

## A.10: Fabrication of Fresnel zone plate in tungsten for hard x-ray applications

Diffractive optical elements (DOEs) are important optical element where any other type of conventional optics cannot be used due to either space requirements or refractive index considerations. A Fresnel zone plate (FZP) is a DOE, designed as per Fresnel half period zones in such a way that it focuses electromagnetic radiation diffractively. FZPs are generally utilized for the specific class of experiments where micro-nano synchrotron probes are required for focusing or imaging experiments related to micro-analysis, micro spectroscopy, micro diffraction and x-ray telescopes. These optical elements are far away from the routine ones due to the fabrication difficulties and material limitations. In fact, the material of zones of FZP should ideally introduce a strong phase shift in the incoming radiation with minimum absorption. As photon energy increases, the amount of material required to introduce ' $\pi$ ' phase shift also increases. To avoid very high aspect ratio and also to achieve acceptable diffraction efficiencies, the zone plate structures are preferred to be made from high atomic number elements. Even then, the required aspect ratio is 10-20 for obtaining both high resolution and efficiency. The choice of materials becomes limited and fabrication of such high aspect ratio becomes difficult. Tungsten is one of the well-suited materials for high resolution fabrication.

A novel and scalable process to fabricate tungsten FZP structures with high aspect ratio using wet etching method has been developed. FZPs with an outermost zone width of 370 nm and a height of 1.1  $\mu$ m were fabricated in tungsten on 35  $\mu$ m polyimide film. It was designed for a focal length of 50 cm at 8 keV such that the radius is 100  $\mu$ m for 142 zones, with an opaque center zone. The fabrication method is a single step resist process where the design has been suitably modified with interconnects to impart strength to the structure (Figure A.10.1). The patterning was carried out using electron beam lithography at 20 keV.



Fig. A.10.1: Dark field image of FZP fabricated in tungsten on polyimide membrane, designed for 8 keV, with (a) regular interconnects, (b) random interconnects to impart strength to the structure. Design parameters were: f = 50 cm, D = 200 $\mu$ m, N = 142,  $\Delta r_n = 370$  nm. Inset shows defocused image, dispersion of colors of visible light with focusing due to FZP was clearly observed.

The testing of FZP was done at Indus-2 synchrotron source, where focusing was observed as per design parameters. It was tested on the beamline BL16 of Indus-2 at energy of 8 keV. An arrangement was made for testing on an optical test bench (Figure A.10.2(a)) where all the optics holders were aligned in the beam path. The incident x-ray beam was limited by a 200 µm platinum aperture so that only the FZP was illuminated. The FZP was placed on a 5 axis mechanical stage followed by a 50 µm platinum order sorting aperture (OSA) after FZP. The transmitted intensity was recorded using a CCD camera having a pixel size of 6.4 µm. Focused intensity was recorded at different distances from the OSA to see the focusing effect. Alignment of the FZP in the beam path was very crucial step and was performed by imaging of the FZP at absorption edge of tungsten at 12 keV without OSA in line (Figure A.10.2). Finally, images were recorded for this geometry with the movement of FZP in x and y directions. The transmission characteristics of polyimide coupled with phase shifting properties of tungsten makes the FZP useful also at lower energies.



(e) 50 µm OSA (f) With FZP

Fig. A.10.2: (a) Test set up at BL16 Indus2. (b, c) Absorption Image of FZP at absorption edge of tungsten at ~12 keV without apertures in path (d) enlarged CCD image of beam profile without FZP but with 200  $\mu$ m aperture (e) with 200  $\mu$ m and 50  $\mu$ m OSA and (f) that with FZP and both the apertures.

An aspect ratio of  $\sim$ 3 was achieved in these structures and they work as partial phase FZPs at 8 keV. It has been concluded that interconnects were important for improving the aspect ratio and mechanical stability. Even though presently there are superior techniques available for the purpose of x-ray diffractive optics, this study is an important improvement in generating an understanding for nano-patterning of tungsten using wet chemistry. The process is very versatile and can be used for other applications, such as high-resolution x-ray test samples and contact masks for x-ray lithography. For details please refer to P. Tiwari et. al., JVST B, **35** (5), 051602, 2017.

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