Towards automation in AFM based nanomanipulation and electron beam induced deposition for microstructuring

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Abstract

To move towards complex assemblies at the micro- and nanoscale, manipulation processes have to be automated to increase throughput and accuracy. First, this paper addresses manipulation at the nanoscale by an AFM and second, automated electron beam induced deposition as a method for structuring at the microscale is presented. Nowadays, AFM based nanomanipulation still requires frequent user interaction and remains a very labor intensive task. Spatial uncertainties are identified as a major problem that prevents reliable automation of AFM based manipulation. Results of a novel particle filter based method for measuring thermal drift in an AFM system is presented and future applications for probabilistic methods are discussed.

The automation of electron beam induced deposition (EBiD) for microstructuring purposes builds a multifunctional tool for additive structuring and also bonding inside an SEM. The presented system has the ability to create EBiD depositions from two different precursor materials by automatically executing predefined sequences. The automation includes the precursor flux control with the possibility to alternate between two materials, the deposition of points and lines at defined positions, as well as the ability to find and track already deposited structures with the use of digital image processing. This assures precise positioning of depositions relative to others even in cases of thermal or electrostatic drifting of the specimen substrate or the electron beam.

Keywords: atomic force microscope, electron beam induced deposition, automation, image processing

1. Automation of AFM based nanomanipulation

1.1. Introduction

Although the atomic force microscope (AFM) is primarily used for imaging, utilizing it as a nanomanipulation device has become of increasing interest in the last decade. Especially due to its high resolution and its flexibility against different types of samples and ambient conditions, it is applicable for a variety of nanomanipulation tasks. Possible applications range from prototyping nanoscale devices [1] to the characterisation and handling of biological samples (i.e. dissection of DNA [2]). However, due to several problems, AFM based nanomanipulation still requires frequent user interaction and remains a labor intensive task. Therefore, automation has been identified as an important research goal to increase throughput of AFM based nanomanipulation.

1.2. Difficulties in AFM based nanomanipulation

Manipulations by an AFM always have to be performed in a "blind" way, because the AFM tip can only be utilized either as the nanomanipulator or the imaging device at the same time. Positioning inaccuracies and the physical characteristics in the nanoworld yield high error rates in the manipulation results. Hence, due to the lack of a real-time capable visual sensor, the result of the manipulation process has to be verified afterwards by scanning the relevant area. Manipulation and image acquisition are therefore mostly performed alternately, resulting in very low throughput.

In AFM based nanomanipulation, and especially for its automation, spatial uncertainties constitute one major cause for erroneous results. These uncertainties are partially caused by hysteresis, creep and other nonlinearities of the piezo scanning stage. The most common way is to compensate these positioning inaccuracies by measuring the actual displacement of the scanner using position sensors and using these data as feedback signal for operating the scanning stage in a closed-loop. However, position sensors are afflicted with noise and for small scan areas this often leads to oscillations in the closed-loop control. Modern approaches exist that are using feedforward strategies by modelling the scanning stage characteristics [3][4].

More critical and less straightforward to counteract are spatial uncertainties that are induced by thermal drift. Even small changes in temperature cause all AFM's components to vary slightly in size (due to thermal expansion and contraction) which result in an unknown displacement between AFM probe and sample. By operating the AFM under homogeneous environmental conditions, the effect of thermal drift can be reduced, but even in highly temperature stable conditions thermal drift is still observable and amounts from 0.01 to 0.2 nm/s [5]. Even though this motion is very slow, it can obviously be detrimental to the success of nanomanipulation in the long run.

Especially when dealing with objects in the order of magnitude of a few nanometres (like shown in Fig. 1), this effect becomes a crucial issue for the success of manipulation (i.e. pushing a certain object). Unfortunately, the spatial displacement between AFM tip and sample cannot be observed directly due to the lack of a real-time-capable and high-resolution visual sensor. Hence, the only means to measure this displacement is the AFM tip itself.

A very common approach is based on cross-correlating successively recorded AFM images to measure the drift inside the AFM system. However, this technique is too slow to be applicable for nanomanipulation, since the
AFM tip (which is also needed for the manipulation itself) is occupied for a couple of minutes to acquire images, and drift velocity and direction may change even in a small time frame. More sophisticated approaches try to track certain features on the sample (i.e. the center of a nanoparticle [6]). Even though these techniques are able to measure drift reliably with update rates in the order of seconds, knowledge about the sample (i.e. the shape of a particle) is a necessity.

### 1.3. Sample independent drift estimation

To allow for robust drift compensation even with unknown and sparsely structured surfaces, a novel algorithm to measure drift was developed. Instead of scanning an area or tracking certain features on the sample to gain information about the true lateral position of the AFM probe, the developed algorithm periodically records short height profiles that can be recorded in a small time-frame (~0.1s). These scanned lines are considered as sensor data containing some information about the tip position in relation to the sample. Although it seems obvious that the tip position cannot be extracted directly from a single height profile, the developed algorithm use these data to update its belief about the current system state, namely drift. This is performed by comparison of the height profiles recorded at arbitrary positions with line scans that are extrapolated from a previously recorded topography image.

Due to a low signal-to-noise ratio and sporadic faulty measurements the recorded line scans only represent the true height profile of the sample roughly. Additionally, depending on the type of sample used, these line scans may also contain only little information. To account for these perceptive uncertainties, a particle filter based algorithm was developed for drift estimation.

Since several years, particle filter based localization has successfully been applied in the domain of mobile, autonomous, macroscale robotics [7]. It is part of the superordinate concept known as probabilistic robotics. The key idea behind probabilistic robotics is to explicitly deal with the uncertainties that exist in a robotic system using the calculus of probability theory. It has been shown that probabilistic algorithms are very robust against noisy or faulty sensor data and that they perform well even when the system's behavior can only be poorly modeled [8]. According to algorithms used in mobile robotics, the task of estimating drift can be reduced to a limited global localization problem: In terms of mobile robotics, the sample surface can be considered as an environment in which a moving robot, in the discussed case the AFM tip, has to be localized. To validate the algorithm, drift measurements were conducted using several types of samples. However, since the algorithm was mainly developed to work even with almost unstructured sample surfaces, most interesting results were obtained with gold coated silicon substrate, which roughness parameters were determined to be $R_{\text{rms}} = 0.765$ nm and $R_s = 0.592$ nm. A topography image of the Au/Si substrate is shown in Fig. 2.

The experiments (see Fig. 3) have shown that even with the highly unstructured substrate used as a sample, drift could be reliably measured over a period of several hours. To prove correctness of the obtained results, a topography image taken before the measurement was cross correlated with an image taken directly after the measurements. The results obtained from this cross correlation have shown to be identical to the drift induced displacement measured by the newly developed algorithm.

Compared to existing state-of-the-art algorithms for drift measurements, the developed algorithm is both fast (with update rates of 0.1Hz) and independent of the type of sample.

### 1.4. Probabilistic approaches for automation of AFM based nanomanipulation

The results discussed above have shown exemplarily the strength of probabilistic approaches and their applicability for nanoscale applications. Especially for
In today's technical applications the EBiD process is generally used for mask repair [9], for production of AFM supertips [10] or as a bonding technique in the nano- and micrometer scale [11]. The EBiD process is also comparable with focused ion beam (FIB) deposition, where accelerated ions cause the release of secondary electrons from the substrate material, again inducing the chemical decomposition. The setup developed in this work uses two self-made precursor evaporation systems inside the SEM chamber with advanced control software. This makes it possible to create depositions from two different precursor materials without any setup changes. The description of work contains the description of the setup with its precursor evaporation systems and its automation software. Additionally some applications for this setup are explained and evaluated.

2.1. Introduction

Electron beam induced deposition (EBiD) is a technique that allows creating three dimensional structures with sizes down to a few nanometers by directly “writing” them with an electron beam onto a substrate material, inside the vacuum chamber of a scanning electron microscope (SEM). The secondary electrons released by the electron beam of the SEM when hitting a substrate cause a chemical decomposition and partial deposition of a precursor gas, which is evaporated into the vacuum chamber.

2. Automated nanostructuring with EBiD

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Depositions for the experiments in this paper were made at a controlled vacuum chamber pressure of $2 \times 10^{-5}$ mbar which corresponds to a molecular flow of about $1.8 \times 10^{21}$ molecules per m² and second at a mean distance of the electron beam to the capillary of 400 µm (Fig. 4). The electron beam was accelerated with a voltage of 20 kV and a beam current of 90 nA.

2.4. Software architecture

The software control architecture is based on the Distributed Control Architecture for Automated Nanohandling (DCAAN) presented in [12, 13]. The flexible architecture is based on several server programs written in C++ connected via Ethernet using the platform independent Common Object Request Broker Architecture (CORBA) as communication framework, which is an object oriented middleware defined by the Object Management Group (OMG). The programs can be distributed onto several PCs, however in our setup all servers were running on the same machine. For the task of automating the EBID process the following software servers were involved (Fig. 5):

- Several sensor programs (e.g. pressure sensor program and vision) are pushing data to the SensorServer at independent update rates. Each sensor data carries the timestamp of the reading. All servers do have a synchronized clock. This way a server can decide whether or not a datum is outdated. The SensorServer collects the most current sensor data and provides them on request to other programs (e.g. PressureControl and HiLeC). The collection of all SensorServer data makes up the current world state of the system.
- HILeC coordinates the different parts for different automation tasks. Set values are sent to actuator specific low-level controllers (e.g. set pressure) and timing between different system parts gets controlled. HILeC is capable of tele-, semi and full automation depending on the task requirements. For semi- or full automation all servers connected to HILeC have a common interface defined in CORBA’s interface definition language (IDL). At connection time HILeC queries the command set of each server. This command set can be directly used at command line or to write automation sequences.

2.4.1 Vision

The vision software [14] tracks objects in visual sensor data (e.g. the SEM life images) and publishes the objects’ positions to the sensor server. In many applications this is a key ability since most actuators do not have integrated position sensors or the accuracy of the integrated ones is not high enough for nanopart manipulation. Even though this key ability is used in our application too, Vision does have a second function. For fast position updates it is necessary to set a region of interest (RoI), such that only this section is scanned by the electron beam. This ability of controlling the scan area is exploited in our setup such that Vision acts as a sensor and as an actuator. For point depositions a RoI of width and height one is used and for horizontal or vertical lines a rectangular RoI of width one ore height one is used. This way all 1024x768 points of the displayed scan field can be addressed.

2.4.2 Pressure control software

Deposition rate of Electron Beam induced Deposition is mostly dependent on electron beam parameters and the precursor density and thus the precursor flux through the ES’s capillaries [15]. To assure a constant precursor flux while making an electron beam induced deposition it is necessary to control the vaporization of each precursor continuously. In the presented desktop station this is provided by control software where two PID-controllers are implemented, one for each ES. For each controller the control variable is selectable between gas pressure inside the vacuum chamber, which is the most suitable variable to assure a constant precursor flux and temperature of the ES precursor reservoir, which is necessary when both precursors should be vaporized at the same time.

2.5. Automation

The major automation objective for this setup is to be able to grow pins and vertical and horizontal lines in a defined and reproducible manner. In addition it should be possible to change the precursor and start a deposition again on top of an existing pin. This allows i.e. deposition of combined pin structures of two different materials in one production step. Before starting the automation sequence a few preparation steps have to be performed. First a default pin model has to be created in Vision for tracking of the pins. Brightness, contrast, focus and magnification of the SEM have to be adjusted properly. The magnification level in our experiments was fixed to a level of 2000 which corresponds to a displayed image width of 25 µm. With an image resolution of 1024x768 this corresponds to an addressable resolution of about 24.4 nm a pixel. So the process resolution depends on...
image resolution and the magnification level. Fig. 6 illustrates the single automation steps used for our experiments. The sequence starts with heating up ES 1 (filled with W(CO)₆ precursor) for this HiLeC sends a start signal to PressureControl with the predefined set pressure of 2·10⁻⁵ mbar. At the time the goal pressure is stable PressureControl sends a signal to HiLeC. Up to that time the electron beam is blanked to avoid unnecessary contaminations of the Si substrate. At that time HiLeC signals Vision to set the RoI to the upper left deposition point for 7.5 min before switching to the next point. The points 2-4 have been done accordingly. Afterwards the two lines are grown in step 5 and 6 by setting a rectangular RoI with height one for 10 min and the same with width one for the vertical line. Now HiLeC stops the closed-loop control of PressureControl for ES 1 and blanks the beam again. HiLeC waits for 3 min to let the peltier element cool down and reestablish the standard high vacuum pressure of the chamber. After that time HiLeC signals PressureControl to start closed-loop control of ES 2 with the same set pressure as before ES 1.

In steps 7 and 8 two pins get grown on top of the pins 1 and 3. Before starting the deposition step drift effects have to be compensated. This is done by setting a RoI at the expected position of the pin that should be modifed. The RoI needs to be large enough to enclose the real position of the point and small enough to ensure that only one pin is in the RoI. With the predefined default pin model the exact position of the pin gets tracked and the deposition with ES 2 starts. The pin deposition is performed as the pins 1-4 before just having the drift compensation step before. The whole sequence has been written as part of the HiLeC program using the commands provided by the connected servers. The execution time of the experiments is about 75 min. Deposition time is about 65 min. Most of the residual time is used for heating and cooling.

2.6. Experimental results

The results of the automated deposition sequence were analyzed by an SEM image in a side view by tilting the specimen substrate to an angle of 70°. Fig. 7 shows The SEM image of one complete sequence. Because of the tilt direction of the SEM stage the pattern is turned 90° clockwise so that the first deposited pin is in the upper right corner. As one can see, the second deposition on top of the first and third pin with the Co₂(CO)₈ precursor are exactly matching with the first deposited part so that there is no displacement in x or y direction. Experiments with different timings during the automated sequence indicated the importance of a constant temperature of the activated evaporation system before starting the deposition. Alternating temperature during a single pin deposition causes drift effects of the substrate material which leads to a displacement of the deposition and thus to sloping pins. Also the absolute positioning accuracy suffers from these drifts during the process. To avoid these problems it is necessary to wait after activating a precursor evaporating system until the temperature is on a constant level before starting the deposition sequence.

2.7. Applications

The aimed application of the presented desktop station in general is the automated electron beam induced deposition of micro and nano scale pin and line structures from different precursors in predefined geometries. Several applications are possible to be automated with this setup. Due to the visual feedback by the SEM image and the implemented object tracking algorithms, one spot on the specimen surface can be approached several times even in cases of thermal or electrostatic drift effects in the SEM image.

In the field of nanorobotics this setup is applicable to build free-standing mechanical structures where different precursors cause various mechanical or electrical properties of the structure. To achieve pin-like structures from different precursor materials it is essential to control the deposition spot because even the changing precursor flow while “switching” the precursor can cause thermal drift of the specimens of about ten times of the diameter of a deposition. The resulting depositions can be cantilevers where a small section is made of a material which is more elastic than the rest of the structure whereby it acts as a solid hinge. As an alternative the different thermal expansion coefficient of depositions from two different precursors can be used for actuation applications where the deposited structures are heated by an electric current.

Another aimed application for the automated desktop station is the EBId based bonding of micro and nanostructures, i.e. carbon nanotubes or nanowires onto surfaces or STM tips. To do this, the automated sequence will start after positioning the objects that have to be bonded in contact to each other in the SEM image. The sequence itself contains the heating of the evaporation system, automatically finding the couple point of joined objects and than the deposition of a short line across this couple point.

2.8. Conclusions and outlook

We proved the functionality of the automated desktop station with a deposition sequence using the precursors W(CO)₆ and Co₂(CO)₈. The experimental results have demonstrated, that a precise positioning of depositions relative to each other is possible without manual interaction. Temperature changes due to alternating precursor supply caused by thermal drifts of the specimen substrate have been compensated. 

With the developed desktop station it is possible to
deposit point structures and horizontal and vertical lines by electron beam induced deposition. When doing this conventionally, it is essential to set up every line and point manually and to examine the correct time for each deposition process. Additionally the change of the precursor is very time consuming. The main advantage of our automated system is the possibility of predefining complex deposition sequences that can be executed several times without manual interaction.

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