

Development of a hard x-ray imaging microscope

B. Lai, W. Yun, Y. Xiao, L. Yang, D. Legnini, and Z. Cai
Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439

A. Krasnoperova and F. Cerrina
Center for X-Ray Lithography, University of Wisconsin-Madison, Stoughton, Wisconsin 53589

E. DiFabrizio, L. Grella, and M. Gentili
CNR-IESS, Via Cineto Romano 42, 00156 Roma, Italy

(Presented on 21 July 1994)

A hard x-ray imaging microscope based on a phase zone plate has been developed and tested. The zone plate, with a 5 cm focal length and a $0.2\ \mu\text{m}$ smallest linewidth, was used to image 8 keV x rays from the samples. The imaging microscope can be used to obtain nearly diffraction-limited resolution over the entire imaging field, and its resolution is almost independent of source size and source motions. We have tested such an imaging microscope, and a resolution of about $0.4\ \mu\text{m}$ was obtained. The images were obtained with an exposure time of less than 1 min, for a magnification factor of 30 in the x rays. The x rays were then converted into visible light, and another 7 times magnification were obtained by using a lens system coupled to a charge coupled device camera. The results from the imaging microscope, and possible applications, will be discussed. © 1995 *American Institute of Physics*.

INTRODUCTION

Two types of x-ray microscopes have been developed using zone plates as basic focusing elements in the soft x-ray regime: the scanning x-ray microscope and the imaging x-ray microscope.¹ In the hard x-ray regime, only the scanning x-ray microscope has been developed, because of the lack of suitable imaging devices. In the soft x-ray regime, because the focusing efficiency of a zone plate is typically around 10% and the detector detection efficiency is 20%–50%,¹ the scanning microscope² may be more advantageous for limiting radiation damage to a sample, which is particularly important for biological specimens.³ On the other hand, an imaging microscope⁴ can be used to image all the pixels in a sample simultaneously (parallel detection) and thus high speed imaging is possible.

For hard x rays, some of the drawbacks of a soft x-ray imaging microscope may be substantially minimized. The focusing efficiency of the zone plate, as high as 33%,⁵ has been demonstrated and is much higher than that for soft x-ray zone plates. The higher efficiency also means reduced background from zero and unwanted orders. A hard x-ray imaging microscope is then practical, and its potential benefits such as parallel detection, reduced sensitivity to source size and source motions, and diffraction-limited resolution over a large field, may be exploited. In the hard x-ray regime, zone plates may be the only functional optics with good imaging properties: no distortion term, and all other third-order aberration terms are small over a very wide field.⁶ The first results of using a zone plate in a hard x-ray imaging setup are presented in the following.

EXPERIMENT

The optical scheme is showed schematically in Fig. 1. The test object was located at $\approx 22\ \text{m}$ from the source. A pinhole of $50\ \mu\text{m}$ in diameter was used to collimate the beam and to define the object size. The zone plate is used as a lens in this setup. By properly arranging the distances between

the object, the zone plate, and the detector, a magnified image of the object may be obtained. Ideally, a beam stop of the size of the zone plate should be placed at the image plane to eliminate the zero order from the image. In our experiment, we mainly want to study the imaging properties of the zone plate; thus a lead sheet was used to block out slightly more than half of the image in order to simplify the alignment process.

The zone plate has a focal length of 5 cm for 8 keV x rays and is $40\ \mu\text{m}$ in diameter. It was patterned onto $1.6\ \mu\text{m}$ of Au, by using x-ray lithography to replicate a high precision mask written with an e beam.⁷ The efficiency of similarly made zone plates was measured previously to be about 25%–30%.⁷ With a minimum linewidth of $\Delta r = 0.2\ \mu\text{m}$, the diffraction-limited resolution due to the zone plate should be $\approx 0.25\ \mu\text{m}$. The physical size of the experimental station limited the distance between the zone plate and the detector to 1.5 m, and thus the maximum magnification of the imaging microscope in our experiment is $30\times$ ($1.5\ \text{m}/5\ \text{cm}$). That means a $0.25\ \mu\text{m}$ feature on the object will be enlarged to $7.5\ \mu\text{m}$ in size at the image plane, which is smaller than the $20\ \mu\text{m}$ pixel size of our charge coupled device (CCD) camera. In order to observe a diffraction-

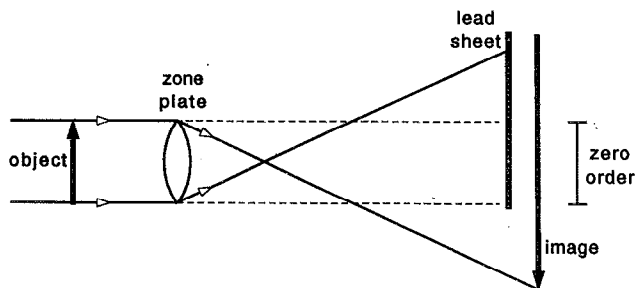


FIG. 1. Schematic of the imaging setup in the vertical direction. The optical scheme was different in the horizontal direction (see the text).

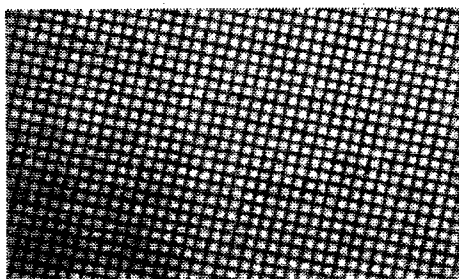
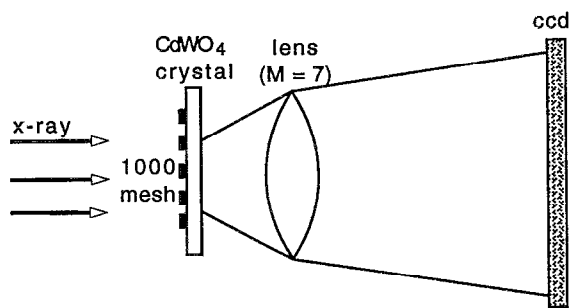


FIG. 2. The scintillator-lens-CCD system was tested by placing a 1000 mesh gold ($25\ \mu\text{m}$ period) in an x-ray beam to simulate an x-ray image of high spatial frequency. The image thus recorded by the CCD is shown at the bottom.

limited element, a scintillation crystal (CdWO_4) was placed at the image plane, and a $7\times$ lens system ($f/0.82$) was used to produce an enlarged optical image onto the CCD. Figure 2 shows a test image of a 1000 mesh recorded by this scintillator-lens-CCD system, which demonstrates an overall resolution of better than $4\ \mu\text{m}$. Using this optical system together with the zone plate, a magnification of $210\times$ total was attained, and a $0.25\ \mu\text{m}$ feature on the object can be recorded by the CCD without distortion.

The imaging setup was tested at beamline X-26C of the National Synchrotron Light Source (NSLS). For an imaging microscope, proper illumination of the sample must be considered. The zone plate has a numerical aperture of about $0.4\ \text{mrad}$ ($\text{NA}=\lambda/2\Delta r$). The NSLS source size is about $1\ \text{mm}$ horizontally; thus an unfocused beam would produce only a divergence of $0.05\ \text{mrad}$ at the sample location, $22\ \text{m}$ from the source, which does not fully illuminate the zone plate. A condenser optics is necessary, and one solution in the soft x-ray regime is to employ a condenser zone plate. It has the advantage that two zone plates fabricated with the same tolerance will have similar numerical apertures, and that the first zone plate may also be used as a monochromator. However, a condenser zone plate for hard x rays would occupy too much space inside the hutch due to the longer focal length (from $\approx 0.4\ \text{m}$ to several meters). Instead, the beamline had a 1:1 horizontally focusing Pt mirror which accepted $2.6\ \text{mrad}$. This overfilled the zone plate horizontally, while the unfocused beam still underfilled the zone plate vertically. This is not the optimal geometry, but will provide sufficient flux for testing the imaging microscope. The spot size at the experimental hutch was approximately $1\times 1\ \text{mm}^2$. The x rays

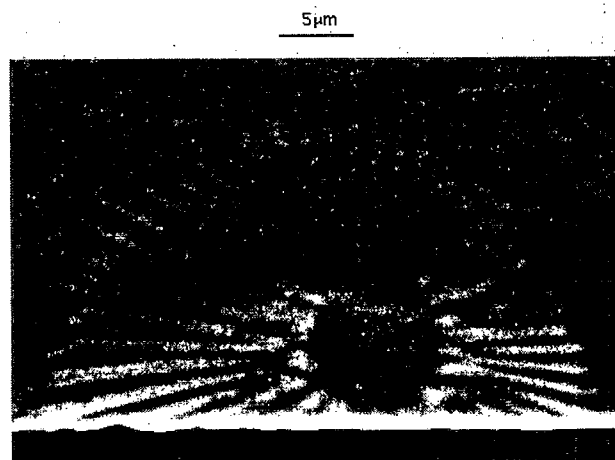


FIG. 3. The image of a test spoke pattern recorded on the CCD camera, using $8\ \text{keV}$ x rays. The bottom part of the image was blocked by a lead sheet acting as beam stop.

were monochromatized by reflecting off W/C multilayers ($d = 29.88\ \text{\AA}$, 50 layer pairs) mounted on a water-cooled substrate located before the test object. The multilayers satisfied the bandwidth requirement of the zone plate ($\approx 2\%$), while delivering plenty of x rays to the sample (55% peak reflectivity).

RESULTS

Due to contributions from zero and high orders, the x-ray image was underlain by a nonuniform background. In order to bring out the details, the image needed to be first normalized to its own background. An empirical solution is to smooth out the original CCD image and obtain a map of the background. This is then used to normalize the original image for removing the nonuniform background. Such a normalized image is shown in Fig. 3, with the image being 210 times larger than the test object. The spoke test pattern was generated using x-ray lithography, with the finest lines about $0.25\ \mu\text{m}$ near the center. However, some defects were introduced during the lithographic process and irregularities were present in the finest line region, which are readily observed in Fig. 3 also. The important point to note is that some lines as fine as $0.4\ \mu\text{m}$, comparable to the predicted diffraction-limited resolution, are clearly visible from the image. It also shows that an object field as large as $50\ \mu\text{m}$ was fully imaged by the zone plate. This confirmed that the aberration due to the zone plate is small even for an object field of the order of the size of the zone plate.

One factor that degrades the image quality in Fig. 3 is that the test pattern is thick enough ($4\ \mu\text{m}$ of Au) only to stop $\approx 80\%$ of the x rays and so the contrast is reduced. Also, the focusing mirror created a diverging zero order beam horizontally, different from that for the vertical direction depicted in Fig. 1. This caused a strong background to superimpose on the magnified image, and increases the noise of the image.

Another important result is that the image was captured with $50\ \text{s}$ of exposure time. This is reasonable, considering the low efficiency of the scintillation crystal, the large refrac-

tive index of the crystal ($n=2.2$), and that the final image was distributed over $\approx 6 \times 10^4$ pixels on the CCD.

CONCLUSION

A zone-plate-based imaging microscope and a scintillator-lens-CCD detector system were developed and tested. The combined resolution is about $0.4 \mu\text{m}$, with a field size as large as $50 \mu\text{m}$. The results are encouraging, particularly with the short exposure time required (50 s). With improved optics and higher brightness from the new generation of synchrotron sources, some real time applications may be feasible in the near future. Another potential application is in microtomography, where a zone plate imaging setup may provide submicron resolution and a fast data acquisition rate.

ACKNOWLEDGMENT

This work is supported in part by the Dept. of Energy, BES-Materials Science, under Contract No. W-31-109-ENG-38.

- ¹C. Jacobsen, J. Kirz, and S. Williams, *Ultramicroscopy* **47**, 55 (1992).
- ²J. Kirz, H. Ade, C. Jacobsen, C. Ko, S. Lindaas, I. McNulty, D. Sayre, S. Williams, X. Zhang, and M. Howells, *Rev. Sci. Instrum.* **63**, 557 (1992); B. Niemann, *X-ray Microscopy III*, Springer Series in Optical Science Vol. 67, edited by A. Michette, G. Morrison, and C. Buckley (Springer, New York, 1992), p. 143; G. Morrison, P. Anastasi, M. Browne, C. Buckley, R. Burge, P. Charalambous, G. Foster, A. Michette, D. Morris, J. Palmer, G. Slark, P. Bennett, and P. Duke, *ibid.* p. 139.
- ³M. Folkard, *X-ray Microscopy III*, Springer Series in Optical Science Vol. 67, edited by A. Michette, G. Morrison, and C. Buckley (Springer, New York, 1992), p. 306.
- ⁴D. Rudolph, G. Schneider, P. Guttman, G. Schmahl, B. Niemann, and J. Thieme, in Ref. 3, p. 392; Y. Kagoshima, S. Aoki, M. Kakuchi, M. Sekimoto, H. Maezawa, K. Hyodo, and M. Ando, *Rev. Sci. Instrum.* **60**, 2448 (1989).
- ⁵B. Lai, W. Yun, D. Legnini, Y. Xiao, J. Chrzas, P. Viccaro, V. White, S. Bajikar, D. Denton, F. Cerrina, E. DiFabrizio, M. Gentili, L. Grella, and M. Baciocchi, *Appl. Phys. Lett.* **61**, 1877 (1992); W. Yun, B. Lai, D. Legnini, Y. Xiao, J. Chrzas, K. Skulina, R. Bionta, V. White, and F. Cerrina, *Proc. SPIE* **1740**, 117 (1992).
- ⁶M. Young, *J. Opt. Soc. Am.* **62**, 972 (1972).
- ⁷A. Krasnoperova, J. Xiao, F. Cerrina, E. DiFabrizio, L. Luciani, M. Figliomeni, M. Gentili, W. Yun, B. Lai, and E. Gluskin, *J. Vac. Sci. Technol. B* **11**, 2588 (1993).