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Tomographic scanning microscope for 1-4 KeV x-rays

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ABSTRACT

X-ray microtomography enables three-dimensional imaging at submicron resolution with elemental and chemical state contrast. The 1-4 KeV energy region is promising for microtomography of biological, microelectronics, and materials sciences specimens. To capitalize on this potential, we are constructing a tomographic scanning x-ray microscope for 1-4 KeV x-rays on a spherical grating monochromator beamline at the Advanced Photon Source. The microscope, which uses zone plate optics, has an anticipated spatial resolution of 100 nm and an energy resolution of better than 1 eV.

Keywords: x-ray microscopy, tomography, three-dimensional imaging, microprobe, x-ray optics, zone plates, beamlines

1. INTRODUCTION

X-ray microtomography is a powerful technique for high-resolution three-dimensional imaging[1,2] that offers elemental and chemical state contrast[3] at greater resolution than that obtainable with visible light, and less radiation damage than electron probes.[4] Applications of this method include 3D microscopy of biological, microelectronics, and materials sciences specimens. Use of 1-4 KeV x-rays for microtomography is especially attractive due to the number of K, L, and M absorption edges in the middle of the periodic table that are accessible, the capability to match the penetration depth to many interesting and industrially important samples, and possibilities for low-dose phase contrast imaging of biological specimens.[5] Third-generation synchrotron facilities, such as the Advanced Photon Source, will open up new opportunities for x-ray microscopy in this energy region that, so far, have been relatively unexplored.

Until recently, the imaging resolution by x-ray microtomography has been limited to about 1 μm by the detector resolution or by the ability to magnify the image before it reaches the detector, e.g., with crystal optics. One way around this limitation is to use a zone plate lens to form a high resolution x-ray microprobe in a tomographic *scanning* microscope.[6,7] We are constructing a microscope based on this principle for operation with 1-4 KeV x-rays. The instrument will be installed at the Advanced Photon Source on the 2-ID-B beamline, which is optimized for this purpose. This paper describes the beamline as well as the microscope and its expected performance.

2. THE 2-ID-B BEAMLINE

The 2-ID-B beamline (fig. 1) is designed to deliver spatially and temporally coherent radiation in the 1-4 KeV region.[8] A dedicated undulator (5.5-cm period), continuously tunable over this range, provides the x-ray beam. Its spectral brightness exceeds 10^{18} ph/s/mm^2/mrad^2/0.1% bandwidth. Water-cooled grazing incidence mirrors handle the substantial power density of the undulator beam and focus it for stigmatic performance and optimum monochromator throughput. The mirrors also match the beam dimensions to the microscope, which requires spatially coherent illumination over an area approximately 50 μm in diameter. The effective horizontal and vertical sources for the microscope are located within 0.8 m of each other, approximately 7 m upstream, which minimizes astigmatic broadening of the microprobe focus to less than 1%. A spherical grating monochromator with adjustable slits and interchangeable water-cooled gratings provides the spectral resolution needed for elemental and chemical specificity (fig. 2); our calculations indicate the peak resolution is better than 0.1 eV at 1 KeV and 1 eV at 4 KeV, with the exit slit position optimized for minimum defocus. The mirrors and gratings have multilayer coatings for higher efficiency above 2 KeV. A differential pump and Si_3N_4 exit window isolate the beamline from contamination and permit the x-ray beam to be used in air in the experimental station.

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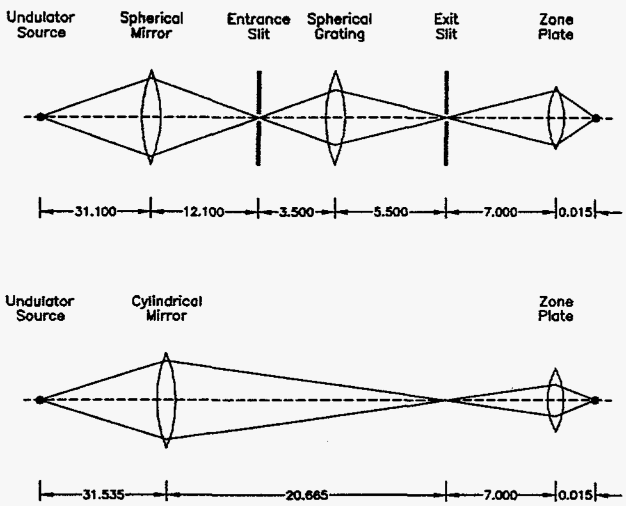

Fig. 1. Schematic of the 2-ID-B beamline and microprobe, depicting the optics as thin lenses in the horizontal (upper) and vertical (lower) planes. Distances are given in meters.

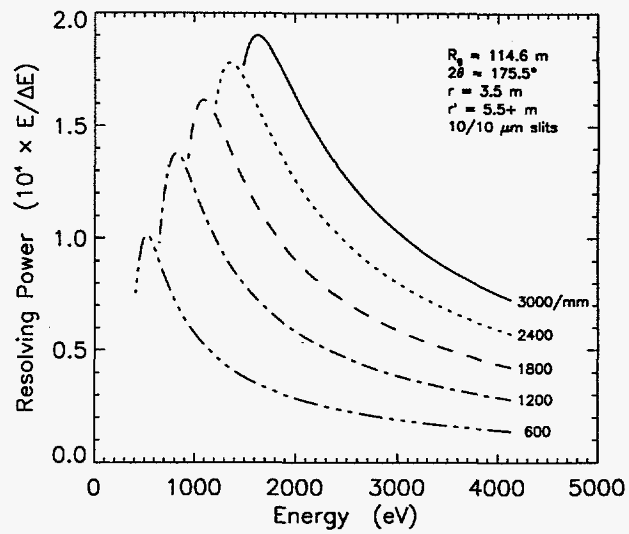

Fig. 2. Calculated spectral resolving power at 2-ID-B for several grating densities using 10-µm entrance and exit slits. Figure error and aberrations are included.

3. THE MICROSCOPE

The microscope (fig. 3) is of the scanning type pioneered by Rarback.[9] Coherent x-rays are focused by a phase zone plate to a diffraction-limited spot onto the sample. An order-sorting aperture preceding the sample screens out unwanted diffraction orders from the zone plate. The sample is scanned in the zone plate focus under computer control while the transmitted x-rays are detected by an avalanche photodiode. It is then rotated incrementally to a new orientation and the process is repeated until a set of scanned projections is obtained. The projections are numerically processed and reconstructed to yield a volumetric representation of the sample, from which arbitrary views may be rendered. Commercially available components are used for the sample stage (see table 1). A two-axis piezoelectric-driven flexure stage on a two-axis stepper motor stage provides fine, in addition to coarse, linear scans. The rotation stage is a low-mass, dc-motor driven, precision spindle mounted to the piezo fine stage. Prealignment of the zone plate, order-sorting aperture, sample, and detector is accomplished with visible-light optics. The microscope is enclosed by a small box, which may be backfilled with helium to minimize absorption in the x-ray flight path. The assembly is supported on an acoustically and vibration isolated optical bench.

Initially, we will use existing gold zone plates capable of 200-nm resolution, then aim for 50-100 nm with improved zone plate designs. Fig. 4 shows the calculated diffraction efficiency of four candidate zone materials: aluminum, titanium, and chromium, and unmetallized PMMA photoresist. The zone plate substrates, typically consisting of 100 nm of Si_3N_4, are at least 90% transparent to 1-4 KeV x-rays. It is therefore apparent that efficiencies of ~30% should be obtainable over this energy range; even higher efficiencies are possible with stepped zone profiles. Moreover, 50-200 nm zone widths and 0.1-2 µm zone thicknesses are technically feasible to make with these materials by e-beam[10] and x-ray[11] lithography. Finer-resolution, thinner zone plates can of course be used, but at concomitantly lower efficiency. Table 1 lists the optical parameters of zone plates to be used in the microscope.

Numerical reconstructions of the tomography data will be obtained by standard means. We are also developing special reconstruction codes optimized for limited data sets (<20 projections, by comparison to >100 which is typical of computed tomography), to minimize the total dose and therefore the effects of radiation damage to the specimen. Codes using a modified algebraic reconstruction technique[12,13] show the most promise in this regard and are under active investigation.

Installation and testing of the 1-4 KeV tomographic scanning microscope will begin in early 1996 following commissioning of the 2-ID-B beamline. We look forward to many exciting applications and results with this new instrument.

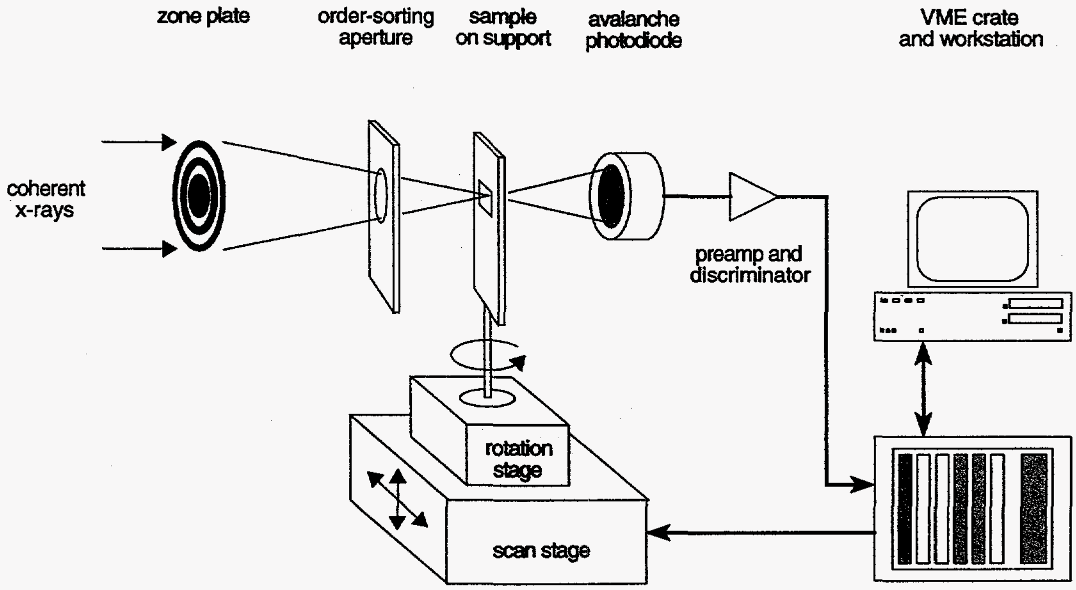

Fig. 3. Schematic drawing of the scanning tomographic microscope. Coherent x-rays are focused by the zone plate through the order-sorting aperture onto the sample, which is raster-scanned in the focus on the two-axis stage. Scanned projections through the sample are obtained at various orientations via the rotation stage. The flux transmitted by the sample is detected by the avalanche photodiode, amplified, then digitally acquired and stored. The scan motion, rotation, and data acquisition are controlled by a VME crate and small computer workstation.

Sample stage		coarse	fine	
linear range		± 10	± 0.1	mm
linear resolution		1000	5	nm
linear velocity		4	20	mm/s
angular range		360		°
angular resolution		0.0001		°
Zone plates		**current**	**future**	
material		Au	Al, Cr	
radius		30	50	µm
zone thickness		1.0	1.4	µm
finest zone width		200	50	nm
transverse resolution		244	61	nm
focal length	(1, 4 KeV)	9.7, 39	4.0, 16	mm
depth of field	(1, 4 KeV)	158, 631	9.9, 40	µm
efficiency	(1, 4 KeV)	8, 14	32, 31	%

Table 1. Sample stage and zone plate specifications.

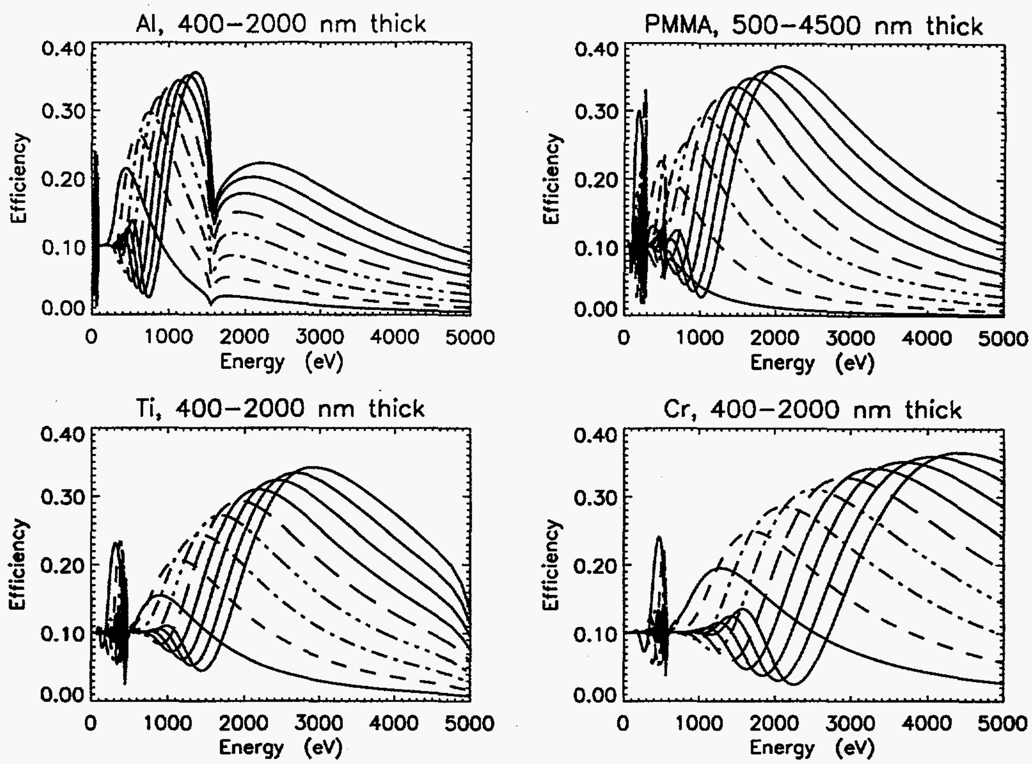

Fig. 4. Calculated diffractive efficiencies of zone plate lenses made of Al, PMMA, Ti and Cr.

4. ACKNOWLEDGEMENTS

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