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Research paper

Systematic efficiency study of line-doubled zone plates

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ABSTRACT

Line-doubled Fresnel zone plates provide nanoscale, high aspect ratio structures required for efficient high resolution imaging in the multi-keV x-ray range. For the fabrication of such optics a high aspect ratio HSQ resist template is produced by electron-beam lithography and then covered with Ir by atomic layer deposition (ALD).
The diffraction efficiency of a line-doubled zone plate depends on the width of the HSQ resist structures as well as on the thickness of the deposited Ir layer. It is very difficult to measure these dimensions by inspection in a scanning electron microscope (SEM) without performing laborious and destructive cross-sections by focus ion beams (FIB). On the other hand, a systematic measurement of the diffraction efficiencies using synchrotron radiation in order to optimize the fabrication parameters is not realistic, as access to synchrotron radiation is sparse.
We present a fast and reliable method to study the diffraction efficiency using filtered radiation from an x-ray tube with a copper anode, providing an effective spectrum centered around 8 keV. A large number of Fresnel zone plates with varying dimensions of the resist structures and the ALD coating were measured in an iterative manner. Our results show an excellent match with model calculations. Moreover, this systematic study enables us to identify the optimum fabrication parameters, resulting in a significant increase in diffraction efficiency compared to Fresnel zone plates fabricated earlier without having feedback from a systematic efficiency measurement.

1. Introduction

Fresnel zone plates (FZPs) serve as diffractive lenses in a variety of x-ray microscopy techniques [1–4]. They can either be used in scanning x-ray microscopes to produce a small, intense focal spot that is scanned across a sample, or as objective lenses in full-field x-ray microscopes to create a magnified x-ray image. FZPs consist of many concentric rings with pitches that decrease from the center to the outer edge. A coherent incident plane x-ray wave will be diffracted by this pattern in such a way, that the first order diffraction interferes constructively in a focal point. Apart from the fact that several diffraction orders exist, a FZP acts very similar to a normal lens. The achievable spatial resolution is limited to approximately the half pitch of the outermost zones. Therefore, FZPs have to be made with small lateral structure sizes, when high resolution is needed. Presently, FZP-based x-ray microscopes routinely achieve sub-100 nm resolution, and in some cases, the resolution is approaching the 10 nm level [5–8].

Apart from providing sufficiently high resolution, FZPs used in the x-ray range should also give sufficient diffraction efficiency. The optimum efficiency is obtained when the zone plate structures induce a phase shift close to π to the x-ray wave. For x-ray optics used at multi-keV photon energies, this means that zone structures should be made of heavy materials and be several 100 nm high. Consequently, nano-structures with very high aspect ratios need to be made by nanolithography techniques. These include the generation of high aspect ratio polymethyl-methacrylate (PMMA) resist structures, which are then filled by gold by electroplating. FZP structures with aspect ratios up to 15 have been achieved using this method [9–11].

A successful way produce metallic nano-structures with smaller line widths and higher aspect ratios is the so-called line-doubling technique [12,13]. It is based on the fabrication of a template structures made of a light material having only a weak effect on x-rays, which is coated with a heavy metal by ALD, as shown in Fig. 1. As the x-ray mainly interact with the metal deposited on the side walls of the template structures, the effective number of lines is doubled. In our case the template is written into HSQ (Hydrogen silsesquioxane) and then coated with Ir (see Fig. 2).

High aspect ratio nano-structures can also be fabricated by other techniques like MACE (Metal Assisted Chemical Etching) in combination with line-doubling [14], multilayer lenses [15–18], by stacking [19] or other techniques [20]. However, reaching the highest possible

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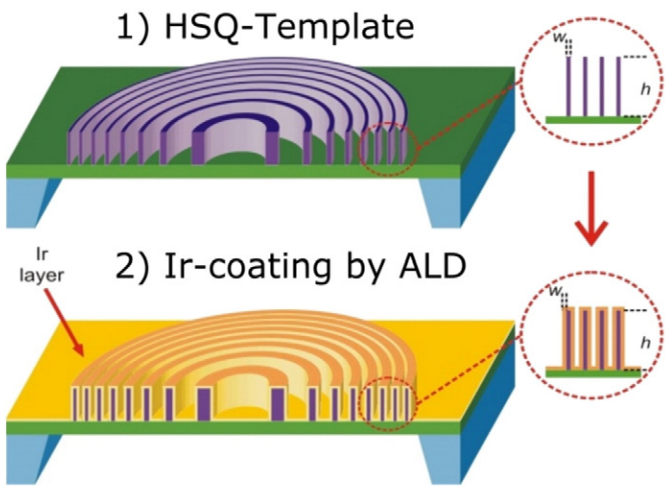

Fig. 1. Schematic of the fabrication process of line-doubled zone plates (blue: Si-frame, green: Si_3N_4, purple: HSQ, yellow: Ir) [13].

aspect ratio is not in the focus of this manuscript. Here we will focus on the effect of the duty cycles in line-doubled FZPs, which is independent from its actual line width and structure height.

2. Efficiency calculations

In order to investigate the effect of the geometry of a diffractive x-ray optical element, we first consider the case of a diffraction grating with constant period. Its efficiency can be calculated using the x-ray optical constants tabulated by Henke et al. [21]. It mainly depends on the height of the structures and the duty cycle, which is defined as the ratio of the pitch to the line width. In this study we focus on the effect of the duty cycle. We consider zone plates consisting of 550 nm high HSQ template structures that are conformally coated with an Ir layer. This geometry gives maximum diffraction efficiency at a photon energy of 4.5 keV. However, in our study we consider a photon energy of 8 keV, which still results in appreciable diffraction efficiencies and allows for convenient characterization with radiation from an x-ray tube source. It should be noted that the actual height or photon energy has no impact on the optimum duty cycle.

For a binary grating, with alternating empty and filled lines, the efficiency is highest for a duty cycle of 0.5. In a line-doubled grating, the situation is more complex, as the structure is a periodic arrangement of zones, that each consist of four parts: air, Ir, HSQ, Ir. Taking into account that the two Ir parts are of equal width, such a grating has two duty cycles, one concerning the HSQ structure width and one concerning the Ir layer thickness.

Fig. 3 shows the effect of both duty cycles on the diffraction efficiency, assuming perfectly shaped zones. The first order diffraction efficiency of a line-doubled grating is highest, when all parts have the same size, which means that both, Ir duty cycle and HSQ duty cycle, are 0.25. All deviations from this optimum lead to decrease of the first order diffraction efficiency and to increase of other orders. Especially the half order, which is just the first order of the HSQ template, can become significant if wrong fabrication parameters are chosen.

The situation is again somewhat different when considering the situation of a FZP, as the pitch of a FZP is changing over its radius. As Ir is deposited by ALD, its thickness is constant over the entire FZP. Thus, Ir duty cycle is changing with the radius. Consequently the efficiency of a line-doubled FZP is changing over its radius, as shown in Fig. 4. Depositing a little more Ir as optimum for the outermost zones, shifts the maximum diffraction efficiency towards the center of the FZP. Thus the overall diffraction efficiency of a line-doubled FZP can be increased.

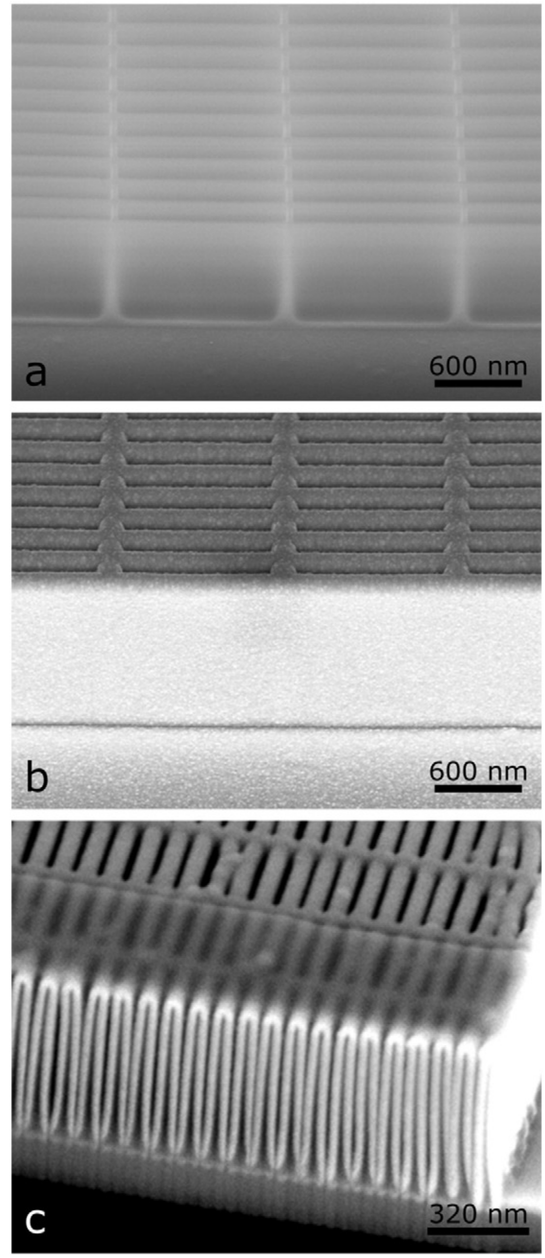

Fig. 2. SEM images of a line-doubled zone plate with 30 nm half Ir pitch and 60 nm half HSQ pitch: a) HSQ-template, b) after Ir-coating, and c) FIB cross section of the Ir coated HSQ template with 20 nm half Ir pitch.

Fig. 5 shows the integrated diffraction efficiency of a line-doubled FZP dependent on both duty cycles. The result of these calculations is, that for a FZP the highest diffraction efficiency can be achieved by an HSQ duty cycle of 0.21 and an Ir duty cycle of 0.33.

3. Fabrication of Fresnel zone plates (FZPs)

For the experiment we produced membrane chips each having 16 zone plates. All zone plates had the very same diameter of 150 µm, and an effective outermost half pitch of the Ir structures of 30 nm. This is not the smallest achievable structure size using the line-doubling technique, but was very suitable for this study, as the smallest achievable structure size was not goal of the optimization.

The FZPs for our experiment were fabricated on silicon nitride membranes. For the membrane fabrication, double side polished silicon wafers with 250 µm thickness were coated with 250 nm low stress silicon