Sub-25-nm laboratory x-ray microscopy using a compound Fresnel zone plate

Olov von Hofsten,* Michael Bertilson, Julia Reinspach, Anders Holmberg, Hans M. Hertz, and Ulrich Vogt

Biomedical and X-ray Physics, Department of Applied Physics, Royal Institute of Technology, Albanova, SE-10691 Stockholm, Sweden

**Corresponding author: olov.hofsten@biox.kth.se*

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Improving the resolution in x-ray microscopes is of high priority to enable future applications in nanoscience. However, high-resolution zone-plate optics often have low efficiency, which makes implementation in laboratory microscopes difficult. We present a laboratory x-ray microscope based on a compound zone plate. The compound zone plate utilizes multiple diffraction orders to achieve high resolution while maintaining reasonable efficiency. We analyze the illumination conditions necessary for this type of optics in order to suppress stray light and demonstrate microscopic imaging resolving 25 nm features. © 2009 Optical Society of America

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High-resolution x-ray microscopy using zone plates as the objective lens has emerged as a valuable tool for nanoscale imaging. With applications in biology, soil science, and nanomagnetism, the technique has been shown to complement such existing imaging methods as electron microscopy and scanning-probe microscopy [1]. So far, 15 nm lines have been resolved [2], and much effort is now taken to achieve sub-10-nm resolution, a major challenge for both the nanofabrication of the optics as well as the optical system design. Most x-ray microscopes are synchrotron based, but laboratory instruments also exist [3,4]. Here, the photon flux is limited, which makes efficient optics important. We present the implementation of a so-called compound zone plate in a laboratory x-ray microscope. A compound zone plate is capable of both high resolution and reasonable efficiency without placing extreme demands on the nanofabrication process. The technique is compared to conventional zone-plate imaging.

The key imaging element in a soft x-ray microscope is the zone plate, which is a circular diffraction grating with radially decreasing grating period. The full-period resolution of such an optic can be shown to be $\sim\Delta r/m$ [5], where Δr is the outermost zone width and m is the diffraction order of the grating. In conventional zone-plate imaging, the zone plate is used in the first diffraction order, which places high demands on the nanofabrication of the optics, since Δr is typically 20–30 nm. Alternatively, the third diffraction order can be used to achieve high resolution [6]. However, the efficiency of a zone plate scales with $1/m^2$, which makes third-order imaging difficult in laboratory microscopes.

The use of a compound zone plate in x-ray microscopy was first proposed by Simpson and Michette [7] and later tested by Kuyumchyan *et al.* [8], in a nonimaging setup. The concept of a compound zone plate is illustrated in Fig. 1. It consists of a zone plate used in the third diffraction order, with a diameter D, where the central area, with a diameter $D/3$, is replaced by a zone plate used in the first diffraction order. Since the focal length of a zone plate is given by $f=(D\Delta r)/(\lambda m)$, where D is the diameter and λ is the wavelength [5], the two integrated zone plates with the given diameter ratio will yield the same focal length if they have the same outermost zone width, which is set by fabrication limits. The result is an optic with a larger NA without a decreased outermost zone width and an overall efficiency that is higher than when using only the third diffraction order.

In this Letter we analyze the illumination requirements for compound zone-plate imaging in our laboratory microscope and evaluate the technique. Most importantly, nonimaging orders of the zone plate will create stray light that must be removed or reduced. This is especially important when using a laboratory x-ray microscope, since the available photon flux is limited.

The most prominent nonimaging order is the zeroth order, which is typically removed from the field of view by a central stop placed immediately af-

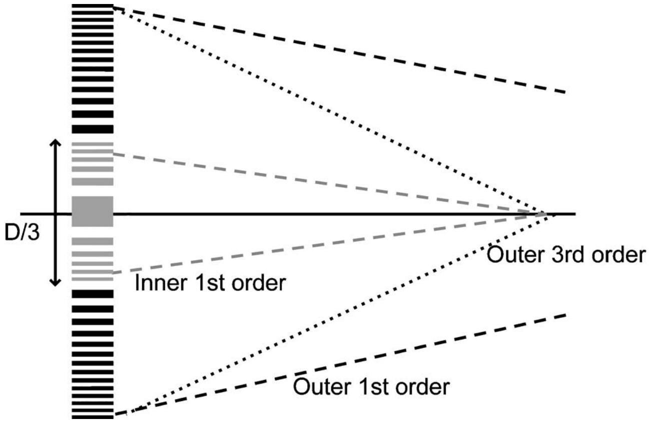

Fig. 1. Illustration of a compound zone plate. The inner first order and outer third order combine in the focus to yield a high NA. The inner and outer parts have equal outermost zone width, and the diameter of the inner part is one third of the entire zone plate.

ter the source, giving a hollow-cone illumination. This is illustrated in Fig. 2, where a schematic of the x-ray microscope is shown. In order to analyze and evaluate the role of stray light resulting from other nonimaging orders, ray-tracing simulations were made. The simulation starts on the 58 mm diameter condenser, which images the source onto the sample, and rays are traced through the optical system. The distance from source to the condenser mirror is 282 mm, and the distance from mirror to sample is 462 mm. The x-ray source has a diameter of 25 μm and is assumed to be Gaussian, and the intensity of each ray is corrected accordingly. The results for a compound zone plate when using a 5 mm central stop, which is our standard arrangement, are shown in Fig. 3(a). The two imaging orders (inner first and outer third) are separated to show that the amount of stray light exceeds the light focused by the third order. The stray light consists of three contributions: (A) the first order from the outer part of the zone plate, (B) the −1st order from the outer part, and (C) the −1st order from the inner part. The last contribution is not inherent of the compound zone plate but is always present when using a zone plate with a short focal length.

A solution for the reduction of the stray light was developed with the help of the simulations. Contributions (B) and (C) can be eliminated by using a larger central stop. There is a trade-off between a larger field of view and a reduction in photon flux that must be considered. A suitable configuration was to increase the diameter of the central stop from 5 to 10 mm. The result of this arrangement is shown in Fig. 3(b). As seen, the inner first-order light is reduced, as the central stop is blocking most of the rays hitting the inner part of the zone plate. Stray light is also significantly reduced, since a large central stop will remove rays heading for the back focal planes of the −1st orders. It will also remove most of the stray light produced by (A). The remaining stray light originating from (A) may be removed by placing an absorbing plate in the front focal plane of the first order of the outer part. This OB is imaged by the order onto the CCD and will therefore remove any remaining stray light in the corresponding area. This is illustrated in Fig. 3(c), showing that the combination of a larger central stop and an OB results in a nearly stray-light-free illumination. In the central area of the CCD, only the imaging orders are present. This is important for high-resolution imaging in laboratory x-ray microscopy.

Two types of zone plates were manufactured: one compound zone plate, where the inner and outer part have an outermost zone width of 50 nm; and one standard zone plate, also with 50 nm outermost zone width. The diameter of both zone plates is 60 μm. The fabrication process involves nickel electroplating of an electron-beam written mask and is described in detail elsewhere [9]. The zone plates were then used as objectives in our laser-plasma-based laboratory soft x-ray microscope [10,11], following the arrangement shown in Fig. 2. The wavelength used in the experiment was 3.37 nm, and a multilayer mirror was used as condenser. The compound zone plate has a focal length of 300 μm, giving a magnification of 2430. For comparison, the standard zone plate had a focal length of 900 μm, and the magnification was 1160. Note that, since the compound zone plate uses third-order imaging, it has an effective outermost zone width of 16.7 nm. The OB (15 μm diameter) and the test samples were electroplated in gold with a thickness of 100 nm.

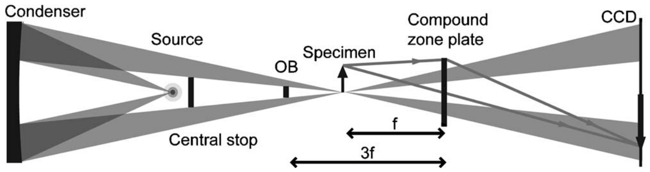

Fig. 2. Schematic of the microscope arrangement when using a compound zone plate. The order blocker (OB) is placed in the outer part's first-order focal plane.

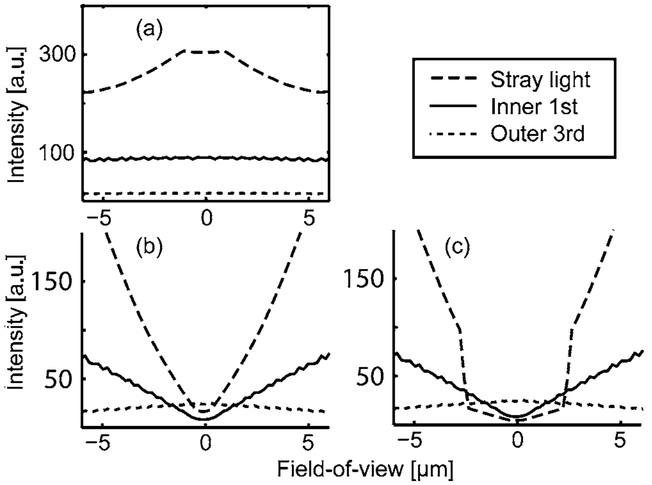

Fig. 3. Results showing the stray-light simulations for a compound zone plate: (a) situation without a large central stop and order-blocker, (b) using the larger central stop, (c) using the large central stop and the order blocker, where the central area of the detector is stray-light-free.

Figure 4 shows the images collected using the compound zone plate and the standard zone plate. The object consists of 25 and 30 nm gold lines. The OB

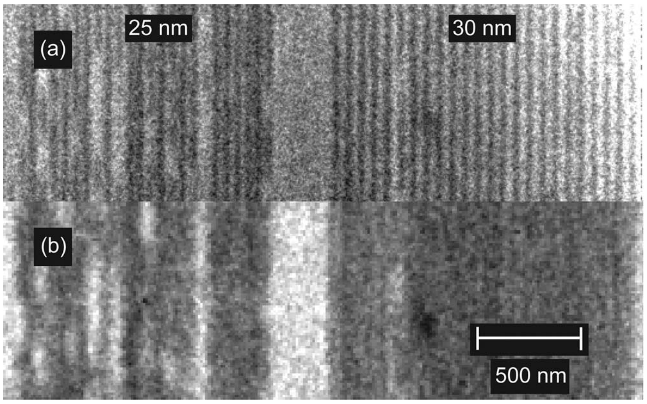

Fig. 4. Images of Au gratings taken with the laboratory soft x-ray microscope: (a) the compound zone plate resolves the (left) 25 nm lines and (right) 30 nm lines, (b) image using a conventional zone plate with 50 nm outermost zone width.

was used together with the compound zone plate, and the size of the central stop was 10 mm. The compound zone plate image [Fig. 4(a)] shows clearly resolved 30 and 25 nm structures. To reduce noise and drift in the image, 15 individual 1 min exposures have been aligned and averaged. The same structures imaged with the standard zone plate are shown in Fig. 4(b). In this case the central stop was 5 mm, and the total exposure time was 5 min. The 30 nm lines are barely visible, which is close to diffraction-limited performance for this zone plate. There is a clear improvement when using the compound zone plate owing to the third-order imaging of the outer zones.

To illustrate the effect of the order-blocker, Fig. 5 shows an image of a Siemens star using the compound zone plate. The 15 μm OB defines the field of view and limits it to about 5 μm, which is a drawback of the technique. Outside the OB, the stray light is so prominent that the contrast of the thicker lines in the object is lower than the smaller lines imaged within the OB area. The OB is therefore necessary for high-resolution soft x-ray imaging when using a compound zone plate.

Images were also acquired using the standard zone plate in third order, but owing to the low efficiency of this imaging technique it is difficult to make a valid comparison. However, a zone plate used in third order also has stray light sources originating from the −1st and first orders, and therefore an OB in combination with a larger central stop can be used to suppress stray light and improve the image contrast.

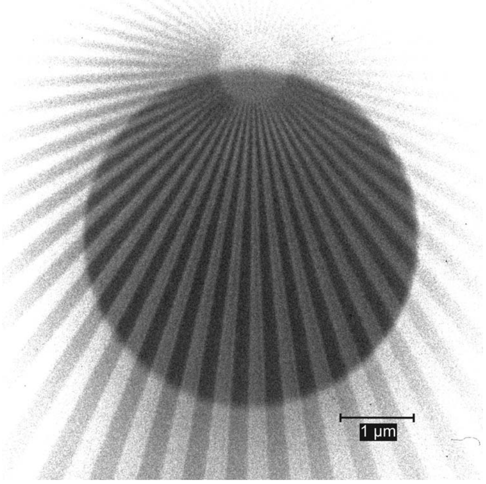

Fig. 5. Image of a Siemens star using the compound zone plate. The order blocker is imaged by the outer part's first order. The stray light outside the OB is significant, and the field-of-view is limited to about 5 μm.

We have shown successful high-resolution imaging using a compound zone plate in a laboratory soft x-ray microscope. By evaluating the illumination and stray-light conditions, we could show that a larger central stop combined with an OB is necessary for high-resolution imaging. This result also applies to synchrotron-based instruments, where third-order imaging is of interest, but since stray-light conditions may vary, a full evaluation must be carried out for each microscope. The compound zone plate had an outermost zone width of 50 nm, and 25 nm features were observed, that were higher than previously demonstrated in any laboratory arrangement. A compound optic has a potential for even higher resolution, e.g., with 25 nm outermost zone width, although this will require a more-stable imaging arrangement and a brighter source. Improvements are currently being made to the microscope in order to fulfill these requirements.

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