9th International Conference on X-Ray Microscopy IOP Publishing
Journal of Physics: Conference Series **186** (2009) 012078 doi:10.1088/1742-6596/186/1/012078

Advanced X-Ray Diffractive Optics

J Vila-Comamala[1], K Jefimovs[1,2], T Pilvi[3], M Ritala[3], S S Sarkar[1], H H Solak[1], V A Guzenko[1], M Stampanoni[1,4], F Marone[1], J Raabe[1], G Tzvetkov[1], R H Fink[5], D Grolimund[1], C N Borca[1], B Kaulich[6] and C David[1]

[1]Paul Scherrer Institut, CH-5232 Villigen PSI, Switzerland
[2]EMPA, CH-8600 Dübendorf, Switzerland
[3]Department of Chemistry, FIN-00014 University of Helsinki, Finland
[4]Institute for Biomedical Engineering, CH-8092 University and ETH Zürich, Switzerland
[5]Friedrich-Alexander Universität, D-91058 Erlangen, Germany
[6]ELETTRA Sincrotrone, I-34012 Basovizza, Italy

E-mail: `joan.vila@psi.ch`

Abstract. X-ray microscopy greatly benefits from the advances in x-ray optics. At the Paul Scherrer Institut, developments in x-ray diffractive optics include the manufacture and optimization of Fresnel zone plates (FZPs) and diffractive optical elements for both soft and hard x-ray regimes. In particular, we demonstrate here a novel method for the production of ultra-high resolution FZPs. This technique is based on the deposition of a zone plate material (iridium) onto the sidewalls of a prepatterned template structure (silicon) by atomic layer deposition. This approach overcomes the limitations due to electron-beam writing of dense patterns in FZP fabrication and provides a clear route to push the resolution into sub-10 nm regime. A FZP fabricated by this method was used to resolve test structures with 12 nm lines and spaces at the scanning transmission x-ray microscope of the PolLux beamline of the Swiss Light Source at 1.2 keV photon energy.

1. Introduction

New developments in x-ray optics are improving the performance of x-ray microscopes, which in turn benefit a lot the investigation of both inorganic and biological materials. At the Paul Scherrer Institut, research activities in x-ray diffractive optics include the manufacture of Fresnel zone plates (FZPs) and diffractive optical elements for both soft and hard x-rays by several methods: Au-electroplated FZPs useful in the multi-keV range have been produced [1, 2] and a new type of diffractive optical elements to be used as condenser in full-field transmission x-ray microscope has been prepared [3, 4]. For soft x-rays, silicon-etched FZPs that can withstand high radiation heat loads have been manufactured [5] and a new technique to produce the FZP patterns using extreme ultraviolet holographic lithography is under development [6]. In particular, we want to introduce here a novel technique for the fabrication of FZPs with extremely narrow outermost zone widths. In a FZP-based microscope, there exists an intimate relationship between the spatial resolution and the outermost zone width of the FZP, the two of them being essentially comparable. Due to manufacturing limitations, the spatial resolution of x-ray microscopes is currently stagnating at 25-20 nm, and higher resolution has only been reported in very few exceptional cases [7, 8]. The method we present can overcome one of the main limitations during the manufacture of FZPs and it provides a clear route to push the spatial resolution of x-ray microscopes to the sub-10 nm regime.

2. Zone-doubling technique for high resolution FZPs

Up to date, x-ray FZPs are mostly prepared by electron-beam lithography tools. Even though these systems are capable of writing patterns with spot sizes and position accuracies in the sub-10 nm range, the final size of the patterned structure is mainly depending on the range of the secondary electrons created in the sensitive resist layer. This effect is particularly detrimental when writing dense patters of lines such as gratings or FZPs, and it explains the stagnation at 25-20 nm in terms of spatial resolution of FZP-based x-ray microscopy. Here we report on a simple nanofabrication method that overcomes the difficulty of high density patterning. Since this approach only requires a single lithography step and no need for alignment, this method is very reproducible. Moreover, it allows the fabrication of structures with extremely high aspect ratios. It is based on a deposition of thin layer of a high refractive index material onto the sidewalls of template made of a low-index material. This leads to a doubling of the effective line density of the deposited material comparing to that of the template, which in turn improves the spatial resolution of the FZP by a factor of two.

The fabrication steps are schematically shown in Figure 1. The process starts with the electron-beam exposure of the FZP pattern on a high resolution negative tone resist. Then, the pattern is transferred to an intermediate chromium layer which is used as mask during the reactive ion etching process to create the template structure on a previously prepared silicon membrane. The final step in this fabrication technique requires a highly conformal deposition of the high refractive index material with accurate control of the thickness. Because of its unique self-limiting growth mechanism, atomic layer deposition [9] is the best choice for this purpose and was employed in this work. A cross section of the 15 nm wide outermost zones of a zone-doubled FZP is shown in Figure 2a. The aspect ratio of these structures is around 12.

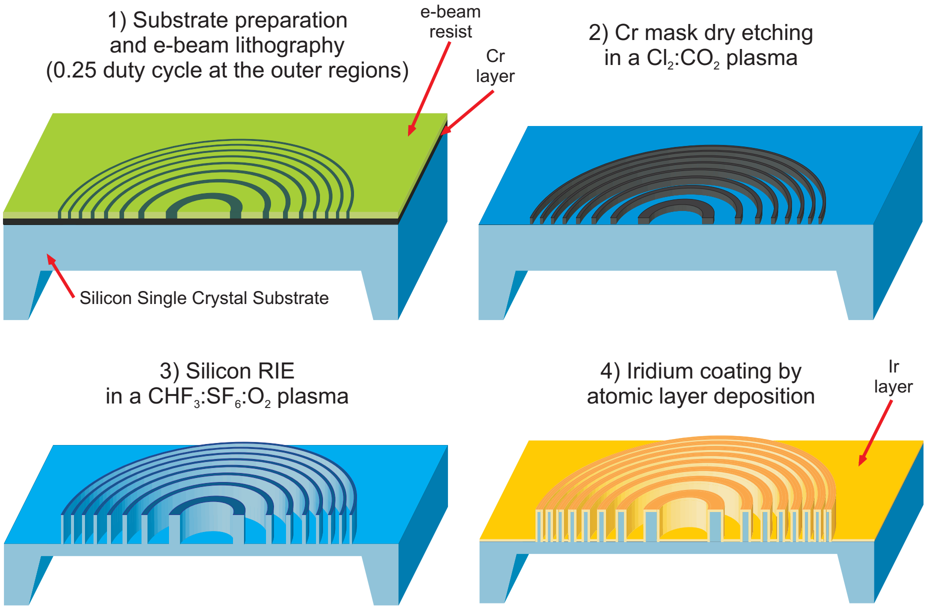

Figure 1. Zone-doubled FZP manufacturing steps: 1) electron-beam lithography, 2) pattern transfer to chromium layer by reactive ion etching, 3) pattern transfer to silicon by reactive ion etching and 4) iridium coating by atomic layer deposition.

In addition, theoretical calculations [8] show that the proposed focusing zone-doubled structures can provide diffraction efficiencies slightly below but comparable to those of ordinary FZPs (∼10—20 %). For instance, 9.1% diffraction efficiency is expected for a zone-doubled FZP made of silicon and iridium, considering a diameter of 100 μm and an outermost zone width of 20 nm at 1.0 keV. Further calculations also show that the zone-doubled structures are more efficient at higher energies, for which the absorption of the silicon template is highly reduced.

3. High resolution scanning transmission x-ray microscopy

To demonstrate the feasibility of the proposed fabrication technique a zone-doubled FZP with an outermost zone width of 15 nm and a diameter of 100 μm was produced and tested at the scanning transmission x-ray microscope (STXM) of the PolLux beamline of the Swiss Light Source at 1.2 keV photon energy. During the experiments, a GaAs/AlGaAs heterostructure containing several line widths ranging from 40 nm down to 9 nm was used as a test sample. Figure 2b shows the STXM image of the sample where the smallest visible features consist of 3 lines of 12 nm width.

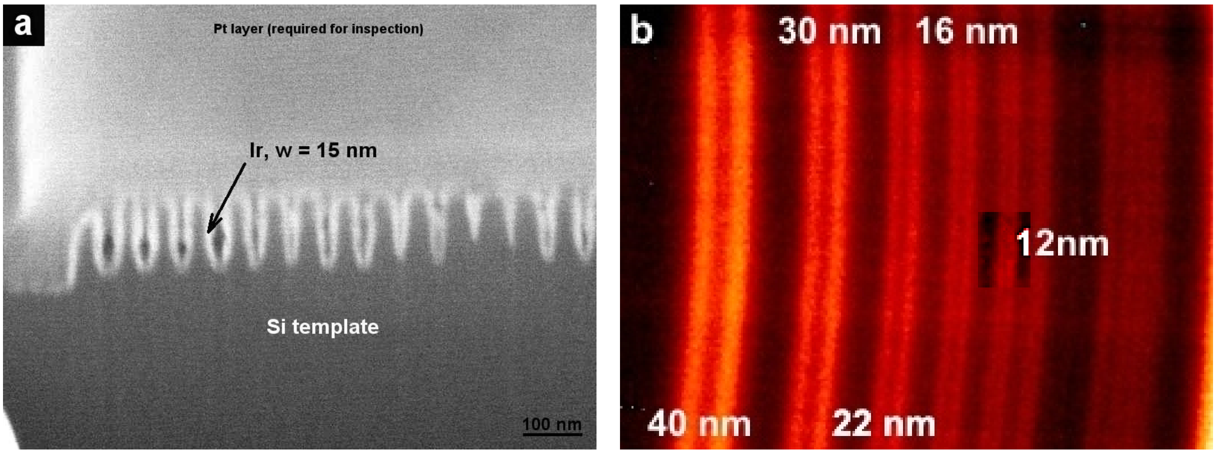

Figure 2. a) Cross section of zone-doubled FZP with an outermost zone width of 15 nm. b) STXM image of GaAs/AlGaAs heterostructure with several line widths. Smallest visible features consist of 3 lines of 12 nm width.

In conclusion, we have developed a novel technique that frees us from the present limitation of electron-beam lithography in the production of FZPs. It improves the spatial resolution by a factor of 2 and provides a clear route to push the resolution of FZP-based x-ray microscopy into the sub-10 nm regime.

References

[1] Jefimovs K, Bunk O, Pfeiffer F, Grolimund D and van der Veen J F 2007 *Microelectron. Eng.* **84** 1467
[2] Borca C N, Grolimund D, Willimann M, Meyer B, Jefimovs K, Vila-Comamala J and David C 2008 *These proceedings*
[3] Jefimovs K, Vila-Comamala J, Stampanoni M, Kaulich B and David C 2008 *J. Synchrotron Rad.* **15** 106
[4] Stampanoni M, Marone F, Mikuljan G, Jefimovs K, Trtik P, Vila-Comamala J, David C and Abela R 2008 *These proceedings*
[5] Vila-Comamala J, Jefimovs K, Raabe J, Kaulich B and David C 2008 *Microelectron. Eng.* **85** 1241
[6] Sarkar S S, Sahoo P K, H H Solak C D and van der Veen J F 2008 *J. Vac. Sci. Tech. B (accepted)*
[7] Chao W, Harteneck B D, Liddle J A, Anderson E H and Attwood D T 2005 *Nature* **435** 1210
[8] Jefimovs K, Vila-Comamala J, Pilvi T, Raabe J, Ritala M and David C 2007 *Phys. Rev. Lett.* **99** 264801
[9] Leskelä M and Ritala M 2003 *Angew. Chem., Int. Ed.* **42** 5548