Towards 5-nm-Focus-Size Fresnel Zone Plates for Hard X-rays

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X-ray microscopy, tomography, and volumetric elemental analysis with few-nanometer resolution are important techniques for probing natural and synthetic materials at the nanoscale. Photon energies larger than ~20 keV enable access to the K, L and M absorption edges of medium-heavy elements, offer additional spectral capabilities, and provide increased penetration depths over lower energy X-rays. Additionally, many table-top microstructured anode X-ray tube sources must operate at high energies to take advantage of large emission intensities from refractory metals used as anodes. While synchrotron radiation facilities are undergoing costly upgrades, and X-ray tube manufacturers are finding new solutions to create high brilliance and high coherence sources [1], these sources are lacking in focusing optics for high energy X-rays. To take maximal advantage of improvements in X-ray beam quality for focusing applications, advanced zone plates need to have zone widths less than 10 nm, and aspect ratios greater than 100:1. Currently, high resolution diffractive focusing optics, traditionally fabricated *top-down* by electron beam lithography and etching or electroforming, have reached a hard limit with zone widths and aspect ratio of 25 nm and 40:1, respectively. *Bottom-up* approaches, such as sputter-and-slice [2,3] or even atomic layer deposition (ALD)-and-slice [4] were proposed, but encountered difficulties in zone width control and slicing, since they were based on focused ion beam (FIB) machining.

The present work demonstrates the feasibility of an approach for fabricating zone plates that will satisfy the current and future needs of the X-ray community. Our approach enables the batch processing of zone-plate devices using ALD of two layers of different X-ray optical properties on the outer surface of batch-fabricated Si pillars. It has the potential to enable the production of hundreds of Fresnel Zone-Plate (FZP) devices from a single Si wafer, with 5 nm zones and aspect ratios >100:1. The nanofabrication starts by fabrication of thousands of cylindrical pillars by Bosch-etching of Si (Fig.1), followed by a side wall smoothing procedure capable of reducing the roughness from >50 nm to <1 nm (rms). Side wall slope can be also controlled by this procedure. Sequential ALD follows, resulting in well-controlled-thickness nanolaminate layers of Al_2O_3 and Ta_2O_5, deposited following the Fresnel zone width rule. Filling the remaining gaps with electroplated metal (Cu in our example), chemical-mechanical planarization, bonding to a carrier wafer and polishing the back side of the device wafer to reach the structures follow. Finally, windows are formed in the carrier wafer, which are spanned by membranes containing embedded FZPs.

As an initial demonstration of our fabrication technique, nanolaminates containing only 50 layers of Al_2O_3 and Ta_2O_5 were used as a proof of principle for process integration capabilities. Layers with thicknesses ranging from 25 nm to 5 nm were deposited and characterized after deposition as well as after completing the fabrication. While modest deformations of the finest nanolaminates were noticed at the exterior of the Cu cylinders next to the Si interface, no deformations happened at the interior of the nanolaminate near the Si-pillars. The deformations at the exterior of Cu cylinders were attributed to the thermal stress due to the higher coefficient of thermal expansion of Cu (17 ppm/^{o}C) as compared to Si (2.6 ppm/^{o}C), leading to compressing of the nanolaminates during heating such as for bonding or resist curing processes. The expansion of Cu during heating had little to no effect on the central Si pillars with nanolaminates, where the stress on the nanolaminates was tensile. Future work will focus on fabricating fully usable FZP with 10-15 μm of nanolaminate "zoned" material. Using more than two materials for the nanolaminate would allow graded index FZPs to be produced.

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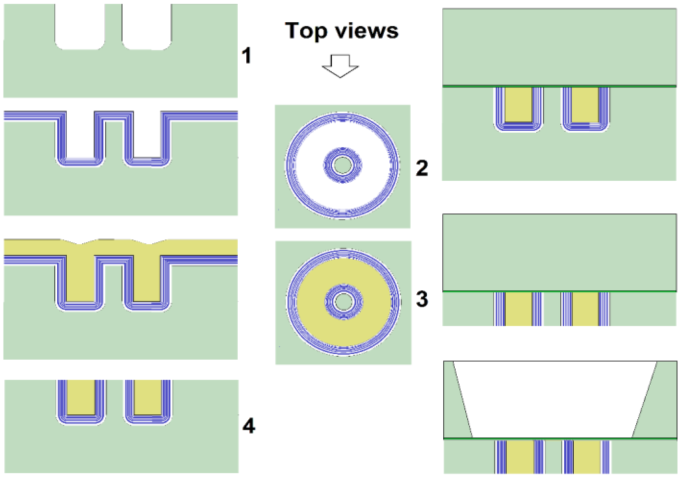

Fig.1. Fabrication sequence for the ALD-and-batch processed FZPs. 1-Fabrication of Si cylindrical pillars by Bosch etching; 2- side wall smoothing layer deposition and ALD of nanolaminates; 3-filling with electroplated metal (Cu); 4- Planarization polishing; 5- Bonding of a carrier wafer; 6- back side thinning and polishing; 7- formation of membranes with embedded FZP by KOH etching.

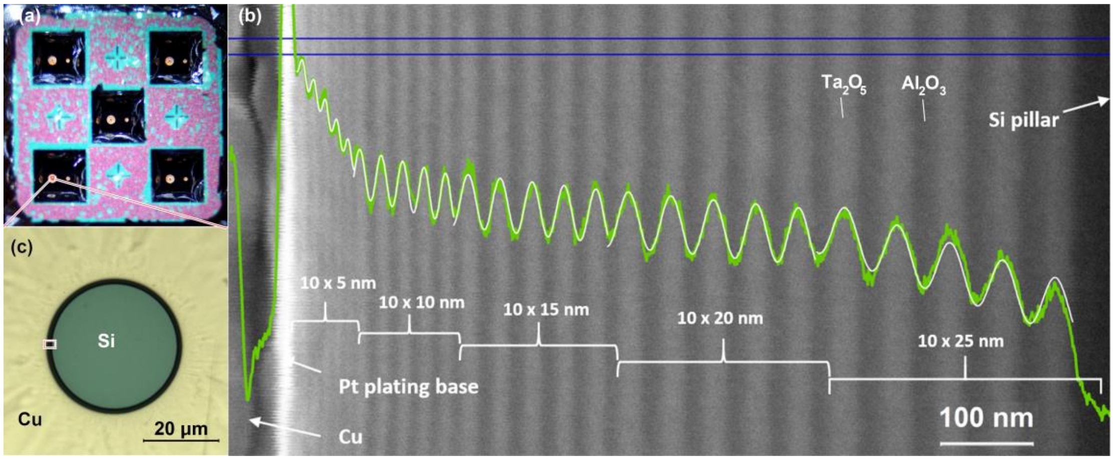

Fig. 2. Details after complete integration: (a) 6 mm x 6 mm chip with 5 membranes containing FZP structures; (b) zoom in into a FZP structure; (c) - high resolution SEM image of Al_2O_3/Ta_2O_5 nanolaminates on the edge of a Si pillar marked with a rectangle in (b), with the 5-nm-thick layers visible. The curves represent intensity line profiles as used for measurements.

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