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High-resolution hard-x-ray microscopy using second-order zone-plate diffraction

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Abstract
Odd-order diffraction of zone plates (ZPs) is already used for x-ray microscopy but the potential offered by even-order diffraction must still be fully exploited. Width differences between lines and interline spaces transfer intensity from odd-order to even-order diffractions. Here we show that the resulting intense second-order diffraction provides a reasonable tradeoff between spatial resolution and intensity—and constitutes a viable strategy for x-ray microscopy to reach sub-20 nm resolution, in spite of the imperfections of high-aspect-ratio ZPs and of other difficulties.

(Some figures in this article are in colour only in the electronic version)

The resolution of zone-plate (ZP) transmission x-ray microscopy is ideally given by $\alpha\Delta r/m$, where Δr is the outmost zone width, m the diffraction order and α a factor determined by the optical aberrations and the coherence level of the radiation [1]. The first-order diffraction resolution can be increased by decreasing Δr [2–4]. However, the production of ZPs with zone widths below 20 nm is a real nanofabrication challenge. The resolution can also be enhanced by optimizing the ZP illumination with a numerical aperture (NA) matching that of the ZP: this can yield a resolution twice better than parallel illumination [5].

Finally, the resolution increases when using high diffraction orders [6, 7]. However, this occurs at the expenses of intensity. Indeed, using the optically thin grating approach and the Kirz formula [8], the diffraction efficiency for the $2n$ order (n = integer) for a perfect ZP is zero and that for the $2n+1$ order is $\eta_{2n+1} = \eta_1/(2n+1)^2$. If the ZP has fabrication imperfections, there is also second-order intensity [9].

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High-resolution ZP imaging with third-order diffraction was experimentally demonstrated for both soft [5] and hard [6] x-rays. However, the low intensity and the stringent requirements for the ZP structure accuracy create problems.

Here we report on a strategy based on high-efficiency second-order ZP diffraction and demonstrate that the imperfections in the ZPs can be so exploited as to obtain significant advantages compared with third-order imaging. The experiments were performed with a full-field transmission hard x-ray microscope (TXM) facility at the Advanced Photon Source (APS), Argonne National Laboratory [10]. A standard APS A-type undulator supplies x-rays filtered by a Si(1 1 1) double-crystal monochromator and a pair of parallel flat total-reflection mirrors. The setup includes a capillary condenser focusing x-rays onto the sample, a pinhole eliminating unwanted illumination, a precision sample stage, a ZP objective magnifying the sample images, the detector (a thin CsI scintillator and a 10× lens with a CCD detector) and a phase ring which is used to obtain Zernike contrast [10, 11].

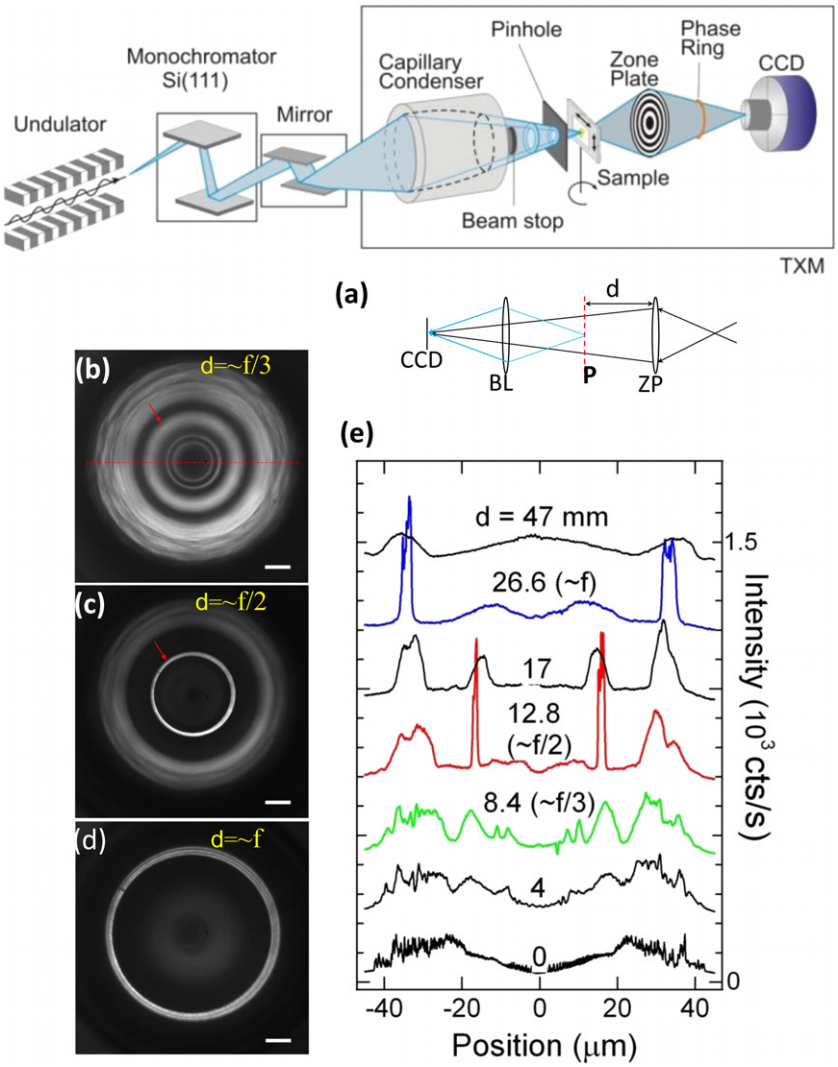

Figure 1. Measurements of the diffraction of a ZP objective: (*a*) schemes of (top) the optical layout of the transmission x-ray microscope and (bottom) the technique using a BL, (*b*)–(*d*) representative condenser conjugate images for the third, second and first diffraction orders, and (*e*) intensity profiles across the diameter of the annulus (see (*b*)) as a function of the distance d after the ZP (focal distance f). Scale bar: 10 μm.

We first discuss the efficiency of second-order diffraction. To effectively measure the diffraction intensity, we developed a direct imaging method using a Bertrand lens (BL) [12]. The optical layout is shown in figure 1(*a*): the lens was located between the ZP and the CCD, so that it can directly image the illuminations at a given plane (P) distant d from the ZP. Combined with the translation of the lens, this setup can effectively investigate the ZP diffraction properties.

An example is shown in figures 1(*b*)–(*e*) for a ZP made of Au with an outmost zone width $\Delta r = 45$ nm, an 85 μm diameter (D) and a thickness (t) of 800 nm at 8 keV [13, 14]. The three images at (*b*) $d = \sim f/3$, (*c*) $\sim f/2$ and (*d*) $\sim f$ (f = the focal distance of the ZP) correspond to the third, second and first-order diffraction. Note the variations in brightness and width of the annular illumination with d. The illumination at the third-order conjugate image (figure 1(*b*)) exhibits four annuli, corresponding the first order and higher orders. Their origin can be derived from their focal properties. The widest annulus is focused at the first-order conjugate plane as shown in figure 1(*d*), demonstrating that it is due indeed to first-order diffraction. Likewise, figure 1(*c*) shows that the second widest annulus is due to second-order diffraction; its diameter is 34 μm, exactly half of the first-order annulus. Note that its intensity is quite high for an even-order diffraction [7].

Figure 1(*e*) shows the intensity profiles across the diameter of the annuli as a function of the distance d. Note in particular that the second-order peak ($d \approx f/2$) is higher than the third-order peak ($d = \sim f/3$).

High-intensity second-order diffraction was confirmed for different ZPs including commercial devices ($\Delta r = 40, 45$ nm). Figure 2 shows the results. The diffraction efficiency was obtained by integrating the intensities of the condenser-focused incident illuminations and the ZP-transmitted and diffracted illuminations in the BL images. The values for five different ZPs used to obtain second-order images were evaluated and compared with calculated value based on ideal ZP structures. The absorption is shown by the data points on the left-hand side; the last data on the right-hand side (marked as 'Etc')

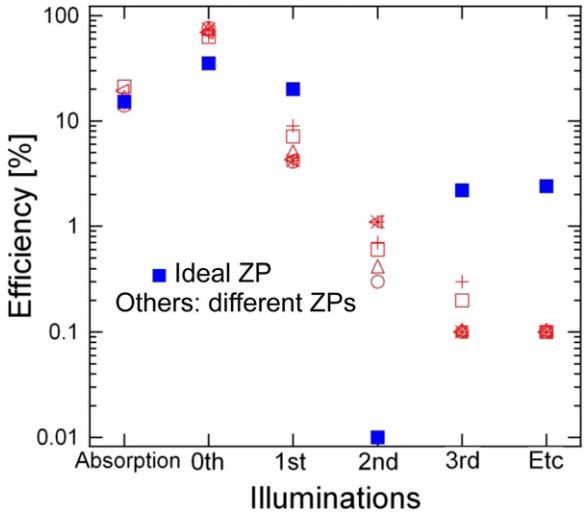

Figure 2. Measured and calculated diffraction efficiency. The ZP thickness was 800–900 nm of Au and the outmost zone width 40 and 45 nm. Note the high efficiency of the second-order diffraction.

correspond to the transmitted intensity minus the intensities of the zeroth, first, second and third-order diffraction. All ZPs exhibit a high efficiency (0.3–1.2%) of the second-order diffraction, again much higher than the third order (0.1–0.3%).

Why is second-order diffraction, theoretically zero for perfect ZPs, actually more intense than third-order diffraction? Dynamical analysis for soft x-rays indicated that a high ZP aspect ratio (>30) can transfer intensity into higher diffraction orders [15–17]. However, this does not apply to our case since we use hard x-rays for which the effect is negligible. Instead, the likely cause of the intensity transfer is the difference between the widths of the ZP lines and spaces. Deviations from a 1 : 1 line-to-space ratio move intensity into even orders at the expense of odd orders, in particular the first order [18].

The high-intensity second-order diffraction can be used for high-resolution imaging. A ZP ($D = 85\,\mu$m, $\Delta r =$ 45 nm) was positioned so that the sample was at $f/2$. An aperture (50 μm in diameter) at the first-order conjugate plane was used to block the first-order illumination. The x-ray magnification so achieved was 140: combined with the 10× magnification of the detector lens, this resulted in a combined total magnification 1400 and a pixel resolution of 4.9 nm.

Figures 3(*a*) and (*b*) compare x-ray micrographs of a 50 nm thick resolution test pattern obtained with first and second-order diffraction. The resolution improvement with second-order diffraction is quite evident—note, for example, the features marked by arrows. The separation of detailed structures is clearly visible with second-order diffraction. Furthermore, second-order diffraction provides better contrast and image quality. This can be explained by the fact that the large NA of the second order makes it possible to collect a larger angular range of scattered signal from sample than the first order.

The image improvement with second-order diffraction was quantitatively characterized by power spectrum analysis. The result in figure 3(*c*) shows that the cutoff feature width is 22 nm, better than the first-order value of 29 nm. In addition,

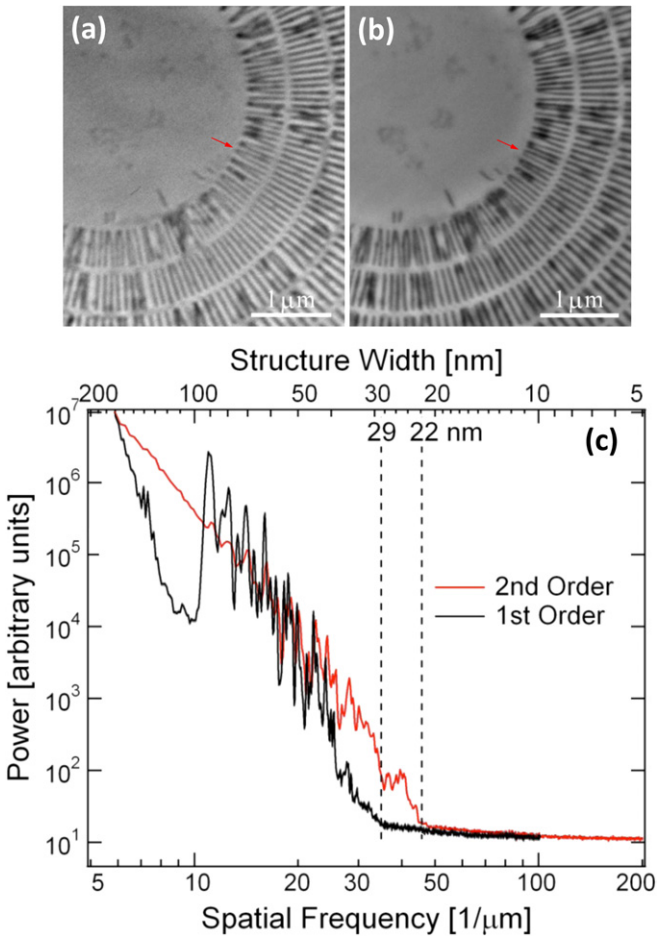

Figure 3. (*a*), (*b*) X-ray micrographs of a 50 nm thick Au resolution pattern with an innermost feature width of 30 nm fabricated by Academia Sinica : (*a*) first-order image and (*b*) second-order image. (*c*) Power spectra obtained for (*a*) and (*b*). The cutoff frequency corresponds to a 22 nm feature size for the second order, and to 29 nm for the first order.

the power intensity is much higher than that of first order over the entire spatial frequency range of interest. These results confirm the qualitative conclusion that second-order diffraction enhances both the resolution and the contrast. The measured enhancement in contrast between figures 3(*a*), ∼7%, and, (*b*), ∼4%, is ∼75%.

Theoretically, the second-order imaging should double the spatial resolution. However, we reached only ≈50% of this theoretical value. The main reason is the NA of our condenser illumination. Indeed, we used the same capillary condenser designed for first-order imaging—whose NA (1.4 mrad at 8 keV) was ≈41% of that of the ZP (3.4 mrad for second-order diffraction). Even better resolutions, closer to the theoretical values, should be possible with better NA matching [6, 7].

Second-order ZP diffraction imaging offers several advantages. First, due to its high efficiency, second-order diffraction can provide a reasonable balance between resolution and intensity. Third-order diffraction can give higher resolution, but its low diffraction intensity is a problem. The separation of the second-order illumination from higher orders is large enough for easy control, while for third order it is more problematic. Finally, the tolerance of the

ZP profile for second-order diffraction is more relaxed than for third-order diffraction—making it more robust for high-resolution imaging.

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References

[1] Attwood D 1999 *Soft X-Rays and Extreme Ultraviolet Radiation: Principles and Applications* (Cambridge: Cambridge University Press)
[2] Chao W, Chao W, Harteneck B D, Liddle J A, Anderson E H and Attwood D T 2005 *Nature* **435** 1210
[3] Chen Y T *et al* 2008 *Nanotechnology* **19** 395302
[4] Yun W, Lai B, Krasnoperova A, Di Fabrizio E, Cai Z, Cerrina F, Chen Z, Gentili M and Gluskin E 1999 *Rev. Sci. Instrum.* **70** 3537
[5] Jochum L and Meyer-Ilse W 1995 *Appl. Opt.* **34** 4944
[6] Rehbein S, Heim S, Guttmann P, Werner S and Schneider G 2009 *Phys. Rev. Lett.* **103** 110801
[7] Yin G-C, Song Y-F, Tang M-T, Chen F-R, Liang K S, Duewer F W, Feser M, Yun W and Shieh H-P D 2006 *Appl. Phys. Lett.* **89** 221122
[8] Kirz J 1974 *J. Opt. Soc. Am* **64** 301
[9] Lima E and McNulty I 2010 *Poster presentation in XRM, Chicago, IL*
[10] Chu Y S *et al* 2008 *Appl. Phys. Lett.* **92** 103119
[11] Chen Y T, Chen T Y, Yi J, Chu Y S, Lee W-K, Wang C L, Kempson I M, Hwu Y, Gajdosik V and Margaritondo G 2011 *Opt. Lett.* **36** 1269
[12] Murphy D B 2001 *Fundamentals of Light Microscopy and Electronic Imaging* (New York: Wiley-Liss)
[13] Lo T N *et al* 2007 *J. Phys. D: Appl. Phys.* **40** 3172
[14] Chen Y T *et al* 2008 *J. Synchrotron Radiat.* **15** 170
[15] Schneider G 1997 *Appl. Phys. Lett.* **71** 2242
[16] Hambach D, Schneider G and Gullikson E M 2001 *Opt. Lett.* **26** 1200
[17] Kurokhtin A N and Popov A V 2002 *J. Opt. Soc. Am.* A **19** 315
[18] Michette A G 1986 *Optical Systems for Soft X-rays* (New York: Plenum)