

Enabling sub-nm 3D metrology inside an Electron Beam Lithography (EBL) tool by integration of an in situ Atomic Force Microscope (AFM)

Characterization of EBL exposed PMMA resist using a compact sample holder-mounted AFM

H. Koop, M. Zech, C. DalSavio, F. Otto and K. Karrai, attocube systems AG, Germany
 D. Schnurbusch, M. Müller, T. Gründl, M.-C. Amann, and A. W. Holleitner,
 Walter Schottky Institut and Physik-Department, Technische Universität München, Germany
 F. Nouvertné, Raith GmbH, Germany



Abstract

This application note describes a new setup that adds 3D metrology to an eLiNE Plus electron beam lithography and nanofabrication system. Based on an atomic force microscope (AFM) integration and subsequent in situ characterization of PMMA resist exposed by standard electron beam lithography, the paper demonstrates sub-nm surface topography resolution in this setup. The interesting observation of sub-nm resist shrinking without any ex situ process yields high potential for better understanding of focused electron beam induced energy deposition process characteristics. Moreover, an in situ AFM ideally complements the range of applications within an EBL and nanoengineering tool regarding the determination and control of material growth/etching rates or surface topography analysis - not only for the sake of inspection and metrology but also for nanolithography on non-planar surfaces.

Introduction

Conventional 2D nanofabrication is still typically using electron beam lithography (EBL) as an initial step. Identified as a clear trend however, 2D nanofabrication is advancing into 3D rapid prototyping processes along a complex technology chain. That is why Raith has pioneered the development and commercial offering of nanoengineering tools inside an EBL system for in situ 3D material deposition/etching or nanomanipulation, thereby exceeding the capabilities of traditional EBL tools.

In addition to the resist exposure characterization described above, an in-situ AFM is an ideal complement to the eLiNE Plus's 2D SEM functionality as it

introduces the third dimension to any measurement. This enables, for example, 3D surface topography analysis for nanolithography on non-planar surfaces. The AFM can go beyond the inspection capabilities of the eLiNE Plus operated in SEM-mode if ultimate performance for high resolution imaging or for precise metrology is required. Moreover, immediate determination of growth or etching rates when using a gas injection system for focused electron beam induced processing such as material deposition or etching becomes possible.

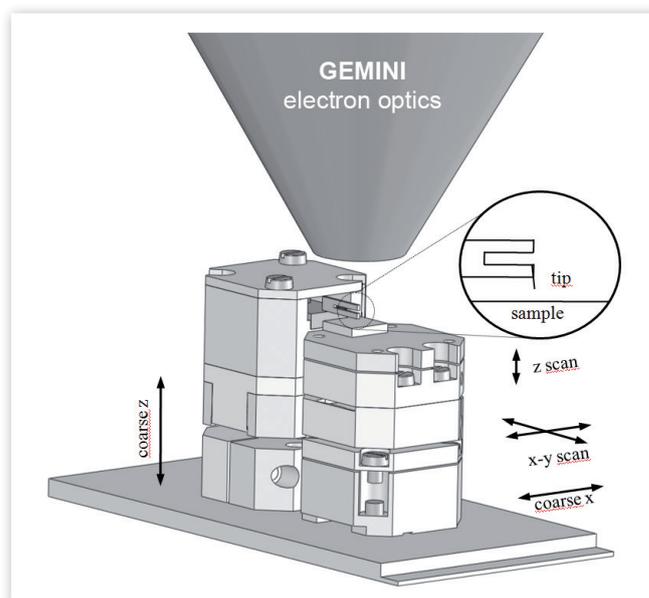


Figure 1a: Schematic drawing of the tuning fork-based atto AFM III as used inside a Raith eLiNE Plus EBL system. The system allows the in situ characterization of the quality of exposed electron beam sensitive resist without the need of a development process.

For driving EBL to its ultimate limits, a better understanding and control of EBL processes along with resist systems is necessary in order to further improve on minimum EBL feature size or line edge roughness. In this context, the atomic force microscope (AFM) could potentially help to better control the processes taking place during EBL on resist systems such as PMMA at the nanometer scale - preferably in situ.

Indeed, it has been observed that, upon electron beam irradiation, these resists exhibit several changes in their physical properties, one of which is a slight decrease in thickness of the irradiated resist regions relative to the unexposed ones. This shrinkage can be visualized and characterized as a topographic image by an AFM [1]-[4]. So far, this has only been done ex-situ after exposing PMMA to ambient conditions, but adsorption of water and subsequent swelling of the resist and other changes in environmental conditions, such as pressure and temperature, can change the chemical properties of the irradiated resists [3]-[5]. Here, we introduce an in situ AFM characterization method of these 'latent' images in electron-beam resists [6]. We demonstrate that for PMMA the beam dose can be detected with a sensitivity of $6.5 \pm 0.2 \mu\text{C}/\text{cm}^2$ in the positive tone regime.

Experimental Setup

In line with the philosophy of the eLiNE Plus as an expandable platform for nanoengineering, we describe how an AFM has been adapted onto a conventional eLiNE Plus sample holder.

The eLiNE Plus's unique laser interferometer controlled stage setup allows a large vertical travel, making it possible to integrate an AFM directly below the electron optical column. This enables studying the impact of electron beam exposure on PMMA (polymethyl methacrylate) by in situ characterization with the tuning fork-based attoAFM III from attocube systems.

Small samples are placed such that advanced EBL can be performed in the presence of the AFM, allowing a direct feedback of any sample or surface modification in the 3rd dimension. Without the need for ex situ resist development, an analysis of the AFM data could potentially supersede iterative exposure, development, and dose distribution optimization loops.

As depicted in Fig. 1a) and b), a compact AFM is mounted directly below the electron optics onto a conventional sample holder of the eLiNE Plus system. The relative position of the sample with respect to the electron beam as well as the coarse distance of the AFM tip to the sample are both independently controlled by piezopositioners. A small tilt angle of the AFM tip with respect to the electron beam allows simultaneous imaging of every location on the surface with both the AFM and the eLiNE Plus's SEM mode.



Figure 1b: AFM adaption onto a conventional eLiNE Plus sample holder

Measurements

In the experiment described, two PMMA resists (A2 and A6) are spin coated onto SiO₂/Si wafers; the thickness of the SiO₂-layer is 160 nm, and after spin

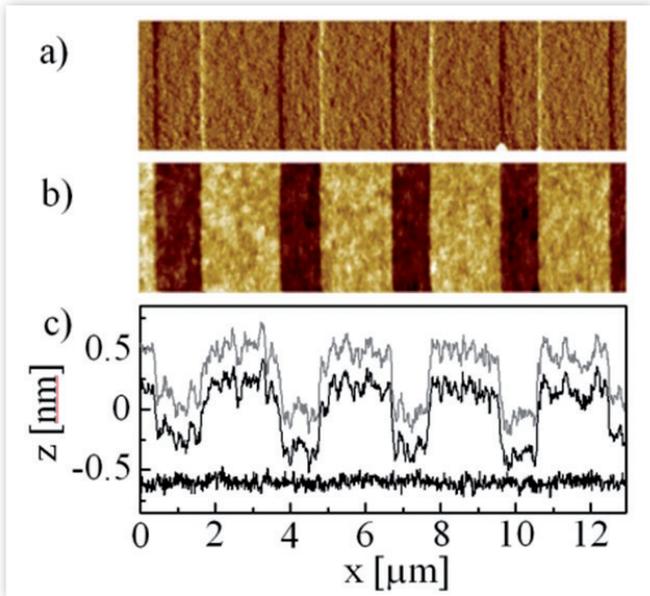


Figure 2: a) Phase and b) topography image of an undeveloped resist measured in situ by AFM. The topographic profiles of two neighboring line cuts are shown in c). The two line cuts are shifted vertically for better visualization. The bottom graph shows the subtraction of the (non-shifted) line cuts, demonstrating a vertical noise on the order of 40 pm rms on electron beam resist without the need of a development process.

coating the resists have thicknesses of 60 nm (A2) and 360 nm (A6). Lines-and-spaces pattern are subsequently exposed at different electron doses while keeping the electron beam energy constant at 20 keV. Fig. 2a) and 2b) show AFM phase and topography images of the resist after it is exposed to a 1 μm / 2 μm lines-and-spaces pattern at a dose of 190 μC/cm², indicating the shrinkage in resist thickness at exposed locations. In this specific example, exposed areas show a shrinkage of 0.45 nm compared to unexposed locations on the same resist.

In order to demonstrate the AFM’s achievable mechanical stability within the EBL tool environment, the vertical resolution/noise limit of the AFM apparatus is determined by subtracting two neighboring topographic line cuts yielding a **vertical noise figure of only 40 pm**, see Fig. 2c).

In a separate set of experiments, the dependence of resist shrinkage on exposure dose was investigated. Figs. 3 (a)-(d) depict in situ AFM images immediately after the exposure of resist A2 with increasing electron doses. Higher electron-beam doses give rise to a larger shrinkage of the exposed areas with respect to the unexposed areas: The probability $\rho(z)$ of having a pixel at a topographical height z is plotted in histograms for each corresponding image in Figs. 3 (e)-(h). The two peaks appearing in each histogram $\rho(z)$ are perfectly well fitted by two Gaussians (see [6] for details).

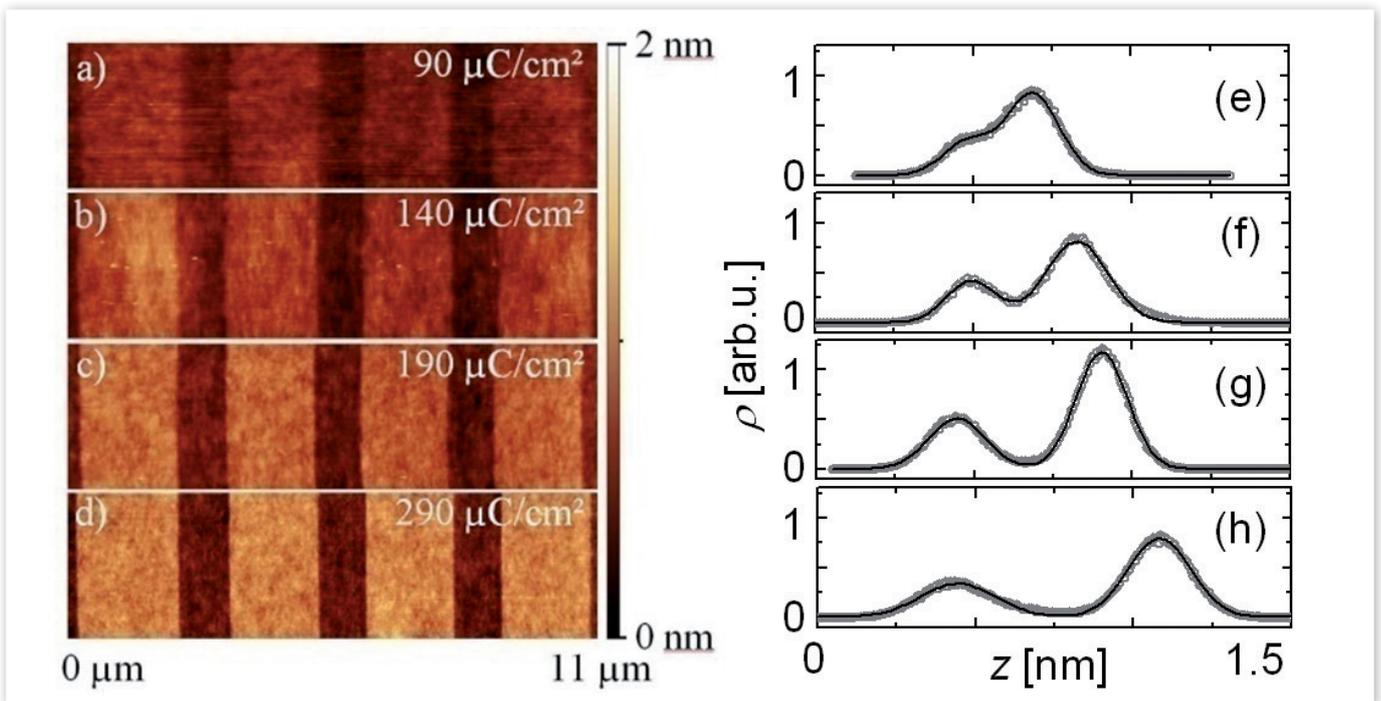


Figure 3: Topographic images of undeveloped resist, exposed to doses ranging from 90 μC/cm² up to 290 μC/cm². The dark areas are corresponding to exposed regions, where shrinkage of the PMMA has taken place.

This also allows to extract the relative shrinkage Δz between unexposed and exposed regions for all gratings with a total standard deviation on the order of 2 pm. Fig. 4 (a) shows the shrinkage Δz of the resists A6 (black dots) and A2 (grey dots) as a function of the electron beam dose D . We can approximate both dependencies with a linear function $\Delta z = \alpha D$, with slopes α of $6.11 \pm 0.18 \text{ pm} / \mu\text{Ccm}^{-2}$ (A6) and $2.43 \pm 0.05 \text{ pm}/\mu\text{Ccm}^{-2}$ (A2). The knowledge of the slope α for a specific resist allows us to judge the quality and the real value of the absorbed electron-beam dose in situ. This can be done by the following estimate:

$$D \approx z_{\text{rms}} / \alpha = 6.5 \pm 0.2 \mu\text{C}/\text{cm}^2 \text{ for A6 and}$$

$$D \approx 16.5 \pm 0.3 \mu\text{C}/\text{cm}^2 \text{ for A2.}$$

The gradient α , however, needs to be calibrated with respect to other parameters, such as the beam energy, the resist thickness, the type of substrate, and the geometry of the irradiated pattern^[7]. The latter is demonstrated for resist A6 in Fig. 4 (b).

Generally, Δz depends on the spacing of the gratings. For the data in Fig. 4 (b), the gratings in the resist A6 have a width ratio of exposed and unexposed areas of 1:1. In Fig. 4 (b), the Δz data can be fitted with a linear function extrapolating to the origin. In other words, for smaller feature sizes, the unexposed area absorbs almost the same energy per unit area as the exposed area, although this area was not exposed on purpose.

Conclusion

In this application note, we have demonstrated a novel technique^[6], which allows the in situ characterization of EBL processes. Instead of the traditional approach of imaging the post-processed resist in a scanning

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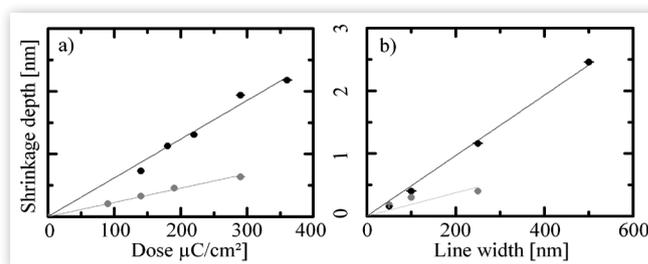


Figure 4: (a) Resist thickness shrinkage after exposure plotted as function of exposure dose D . Resists A2 (open circle) and A6 (closed circle). (b) Thickness shrinkage for grating with a line:space ratio of 1:1 as a function of the line width for resist A6 at a constant dose of $190 \mu\text{C}/\text{cm}^2$. The linear fits are empirical.

electron microscope (SEM), we successfully integrated an easy-to-handle AFM into the Raith eLINE Plus, a flexible platform which did not need any modification to accommodate the AFM. In-situ measurements of the EBL resist immediately after the exposure deliver more physically valuable information, such as polymer grain size and distribution. We observe a resist shrinkage effect for 2 different resist types, which increases linearly with the exposure dose. This enables a step-wise proximity correction or a re-exposure of under-exposed spots.

We demonstrated that for PMMA the beam dose can be detected with a sensitivity of $6.5 \pm 0.2 \mu\text{C}/\text{cm}^2$ in the positive tone regime. This sensitivity corresponds to approximately $0.5 \text{ e}^-/\text{nm}^2$, making this a tantalizing setup for investigating the possibility of directly measuring shot-noise effects in electron dose. In principle, once measured, the shot-noise effects may be corrected in situ by an additional overlay exposure with the aim of further pushing the minimum feature size and line edge roughness achievable in electron beam lithography.

Raith GmbH, Konrad-Adenauer-Allee 8, PHOENIX West, 44263 Dortmund, Germany
phone +49 231 / 95004 - 0, fax +49 231 / 95004 - 460, e-mail: sales@raith.com, website: www.raith.com

Raith America Inc., 1377 Long Island Motor Parkway, Suite 101, Islandia, New York 11749, USA
phone +1 631 738 9500, fax +1 631 738 2055, e-mail: sales@raithamerica.com

Raith Asia Ltd., Two Chinachem Exchange Square, No. 338 King's Road, Floor 11, Unit 03, North Point, Hong Kong
phone +852 2887 6828, fax +852 2887 6122, e-mail: sales@raithasia.com

Raith China Ltd., Room 1409, Tower B, Techart Plaza, No.30 Xue Yuan Road, Haidian District, 100083 Beijing, P.R.China
phone: +86 10 828679 20/21/22, fax: +86 10 828679 19, e-mail: sales@raithchina.com

Raith India Pvt. Ltd., Sri Krishna Complex, No 36, 2nd Floor, Opposite Mother Theresa School, M. E. S. Ring Road, Bangalore 560054, India
phone +91 80 2838 4949, Fax +91 80 2838 4949, e-mail: sales@raithindia.com