SCANNING ELECTRON MICROSCOPY BASED MANIPULATION AND CHARACTERISATION OF NANO-SCALE OBJECTS

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Abstract: Two scanning electron microscopy (SEM) based devices for positioning, manipulation and imaging at the nano-scale have been developed. The control and vision system is based on both a commercial scanning probe microscopy (SPM) controller and a client-server approach to ensure that nanopositioning and SEM image processing are executed in real-time. The evaluation of the two devices has been performed by implementing three different applications: (i) attachment of carbon nanotubes on SPM tips, (ii) investigation of mechanical properties of nanowires and (iii) tensile strength measurements for focused electron beam deposits.

Keywords: Cartesian manipulators, image processing, manipulation tasks, mechanical properties, robot control, robotic manipulators, telemanipulation.

1. INTRODUCTION

In recent years, the manipulation and characterisation of nanowires (NWs) and carbon nanotubes (CNTs) became a matter of particular interest in research. Due to their unique structure, small size, high aspect ratio and low density as well as excellent mechanical and electrical properties, nanotubes are expected to find use in a wide range of applications. Mounted on tungsten tips they can be applied as field emitters in displays or in high-resolution electron beam instruments (Bonard, *et al*., 1998; De Jonge, *et al*., 2003). CNTs mounted on atomic force microscopy (AFM) tips promise to overcome the limitations of standard tips regarding resolution and wear (Stevens, *et al*., 2000). Nanowires are of interdisciplinary interest to applications in the fields of bio-medical sensing, nano- and optoelectronics and photovoltaics due to their electrical, optical, mechanical and geometrical properties that deviate quite substantially from bulk (Law, *et al*., 2004).

For all these applications dedicated tools are essential to functionalise, manipulate and characterise – both mechanically and electrically – CNTs and NWs and assemble them into nano-devices. In addition, novel processes and strategies for nano-scale visualisation have to developed based on high spatial resolution imaging instruments like scanning electron microscopes (SEMs).

The manipulation and characterisation of nano-scale objects and of matter even at the atomic level has been opened up by the invention of the scanning tunneling microscope by Binning and Rohrer in the early eighties, and the subsequent invention of the atomic force microscope by Binning, *et al*. (1986). Scanning probe microscopy (SPM) based nanomanipulation has rapidly gained in importance during the last ten years, and several kinds of manipulation systems have been developed (Schaefer, *et al*., 1995; Requicha, *et al*., 1998; Theil-Hansenyk, *et al*., 1998; Li, *et al*., 2003). To overcome the main drawback of these systems, i.e. the lack of visual feedback in real-time, SPMs have been combined with haptic devices (NanoManipulator, NanoFeel, Omega Haptic Device), and virtual reality interfaces have been developed, for instance (Sitti and Hashimoto, 1998; Guthold, *et al*., 2000). Further on, SPMs have been integrated into SEMs combining the advantages of both instruments, e.g. allowing the combination of nano-scale chemistry, crystallography imaging via electron-matter interactions with information from tip-sample interactions like topography or magnetic/electrostatic force imaging. Several of such hybrid SPM/SEM systems have been developed, e.g. (Stahl, *et al*., 1994; Ermakov and Garfunkel, 1994; Troyon, *et al*., 1997; Joachimsthaler, *et al*., 2003).

In parallel to the use of SPMs, dedicated nanomanipulation systems have been developed and integrated into SEMs. This work was pioneered at the University of Tokyo by Hatamura and Morishita (1990). Subsequently, worldwide research activities in this field have been started (Yu, *et al*. 1999; Schmoeckel and Fatikow, 2000; Dong, *et al*., 2001). Compared to SPMs, these systems offer a larger workspace, greater flexibility, more degrees of freedom and dedicated control systems. However, their automation level is still low. SEM image processing, for instance, is hardly used for positioning or pick-and-place operations. Most of the systems still rely on the operator's attention to the movement of the positioning tables as shown on the SEM screen, and closed-loop nanomanipulation is still an exception.

2. SEM-BASED NANOMANIPULATION AND CHARACTERISATION SETUPS

Two different systems for the manipulation and characterisation of nano-scale objects have been developed and integrated into an SEM: (i) an atomic force microscopy setup, (ii) a six-axes Cartesian nanomanipulator. Both systems are described in the following.

2.1 AFM integrated into an SEM

The AFM setup can be used in scanning, i.e. imaging, and manipulation mode. It is composed of two different manipulators (Fig. 1). The sample is mounted on a three-axes Cartesian nanopositioning stage with integrated capacitive position sensors (P-620 series, Physik Instrumente, Germany). Piezoelectric actuators and a flexure guiding system provide a travel range of 50 μm for each axis and a sub-nanometer resolution. This stage is used to generate the scanning trajectory in scanning mode and for fine positioning of the sample in manipulation mode. For coarse positioning towards the sample, the AFM tip is mounted on a three-axes nanomanipulator with two rotational and one linear degree of freedom (MM3A, Kleindiek Nanotechnik, Germany). The manipulator is driven by piezo actuators with sub-nanometer precision; by operating the actuators in slip-stick mode a working range of more than 100 cm^3 can be achieved. For AFM imaging as well as manipulation and characterisation of nano-scale objects a piezoresistive AFM

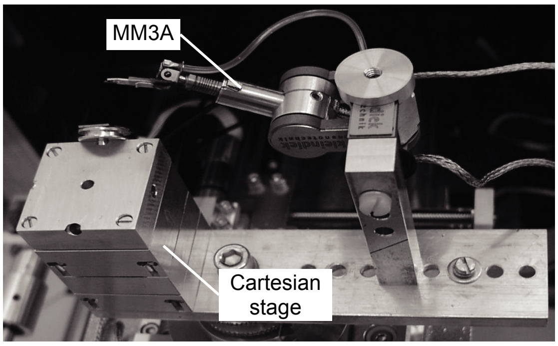

Fig. 1. AFM setup inside SEM.

cantilever can be mounted on this manipulator. Additionally, standard or specially shaped AFM cantilevers, e.g. Arrow™ probe (NanoWorld, Switzerland), can be used for nanomanipulation.

The whole setup is mounted inside an SEM such that the sample is at an angle of 60° with the electron beam. With the SEM sample stage, the sample's area of interest can be moved into the field of view. In scanning mode, the AFM is controlled using a fully digital control system for SPMs (Nanonis GmbH, Switzerland, www.nanonis.com). In manipulation mode, both nanomanipulators are controlled using the SPM controller's graphical user interface and a teleoperation device.

2.2 Cartesian nanomanipulator

The second system is based on a six-axes Cartesian nanomanipulator. This approach allows for semi-automated manipulation and characterisation of nano-scale objects and samples using different tools inside an SEM. The setup consists of four main components: nanomanipulator, nanotools, control system and SEM vision system.

Nanomanipulator. The nanomanipulator is driven by piezoelectric actuators combined with a slip-stick motion principle. The stepping-mode allows long displacements at a relatively high velocity of typically 5 mm/s. The resolution is limited to one step, typically 200-400 nm. Once the position is within less than one step distance of the target, the piezo actuators are deformed slowly until the final position is reached. In this so called *scanning mode*

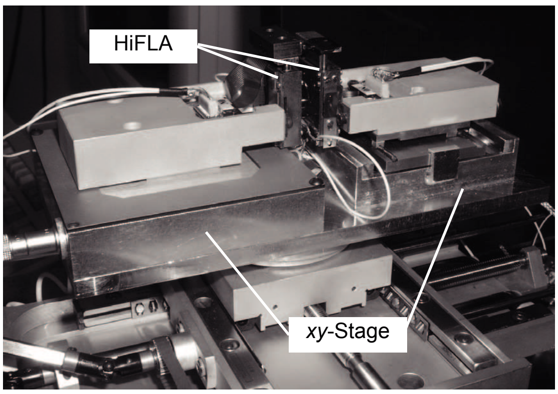

Fig. 2: Six-axes Cartesian nanomanipulator mounted on an SEM stage.

the resolution is a fraction of a step (typically better than 5 nm). In the setup shown in Fig. 2, two three-axes Cartesian positioning stages have been combined into a nanomanipulator with six axes and three degrees of freedom. Each positioning stage is built of an *xy*-stage and a so-called *High thrust Force Linear Actuator* (HiFLA) for *z*-positioning. The HiFLA has especially been designed to avoid electromagnetic interferences for operating inside an SEM (Mazerolle, *et al*., 2004). By implementing a pre-load mechanism, thrust forces of 1.6 N in vertical direction could be realised. The travel range of the nanomanipulator is 10 mm in *x* and *y*, and 8 mm in *z* direction; the maximum velocities are 5 mm/s. Incremental optical encoders with a grating pitch of 20 µm are embedded into each axis. A 50-fold interpolation yields a final resolution of 100 nm. By using the SEM image for fine position feedback, a resolution down to approximately 5 nm or even better can be expected – depending on the resolution of the SEM and the driving circuitry of the actuators.

Nanotools. For imaging, manipulation and characterisation of nano-scale objects, different AFM cantilevers are mounted onto the nanomanipulators described above. These AFM tips are also used as a tool for rapid prototyping at the nano-scale by nanolithography (Michler, *et al*., 2004). Furthermore, tasks like pushing, cutting, or indenting can be performed. By using an AFM cantilever with integrated piezoresistive sensors, the deflection of the cantilever can be measured with nanometer resolution. Thus, the cantilever can be used not only for AFM imaging but also as a force sensor in sub micro-Newton ranges.

Control system. The control system of the SEM-based nanomanipulator has to perform numerous tasks such as closed-loop control of the nanomanipulators' motions to generate the appropriate driving voltages for the actuators, trajectory planning, controlling tools and grippers, acquisition and processing of the SEM images as well as to control the peripherals (graphical user interface, teleoperation device, etc.). To get the required computational power, the control system is based on a client-server approach. A vision server acts as sensor and sends its data to a client. The data between client and server is sent through an Ethernet connection. For data transmission, the connectionless user datagram protocol (UDP/IP) has been chosen.

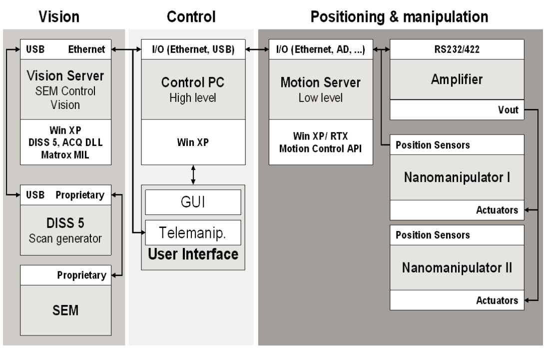

Fig. 3. Schematic diagram of the control system.

The control system implemented consists of a motion control server, a vision server, a vision and motion control client, electronics for signal generation and a teleoperation device, Fig. 3. The motion control server performs trajectory planning and closed-loop control of the nanomanipulator based on the position information of integrated linear encoders. It runs Windows XP with a real-time extension (RTX, Ardence Inc., USA). All time-critical operations, like trajectory generation, control and I/O operations, are implemented as real-time processes. Processes which are not time-critical, mainly communication via Ethernet with the motion control client, and setting of controller parameters, are realised as standard Win32 application. The vision server and the motion control server are connected via a LAN (Ethernet) to a higher-level control PC which collects all relevant data of the nanomanipulation system and provides the user with a graphical user interface (GUI) and a teleoperation device. The motion control client is a module of the high-level application software on the client PC. One main task of this client is to provide the operator with user interfaces to configure and operate the nanomanipulator system, either in semi-automatic or in teleoperation mode. Furthermore, closed-loop control of the nanomanipulator's motion based on SEM images is implemented at this client.

SEM vision system. The performance of the nanomanipulation system depends to a great extent on the performance of the sensors, which have to be able to provide the control system with accurate sensor signals in real-time. The integrated position sensors offer a resolution of 100 nm, which is not sufficient for some applications. Hence, this setup incorporates SEM-based visual control to realise flexible and secure applications. The vision signal can be either used as feedback information for a user in teleoperation mode controlling his actions or as sensor information for a control task. The following tasks are implemented: acquisition of SEM images, image analysis, definition of the needed actions and visualisation.

The basic tasks of the vision server are image acquisition and image analysis; they have been realised using third-party tools. SEM image acquisition and control is achieved through a control program (DISS 5, point electronic, Germany). A DLL provides an easy to use digital interface to collect SEM image data into computer system memory and to control SEM settings. For image analysis the Matrox Imaging Library MIL (Matrox, Canada) has been chosen. The most important operation on images is the localisation of features within an image based on pre-defined models. For noisy SEM data, edge detection provides a stable and fast method for object detection and tracking. The MIL has also been integrated through a DLL into the image server software. The image server acts as a sensor. Sensor data (e.g. position, scale, angular orientation of an object) is provided, and sensor settings (e.g. feature to track, threshold parameters for object detection) may be set.

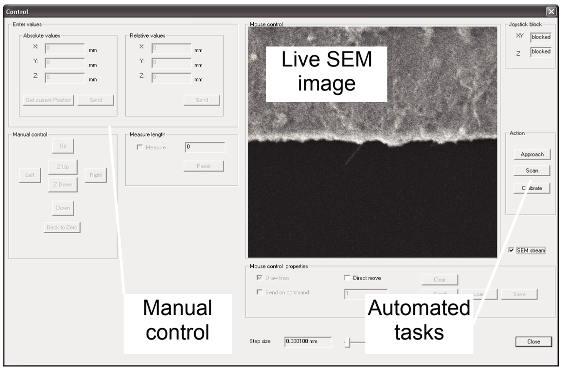

Fig. 4. Graphical user interface.

The image client is a module of the high-level application software and interacts with the graphical user interface, Fig. 4. The main tasks of the image client are collection of image server data (image data, position data) and definition of image server settings (object features, object detection properties).

The graphical user interface visualises SEM image features and enables interaction between the user and the application. Its main functions include: (i) visualisation of image data (live image of SEM, image of pre-defined features that are to detect), (ii) selection of image regions that define new models to be detected and (iii) interactive control, i.e. moving nanomanipulators by clicking in the image.

The definition of models that shall be detected by the image server is carried out using a live image of the SEM, Fig. 5. Within a running application the user can select an image region. The features within this region will be localized by the image server. For example the task of tracking an AFM tip can easily be optimised for different tip shapes by defining the appropriate model. Model settings such as a reference orientation, origin position, acceptance values, etc. can be adjusted. If the object detection fails (e.g. due to noisy SEM images) a refinement of the model is possible.

Motion control and the calibration of the actuators for various settings of the SEM magnification are also carried out with the help of vision features. The application measures the position of image details during motion and after a calibration step.

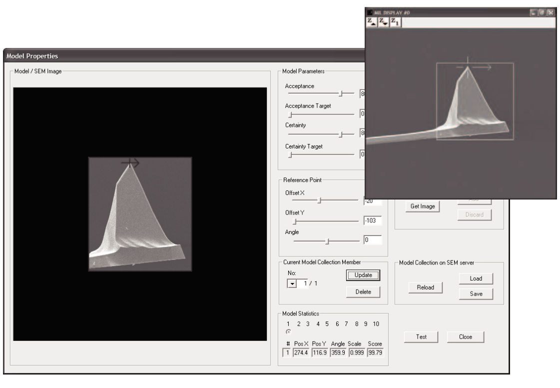

Fig. 5. Definition and tracking (inset) of the model of an AFM tip in an SEM image.

3. EXPERIMENTS ON NANOMANIPULATION AND NANOCHARACTERISATION

With the two setups described, various experiments on nanomanipulation and nanocharacterisation have been carried out. Three of them are described subsequently: (i) attachment of carbon nanotubes on AFM tips, (ii) investigation of mechanical properties of nanowires and (iii) tensile strength measurements for focused electron beam (FEB) deposits.

3.1 Attachment of carbon nanotubes on AFM tips

The benefit of attaching carbon nanotubes on AFM tips has been described before. The automation of this process is of great advantage compared to the manual assembly (Dai, *et al*., 1996; Yamamoto, *et al*., 1998; Yu, *et al*., 1999; De Jonge, *et al*., 2003) and a promising alternative to parallel processes (Cheung, *et al*., 1999). Therefore, we implemented object recognition and tracking of the AFM tip by processing the SEM image as described in section 2.2. Multi-walled carbon nanotubes with diameters of several tens of nanometers produced by an arc-discharge method were used. The nanotubes were aligned on a carrier using electrophoresis in a way that they protruded from the edge of the carrier. The nanotube carrier and the AFM tips were both mounted on the nanomanipulator (Fig. 6). The nanomanipulator was mounted on the SEM sample stage.

A suitable nanotube and the AFM tip were brought into contact by a combination of teleoperation and semi-automatic approach, Fig. 7c. When in direct contact, the nanotube stuck to the AFM tip due to adhesion forces. To ensure a rigid connection between nanotube and tip, amorphous carbon was deposited on the joint by irradiation with the electron beam. Afterwards, the nanotube could be retracted from the carrier To compare these AFM tips with standard ones, images of an Si wafer with a 50 nm granular Al coating have been taken. The results prove the better scanning performance of CNT-decorated AFM tips compared to standard tips (Keles, *et al*., 2004).

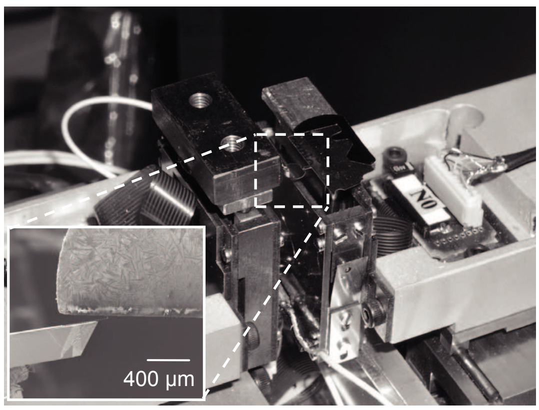

Fig. 6. Carrier with nanotubes and AFM tip mounted on the nanomanipulator.

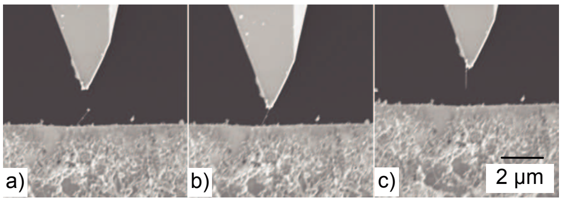

Fig. 7. Experiment on AFM tip decoration:
a) approach, b) touching, FEB fixation and
c) withdrawal of decorated tip.

3.2 Characterisation of nanowires

The mechanical stability of nanowires is essential either for their nanomanipulation, functionalisation or for integration into nano-devices. Several methods were used in the past to access mechanical properties of silicon NWs and nanobeams. The AFM setup inside the SEM described above was used to bend and move nanowires and perform mechanical testing experiments. From the acquired images, mechanical properties of NWs could be extracted. Bending experiments on silicon NWs grown vertically and epitaxially on a [111] oriented silicon substrate have been performed. By direct visual observation and using elasticity theory as well as simulations applying the finite element method, the fracture strength of the NWs could be determined. An AFM tip was used to bend a NW standing perpendicular to the substrate until it breaks (Fig. 8). A permanent deformation of the NW has not been observed. Even strongly deflected NWs snapped back to their original position when released. This allowed to calculate the maximum stress from the NW deflection just before fracture.

The AFM tip was mounted on the MM3A nanomanipulator, the substrate with the NWs was mounted on the Cartesian nanopositioning stage. The NWs were bent perpendicular to the electron beam, so that the deflection could be read out directly from the SEM image. The whole process was recorded in a video file. From the last image before fracture length l, diameter d and deflection s were extracted (Fig. 8). With these parameters, the maximum stress could be calculated. The average fracture strength of the NWs was (11±3) GPa, which is around 6% of their Young's modulus.

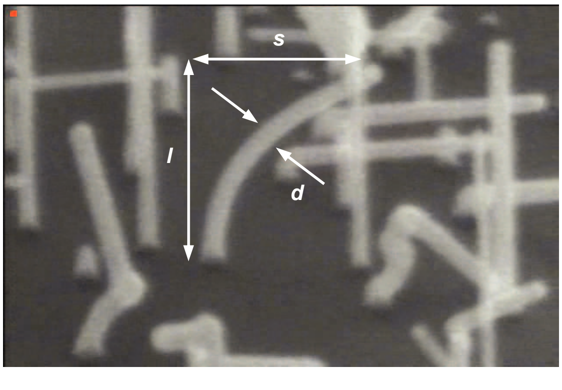

Fig. 8. Bending of a nanowire with an AFM tip.

3.3 Tensile strength measurements for FEB deposits

When the mechanical properties of nanotubes and nanowires are investigated, mechanically stable attachments are needed. This can be achieved using contamination writing in an SEM: the focused electron beam decomposes hydrocarbons present as contaminants on the surface of the substrate and forms joint deposit. The hydrocarbon molecules are supplied from the background pressure inside the SEM and by surface diffusion. Introducing locally organic precursors into the SEM chamber allows to control the deposit chemistry and to study its mechanical properties. By tailoring the metal/carbon content of the FEB deposits they can become the ideal bond material providing simultaneously electrical conductance and mechanical stability.

With the experimental setup described in section 2.2 tensile strength measurements for FEB deposits from tungsten and cobalt carbonyls have been performed. The substrate and an AFM cantilever were each mounted on a positioning stage of the nanomanipulator and put into the SEM together with the precursor supply. Thus, full control was achieved over mutual alignment of precursor supply, substrate and cantilever with respect to the electron beam. Freestanding rods were pre-deposited at the inclined edge of the Si-substrate by slow single line scans with the FEB. At low scan speed wall-like structures were obtained which separated into individual freestanding rods with increasing scan speed (Fig. 9a). These rods were used for tensile strength assessment as shown in Fig. 9b. The AFM tip was brought into contact using the nanomanipulator and bonded with a thin film FEB deposition (about 100 nm thick). Then a tensile force is generated by retracting the cantilever in small steps along the rod axis, being equivalent to the product of force constant k and cantilever deflection Δx. Monitoring the rupture by the SEM permits to read out the cantilever deflection directly and to repeat bonding and tensile tests. The tensile strength is calculated as $\sigma = k \cdot \Delta x / A$, A being the rupture surface. Cantilevers with nominal force constants of 45 N/m (Nanoworld, Switzerland) and 30 N/m (Nascatec, Germany) were used. With the rupture areas observed ex-situ using an FE-SEM, tensile strengths of (2±0.5) GPa for $W(CO)_6$ and (1±0.5) GPa for $Co_2(CO)_8$ were obtained. The error in the measurement mainly evolves from the determination of the rupture areas.

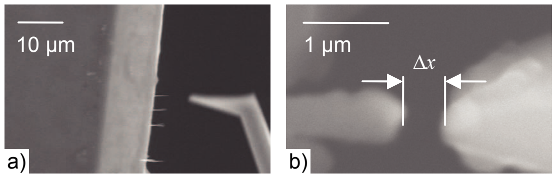

Fig. 9. Tensile strength experiment inside SEM:
a) AFM cantilever approaching a FEB pre-deposited freestanding rod; b) After bond rupture the FEB rod snaps back to its initial position, and the cantilever resumes its unstrained position.

4. CONCLUSION

Nano-objects, such as nanotubes and nanowires, are applied in key application areas like nanoelectronics and nanotechnology. A promising approach to built nano-devices from the bottom with these nano-building blocks is the use of nanomanipulation systems which operate inside an SEM. This approach has been implemented by the development of two different setups for the manipulation and characterisation of nano-scale objects: an AFM with imaging and manipulation mode and a six-axes Cartesian nanomanipulator. The AFM can be used as a manipulator to bend and move nanowires and perform mechanical testing experiments. From the acquired SEM images, mechanical properties of the nanowires can be extracted. With the Cartesian nanomanipulator setup, the teleoperated and semi-automatic attachment of carbon nanotubes on AFM tips inside an SEM has been demonstrated. This setup has additionally been used for tensile strength measurements on FEB deposits using a cantilever-based force sensor. In a next step, the automation of manipulation and assembly tasks will be advanced by implementing improved image processing algorithms for the detection and tracking of nanotools and nano-objects within the SEM images.

ACKNOWLEDGEMENTS

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