Soft X-ray Scanning Microtomography with Submicron Resolution

I. McNulty

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

W.S. Haddad and J.E. Trebes

Lawrence Livermore National Laboratory, Livermore, CA 94550

E.H. Anderson

Center for X-ray Optics, Lawrence Berkeley Laboratory, Berkeley, CA 94720

Scanning soft x-ray microtomography was used to obtain high-resolution three-dimensional images of a microfabricated test object. Using a special rotation stage mounted on the scanning transmission x-ray microscope at the X1A Beamline at the National Synchrotron Light Source, we recorded nine two-dimensional projections of the 3D test object over an angular range of -50° to +55°. The x-ray wavelength was 3.6 nm and the radiation dose to the object per projection was approximately $2 \times 10^6$ Gy. The object consisted of two gold patterns supported on transparent silicon nitride membranes, separated by 4.75 μm, with 100 to 300-nm wide and 65-nm thick features. We reconstructed a volumetric data set of the test object from the two-dimensional projections using an algebraic reconstruction technique algorithm. Features of the test object were resolved to ~100 nm in transverse and longitudinal extent in three-dimensional images rendered from the volumetric set.

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I. Introduction

A wealth of x-ray microscopies have been developed in the past decade, broadening the range of applications in both the soft and hard x-ray energy regions. Soft x-rays of 2.3 - 4.4 nm wavelengths are well suited to imaging of biological objects in aqueous environments [1]; higher energy x-rays are better suited to imaging denser, thicker specimens [2]. Substantial technological progress with x-ray sources, optics, detectors, and numerical methods have driven this progress and, in many instances, opened the door to previously inaccessible areas. One of these techniques is three-dimensional (3D) x-ray microscopy [3-5]. The ability to visualize microscopic 3D structure has long attracted biologists, materials scientists, and the microelectronics industry. Although complementary approaches such as x-ray holography [6] exist for forming 3D x-ray images, among the most viable is x-ray microtomography. A significant limitation to these techniques is the achievable imaging resolution, especially in the longitudinal or depth direction through the sample. In this paper we report on the technical aspects of a soft x-ray scanning microtomography experiment at submicron resolution using zone plate optics and a microfabricated 3D test object. A more general description can be found elsewhere [7]. The work was performed using the high resolution scanning transmission x-ray microscope (STXM) at the X1A Beamline at the National Synchrotron Light Source (NSLS).

II. Scanning microscopy with a new twist

The X1A STXM utilizes a zone plate to form a diffraction-limited focal spot under which the sample is scanned [8]. A central stop on the zone plate and an order-sorting aperture (OSA) serve to isolate the first-order focus. For this experiment we used a nickel phase zone plate [9] with a radius $R$ of 70 $\mu$m and finest zone width $\delta r$ of 60 nm. The zone plate delivered a focal spot with radius $1.22\delta r \approx 73$ nm and a focal length $2R\delta r/\lambda = 2.3$ mm at a wavelength $\lambda = 3.6$ nm. Its diffractive efficiency at this wavelength was nearly 15%. Although we have used zone plates with considerably finer resolution [10] in the past, we chose this one because (1), its depth of field was sufficiently large that the test object was always in focus, and (2), the additional working distance provided by this optic allowed the test object to be rotated through large angles without fear of colliding with it.

The STXM was supplied with coherent soft x-rays by the Soft X-ray Undulator and the X1A Beamline at the NSLS [11]. The wavelength and spectral width of the x-ray beam was 3.6 nm and 0.5%, respectively. Adequate temporal coherence for diffraction limited operation of the zone plate was supplied by the beamline monochromator; the monochromator exit slit provided sufficient spatial coherence at the zone plate.

We modified the scanning microscope for this experiment by adding a precision specimen
rotation stage (Fig. 1). This involved several technical challenges. One, the stage had to allow 360°
rotations with minimal radial and angular error. Two, it had to be small enough to fit onto the
STXM scanning stage. Three, it had to have low mass in order not to impede the scanning motion,
which achieves linear velocities up to 4 mm/s. We built a compact rotation stage using two grade-7
ball bearings and a precision ground shaft to address these requirements. A small collet supports
the specimen on the shaft. The specimen rotation angle is adjusted manually. A tiny pointer and
photo-reduced protractor were used to read the shaft angle to ±0.5°. We have since added a 52-
tooth gear to the shaft and a small ball bearing, captured by a strip of spring steel against the gear,
to provide precise 6.9° angular increments of the shaft rotation (Fig. 2).

The scanning hardware and software was the same as that used for 2D imaging [8]. The
user-friendliness of the X1A STXM facilitated rapid alignment and focusing. In particular, we
made frequent use of "focus-mode" scans by which the specimen is scanned transversely and the
zone plate is scanned longitudinally to locate the best focus position for each projection.

The 3D test object was fabricated lithographically by lift-off and electroplating. It consisted
of two gold patterns supported on 100-nm-thick silicon nitride membranes on 2 mm by 12 mm
silicon frames. The frames were glued together such that the gold patterns were separated by 4.75
μm, as shown in Fig. 3. The features in the patterns range from 100 to 300 nm in width and are
65-nm thick. This thickness has 18% transmittance for 3.6-nm x-rays yet still transmits 3% of the
incident radiation when two features overlap in projection. Thus, we were assured of both good
contrast and acceptable counting statistics even through two overlapping layers. In addition to
simple features of various lengths, widths and orientations, we also incorporated a rectangular
alignment pattern into the test object (see Fig. 3). Its purpose was twofold: the incidence angle of
the test object to the beam could be checked by measuring the apparent lengths of two
perpendicular sides of the alignment pattern and comparing with the known fabricated lengths, and
errors in the scanned image were readily visible as measurable distortions in the alignment pattern.
Lastly, the test object included an order-sorting aperture to exclude unwanted diffraction orders
of the zone plate. The OSA consisted of a 10 μm by 15 μm, 700-nm-thick gold mask deposited onto
pattern A of the test object. Because the specimen was scanned in the shadow of the central stop of
the zone plate, the OSA effectively admitted only the first-order focused radiation. By using a
focal-plane OSA instead of an aperture located between the zone plate and the specimen (ordinarily
the case with the STXM), we were afforded considerably more working distance for specimen
rotation.

III. Data collection

Using the modified STXM, nine 2D projections of the test object were recorded at 5-15°
increments, spanning an angular range of -50° to +55° from normal incidence. The object was
centered in the scan field and focused for each projection. The scan field was 250 x 200 pixels and covered an area of 6.25 μm by 5.00 μm using 25-nm pixels.

The flux in the zone plate focus incident on the test object was typically 2 x 10^6 photons/s. In order to obtain images that were not limited by shot-noise statistics, the dwell time per pixel in each scan was typically 10 ms, yielding ~10^4 detected photons per pixel in the open areas of the test object. Scans took approximately 8 min to acquire. Based on the known absorption of soft x-rays in gold [12], the estimated radiation dose to the test object per projection was 2 x 10^6 Gy.

IV. Image reconstruction

The raw scan data were numerically processed with a median filter to reduce noise and the logarithm was taken to obtain the transmittance of the test object. The data were then corrected for spatial distortions, principally non-orthogonality of the scan axes, and occasional misregistration of a scan line. Scan non-orthogonality was most likely due to metal fatigue in the flexure-based scanning stage, whereas misregistration was caused by missed scan-line start pulses. To perform an accurate test of the method, we manually corrected the projection data for these errors prior to reconstruction by requiring the edges of the alignment pattern to be straight and orthogonal. The centers of rotation of the 2D projections were then found and aligned to each other.

An algebraic reconstruction technique (ART) code [13] was used to reconstruct a volumetric data set from the corrected projections. ART codes have proven to be effective for reconstructing a limited number of tomographic projections in the presence of noise, especially in cases where other methods such as filtered back-projection introduce artifacts or break down [14]. Fig. 4 shows four views rendered from the 256 x 256 x 100 pixel volumetric set. The alignment pattern was not included in the reconstruction. The longitudinal resolution is comparable to the transverse resolution, of order ~100 nm, as can be seen by the apparent thickness of the test object features. Figs. 4c and 4d show some blurring of the horizontal bar in pattern B. Because this feature was perpendicular to the rotation axis, insufficient off-axis information about its thickness was available from the 2D projections to reconstruct it as accurately as the other features. The view from directly "above" the two planes of the test object in Fig. 4d most clearly shows how well the features in the test object were resolved longitudinally. Despite the fact that only nine projections were used in the reconstruction, the images exhibit excellent depth resolution and a low degree of artifact.

V. Conclusion

This work demonstrates high resolution three-dimensional x-ray imaging with zone plate optics by scanning microtomography. The 3D reconstructions obtained exhibit comparable depth and transverse resolution near the 73-nm diffraction limit of the zone plate that was used. Scanning
methods offer low specimen dose, and ART is helpful when reconstructing a limited number of projections. Both may be advantageous when radiation damage is at issue. With recent advances in zone plates and high brightness x-ray sources for harder (2-8 KeV) x-rays, scanning microtomography at the submicron scale should soon become possible at these energies as well.

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References

12. XCAL, a software package for calculating the interactions of x-rays with matter, by S. Mrowka, Oxford Research Group, 5737 Clinton Ave., Richmond, CA 94805.
Figure Captions

Fig. 1. Schematic of the scanning microtomography experiment. The specimen is scanned in the zone plate focus at various orientations to obtain a set of two-dimensional projections.

Fig. 2. Specimen rotation stage.

Fig. 3. The 3D test object.

Fig. 4. Four views of the test object rendered from the reconstructed data. (a) On-axis view, showing the two overlapping planes of the test object. (b) View rendered for a rotation $\phi = 42^\circ$ through the vertical axis. (c) View for a similar rotation $\theta$ through the horizontal axis. (d) View for $\theta = 90^\circ$, corresponding to viewing both planes from above.
coherent x-rays

zone plate with central stop

3D test object on silicon nitride windows

proportional counter

rotation stage

scanning stage
specimen

support pin

collet
gear
captured ball bearing

spring steel

mounting bracket

rotation adjustment

12 mm

53 mm

15 mm

15 mm

fig. 2
Pattern A

Pattern B

65 nm

2 μm

4.75 μm

100 nm gap

100 nm

2 μm

fig. 3