the National Enterprise for nanoScience and nanoTechnology (NEST), Dr. Stefano Roddaro and his team investigate the electro-thermal characteristics of nanowires. The manipulation of single nanowires is integral part of their experiments. This section illustrates the process of handling nano-objects in the SEM with their samples.

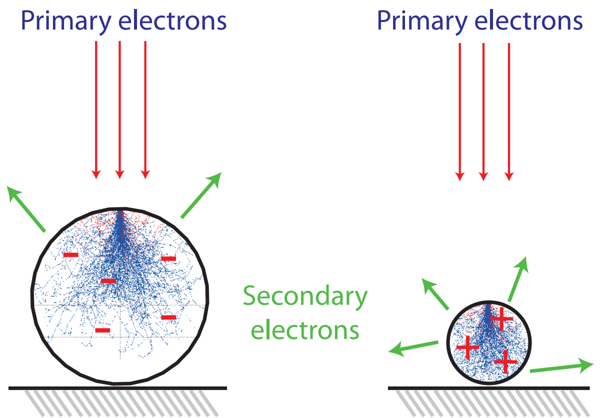

Figure 2. The smaller the particle, the larger the number of electrons leaving a dielectric particle under the effect of the primary electron beam irradiation. This results in small particles charging positively and large particles charging negatively.

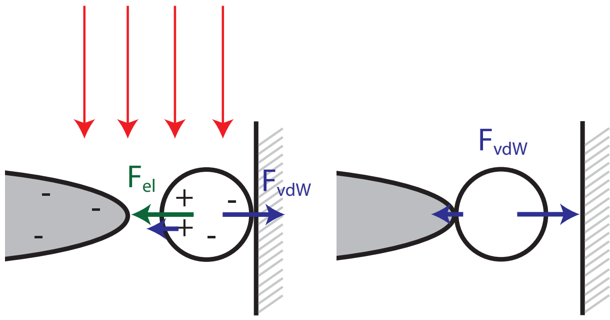

Figure 3. (Left) A particle is picked up when the electrostatic force (F_{el}) is larger than the van der Walls force (F_{vdW}). (Right) To release the particle, when it is close to the substrate, the electron beam is turned off to cancel F_{el} and let the substrate attract the particle by F_{vdW}.

EXPERIMENTAL SETUP

The experiment is carried out in a MERLIN SEM from Carl Zeiss with the Nanoprobing SEM Solution from Imina Technologies loaded with 4 miBot™ nanomanipulators.

To offer a better access to the nanowires, the sample, an InAs substrate on which nanowires are grown by Chemical Beam Epitaxy (CBE), is glued with a carbon conductive tape on an SEM stub with a 45° angled plane. The nanowires' diameter is in the range of 60 - 80 nm and the length between 0.6 to 1 um [Figure 4].

Tungsten probes with a tip radius of 100 nm are inserted on the probe holders of the miBot™ nanomanipulators. The sample is placed at the center of the Nanoprobing stage and the miBots around, very close to it. This gives the possibility to use short probes (15 mm), resulting in an increased mechanical stability of the manipulators and motion without vibration.

The motorized stage of the SEM is used to move the sample and the manipulators at a 15 mm working distance, and the microscope parameters are set to an acceleration voltage of 5 keV with a 190 pA electron beam current.

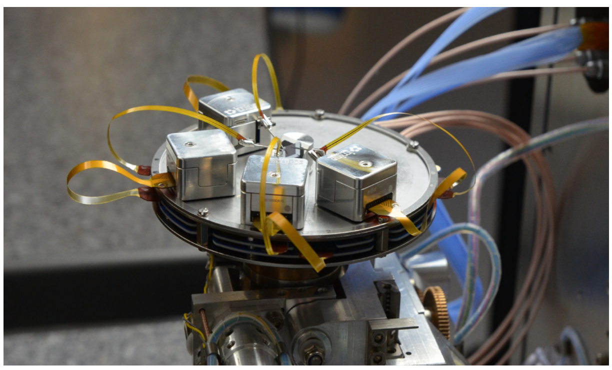

Figure 4. Imina Technologies Nanoprobing SEM platform loaded with 4 miBot™ nanomanipulators and mounted on the SEM translation stage. At the center of the platform, the sample is mounted on a 45° angled stub.

PICKING UP A NANOWIRE

Because of the nanowire elongated shape, the SEM electron beam irradiation is usually difficult to control. Hence, the electrostatic forces can unfortunately not be used at the operator advantage for nanowire pick and place. It is therefore advised to reduce the electrostatic effects by grounding the probes, limiting their time of exposure, and reducing the beam current to less than 200 pA.

Picking a vertically grown nanowire involves to first tear it off from its native substrate and then pick it up with the probe tip. This operation may be delicate if, as in this experiment, the nanowires are very flexible. In fact, they tend to jump away under the effect of the accumulated elastic energy when the breaking point is reached [Figure 5].

The nanowires should therefore be uprooted at their base [Figure 6]. This is achieved by slowly bringing the probe tip in contact with the base. When the contact is made, the probe is gently pressed against the wire until it breaks.

Although the operator only sees the probe tips in the microscope field of view, the control over its position and orientation is intuitive with the axes of Imina Technologies' control pad naturally aligned with the nanomanipulator motion directions. Moreover, as the miBot™ is free to rotate its body (+/- 180°), the probe trajectory can be optimized to navigate between the nanowires.

Approaching a probe tip of the base of a nanowire requires the operator playing with the speed and step size of the nanomanipulator. In this experiment, the approach is made in stepping mode with a horizontal speed ranging from 1 mm/s for the coarse positioning to less than 30 nm/s for the fine positioning.

After the nanowire is uprooted, the probe tip is brought in contact with the nanowire to pick it up with the van der Waals forces. The delicate operation of landing the tip in contact with the nanowire is more easily controlled by setting the miBot™ motion mode in scanning with a vertical speed reduced to less than 10 nm/s for the last steps. The nanowire can then be moved over several millimeters to the destination substrate.

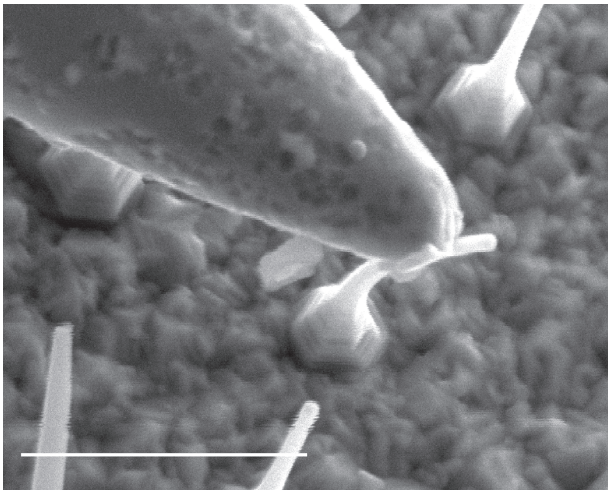

Figure 5. A probe tip is bending a vertical InAs nanowire.

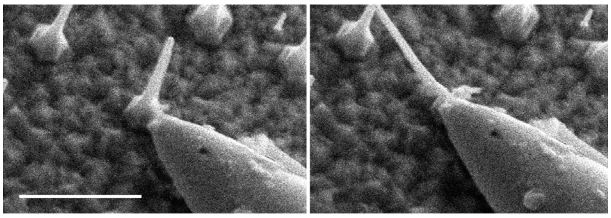

Figure 6. (left) A probe tip uprooting a nanowire at the base. (right) The nanowire is broken from its native substrate and can be picked up with the probe.

DROPPING OFF A NANOWIRE

To drop off the nanowire, the probe tip is brought at slow speed close to the destination substrate. At this moment, the operator should closely observe the nanowire at the tip of the probe. As soon as it moves, a contact between the nanowire and the substrate is established and the motion parameters should be tuned for a smooth landing. When the nanowire is only held by one of its ends, it quickly gets attracted by the field of van der Waals forces emanating from the substrate [Figure 7].

With two probes, it is also possible to orient the nanowire before releasing it by independently moving its ends [Figure 8]. Furthermore, similarly to chopsticks and a grain of rice that sticks, the operation of releasing the nanowire at destination is also much easier with a second probe tip to help push the nanowire on the substrate.

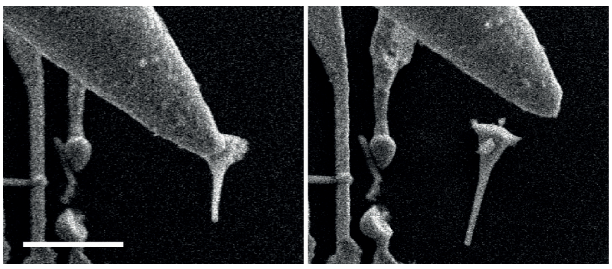

Figure 7. A single nanowire is moved by a probe tip (left) and then released on the substrate (right).

CONCLUSIONS

Nanomanipulation of particles in the SEM is a challenging task which requires training and patience to let the operator acquire a good control over the effects of forces at nanoscale. The operation is however greatly simplified and accelerated with the intuitive to drive miBot™ nanomanipulators. In fact, before the experiment starts in the vacuum chamber, time is saved by the possibility to pre-position the manipulators by hand around the sample. During the experiment, the speed of motion and the positioning resolution of the piezoelectric actuators are easily tuned from a control pad to adapt to the microscope magnification and the dexterity required by the manipulation. Moreover, the miBot™ compactness provide high mechanical stability and avoid vibrations when interacting with nanoparticles. Also, it enables operating the SEM at a working distance lower than 5 mm and, consequently, to decrease the electron beam energy to safeguard delicate samples.

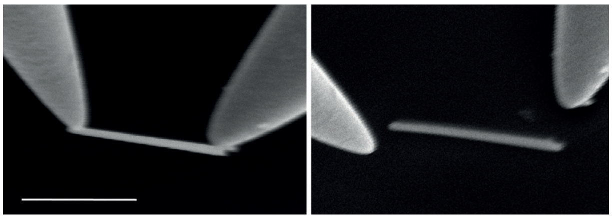

Figure 8. Two probes can be used to orient a nanowire (left). It is also easier to release a nanowire with the help of a second probe.

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