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Speckle analysis as a characterization tool for the focusing properties of diamond X-ray lenses

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Abstract. The availability of intense X-ray beams at megahertz repetition rate allows for new scattering and imaging experiments using the unique pulse train structure of the European X-ray Free-Electron Laser (EuXFEL). However, the resulting heat load can pose challenges for X-ray optics and potentially limit the efficiency. In this context, X-ray optics made of diamond emerge as a promising solution better suited for the extreme beam conditions at EuXFEL. In this article, we demonstrate a simple speckle analysis to determine the size of the focused beam in a caustic scan. The lenses are 1D planar diamond lenses and experiments were performed at the Materials Imaging and Dynamics (MID) station of EuXFEL. We find a minimum beam size of 630 nm and compare the speckle method with the more conventional method of wire scanning.

1. Introduction

The availability of high-brightness hard X-rays at X-Ray Free-Electron Lasers (XFELs) and the unique MHz time structure of pulse trains at the EuXFEL has made time-resolved diffraction and imaging experiments with femtosecond resolution possible [1]. The spatial coherence properties of XFEL beams are excellent and enable coherent scattering and imaging experiments which also can serve as beam characterization methods. Here we demonstrate that static speckle images produced by a static scatterer (porous Vycor glass) can be analysed to evaluate the nanofocused beam size. Recently, diamond has emerged as a promising material for X-ray optics, in particular compound refractive lenses (CRLs) at XFELs, due to its high thermal conductivity and mechanical hardness that are surpassing beryllium, which has been the reference material for CRLs, by orders of magnitude. In addition, by using diamond the toxic by-products from beryllium oxidation [2] can be avoided.

Focusing hard X-rays by refractive optics requires the beam to pass through bulk material of some thickness due to the weak interaction between X-rays and matter. Hence the material's absorption has to be minimized but also the quality of optical surfaces and the precision of the lens shape must be excellent. Only recently, improved lens manufacturing techniques and access to high quality material has made diamond a viable alternative to beryllium. Here we investigate 1D (planar) diamond lenses with nano-focusing capability in the hard X-ray range (9 keV) at the Materials Imaging and Dynamics (MID) instrument of EuXFEL. Due to the large demagnification (short focal length F and a source-to-lens distance that is typically a factor of 10^3-10^4 larger than F) and the principle of refractive lenses, the effect of spatial beam jitter, which is unavoidable for self-amplified spontaneous emission (SASE) radiation, is small in absolute terms but the focusing is chromatic and hence the ultimate performance will depend on the spectral properties of the XFEL beam[3,4].

The lens shape, surface roughness, and other critical factors in lens fabrication should be precisely controlled for an optimum lens quality and the extreme mechanical hardness of diamond makes the fabrication process challenging to achieve a surface roughness below 1 um [5]. Deviation from the ideal lens shape naturally affects the focus quality and the smallest focal spot size that can be achieved is limited by defects that might be introduced during diamond lens fabrication [6]. Polycrystalline lenses, including those made of CVD diamond, have the drawback of creating a diffuse scattering background to the focused beam waist which can be a problem, e.g. in coherent scattering experiments [7]. More generally, when coherent light is scattered from a disordered system, it produces a speckle pattern correlated with the spatial arrangement of the illuminated part of the material. Since the statistical properties of the speckle pattern depend-on the size of the illuminated volume and the coherent properties of the X-ray beam, these can be quantified by such an analysis [8–10]. Here, we demonstrate the use of a statistical analysis to evaluate the speckle size in coherent scattering images from Vycor glass sample. The speckle size is inversely proportional to the illuminating beam size produced by the planar nano-focusing lenses (NFLs) made of CVD diamond. We compare this technique with the wire scan method for characterizing the X-ray spot size.

2. Methods

2.1 Lens Manufacturing and Test Details

Experimental data on 1D NFL were collected at the MID instrument of EuXFEL [3,11]. The parabolic lens stack was fabricated in polycrystalline diamond by reactive ion etching as described in reference [5]. In this work, we present results obtained from a lens stack, consisting of 4 lenses with a 4.8 μm radius of curvature R and geometrical aperture of 30 μm. The refractive index of CVD diamond is $n = 1 - \delta$ with δ = 9.02E-6 at 9 keV [12]. The selected lens stack has a theoretical focal length of 66 mm which result in a numerical aperture of 2.3E-4 at 9 keV and 300 nm as the minimum theoretical value of spot size. During the fabrication a special tungsten (W) mask was applied to the parts of the diamond wafer that were not intended to be etched, and it remained as a top layer afterwards. The NFL was aligned vertically on a specially designed holder made of two main pieces: the main part, a copper block containing a water-cooling channel and an aluminum holder mounted to the copper block (Figure. 1b,c). For full position control the lens assembly was mounted on a hexapod (PI H.811) using magnetic kinematic base (Thorlabs). A Vycor glass (~30% porosity) piece of ~100 μm thickness was used as sample to generate the speckles in coherent small-angle X-ray scattering (SAXS).

2.2 Measurements, Data Analysis

SASE X-ray pulses of 9 keV photon energy with a relative bandwidth of approximately 0.3% were collimated and pre-focused using upstream CRL optics located in photon tunnel [3] to match the 30 μm entrance aperture of the diamond NFL. The coherent scattering images (speckles) were collected using an ePix detector with a pixel size of 50 μm placed at 10.2 m distance downstream of the sample for SAXS measurements. The NFL was aligned in the X-ray beam with the help of a high-resolution microscope (Optique Peter). Speckle patterns were collected for 3 min at each offset position of the NFL with respect to the sample (caustic scan). For comparison, wire scans were performed separately using a tungsten wire of 250 μm thickness mounted on a linear nano-positioning translation stage (SmarAct). The transmitted X-ray intensity was collected using a thin YAG imager and a Basler camera at the MID diagnostics end-station (DES) [13]. The speckle measurements were performed using 1 pulse per train with varying attenuation and radiation damage tests used unattenuated 50 pulses per train at 2.25 MHz repetition rate in the 10 Hz burst mode of EuXFEL.

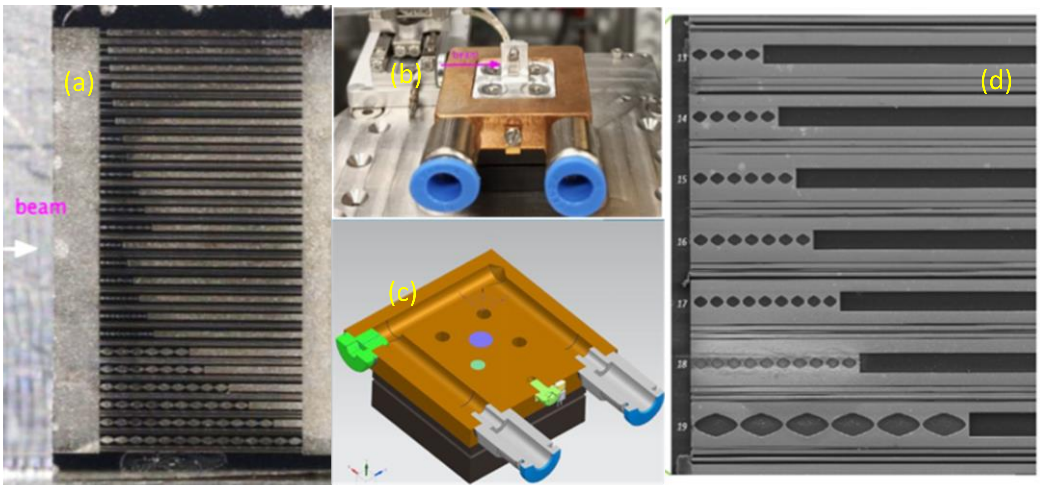

Figure 1: a) Microscopy image of the diamond NFL showing the incoming beam direction. b,c) Photo and CAD drawing of the NFL mounting stage with a cooling copper block attached to a magnetic base. d) SEM image of the NFL where lens row 13, characterized using the speckle method, and row 18, used for damage threshold tests, are visible. For the latter, beam-induced damage of the W top layer is evident.

3. Results and discussion

Figure 1a shows the diamond wafer and direction of the X-ray beam as well as the parabolic shape of the etched lens array. Figure. 1b and 1c show the lens holder and its CAD model, respectively. For the speckle measurements lens row 13 (4 lenses of R=4.8 μm) was used to focus the beam and row 18 (10 lenses of R=5.2 μm) used for damage tests with the full unattenuated beam. Both lens rows are visible in Figure. 1d showing SEM image of NFL wafer obtained after the beamtime. The lens row 13 exhibited no damage after extensive use at 1 pulse/train. For the lens row 18 exposed to damage tests the X-ray beam affected only the W overlayer without any damage to the diamond material underneath. The overlayer damage must be explained by X-ray ablation rather than melting due to the high melting point of tungsten (approximately 3422 °C) that is impossible to reach under these conditions. It is well known that the absorption cross-section per atom defines the ablation threshold and provides a real advantage of light materials in XFEL beams.

Figure 2a shows a representative speckle image recorded by the ePix detector and the corresponding 2d-autocorrelation of the image at the focus point of the NFL. The cross section of the autocorrelation function is fitted with a gaussian profile shown in figure. 2b, and the FWHM is then used to calculate the speckle size. Figure. 2c shows the caustic of a nano focused beam produced by NFL row 13 obtained by speckle measurements and wire scan technique. The measured maximum speckle size of 2.2 mm corresponds to a minimum beam size of 630 nm in the focal point.

The theoretical value of the minimum spot size is 300 nm following equation 1,

$$\Delta l_{min} = \frac{\lambda}{2NA}, \tag{1}$$

where λ is the wavelength and NA is the numerical aperture of the lens. The focal length F of 66 mm for the four lenses combined is calculated using equation 2:

$$F = \frac{R}{2N\delta}. \tag{2}$$

R is the parabola's apex radius, and N is the number of lenses in the stack [6]. The beam size d is estimated from the speckle size using equation 3:

$$d = \frac{L\lambda}{D}, \tag{3}$$

where L is the sample detector distance, d is the beam size, and D is the speckle size. As seen in Figure 2a, the elongated speckle shape in the vertical direction is caused by the focusing that takes place only in one dimension (vertical) for the manufactured planar lens rows. Figure 2b shows a cross section of the 2D-spatial autocorrelation function from which the speckle size can be calculated. As seen in Figure 2b a gaussian fitting curve describes the profile of autocorrelation function well. The beam size is then calculated from the speckle size using (3) as shown in figure. 2c. At the focus point, the focal spot size exceeds a theoretical value by a factor of 2, which can be expected due several factors including imperfections of lens manufacturing [7], spatial jitter and spectral bandwidth of SASE beam. A wire scan was performed for every position of the lens where the transmitted X-ray intensity was recorded as a function of the vertical wire position crossing the focused X-ray beam. The wire scan data were analysed, first by smoothing the intensity fluctuations using a Savitzky-Golay filter, then by differentiation of the smoothed curve which is fitted by a gaussian profile where the FWHM is taken as the beam size. The result is plotted in figure. 2c at different lens positions together with the beam size estimates from the speckle method. The minimum value obtained by the wire scan method is about 1.5 µm which is larger than the one obtained by the speckle analysis. Several factors, including XFEL beam jitter influence the larger spot size from the wire-scan. The primary X-ray beam jitter is minimized in one dimension because of the 1D lens stack's magnification. However, an additional jitter occurs in the focal plane due to the incidence angle not aligning with the lens stack's axis. The dynamic distortion of the beam profile leads to a non-constant intensity distribution during scanning, particularly if the roughness of the scanning wire is included.

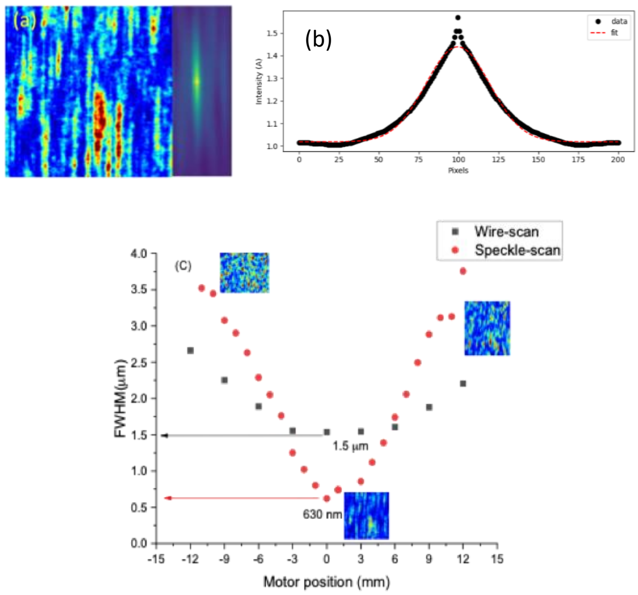

Figure 2: a) ePix detector image of summed frames showing the static speckle pattern from a Vycor glass, and the result of applying a 2d spatial autocorrelation calculation. b) Vertical cross-section of the autocorrelation. The red line is a Gaussian fit to extract the width of the autocorrelation. c) Caustic scan of the planar lens stack, the inset shows the speckles at the corresponding positions. The black squares are corresponding wire-scan results.

4. Conclusion

We have characterized the focusing capabilities of a planar diamond 1D lens for XFEL applications using speckle-based analysis and wire scanning. The difference between the values obtained from the wire scan method and the speckle analysis suggested a higher resolution of the speckle-based method. The speckle analysis of caustic scan provided 630 nm as a minimum spot size in comparison of the wire scan method that provided systematically larger values. The advantage of speckle analysis method as pulse-resolved diagnostic of nanofocused XFEL beam can be further explored using MHz area detectors.

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