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Towards Soft X-Ray Phase-Sensitive Imaging with Diffractive Optical Elements

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In this contribution we present the first diffractive optical elements for soft x-ray differential interference contrast microscopy. Due to an improved calculation method the nanofabrication accuracy of these optics is the same as for comparable normal zone plate optics with the same outermost zone width. Different diffractive optical elements were fabricated with outermost zone width of 100 nm, different spot separation directions and different phase relations between the two spots. The optics were successfully used in experiments both at the synchrotron radiation based TWINMIC microscope and at the Stockholm compact liquid-nitrogen laser-plasma source based microscope.

KEYWORDS: X-ray microscopy, differential interference contrast, diffractive optics

1. Introduction

In the x-ray region, for nearly all elements the real part delta of the complex index of refraction n ($n = 1 - \delta + i\times\beta$) is larger than the imaginary part beta. It follows that the phase shift of any object is stronger than the absorption. Thus, phase sensitive x-ray imaging techniques like Zernike phase contrast x-ray microscopy [1),2)] were developed to access the phase shifting, real part of the refractive index of the object in order to examine thin and weakly absorbing samples with sufficient contrast. Another alternative approach was the use of segmented detectors in scanning x-ray microscopes [3)]. Recently, the differential interference contrast (DIC) method – first introduced by Nomarski [4)] for visible light – was demonstrated with so call twin zone plate optics (TZP) at 4 keV photon energy [5)]. However, these elements are difficult to fabricate. A possible solution to this problem is the combination of the two zone plates into a single diffractive optical element (DOE). First proof of principle experiments with this kind of optics for DIC-microscopy at 4 keV photon energy were successful [6)] and showed the strong potential of DOE x-ray optics.

In this paper we expand the concept of DIC-microscopy in full-field transmission mode with DOE optics into the soft x-ray range. We used an improved calculation method to design high-resolution DIC-objectives. The optics were manufactured by means of nanofabrication methods. First experiments with a compact laser plasma source and with synchrotron radiation using the TWINMIC microscope successfully proofed the functionality of the DOEs.

2. Design and manufacturing of diffractive optical elements

As starting point for the design of our diffractive optical elements we used a concept presented by di Fabrizio et al. [6)]. A single x-ray DOE acts like a normal zone plate but produces two slightly shifted foci acts as diffractive amplitude beam splitter and x-ray objective simultaneously. The result is a differential image division in the detection plane that leads to differential interference contrast. Our calculation of a DOE pattern is similar to the one described in Ref 6). A one dimensional phase function Φ (modulus 2π) is calculated by wavefield propagation of two spherical monochromatic waves originating from two slightly displaced point sources. This point sources can be positioned perpendicular to the optical axis (lateral shear) or along the optical axis (axial shear). Additionally, a phase difference between the two points can be defined (bias). A binary pattern is achieved with the conditions $0 \leq \Phi < \pi \Rightarrow 0$ and $\pi \leq \Phi < 2\pi \Rightarrow 1$. Zero indicates the area of the DOE with no material and 1 indicates the area with material. Afterwards the one dimensional pattern is extrapolated into two dimensions by symmetry considerations. The pattern appears as a normal zone plate pattern but with an overlaid linear phase grating [7)] (see Fig. 1).

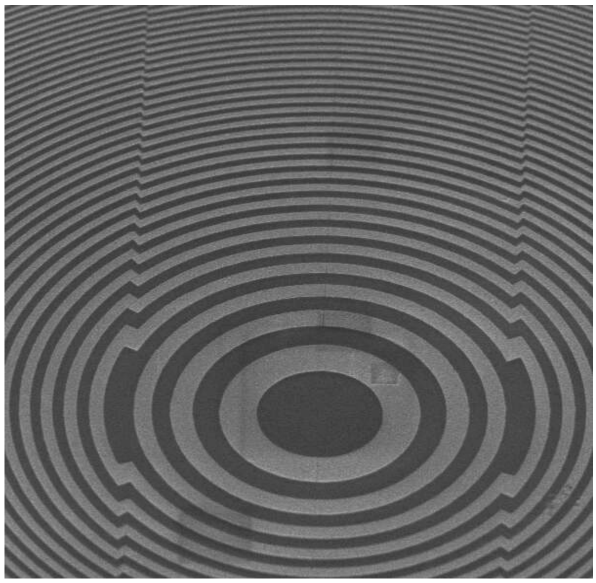

Fig. 1: Electron microscope image of the inner part of a diffractive optical element. Note the phase reversal of the zones in the left and right part of the image.

The DOEs are manufactured by a standard nanofabrication process also used for the production of normal zone plates [8)]. The key steps are the generation of the pattern by means of electron-beam lithography and the electroplating of the final optical element in nickel with a thickness of 150-200 nm. Different DOEs were produced with a common diameter of 75 μm and outermost zone widths of 100 nm, resulting in focal lengths of 3 mm at 2.4 nm wavelength.

In order to test the fabricated DOEs, different experiments were performed. As light sources a laser plasma and synchrotron radiation were used.

2. Experiments with laser plasma source

The general experimental arrangement, also used for the experiments described in the next section, is sketched in Fig. 2.

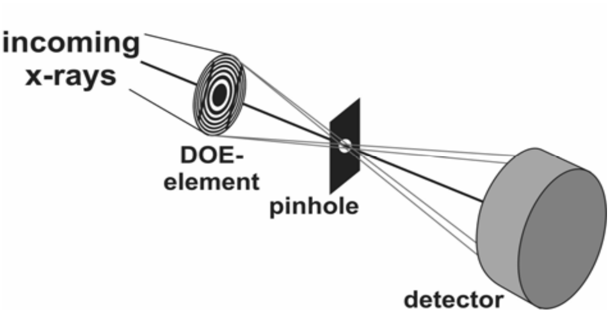

Fig. 2: Experimental arrangement for the testing of the diffractive optical elements.

In case of the laser plasma, the x-ray emission from a liquid-nitrogen jet target was filtered with a 300 nm thick titanium foil to isolate the 2.88 nm emission line from the rest of the spectrum. This resulted in a sufficient degree of temporal coherence needed for the experiment. The necessary spatial coherence was achieved by placing the DOE in a sufficient distance (25 cm) away from the plasma. The used DOE had an outmost zone width of 100 nm and a lateral shear spot separation of 600 nm. The pinhole, located in the focal plane, blocked away all other diffraction orders except the $+1^{st}$ order. The light was detected by an x-ray CCD camera in a distance of 25 cm from the pinhole. To avoid absorption of the radiation, the experiment was performed under vacuum conditions.

An image obtained on the CCD, which can be considered as an interferogram produced by the focal spots in the focal plane, is shown in Fig. 3. It exhibits a basic modulation with a period of 1.2 mm that is the expected period given the spot separation and distance to the detector. Moreover, the interferogram contains also additional modulations with higher frequencies (see line plot 3). These modulations are a direct consequence of the binary nature of the produced DOE. In the focal plane of the binary DOE a number of side peaks appear in addition to the two main focal spots. Thus, these side peaks lead to the observed modulations with higher frequencies. This explanation could be confirmed by theoretical wavefield propagation calculations. Additionally, an imaging experiment clearly showed enhanced edge contrast [9].

3. Experiments with TWINMIC

The experiments described in the previous section proofed the functionality of the DOE designing and fabrication method. However, this optic cannot be used for standard differential interference contrast microscopy. For this the spot size separation in the focal plane must be smaller than the outermost zone width [5] which was not achieved with the used DOE. Therefore new sets of DOEs suited for DIC experiments were designed and manufactured.

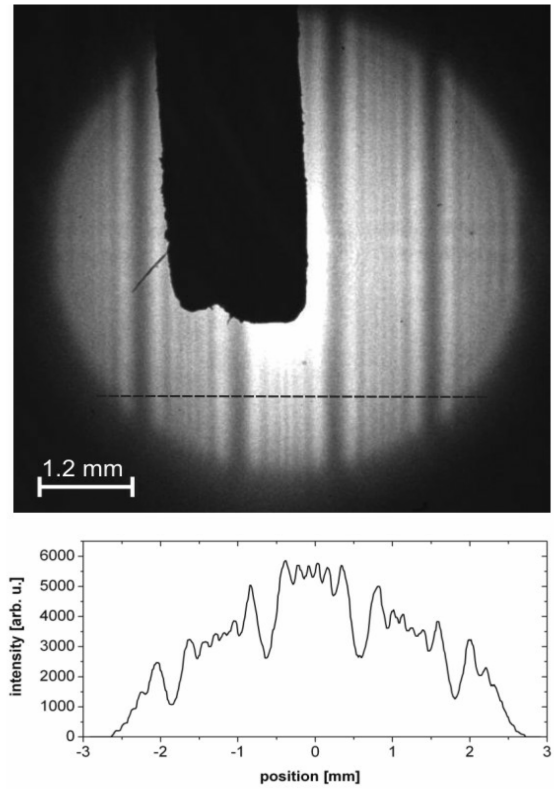

Fig. 3: Interferogram obtained on the CCD detector with an exposure time of 120 s. The dashed line indicates the position of the line plot through the image which is shown below. The large black shadow is the central stop to block the zeroth order [9].

One set consists of nine optics in total, three normal zone plates, three DOEs with lateral spot separation of 75 nm and three DOEs with axial spot separation. The bias was set to π. The common outermost zone width was again 100 nm The advantage of having a normal zone plate and a DIC-DOE on the same wafer is that they can easily be exchanged during an microscopy experiment and a direct comparison of the results can be made.

The optics were tested using the principle setup of Fig. 2, but with synchrotron radiation using the TWINMIC x-ray microscope [10] located at the BACH beamline of the ELETTRA storage ring in Trieste, Italy. The undulator radiation at 1.72 nm was monochromatized by means of a grating monochromator to $\lambda/\Delta\lambda > 6000$ which is sufficient for the temporal coherence needed. A secondary source was formed after the beam is focused by a toroidal mirror, and the distance of 2.5 m between the intermediate focus and the DOE set resulted in a complete spatial coherent illumination of the optics. To detect the interferograms, a phosphor screen in combination with a visible light optics and fast read-out CCD camera was employed.

Fig. 4 shows the results obtained for the three types of optics. In Fig. 4 a) the $+1^{st}$ diffraction order cone of the normal zone plate can be seen. The bright area in the center is the unblocked 0^{th} order. Fig. 4b) is the interferogram of the lateral shear DOE. It exhibits only a single interference fringe in the center. The position of the fringe in the center is a direct consequence of the shear value of π, while the small

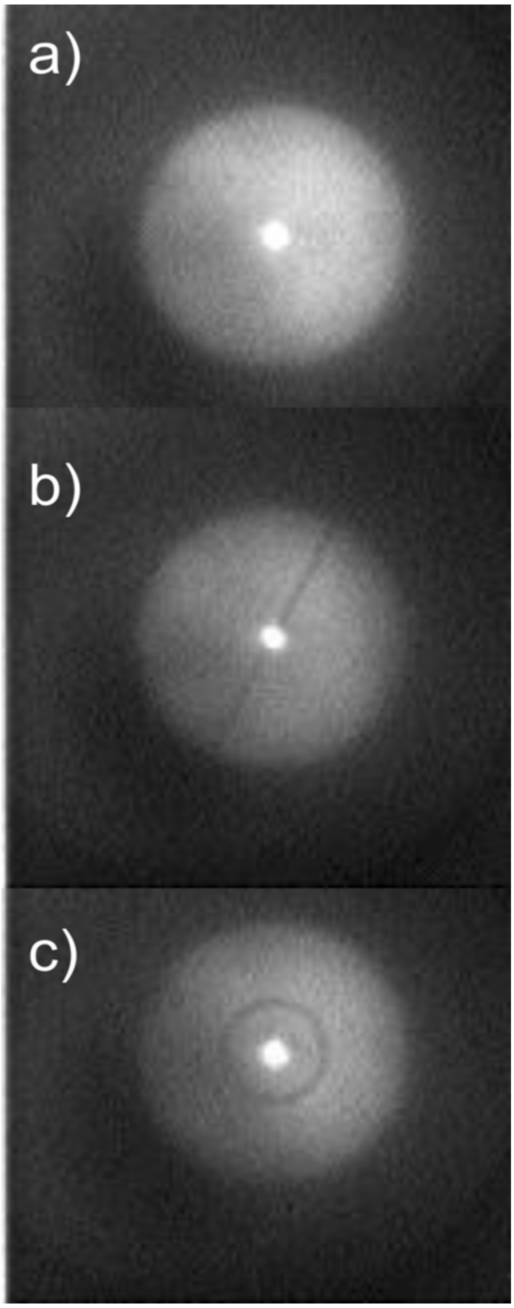

Fig. 4: Interferograms obtained with DOE suited for DIC microscopy: a) normal zone plate +1st order cone. b) lateral shear DOE, bias π. c) axial shear DOE, bias π. For details see text.

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width is again due to the binary DOE pattern. Consequently, for the axial shear DOE also a single small interference fringe is visible in Fig. 4c), but now as a ring around the center. These are exactly the interference patterns that are theoretically expected for these DOE designs.

4. Conclusions

In summary, we present the successful design, manufacturing and testing of a diffractive optical elements for operation at water-window soft x-ray wavelengths. The DOE with small shear distances will be the basis for advanced phase-sensitive differential interference contrast imaging experiments in the soft x-ray range, both in combination with laser-plasma based microscope and the TWINMIC using synchrotron radiation.

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