

# Instrumentation Advances And Detector Development With The Stony Brook Scanning Transmission X-ray Microscope

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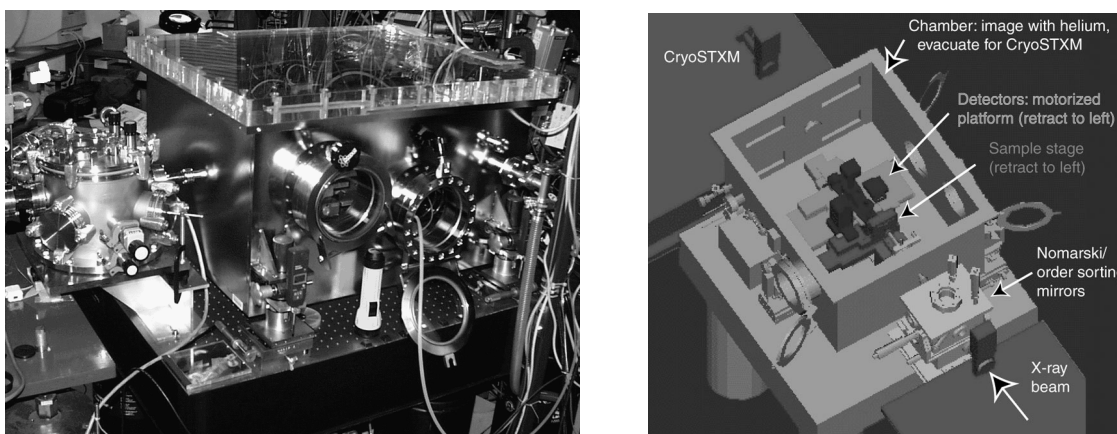
**Abstract.** Driven by the requirements of new x-ray microscopy instrumentation the Stony Brook microscopy beamline X-1A has undergone considerable evolution [1]. The room temperature scanning transmission X-ray microscope (STXM) has been completely redesigned improving performance, ease of use and compatibility with other experiments. We present the highlights of the new design, the available detectors and the result of early tests of this new microscope.

## INTRODUCTION

In this paper we describe the features of the redesigned room temperature STXM operating at beamline X1 of the National Synchrotron Light Source at Brookhaven National Laboratory. It succeeds a STXM [2] that has seen a lot of use during the last decade and has become a valuable tool for scientists addressing problems in biological, environmental, polymer science and other fields. In contrast to the old STXM, where most of the work has been done at the K-absorption edge of carbon, the new STXM extends that work to the K-absorption edges of nitrogen and oxygen, since it is designed to operate in a closed Helium atmosphere. Matched sets of zone plates (ZP) [3] and order sorting apertures (OSA) can be interchanged rapidly to accommodate for different working distance / resolution requirements. Three detectors can be mounted simultaneously on a platform that is motorized and computer controlled. For maximum signal to noise detection a continuous flow proportional counter is employed. A multi channel silicon detector with segmentation is under development that is matched to the geometry of a STXM. This detector has the potential to record not only the “bright field” (absorption map), but simultaneously record the “differential phase contrast” and “dark field” information that are present in the detector plane. Since many experiments at X1 share the beamlines, the new STXM is designed to allow rapid interchange with a cryo STXM [4] located downstream of the STXM.

## SEALED VACUUM CHAMBER WITH HELIUM ATMOSPHERE

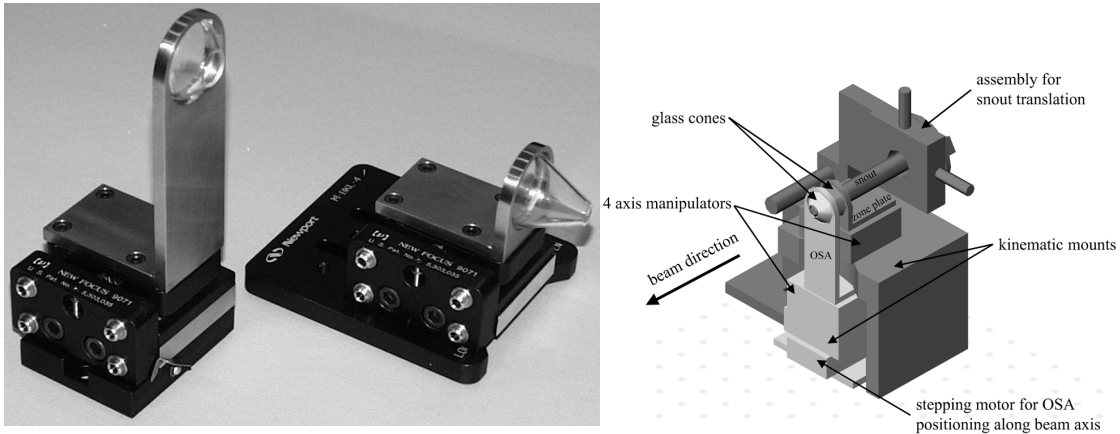
The K-absorption edges of oxygen and nitrogen are within the energy range of beamline X1. For high quality imaging and spectroscopy, oxygen and nitrogen have to be removed from the beam path. This is accomplished by putting all microscope components into a vacuum chamber (see Fig. 1). Since some microscope components are not suited for operation in vacuum, the chamber is evacuated and filled with Helium with sensitive equipment turned off during the evacuation period. First tests have shown that with one evacuation-refill cycle we can get the air content down to less than 0.1%, making quantitative oxygen and nitrogen edge spectroscopy work possible. Oxygen and nitrogen are abundant in samples of interest in biology, material and polymer science.



**FIGURE 1. Left:** The STXM installed at the beamline. **Right:** Schematic View. The vacuum vessel features a Plexiglas top and 3 view ports for accessibility. Evacuation and refilling with Helium takes about 10 minutes. A glove allows access to the interior of the chamber without breaking the seal to make sample changes possible. The chamber is kinematically mounted with 20  $\mu\text{m}$  positioning capability (translation and tilt). An additional vacuum chamber located upstream of the STXM holds a set of 4 mirrors that are used for order sorting and Nomarski differential interference contrast illumination. The Nomarski experiment is done in collaboration with F. Polack (Centre Universitaire, Orsay, France) and D. Joyeux (Institut d'Optique, Orsay, France) [5].

## MECHANICAL DESIGN

For diffraction limited imaging in a STXM it is essential to eliminate vibrations in the mechanical connection between the ZP and the sample. Wiggling of the probe on the sample spoils the spatial resolution. The microscope is mechanically isolated from the experimental floor by an air table. The arm that connects the ZP with the sample is kept as light and stiff as possible to minimize the amplitude of remaining vibrations. Fig. 2 shows the mounts of the ZP and OSA. Little or no adjustments have to be made when switching between matched pairs.



**FIGURE 2. Left:** Photograph of a set of ZP (right) and OSA (left) mounts. The ZP and OSA are glued to the end of the glass cones, which are connected to 4 axis manipulators on kinematic mounts. Once the units holding the ZP / OSA are aligned in the microscope, they can be removed and put back without losing the alignment. Many ZP / OSA pairs can be prepared to match the working distance / resolution requirements of different experiments. We are using circular glass cover slips with laser drilled holes as OSAs. **Right:** Perspective view of the ZP / OSA units mounted in the microscope. The glass cones fit into each other, the ZP is stationary and the OSA is motorized along the beam axis to accommodate for changes in focal length as the X-ray energy is tuned.

## ELECTRONICS AND SOFTWARE

The microscope electronics can acquire simultaneously 8 channels of analog and 6 channels of pulse data at 16 Bit precision. Multi-channel detectors can be used to increase the total flux capability or extract information of the spatial distribution of X-ray intensity in the detector plane.

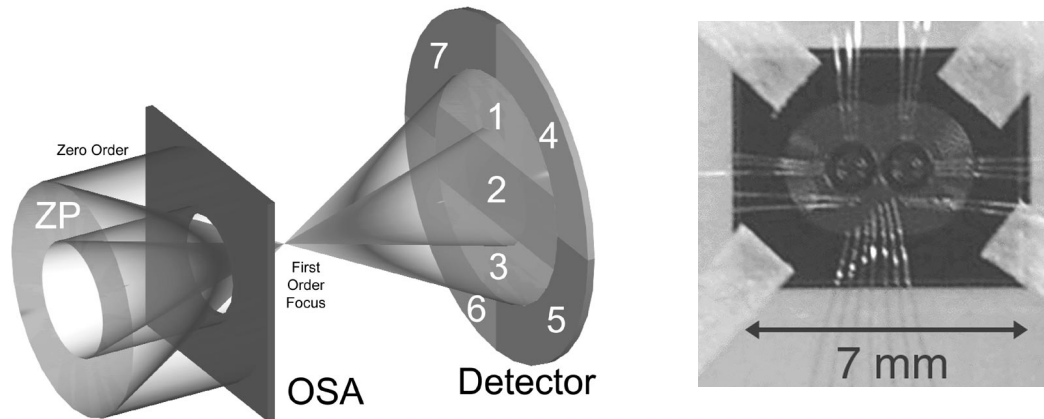
All electronics are running on a general purpose interface bus (GPIB) and communicate with a PII class PC equipped with a GPIB ISA bus card running Linux. The software controlling the microscope electronics is written in C++ making use of freely available toolkits for file I/O (Unidata NetCDF), GPIB communication (Linux Lab Project Driver), graphical user interface (Troll Tech Qt), compilation and programming (GNU make, GNU GCC).

## X-RAY DETECTORS

For maximum signal to noise detection a continuous flow multi wire gas proportional counter will be used. The counter operates with a 16 ns shaping time and has shown linear response to soft X-ray photons for countrates up to 1 MHz for one channel. This detector is not position sensitive and integrates over the intensity in the detector plane.

For high count rate experiments one can afford to accept the electronic noise of a charge integrating detector, since it adds only very little total noise compared to the photon shot noise of the experiment. A novel charge integrating detector with segmentation is being developed. Soft X-ray photons are being absorbed beneath a

shallow P/N junction in high resistivity silicon and create a charge proportional to the energy of the X-ray photon. The charge is integrated over a time window that is matched to the dwell time per pixel of the scanning microscope. Custom built low noise electronics generate a voltage proportional to the collected charge per time and segment that is read by the microscope electronics. The detector will be very attractive for count rates in excess of 100 kHz. The segmentation (see Fig. 3) is designed to pick up information about the spatial distribution of x-rays in the detector plane. Dark field contrast and Nomarski differential phase contrast are two applications of this scheme.

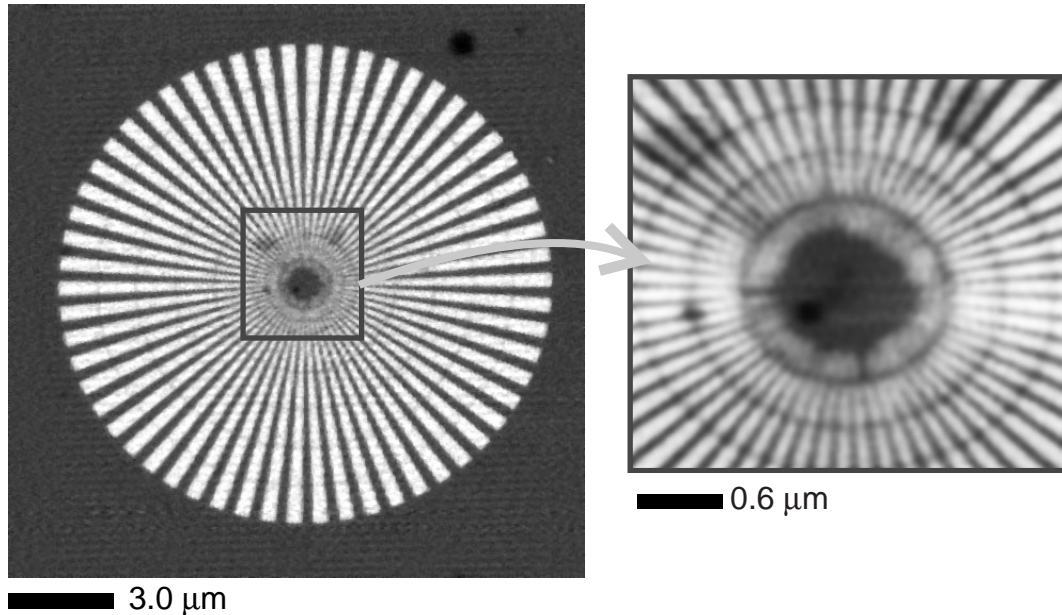


**FIGURE 3. Left:** The detector has 7 active segments (right). Segments 1-3 are matched to the bright field cone of light coming from the zone plate (left). The pinhole (OSA) selects the light focused to the positive first order. Segments 4-7 of the detector can be used for dark field and differential phase contrast measurements. **Right:** The silicon chip mounted on a PC board for first tests. Two detector structures are on each chip. The extra detector can potentially be used to subtract low frequency noise pickup.

The microscope has a detector platform for three detectors that can be positioned accurately and reproducibly with stepping motors. Switching between the visible light microscope (used for sample inspection and alignment) and X-ray detectors is computer controlled.

## HIGH RESOLUTION SCANNING STAGES – FIRST TESTS

For large area scans stepping motors with encoders that offer 0.2  $\mu\text{m}$  resolution and good reproducibility are used. For high resolution scans we employ a piezo actuated flexure stage with capacitance micrometers in closed loop feedback (PI model 731.20). Using the full 100  $\mu\text{m}$  of travel the minimum step size of this stage is 5 nm using a 16 bit digital to analog converter. A scan of a test pattern (see Fig. 4) demonstrates the high spatial resolution and operation without distortions.



**FIGURE 4.** First images of a test pattern taken with a 80  $\mu\text{m}$  diameter, 30 nm outermost zone width zone plate at a photon energy of 535 eV. **Left:** The large overview scan demonstrates the orthogonality and linearity of the fine scanning stage. **Right:** A high resolution scan of the inner part of the test pattern. The images have been recorded with a non-optimized detector that did not allow diffraction limited imaging and thus do not represent the resolution limit of the zone plate used.

## ACKNOWLEDGEMENTS

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