Development of a zone plate-based full field imaging microscope for hard x-rays

W. Yun, M. R. Howells, A. A. MacDowell, H. A. Padmore
Advanced Light Source, Lawrence Berkeley National Lab, Berkeley, CA, 94720

R. O. Richie
Dept. of Materials Science, University of California at Berkeley, Berkeley, CA, 94720

J. Spence
Department of Physics, Arizona State University, Tempe, AZ, 85287

Use of x-ray phase contrast in the hard x-ray region is among the most important developments in x-ray imaging techniques. The relative figure of merit of x-ray phase contrast to amplitude contrast may be approximately given by the square of the ratio of the real (f_1) and imaginary (f_2) part of the atomic scattering factor for "thin" samples. Because the real part of the atomic scattering factor is approximately equal to the number of free electrons in an atom while the imaginary part decreases with the cube of x-ray energy away from an absorption edge, the relative figure of merit can be significantly greater than unity. For example, the relative figure of merit is approximately equal to 3.6×10^5 for a sample consists of mainly carbon for 8 keV x-rays. For a "thick" sample, this relative figure of merit is multiplied by the inverse of the transmission of the sample. The superiority of phase contrast compared to absorption contrast for low-Z samples was dramatically demonstrated in the work of Cloetens et al (J. Appl. Phys., 81, p. 5878 (1997)). The measurements were made at the European Synchrotron Radiation Facility (ESRF) on cracks in polymers and in an aluminum-matrix-silicon carbide composite. An x-ray energy of 25 keV and a 150-meter source-to-sample distance was used to obtain highly coherent (collimated) x-rays to illuminate the sample. By placing a phosphor-CCD-based detector about 1 m beyond the sample, the intensity variations resulting from the phase curvature produced by such features as cracks and voids were recorded and used to reconstruct three-dimensional images of the features at 6-7 microns resolution. Cracks of sub-micron dimension were detected but not resolved due to the limited resolution. It should be noted that cracks and voids with micron dimensions were not detectable by simple absorption imaging (radiography) as they constituted such a small fraction of the total volume sampled.

In collimated-beam experiments, like those of Cloetens, and all others to date, the intrinsic resolution is that of the detector which is never better than a few microns. In order to circumvent the detector spatial resolution limitation, we are developing a zone plate-based x-ray full field imaging microscope capable of obtaining sub low micron spatial resolution. The microscope is a type of Zernike phase contrast microscope, which is an established technology in the soft x-ray region. The zone plate is used as the objective with a $\pi/2$ phase-shifting disk in the back focal plane in exact analogy to the visible light case. The magnification produced by the zone plate allows high resolution to be achieved even when a low-resolution detector such as a CCD detector is used. At an appropriate magnification, the spatial resolution is limited by the intrinsic resolution of the zone plate. The state of the art is about 0.15-μm resolution at high focusing efficiency for 8 keV x-rays and 0.08 μm at low efficiency and still improving.

An initial experiment was done using a prototype microscope on the beamline 7.3.3 with three days of beam time. The schematic of the experiment setup is shown in Fig. 1. The design allows us to use it either in a phase contrast mode or a dark field mode. In the dark field mode,

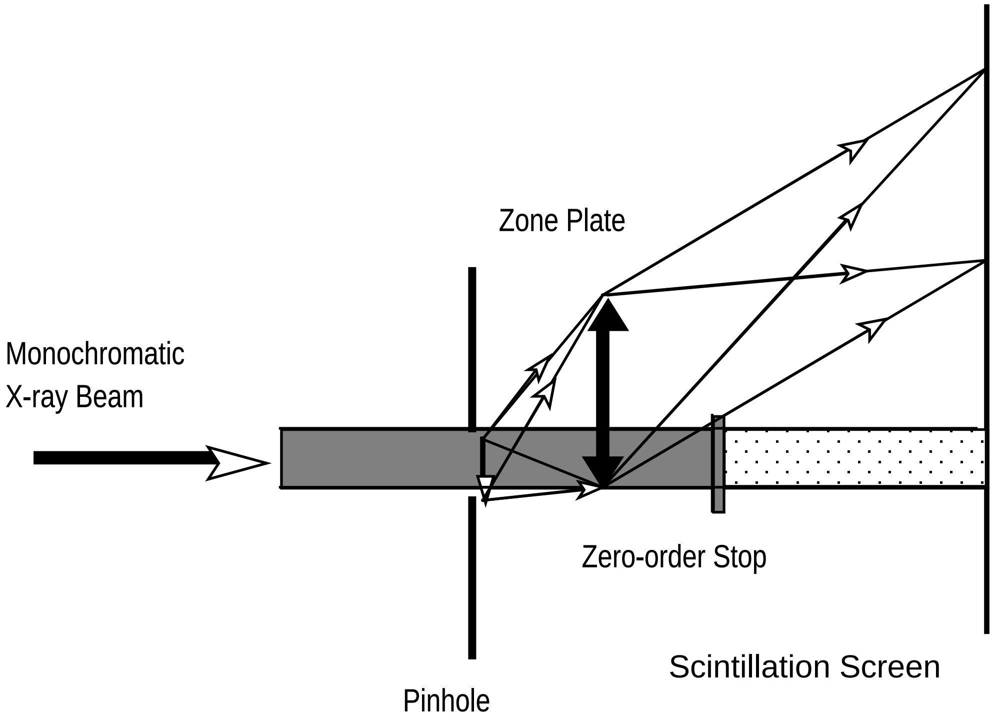

Fig. 1 Schematic of the full-field imaging microscope used in our experiment.

the image arises from the x-ray photons scattered out of the envelope of the incident beam as well as its corresponding image, and the image contrast is proportional to the scattering strength between two neighboring resolution elements, which is proportional to the square of the atomic scattering factor. Because of its low background, the dark field mode may be a preferred imaging technique for some special cases.

The key optical component in this setup is the phase zone plate and it was used as an objective in a light microscope. The phase zone plate used was developed in a long-term collaboration between Argonne National Laboratory, University of Wisconsin at Madison, and Istituto di Electronica dello Stato Solid, Italy. Its key parameters are given below:

Diameter	145 µm
Outermost zone width	103 nm
Thickness of Au	900 nm
Focal length @ 8 keV	10 cm

In theory, the theoretical spatial resolution of the phase zone plate is about 0.12 µm and an experimental value of 0.15 µm was demonstrated. The focal length becomes 9.375 cm at 7.5 keV x-ray energy that was used in the experiment. The zone plate was used to a magnifying objective as a lens is used in the optical region. In order to achieve high spatial resolution with a CCD detector with a 24 µm pixel size, a large sample-detector distance was used to obtain a large x-ray magnification by the phase zone plate, and a scintillator/lens system was used to further magnify an image before it was recorded by the CCD detector. The x-ray and optical magnification were 21 and 14 respectively and the combined total magnification is therefore 294. Each pixel of the CCD detector, therefore, represents a spatial size of 0.081 µm on the objective plane.

In our first experiment, only the dark field imaging was done because of its relative simplicity and the time limitation. In the dark field mode, the pinhole was used to define a field of view to be imaged. In principle, the field of view can be as large as the area of the zone plate in an optimized setup and is limited in our present setup to an area with a linear dimension approximately equal to the radius of the zone plate. A pinhole of 50-µm diameter was used in our experiment and it defined the field of view. With each pixel of the CCD representing of 0.08 µm on the object plane, the corresponding image field at the CCD detector was an area of 625 x 625 pixels.

This setup was used to obtain full field imaging of several samples, which include a human hair, a Cu mesh grid, a pyrolytic carbon piece of about 2 mm thick, and a piece of grass. The exposure time required was about 100 seconds. It was found that once the imaging microscope is aligned, imaging of different samples could be easily done. Fig. 1 shows two images obtained using the imaging microscope in the dark field mode. Because of time limitation, we could not complete a plan of using the setup in the phase contrast mode. A phase shifter made by drilling a 20 µm on a 6.5-µm thin Al foil was prepared before the experiment. We believe it should be straightforward to use this setup to develop the phase contrast imaging technique in the hard x-ray region. We are now developing a more optimized version of this system.

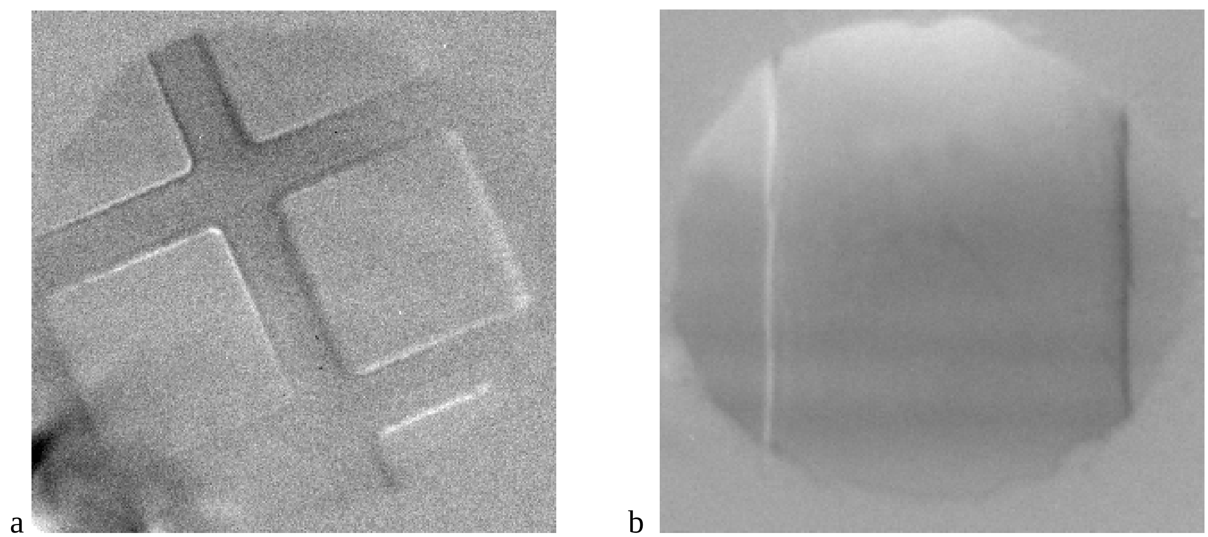

Fig.2 Image of a part of a Cu mesh grid (2000 mesh/inch) (a) and a human hair of 38-µm in diameter (b). The field of view is about 50 µm.

In summary, an x-ray tomographic facility with 0.1-µm spatial resolution can be developed using a bending magnet source at the ALS. This feasibility has been confirmed by a preliminary experiment done on beamline 7.3.3. Such a facility would be a very important tool for materials and engineering research.

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Principal investigator: Wenbing Yun, Advanced Light Source, Lawrence Berkeley National Laboratory. Email: wyun@lbl.gov. Telephone: 510-486-2974.