

## Ion Beam Lithography for Fresnel Zone Plate Fabrication in Gold on Membranes

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### Introduction

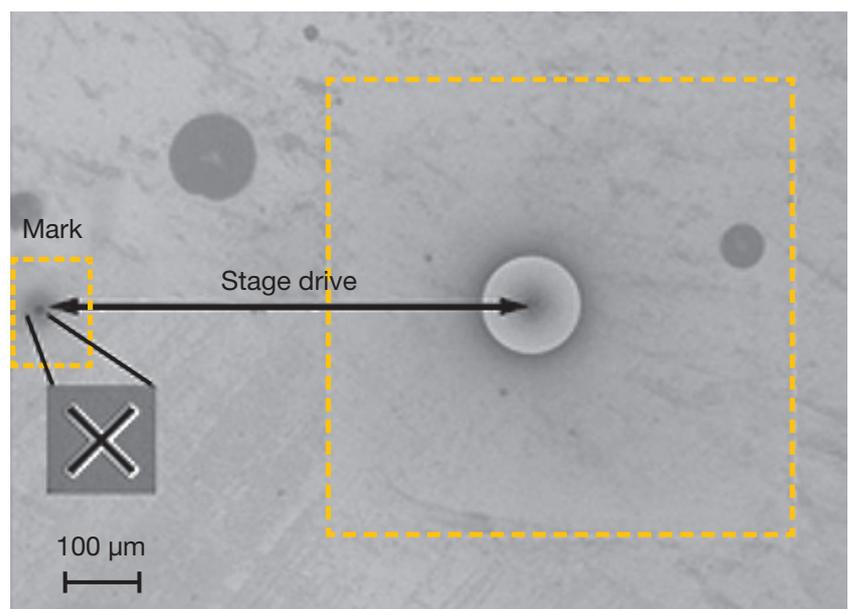
X-ray microscopy allows the investigation of samples with an element of specific contrast and a large penetration depth<sup>[1]</sup> at the nanometre scale. In the soft X-ray range the best resolutions are achieved by using Fresnel zone plates (FZP), a diffractive-based focusing element, which routinely achieves high resolutions down to 25 nm<sup>[1]</sup>. The resolution of a Fresnel zone plate is essentially determined by the width of its outermost zone  $\Delta r$ . Fresnel zone plates are typically prepared by Electron Beam Lithography (EBL) and conventional multiple step fabrication processes<sup>[2]</sup>. Here, we report on how the number of necessary steps can be significantly reduced by employing direct ion beam lithography in a single step. A FIB tool (Raith ionLINE) has been used to fabricate a zone plate with a diameter of 100  $\mu\text{m}$  and an outermost zone width of  $\Delta r = 100$  nm milled on a 500 nm-thick gold layer on top of a  $\text{Si}_3\text{N}_4$  membrane. The system overcomes nanofabrication-specific limitations of analytical FIB or FIB/SEM instruments by means of a dedicated lithography architecture. In particular this includes a laser interferometer stage,

high beam-to-sample stability as well as beam current stability and true automation capabilities (e.g. for additional positioning correction). A more detailed description of this work can be found in [3].

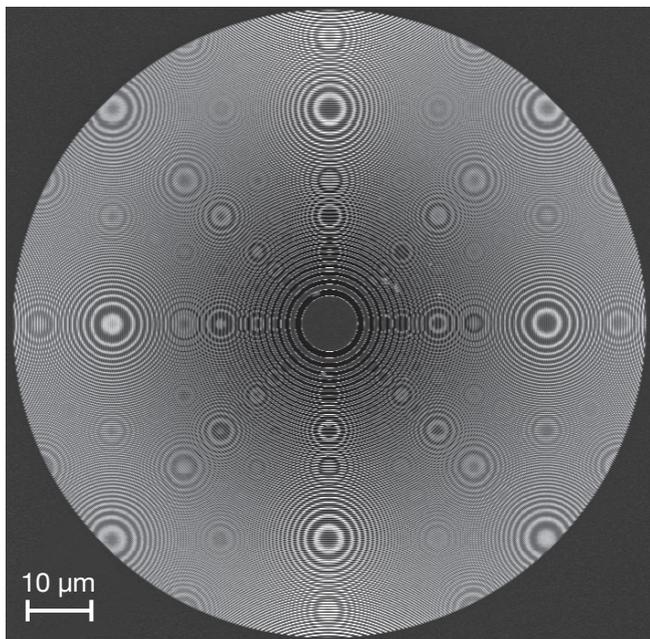
### The Fabrication Process

The FZP design consists of 251 zones with an outermost zone width of 100 nm and a 100  $\mu\text{m}$  diameter. The focal length at a photon energy level of 1.2 keV (wave length 1.03 nm) is 9.64 mm. The patterning of a 500 nm-thick gold layer on a 500 nm thick  $\text{Si}_3\text{N}_4$  membrane with direct ion beam writing using 40 kV acceleration voltage and 50 pA beam current lasted approximately 15 hours. This is longer than the typical exposure time with EBL for exposure of an electron sensitive resistance but no further process steps for pattern transfer are necessary here.

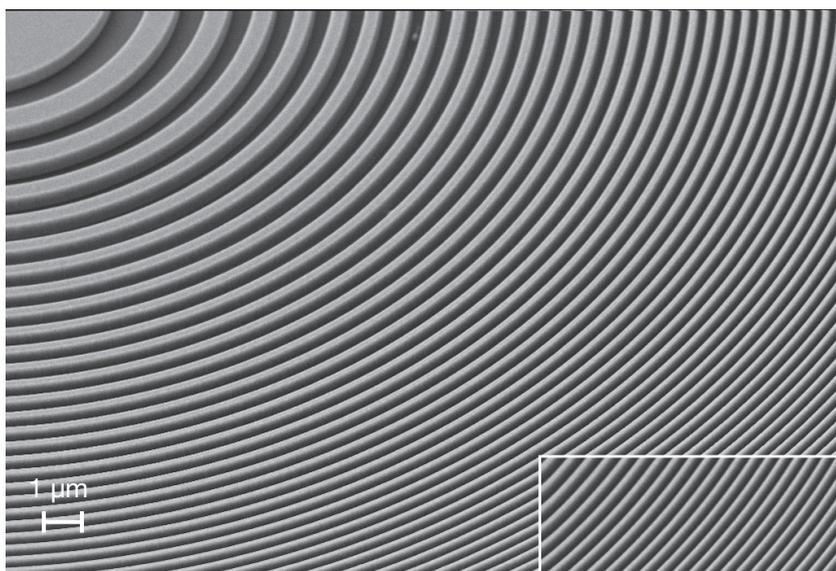
The fabrication process was divided into 100 patterning iterations of the zone plate design with automated drift correction steps in between. For milling of the circle ring elements we used a fill mode with



**Figure 1:** Overview SEM image of the silicon nitride membrane with active area including gold zone plate and position of reference mark for automatic positioning correction on bulk sample.



**Figure 2:** SEM micrograph of the zone plate described here (inner circles are a “Moiré pattern” due to imaging).



**Figure 3:** SEM micrographs showing a 45° tilted view of the zone plate: top) inner zones, bottom) 100 nm wide outer zones.



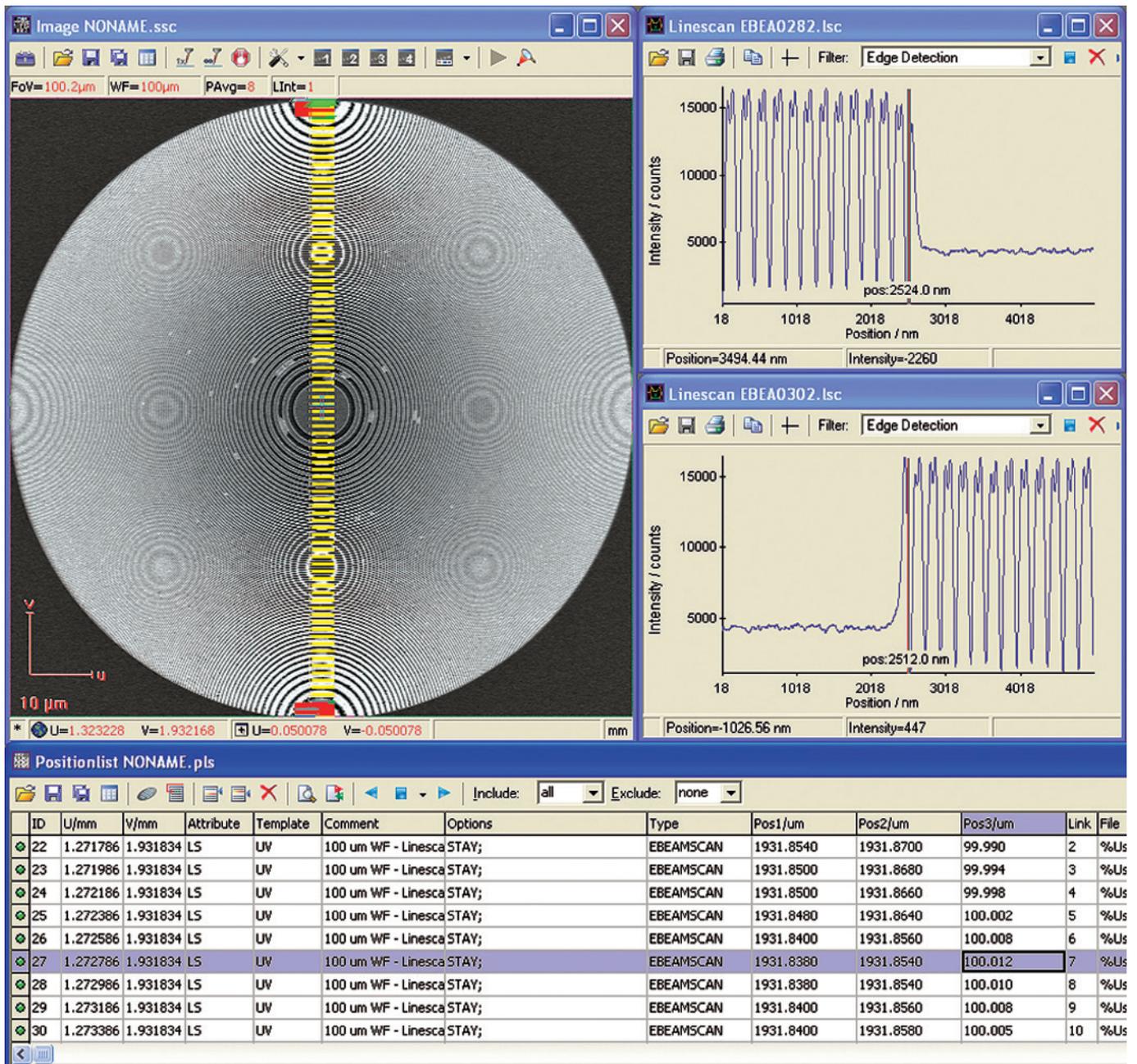
a circular inward beam movement with 10 nm step size, 1.4  $\mu$ s dwell time and five element loops. The drift correction method used is based on an image comparison of the mark scan with a reference image, which was acquired on the mark location before the zone plate fabrication was started. Due to the nm-precise laser stage positioning, the mark for automated drift corrections could be located outside the writing field and even outside the membrane at a distance of 500  $\mu$ m to the zone plate location (Fig. 1).

### Results and discussion

Several prototypes of zone plates have been routinely produced in automated overnight FIB processes by the method described above. To evaluate the quality of the devices we performed SEM imaging, FIB cross-sectioning and e-beam metrology<sup>[3]</sup>. Fig. 2 displays an SEM overview image of the complete zone plate which already gives an impression of the good homogeneity of the zones.

SEM images of inner and outer zones, with a higher magnification and a tilt angle of 45°, are shown in Fig. 3. The high quality and homogeneity of the outer zones indicate the reliability of the applied drift compensation process with an accuracy of better than 20 nm.

Another important parameter for the functionality of a zone plate is its roundness. The diameter should not vary more than 1.4 times the width of the outermost zone<sup>[4], [5]</sup> which can be demanding on the calibration of the writing field. Usually the



**Figure 4:** Example of e-beam line scan measurements to determine the diameter in a vertical direction. The two intensity profiles display the results of one of 20 pairs of line scans on the top and the bottom of the FZP. The red lines indicate the calculated position of the outer edge within the line scans. The calculated distance between these two edges is 100.012  $\mu\text{m}$ .

field of view of conventional FIB/SEM tools is calibrated by means of reference samples with accuracies worse than 100 nm - which is in fact good enough for the analytical applications they are usually used for.

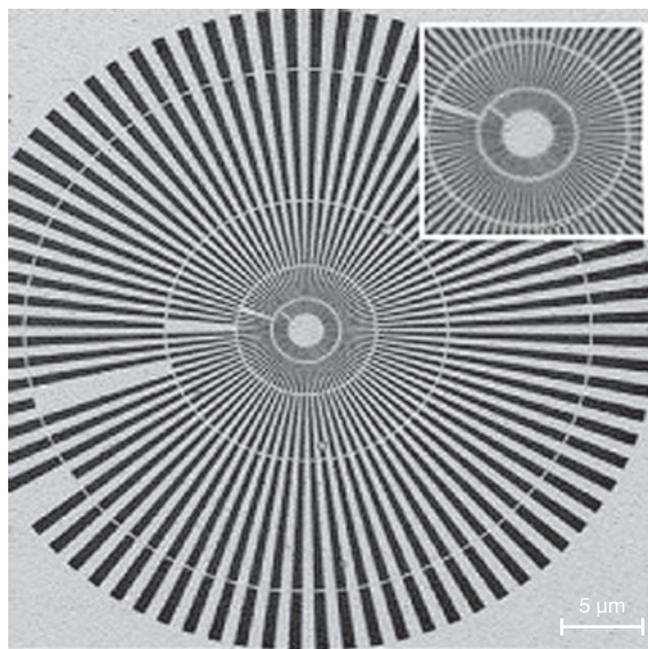
Dedicated lithography tools can use their nm-stage accuracy as a reference standard for the field calibration. This enables scaling errors in horizontal and vertical directions or orthogonality errors to be corrected with accuracy in the nm range. To determine the roundness of the zone plate we measured its diameter in horizontal, vertical and two diagonal di-

rections. In order to measure 100  $\mu\text{m}$  distances with nm accuracy we used the metrology functionality of a RAITH150 Two EBL tool with the ability to calibrate the measurement field with the desired accuracy. An e-beam line scan analysis including an edge detection algorithm was performed to detect the outer edges of the zone plate. The resulting values of the diameter in horizontal, vertical and both diagonal directions differ less than 25 nm from the nominal value of 100  $\mu\text{m}$  in all 4 measured directions. Fig. 4 shows an example of a line scan analysis in a vertical direction including the intensity profiles of a pair

of line scans on opposite edges of the zone plate. First insights into the focusing and imaging properties of the present zone plate could be gained at the scanning X-ray microscope (SXM) Maxymus at Bessy II<sup>[6]</sup> by imaging a commercially available gold Siemens star (X30-30-1, Xradia, USA) presenting a gold thickness of 180 nm and a feature size decreasing from 480 nm to 30 nm. An overall image of the Siemens star pattern (Fig. 5) was acquired at 1200 eV. The inset in Fig. 5 shows features in the outermost part of the second inner ring with a half pitch size of 120 nm. This is in accordance with the expected resolution for an FZP of  $\Delta r = 100$  nm. Further investigations of the imaging capability of the zone plate are on-going and have revealed interesting properties, which are under precise analysis.

## Conclusion

In this application note we have demonstrated the successful production of a functional 100  $\mu\text{m}$  diameter FZP in a 500 nm gold layer with a 100 nm outermost zone width by means of the direct ion beam lithography milling approach. The unique system architecture of the employed true FIB system (Raith ionLINE) with integrated laser interferometer stage, high stability ion optics and the automation capabilities of the system allow a solution to the trade-off between the need for high resolution and long patterning time. To the best of our knowledge this is the first ion beam fabricated zone plate which has



**Figure 5:** Soft X-ray images of a Siemens-star acquired with the FZP described in this manuscript, at 1200 eV in a line by line scan with a step-size of 75 nm and a dwell time of 1.18 ms/pixel. The inset is a close-up view of the centre part of the overall image.

proven its excellent functionality for X-ray imaging. This FIB approach opens up the application to many different materials for FZP, as the parameter optimisation effort is much less than compared with conventional processing.

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