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Ion beam lithography for direct patterning of high accuracy large area X-ray elements in gold on membranes

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ABSTRACT

X-ray elements like Fresnel zone plates (FZPs) are challenging objects for nanofabrication, as they require high accuracy, high aspect ratio and large area structures. In most cases they are fabricated with electron beam lithography (EBL) based techniques plus plasma or wet etching, which deliver routinely zone plates with outermost zone widths down to 25 nm and aspect ratios of 5:1. In this work we present a complementary resistless technique based on ion beam lithography (IBL) using direct milling with a focused Gallium beam and advanced process control. A zone plate of 100 μm diameter and 100 nm outermost zone widths has been produced in a 500 nm thick gold layer on a Si_3N_4 membrane. To achieve the desired resolution we developed a 15 h low current milling process including high accuracy automated drift correction on a mark outside the 500 × 500 μm membrane window. In addition first results of X-ray imaging with the zone plate are presented. To the best of our knowledge this is the first ion beam fabricated zone plate which has proven its functionality for X-ray imaging.

1. Introduction

X-ray microscopy allows the investigation of samples at the nanometer scale with an element specific contrast and a large penetration depth [1]. For the effective focusing of X-rays optics making use of the reflective, refractive and diffractive properties of X-rays have been developed. In the soft X-ray range the best resolutions are achieved by using Fresnel zone plates (FZPs), a diffractive based focusing element, which routinely achieve high resolutions down to 25 nm [1]. The resolution of a Fresnel zone plate is essentially determined by the width of its outermost zone Δr. Fresnel zone plates are typically prepared by electron beam lithography (EBL) based techniques. The conventional fabrication process consists of four steps [2]. In the first stage an electron sensitive resist is coated on a substrate, the zone plate pattern is then written in the resist with the electron beam and in the second step the resist is developed. The structure is filled by electroplating and finally the resist is removed leaving the free standing zone plate on the membrane.

Here, we report on the manufacturing of a Fresnel zone plate, where the number of necessary steps has been significantly reduced as the fabrication is performed by direct ion beam lithography in a single step.

First attempts to produce X-ray FZP by direct writing with a FIB have already been reported in [3,4]. However, the quality of the zones was relatively low and the focusing or imaging properties of the lens were not tested. Besides difficulties in FIB patterning of metals and in particular gold, which is a very popular material for X-ray applications as well, the employed instruments that are mainly designed for material science and analytical applications might have been a limitation so far.

Focused ion beam (FIB) systems and combined SEM/FIB microscopes are widely in use since many years for various analytical tasks such as cross sectioning or TEM lamella preparation which require high ion beam currents [5,6]. Nowadays most commercial FIB columns are integrated in existing SEM platforms which can be also beneficial for such tasks as it allows monitoring the process simultaneously by SEM imaging. However, milling of nano scale patterns on this kind of systems is usually limited to small areas of simple features due to limited stage positioning accuracy and beam deflection distortions or aberrations within large fields of view. Furthermore, compromises within the system architecture like the tilted stage for ion beam processing lead to additional sources of instability (drift) which are contradictory to the need of long process times due to the use of low beam currents for high resolution.

Here a novel IBL tool (Raith *ionLiNE*) has been used to fabricate a zone plate with a diameter of 100 μm and an outermost zone width Δr = 100 nm milled on a 500 nm thick gold layer on top of

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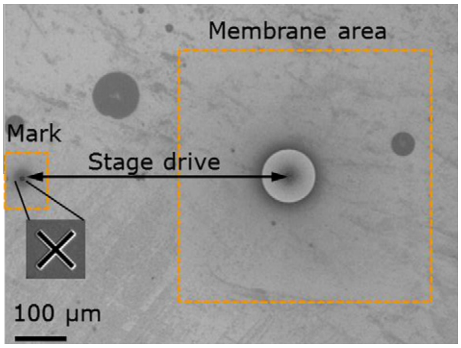

Fig. 1. Overview SEM image of the silicon nitride membrane with active area including gold zone plate and position of reference mark for automatic positioning correction on bulk sample.

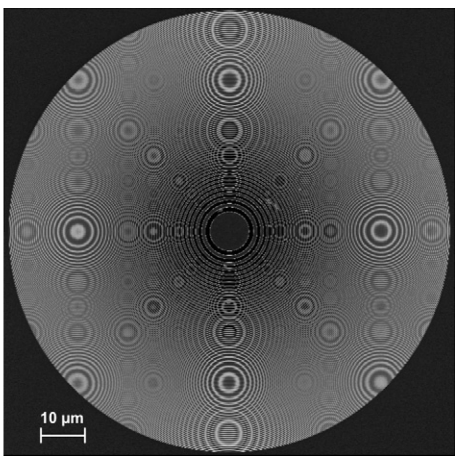

Fig. 2. SEM micrograph of a 100 μm diameter zone plate in a 500 nm thick gold layer on a silicon nitride membrane (inner circles are a "Moiré pattern" due to imaging).

a Si_3N_4 membrane. The system overcomes nanofabrication specific limitations of analytical FIB instruments via a dedicated lithography architecture. This includes in particular a laser interferometer stage, high beam to sample as well as beam current stability and true automation capabilities (e.g. for additional positioning correction).

2. Fabrication process

A 500 nm thick gold layer was deposited by ion beam sputtering on a 500 nm thick Si_3N_4 membrane. The membrane window size is about 500 × 500 μm. The patterning with direct ion beam writing lasted approximately 15 h, which is much longer than the typical exposure time (a few minutes) required by EBL for the writing of the structure in an electron sensitive resist but here no further process steps for pattern transfer are necessary. Thus IBL avoids the usual accumulation of errors typical for multi-step processes and simplifies the overall manufacturing. Furthermore, much less efforts are necessary for IBL process development when changing to other materials. Depending on the zone plate material process development of EBL including hard mask, pattern transfer, plasma/wet etch can take several month whereas IBL parameter optimization will take only days. In addition some materials cannot be processed at all with the conventional techniques.

For the fabrication of the zone plate we chose an acceleration voltage of 40 kV and a beam current of 50 pA, which is small enough to achieve the required resolution and big enough to keep the process time within a reasonable range for an automated overnight patterning. The FZP design consists of 251 zones with outermost zone width of 100 nm, a zone height of 500 and 100 μm diameter. The focal length at a photon energy of 1.2 keV (wave length 1.03 nm) is 9.64 mm.

The fabrication process was divided into 100 cycles with automated drift correction steps between each cycle. Within each cycle the complete zone plate design has been milled from outer to inner zones. For the patterning of the circle ring elements we used a fill mode with a circular wise inwards beam movement with 10 nm step size, 1.4 μs dwell time and five element loops.

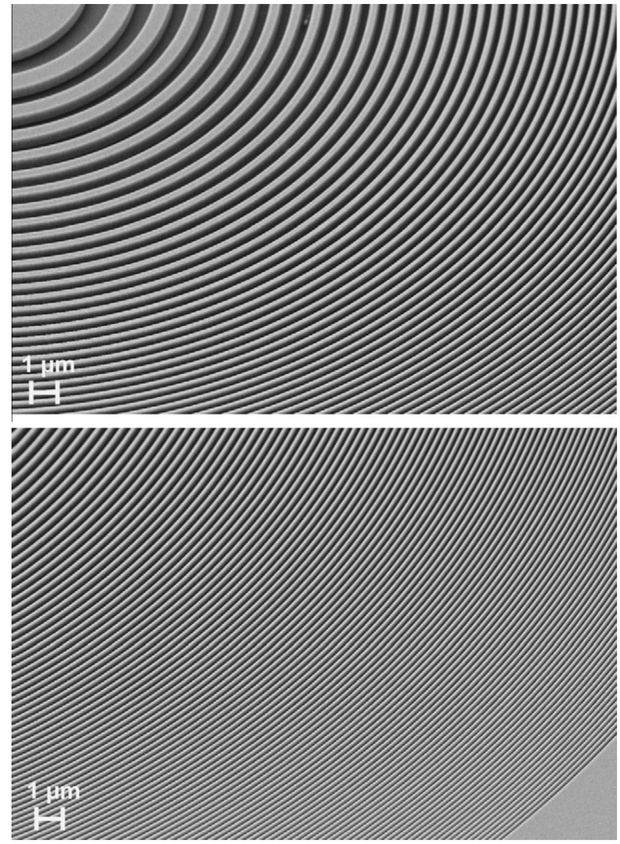

Fig. 3. SEM micrographs showing a 45° tilted view of the zone plate: (top) inner zones and (bottom) 100 nm wide outer zones.

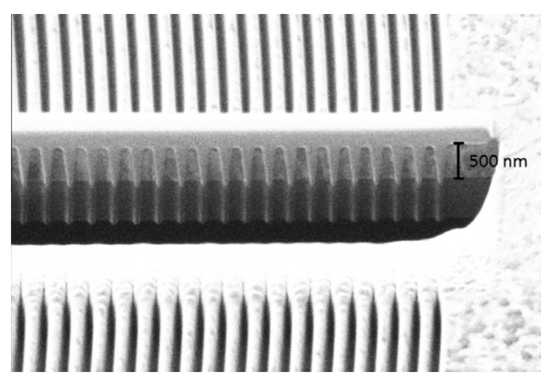

Fig. 4. Ion beam image showing a cross-section of outermost zones (45° tilted view) with the processed gold layer, the Si_3N_4 membrane underneath and a tungsten protection layer on top produced by ion beam induced deposition. The vertical stripes in the membrane are caused by curtaining effects during the ion beam cross-section cut.