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Full-field hard x-ray microscopy with interdigitated silicon lenses

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ABSTRACT

Full-field x-ray microscopy using x-ray objectives has become a mainstay of the biological and materials sciences. However, the inefficiency of existing objectives at x-ray energies above 15 keV has limited the technique to weakly absorbing or two-dimensional (2D) samples. Here, we show that significant gains in numerical aperture and spatial resolution may be possible at hard x-ray energies by using silicon-based optics comprising 'interdigitated' refractive silicon lenslets that alternate their focus between the horizontal and vertical directions. By capitalizing on the nano-manufacturing processes available to silicon, we show that it is possible to overcome the inherent inefficiencies of silicon-based optics and interdigitated geometries. As a proof-of-concept of Si-based interdigitated objectives, we demonstrate a prototype interdigitated lens with a resolution of ≈ 255 nm at 17 keV.

1. Introduction

X-ray microscopy (XRM) is an established family of techniques for imaging embedded and structurally complex specimens with sub-μm resolution. The ability to 'look' inside dense matter has provided crucial insight into phenomena such as ferromagnetic domains [1], nano-scale strain [2] and compositional inhomogeneity [3]. The techniques can be broadly categorized into *scanning*- [4], *projection*- [5] and *objective*-based [6] approaches, which offer different compromises of spatial resolution, acquisition time and sensitivity. Full-field imaging with an objective is particularly relevant to materials and geological sciences, as its efficiency and modalities (e.g. dark-field [7]) enable real-time imaging of complex processes [8]. Performing full-field XRM at hard x-ray energies (>15 keV) would then open a new door to dynamic, three-dimensional (3D) multi-scale studies of denser and more complex samples. However, current x-ray objectives tend to be aberrated or inefficient in the hard x-ray regime, limiting spatial and temporal resolution.

In this paper, we show that Si-based 2D compound refractive lenses (CRLs) constitute a viable approach for improving the numerical aperture (NA) and efficiency of full-field XRM objectives at hard energies. Specifically, we show theoretically that sufficiently miniaturized Si CRLs can outperform the current state-of-the-art at hard energies, and validate this prediction based on the performance of a prototype objective at 17 keV.

Various x-ray imaging optics have been proposed for use at hard energies: reflective optics (multilayer mirrors [9]) are efficient but expensive and delicate; diffractive optics (Fresnel zone plates [10]) have large NAs but are inefficient, while refractive optics (compound lenses [11] or prisms [12]) can be efficient but are prone to aberration and have small NAs. The approach demonstrated here can overcome some of the current limitations of refractive optics at hard energies by utilizing a miniaturized, 2D 'interdigitated' configuration of planar silicon lenses (Fig. 1).

The ideal lenslet geometry and configuration of a CRL-based XRM can be determined by optimizing the NA. From Ref. [6], the NA of an XRM using an ideal, absorption-limited CRL of focal length f, comprising N identical lenslets with apex radius of curvature R, linear attenuation coefficient μ and refractive decrement δ follows the relationship:

$$\mathrm{NA} \propto \left(\frac{\mathcal{M}}{\mathcal{M}+1}\right)\sqrt{\frac{\delta}{\mu}}\sqrt{\frac{1}{f}} = \left(\frac{\mathcal{M}}{\mathcal{M}+1}\right)\sqrt{\frac{N\delta^2}{\mu R}} \tag{1}$$

Eq. (1) implies that for a given magnification $\mathcal{M}$, the NA is greatest in CRLs with a short focal length and lenslets of a low-Z material. Many CRLs therefore utilize lenslets produced by indenting the parabolic lens profile into polycrystalline Be or Al [6].

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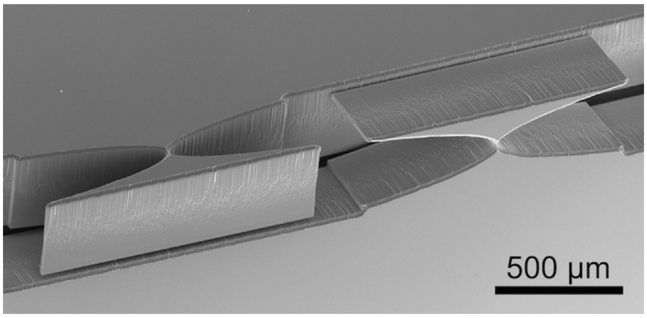

Fig. 1. SEM image of Si interdigitated lenslets within the prototype objective lens tested in this work. The small apex radii of curvature accessible to Si-based lenslets enables shorter focal lengths and higher NAs than existing 2D objectives.

While these materials have favourable values of δ and μ in the hard x-ray regime, the indenting process is expensive and limited to large R due to material (grain structure, porosity, and plasticity) and processing (tool shape and concentricity) issues. At hard energies, this necessitates the use of many lenslets at significant cost to achieve a short f and thus large NA. Polymer CRLs avoid these drawbacks, but are susceptible to radiation damage when flux and energy are high (e.g. in bright-field XRM) [13]. However, Eq. (1) also indicates an alternative route to large NAs by using lenses of inferior refractive medium but with drastically miniaturized dimensions (i.e. small R and lenslet thickness T).

We therefore propose Si-based objectives produced using the same nano-manufacturing methods as for nano-focusing 1D CRLs, which are both cost-effective and capable of apex radii as small as 1 µm [14]. 2D focusing can be achieved by 'interdigitating' the 1D CRLs such that horizontal and vertical lenslets alternate [15] as shown in Fig. 1. This reduces astigmatic aberration compared to sequential chips and potentially enables lens-by-lens optimization of the NA through aberration-corrected [12], adiabatic [16] and kinoform geometries [17]. Ultimately, we show theoretically that the technical benefits of utilizing Si as a refractive medium overcomes its inherent disadvantages in terms of refractive performance to yield higher performing lenses at hard energies.

2. Simulation

The formalism (see Supplementary Material) uses the ray-transfer matrix approach [18–20] to describe the cumulative effects of the individual lenslets in the CRL and predict the aberration induced by misalignment. At its core is the general expression for the transmission function I/I_0. In the 1D case of axisymmetric imaging lenses, I/I_0 describes the attenuation of a ray originating from a Gaussian source with radial position r_s and angle w_s by a CRL comprising N identical parabolic lenslets of web thickness T_0 and linear attenuation coefficient μ, where γ is a geometry-dependent constant defined in the Supplementary Material:

$$\frac{I}{I_0}(r_s, w_s) \propto \exp(-NT_0\mu) \times \exp\left(\frac{-r_s^2}{2\sigma_v^2}\right) \times \exp\left[\frac{-(w_s-\gamma r_s)^2}{2\sigma_a^2}\right] \tag{2}$$

Eq. (2) is a product of three exponential terms: a constant absorption factor, a Gaussian with standard deviation σ_v describing the vignetting at the sample plane (i.e. reduction in image brightness towards the periphery) and another Gaussian describing the angular acceptance of the lens whose standard deviation σ_a is half the NA. We can therefore optimize the NA in terms of N (and consequently the sample-objective distance d_1) for a given lenslet geometry, material, magnification and x-ray energy. This then enables the comparison of the best practically achievable performance of CRLs of different materials geometries and configurations across the hard energy regime. Fig. 2 shows the optimum NA and the total integrated image intensity (calculated by integrating Eq. (2) with respect to r_s and w_s) from 15 to 75 keV for a selection of imaging objectives: axisymmetric lenses of Be and Al (both R=50 µm), and interdigitated lenses of PMMA (R=10 µm) and Si (R=20 µm and R=5 µm). The advantages of small-R interdigitated Si CRLs are evident above ≈ 30–35 keV, where they can be more efficient than the PMMA, Be or Al CRLs.

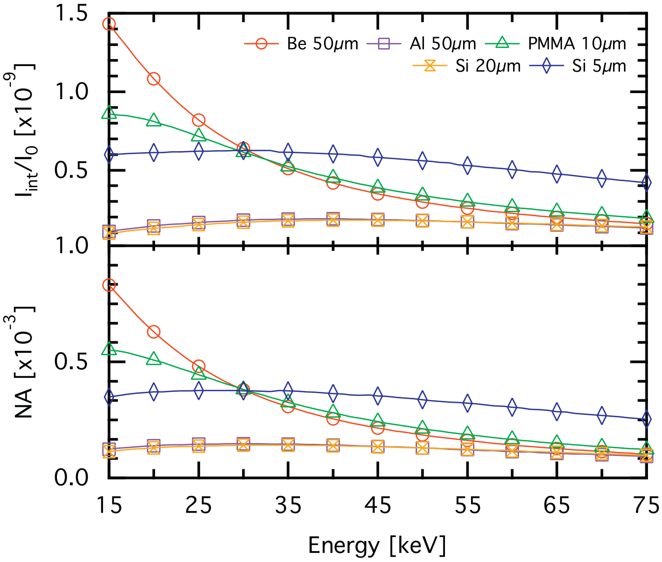

Fig. 2. NA and total integrated image intensity (I_{int}/I_0) of axisymmetric Be and Al lenses compared to interdigitated polymer and Si lenses in the hard x-ray regime. The number of lenses (and hence focal length) is optimized for each CRL and energy to produce the best compromise of NA and I_{int}/I_0. Details of the optimization routine and the CRL geometries are given in the Supplementary Material.

3. Results and discussion

We produced a prototype interdigitated lens comprising two chips of 20 1D Si lenslets with 20.52 ± 0.53 µm apex radii. The chips were produced through the standard contact UV-lithographic and deep reactive ion etching process [21] to a depth of 350 µm [22], laser-cut from the wafer and assembled on a steel gauge block using a micromanipulator and an optical microscrope. The 2D lens was tested in a full-field XRM at beamline ID06 at the European Synchrotron using x-rays of energy 17 keV (wavelength λ=0.7293 Å). The microscope was positioned 60 m from the source and 2 m from a rotating decoherer of amorphous carbon. The microscope had a magnification ratio of $\mathcal{M}$ = 11 and was intentionally diffraction-limited by the objective and oversampled by the CCD detector to reduce the contribution of the detector optics to the optical transfer function and resolution. For more details regarding the experiment, the reader is referred to the Supplementary Material.

A direct and straightforward measure of the resolution and aberration of the microscope can be made by inspecting the image of an absorbing Siemens star with radial features ranging from 5 µm to 50 nm (Fig. 3). The image shows a rapid degradation of contrast between 500 and 200 nm, which is consistent with the NA of the lens. Also visible are horizontal striations and inhomogeneous image contrast over the field of view. Both artifacts can be attributed to consistent periodic lens shape errors. Such errors could act as a phase object, damping medium-to-low spatial frequencies and resulting in this observed phase contrast [23]. While this does not reduce the ultimate resolution of the lens, it is