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X-RAY MICROSCOPY FOR THE LIFE AND PHYSICAL SCIENCES

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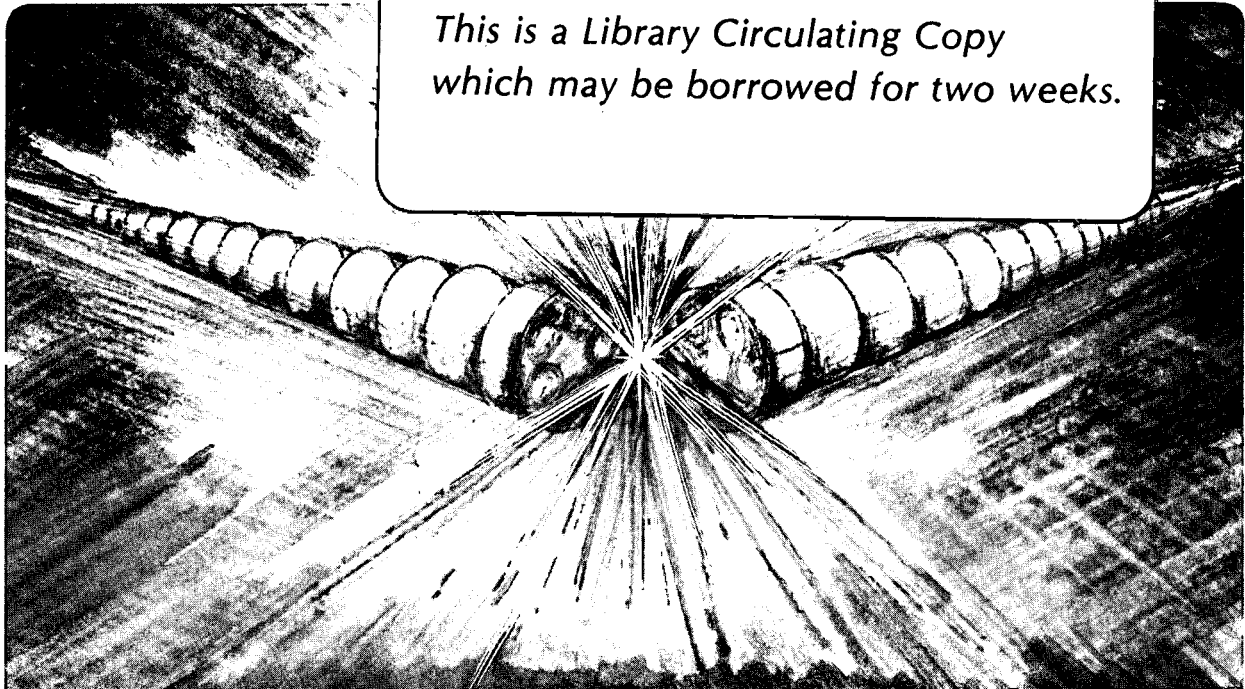
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# X-Ray Microscopy for the Life and Physical Sciences

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Advances in x-ray microscopy coupled with the development of bright, partially coherent radiation at x-ray wavelengths, herald a new period in which scientists in diverse disciplines will use x-ray imaging and probing techniques to see ever smaller structures, write ever smaller patterns, and study physical, chemical and biological systems, not only with an elemental sensitivity, but in many cases with a sensitivity to details of bonding. The combination of x-ray microscopy and various emission spectroscopies will permit the study of surfaces, thin films and material interfaces, as well as biological samples in their natural, unaltered and hydrated state<sup>1,2</sup>.

In this brief report we describe two recent x-ray microscopy experiments which demonstrate achievement of significant milestones in the development of high spatial resolution x-ray microscopy for the life and physical sciences. In one series of experiments it was clearly demonstrated that x-ray optical systems are capable of forming images of nanostructure patterns associated with future microelectronic devices, to a spatial resolution better than 0.1 microns. In the second set of experiments, unaltered biological material was imaged in its natural state without recourse to sectioning, staining, fixing or drying, at a spatial resolution well beyond that of the optical microscope, and with a delivered energy dose well below that of an electron microscope.

The series of experiments<sup>3</sup> in which it was shown that x-ray optical systems are capable of imaging microelectronic circuits, to a spatial resolution better than 0.1 microns, were performed by collaborators from LBL's Center for X-Ray Optics, IBM's Nanotechnology Group, and the Universität Göttingen's Forschungsgruppe Röntgenmikroskopie. The microelectronic pattern imaging experiments were performed using a new 400Å (outer zone width) gold Fresnel zone plate objective lens<sup>4</sup>, in the Göttingen group's x-ray microscope<sup>5</sup>, operated at 45Å wavelength at the Berlin Electron Synchrotron facility (BESSY). The new 400Å zone plate lens was fabricated at IBM as part of a collaborative agreement with the Center for X-Ray Optics. The theoretical resolution of such a lens is 500Å, a value which appears to have been achieved or closely approached in these experiments<sup>3</sup>. Figure 1 shows an SEM photograph of the gold Fresnel zone plate lens.

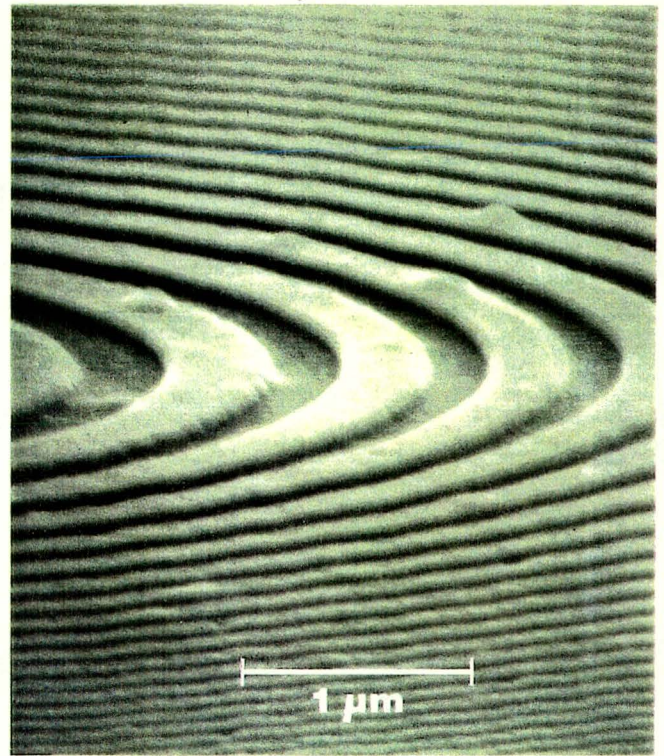


Figure 1. The gold Fresnel zone plate lens used to image nanostructure electronic patterns at BESSY. The lens has 500 zones, with a nominal outer zone width of 400Å, a gold thickness of 700Å, and a bar to space ratio in the outer region of 2:1. The lens has an outer diameter of 80μm, and a focal length of 0.71 mm at the 45Å wavelength used for pattern imaging. The effective aperture is F/9.

Figure 2 shows x-ray images of nanostructure patterns associated with a developmental, fast gating time FET of interest to IBM<sup>6,7</sup>. The opaque gold pattern is supported by a thin (~1,000Å), x-ray transmissive silicon nitride membrane. Pattern features well below 0.1 micron (1,000Å) are clearly resolved, demonstrating an ability to "see" such features albeit on a relatively small transverse field (~30 microns wide). The next step is to "write" patterns with this scale of detail, and then to devise a combination of methods for doing so on a larger transverse field.

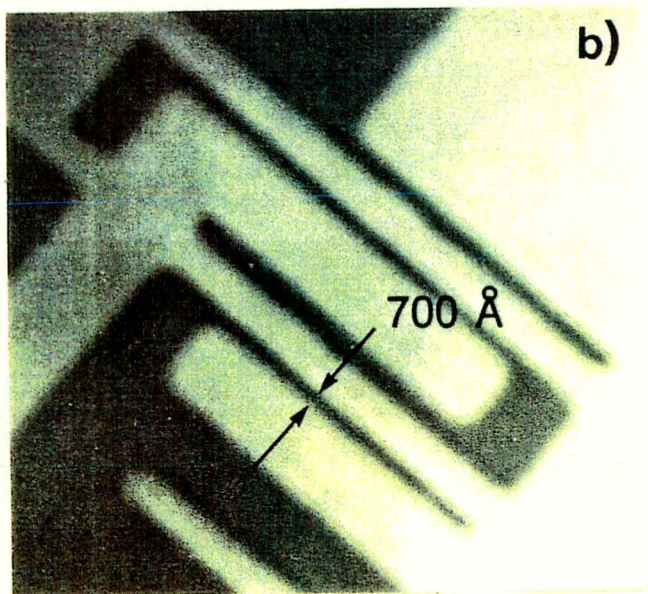
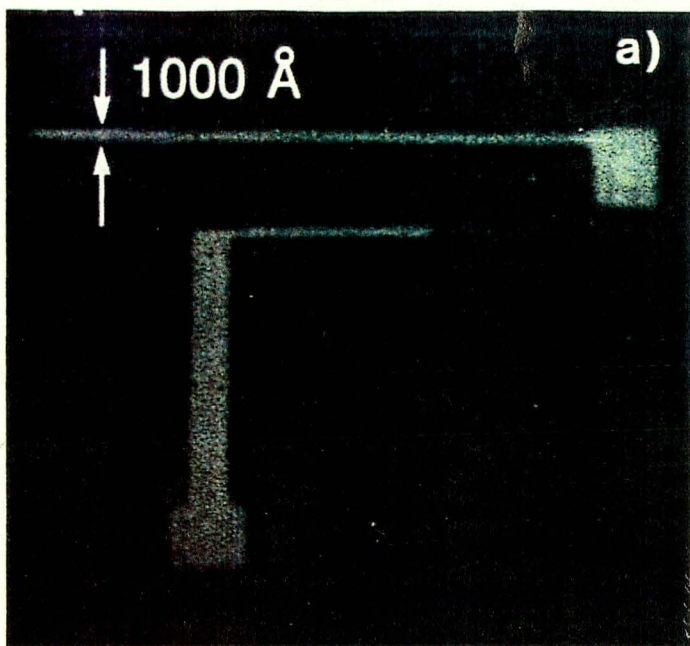


Figure 2.(a) A soft x-ray image, taken at  $45\text{\AA}$  wavelength, of the gate level pattern associated with an experimental  $0.1\text{ micron}$  MOSFET under development at IBM. The  $1,000\text{\AA}$  ( $0.1\text{ micron}$ ) gold bars are clearly resolved. The gold pattern is supported by an x-ray transmissive silicon nitride membrane of approximately  $1,000\text{\AA}$  thickness. The image shown is one portion of a repetitive pattern which is approximately  $30\text{ microns}$  across. (b) A soft x-ray image of two levels of the same MOSFET showing further pattern detail, including a clearly resolved  $700\text{\AA}$  gap ( $0.07\text{ micron}$ ) between gold fingers.

In the second series of experiments<sup>8</sup>, a significant milestone was achieved in that for the first time unaltered biological material, in its wet and natural environment, was imaged with a spatial resolution well beyond that of the optical microscope. In these experiments approximately  $1\text{ micron}$  diameter vesicles containing digestive enzymes were imaged at a spatial resolution better than  $1,000\text{\AA}$ , using IBM-CXRO Fresnel zone plates similar to those discussed above<sup>4</sup>, but having a thick apodized central region, and outer zone widths of  $500\text{\AA}$  or  $700\text{\AA}$ , depending on the particular experiment. These experiments,

involving damage sensitive biological material, were performed with the Stony Brook/NSLS scanning x-ray microscope at Brookhaven National Laboratory's National Synchrotron Light Source (NSLS)<sup>9</sup>, as a part of a collaboration involving SUNY Stony Brook, Brookhaven National Laboratory, UC San Francisco, IBM's Nanotechnology Group, and LBL's Center for X-Ray Optics. Figure 3 shows an image obtained at  $32\text{\AA}$  wavelength of a pancreatic secretion granule, separated from a male Sprague-Dawley rat, but maintained whole and otherwise unaltered in a specifically constructed fixture capable of maintaining an aqueous environment, with thin x-ray transmissive windows suitable for use with soft x-rays, and designed for use with the Stony Brook/NSLS scanning microscope<sup>10</sup>. This particular image was obtained with a  $700\text{\AA}$  outer zone width lens. Image formation in the scanning microscope is obtained on a pixel by pixel basis, by recording variations in transmitted x-ray flux as a function of position as the sample is rastered past the lens focal spot. In figure 3, a color image has been formed from the resultant array of numbers. The quantitative color assignment provides a mechanism for feature recognition through x-ray absorption. Low count rates, and thus high absorption, are represented by red, while blue represents low absorption. The chemical nature of this experiment and the wavelength used, suggest that red to orange appearance indicates varying concentrations of carbon containing proteins, while blue areas indicate prevalence of water. Comparison to electron microscope data is discussed in reference 8. Although the radiation dose was relatively high for this sample, approximately one megarad, the specimen was observed about 20 minutes later in a second image, and except for small scale spatial variations near the resolution limit of the microscope, appeared to be unchanged. Future experiments will attempt to study not only structural integrity and elemental mapping<sup>11</sup>, but will also attempt to study biological function as well, through variation of chemical environment between exposures.

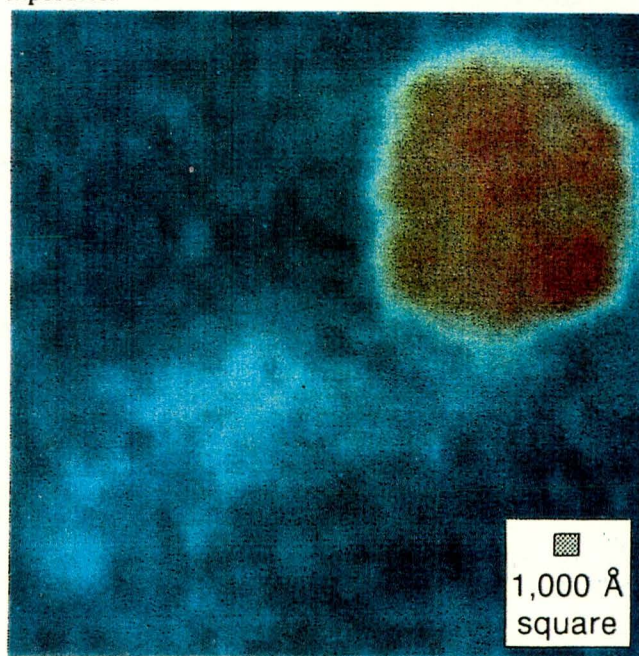


Figure 3. The image of a one micron diameter zymogen granule, obtained at  $32\text{\AA}$  wavelength with the scanning absorption microscope at NSLS. Protein content, indicated by higher absorptivity at this wavelength, is shown in reddish orange. Water, indicated by relatively low x-ray absorption is shown in blue. Color is quantitatively related to count rate, and thus absorption. Spatial resolution in this particular image, is approximately equal to the  $1,000\text{\AA}$  by  $1,000\text{\AA}$  square shown in the figure.

In conclusion, it is evident that x-ray optics and x-ray microscopy are making regular progress in the development of new tools for science and technology. The authors are pleased to acknowledge assistance from the staffs at NSLS and BESSY, use of the Göttingen microscope developed by G. Schmahl and D. Rudolph, and financial support from the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Material Sciences, through contracts DE-AC03-76SF-00098 and DE-AC02-76-CH00016, the U.S. Department of Defense, Air Force Office of Scientific Research through University Research Initiative contract F49620-87-K-0001, the German Federal Ministry for Research Technology, and the U.S. National Science Foundation through grant BBS 8618066.

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