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50. Internationales Wissenschaftliches Kolloquium
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An automated System for Electron Beam induced Deposition

ABSTRACT

Within this article we will show a new set-up for automated production of micro-structures within a Scanning Electron Microscope (SEM) based on Electron Beam induced Deposition (EBiD). We will present experimental results which are the main parameters for later development of an microrobotic system for gluing inside a SEM and present a typical application for EBiD.

Introduction

EBiD is a processing technology which has been explored within the last ten years in order to develop new methods for 3D-nano-structures [1,2]. Within the vacuum chamber of a SEM, a substrate is scanned by the electron beam of the SEM. However, as every vacuum system always has some substances left in the chamber, these substances can react with the probe's surface due to the energy induced by the electron beam. Commonly, the effect of contamination deposition on the probe’s surface can be observed whenever a probe is viewed for a longer time in the SEM. These contaminations derive from substances bound to surfaces inside the vacuum chamber, which evaporate at lowered pressure. However, the effect that small amounts of gases, which are fed into the vacuum chamber of the SEM and lead to deposition on the substrate by means of the electron beam can be exploited for technical applications.

Set-up

The EBiD sytem used for this set-up consists of the a mobile platform, the gas evaporation system, the control software for the pressure control inside the SEM's vacuum chamber and software tool for the positioning of the SEM's electron beam. The single elements will be described in the following paragraphs.

Scanning Electron Microscope For our experiments we used a Zeiss DSM 950 Digital Scanning Electron Microscope. This electron microscope provides the possibility to measure its pressure in the vacuum chamber via external connectors. This pressure connector has been used for setting up a control cycle between pressure and temperature of the evaporation system.

Additionally, we used point electronic's DISS5 hardware [3] for controlling the SEM from an

external PC, which enabled us to control the electron beam for deposition. The hardware is also used for the grabbing pictures and videos from the SEM.

Evaporation System The evaporation system is the main part of the mechanical set-up, as shown in fig. 1. It consists of a thermal mass, which acts as a reservoir for thermal energy. On top of the thermal mass a Peltier element is mounted, which is used to shift the thermal energy between the thermal mass and the precursor reservoir. This set-up has the advantage, that the precursor reservoir can be heated up as well as cooled down, only by changing the polarity of the Peltier element.

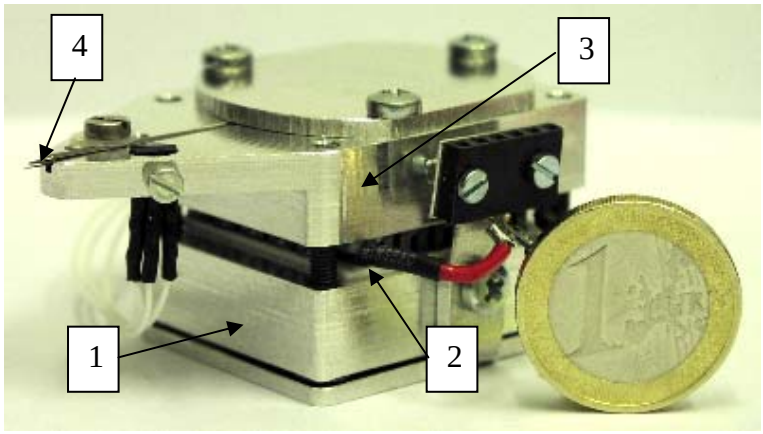

Fig. 1: Set-up of the evaporation system. (1) heat reservoir, (2) Peltier element, (3) precursor reservoir, (4) glass capillary

The precursor reservoir is filled up with the precursor, e.g. Tungsten Hexacarbonyle ($W(CO)_6$). By heating the reservoir up to the evaporation temperature of the precursor the evaporation starts. This leads to difference in pressure between the precursor reservoir and the SEM's vacuum chamber. The precursor vapour flows along the pressure gradient through a small glass capillary with a diameter at its end of approximately 200 µm into the vacuum chamber. In order to prevent condensation of the precursor in the glass capillary, the precursor reservoir has been designed to guarantee a nearly homogenous heat distribution around the capillary. Additionally, in order to prevent electrical charging-up effects of the capillary, it has been sputter-coated with a palladium-gold-layer.

The temperature of the heat reservoir and the precursor reservoir is monitored by three temperature sensors. This is useful for controlling the temperature difference between precursor- and heat-reservoir, for preventing the metallo-organic compound to be disassociated already in the precursor reservoir by overheating and to prevent condensation of the precursor in the capillary.

Mobile Platform The mobile platform carries the evaporation system on its back. This is necessary in order to position the evaporation system close to the substrate where the depositions are to be made. In our experiments we found out, that a defined distance between the substrate and the

capillary's precursor outlet is necessary to obtain good results in deposition. The mobile platform operates on the stick-slip-principle and has in stepping mode a smallest step-size of approximately 100 nm, as described in [4]. These values are good enough for adjusting the evaporation system's position to the substrate.

Electron Beam Control In order to control the deposition, the SEM's electron beam has to be positioned on a spot for a certain time. This feature is provided by point electronic's DISS5 software. By generating a control script-file through MATLAB, which is then read by the DISS5 software, the depositions are easy controllable. These scripts provide the position of the electron beam in x- and y-coordinates and the deposition time. Furthermore, it is possible to deposit lines using the same scripts and the DISS5 software. This is useful for the deposition of aerial elements, which are composed out of single lines.

Pressure Control System The pressure control system consists of a program, which reads the values of the temperature sensors on the evaporation system, the pressure inside the SEM's vacuum chamber and computes via a PID-controller the necessary current respectively voltage to be put out on the Peltier element. Thus, a pressure control and a temperature control loop have been set-up. The system proved to be stable and rugged even for pressure changes over many orders of magnitudes in pressure. However, in order to prevent the precursor from being disassociated by heating the precursor reservoir up to fast, an upper limit for the Peltier element's power has been set.

Experimental Results

All depositions were made under similar conditions at room temperature. In each experiment only one parameter is varied. The other parameters were set to the following default values except the parameter itself was varied. The SEM was set to an acceleration voltage of 20 kV and medium resolution at a working distance of 7 mm. The deposition time was 15 min and the chamber pressure was kept to $1x10^{-5}$ mbar controlled by the described software. The depositions were made on silicon wafers sputtered-coated with a palladium-gold-layer.

Dependence on deposition time First of all, the time dependence of growth will be examined due to the fact that the growth rate plays an important role in every imaginable application. The deposition time was varied between 5 to 20 min in 5 min steps. For each deposition time six tips were grown. The plot tip height versus deposition time (cp. fig. 2) shows a linear growth over the time range from 5 to 20 min. Thus, the linear growth rate is given by 2.5 nm/s. It is also apparent, that the best linear fit will not cross zero point. Hence, a non linear growth at deposition's beginning can be assumed.

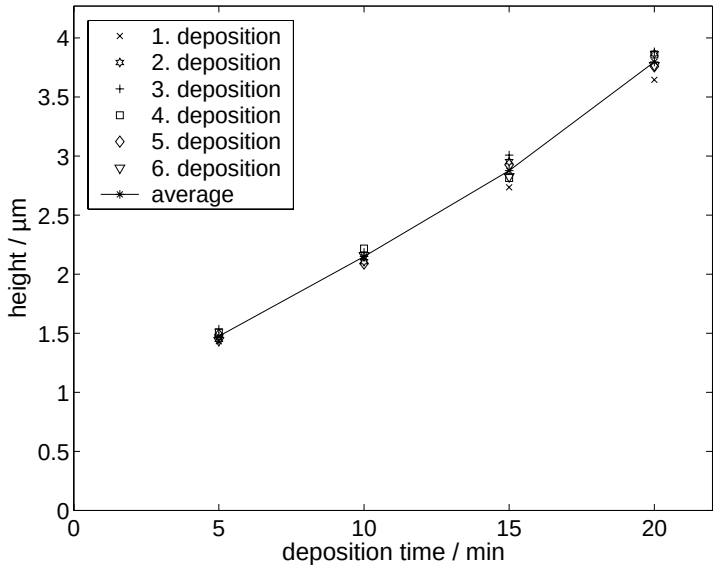

Fig. 2: tip-height in µm vs. deposition time in minutes

Dependence on resolution The resolution is an important SEM parameter. At higher resolution, the probe current is decreased as well as the probe diameter. This can be seen from formula (1), where I_{Probe} is the probe current, J_{Probe} is the probe current density, which is assumed as constant and A_{Probe} is the surface of the electron beam on the substrate.

$$I_{Probe} = J_{Probe} \; x \; A_{Probe} \tag{1}$$

The dependence between resolution setting of the electron microscope and probe current was taken from the SEM's user manual. As a matter of fact the electron beam can be focused much better at lower probe currents. Thus, resolution has a strong influence on the lateral dimension.

During the experiments, the resolution was varied from low to high in six steps. At very high resolution no reasonable depositions could be obtained due to the very small probe current. Three depositions were made for each resolution. Fig. 3 depicts SEM overview image of the depositions. From fig. 3 it is apparent, that the tip's height fluctuates over increasing resolution. The underlying processes leading to non-monotonic vertical growth have not been completely understood yet.

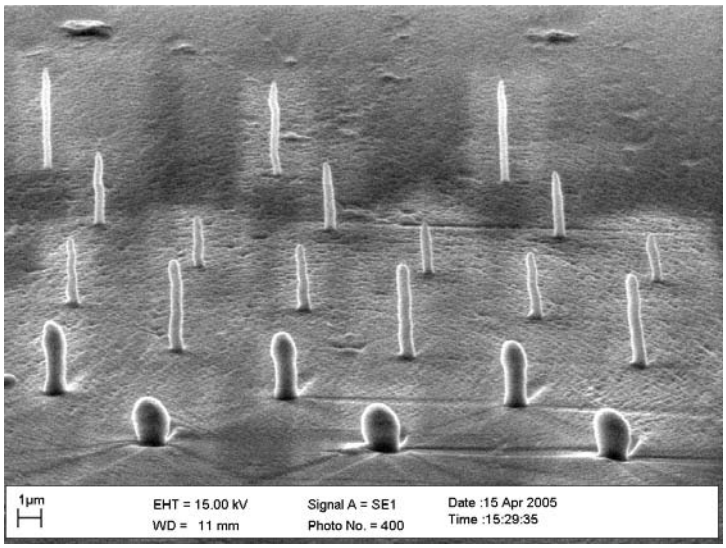

Fig.3: SEM-image providing an overview for the deposition dependence on resolution. The substrate was tilted by 75°. The SEM's resolution parameter increases in each row from front to back.

However, fig. 4 reveals clearly the effect of increasing resolution to lateral dimension. The probe current dependence of tip width is demonstrated in this figure. The width used here is defined as full width at half maximum (FWHM). With decreasing probe current the width first descends quite strong in order to reach smallest lateral dimension at approximately 400 nm below 2.12 nA probe current. Hence, further probe current reduction leads to no advantage of smaller lateral dimension under used conditions.

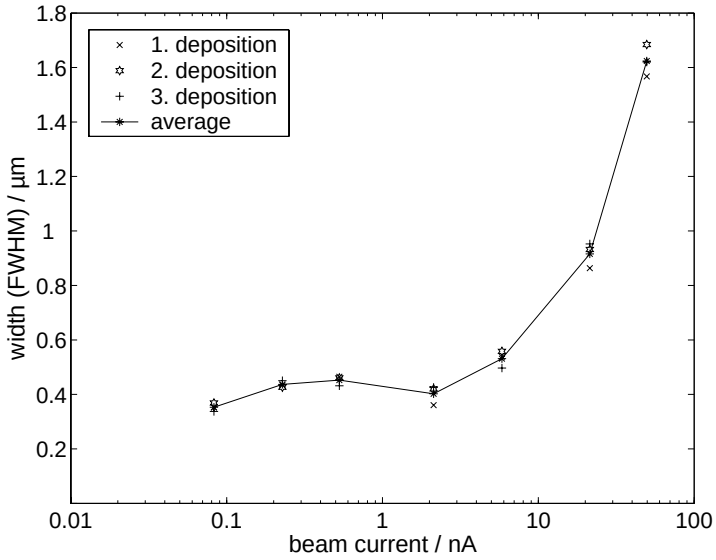

Fig. 4: Tip's width (FWHM) as a function of beam current.

Dependence on pressure The gas pressure in the SEM chamber is a limiting parameter to EBiD reactions. For getting an idea of gas pressure impact on EBiD reactions, three depositions were made for each pressure in the range of $1x10^{-6}$ to $1x10^{-5}$ mbar in four steps.

The vertical respectively lateral deposition rate is defined as height respectively width divided by deposition time. In fig. 5 the vertical deposition rate can be seen. At very low gas pressure the vertical deposition rate rises rapidly with increasing pressure. At higher pressures, the rise in vertical deposition rate with increasing pressure slows down. As described in [2], EBiD can be understood as localized chemical vapour deposition (CVD). Therefore it is obvious, that EBiD process is limited by mass transport at low pressures. Further pressure increase will probably lead to saturation of the vertical deposition rate. Additionally, the pressure is limited upwards due to vacuum system's safety switch off of the SEM.

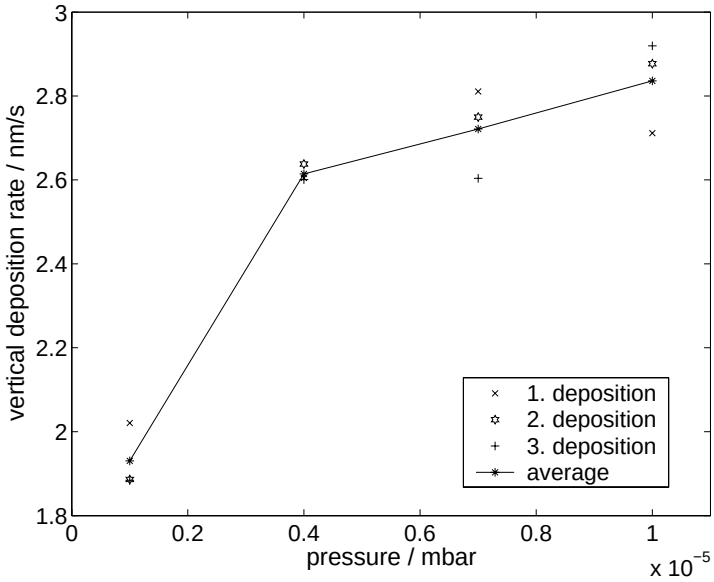

Fig. 5: Vertical deposition rate (height / deposition time) as a function of SEM chamber pressure.

Fig. 6 showing the lateral deposition rate offers an interesting fact. With increasing pressure, the lateral deposition rate decreases. As the deposition time was 15 min for each deposition, this means that the width decreases, although more precursor molecules can participate in EBID reactions.

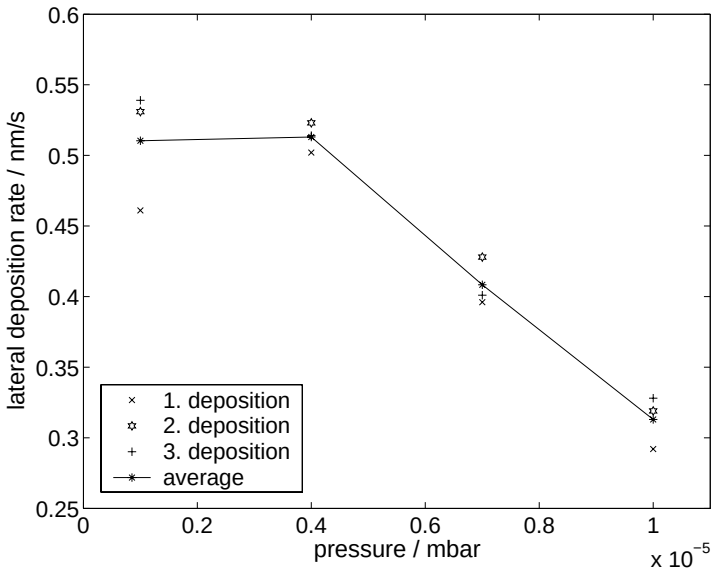

Fig. 6: Lateral deposition rate (width / deposition time) as a function of SEM chamber pressure.

This may be explained by the rising vertical deposition rate. As described in [1], primary electrons (PE) scattering inside the tip generate secondary electrons (SE), which leave the tip sideways. These SE dissociate adsorbed precursor molecules at side flanks leading to lateral broadening. As the vertical deposition rate rises with increasing pressure, a smaller amount of PE enters the tip being able to scatter sideways. Thus, with increasing vertical growth the width becomes reduced.

Example Application

In the following paragraph we will show an example applications where the technique of Electron Beam induced Deposition is useful for research and development applications.

Micro-Gluing inside a SEM With the help of Electron Beam induced Deposition, it is possible to provide a method for gluing micro- respectively nano-parts together. This can be achieved by putting two parts closely together, ideally in that way, that mechanical contact is given. Afterwards, a line of precursor material is deposited on top of the two parts. An example is given in fig. 7, where a STM-tip made of tungsten is glued to a TEM-lamella. The lamella can afterwards be lifted out of its box for examination in a Transmission Electron Microscope (TEM).

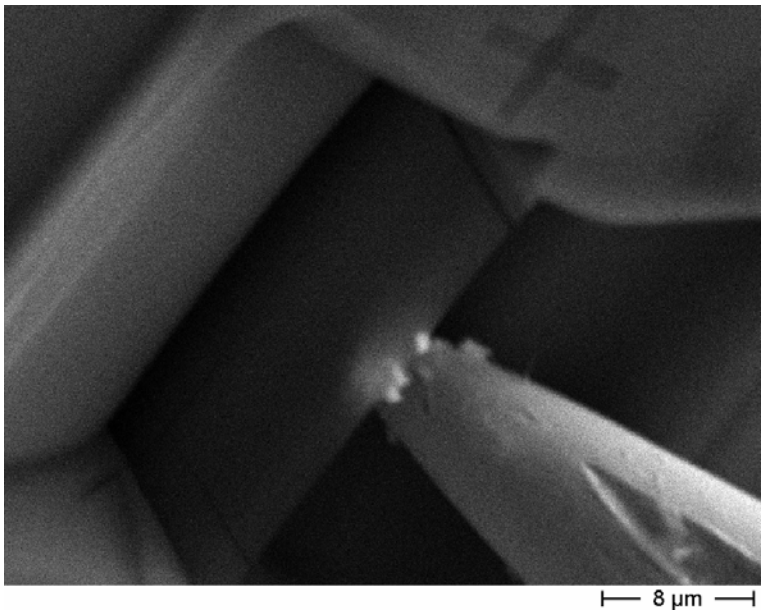

Fig. 7: STM-tip glued to a TEM-lamella by EBiD. The EBiD depositions are visible as light lines.

In [5] a system was proposed for the semi-automatic handling of TEM-lamellae inside a SEM. Therein, we used mechanical grippers for taking out the lamellae out of the boxes. However, this mechanical principle has its drawbacks. One of it is the fact that it is quite easy to grip the lamella, but hard to get the lamella off the gripper due to the parasitic forces like adhesion and electrostatic forces. With the help of the proposed EBiD system, it is possible to glue the TEM-lamella after taking it out of its box with a mechanical gripper to the TEM-grid where it should be deposited in order to overcome the parasitic forces between gripper and lamella.

This system provides also the possibility to glue other micro- and nano-objects to other parts. Another example is the handling of nano-wires and tubes.

Discussion

In section “Experimental Results” we showed that the deposition height of the tips is time dependent and has linear growth factor of 2.5 nm/s. However, the results show that the beginning of the deposition from approximately 0 to 5 min is non-linear. This is caused by layer growth processes.

By varying the resolution of the SEM, we changed the beam current. Increased beam current lead to an exponential increase in lateral width. This is due to the increased spot size and thus to an extend in the area where secondary electrons are generated, which cause the chemical reaction.

Additionally, we varied the pressure inside the SEM's vacuum chamber and found out, that with

increasing pressure the vertical growth rate increases in contrary to the lateral growth rate which decreases.

For the deposition of fine and well defined 3D-nano-structures it is important to deposit at high pressure, this leads to an increased vertical growth rate while decreasing the lateral growth rate. Furthermore, it is necessary to decrease the beam current, which decreases the width of the deposition as well.

If the application aims at big depositions, e.g. for gluing applications or for plane depositions, it is advisable to decrease the pressure and to increase the beam current.

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