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Development of a Novel Apparatus for Experiments in Soft X-ray Diffraction Imaging and Diffraction Tomography

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Abstract. We report the development of a novel experimental chamber for experiments in soft x-ray diffraction tomography, diffraction imaging of single biological objects, and magnetic speckle imaging. The chamber will allow for acquisition of nearly full three-dimensional diffraction data sets as well as high magnification zone plate images of holograms for the diffraction tomography experiment.

1. INTRODUCTION

In order to obtain high resolution 3D images one has to collect several hundreds of views of the specimen. To make this feasible, it is necessary to automate the data collection process. In addition, one has to protect the sample against radiation damage from the incident X-rays.

2. DESCRIPTION OF THE APPARATUS

2.1 Goniometer and specimen holder

To minimize radiation damage during data acquisition, the sample is cooled to liquid nitrogen temperature using a Gatan 630 cryo specimen holder [1]. The geometry of this holder together with the Gatan sample grids (see Figure 1) provide us with a tilt range of ±80°. In order to reach automated data collection, the sample holder is translated and rotated by a motorized 4 axis JEOL FasTEM goniometer system [2] (see Figure 2)

Figure 1: Close-up view of the Gatan 630 ultra high tilt holder with specimen grid.

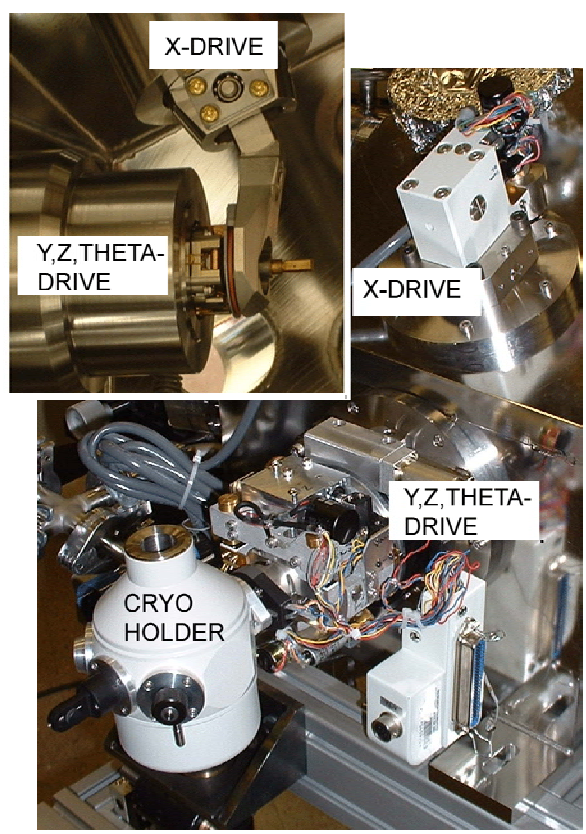

Figure 1: Close-up view of the Gatan 630 ultra high tilt holder with specimen grid.

2.1 Optics and beam defining apertures

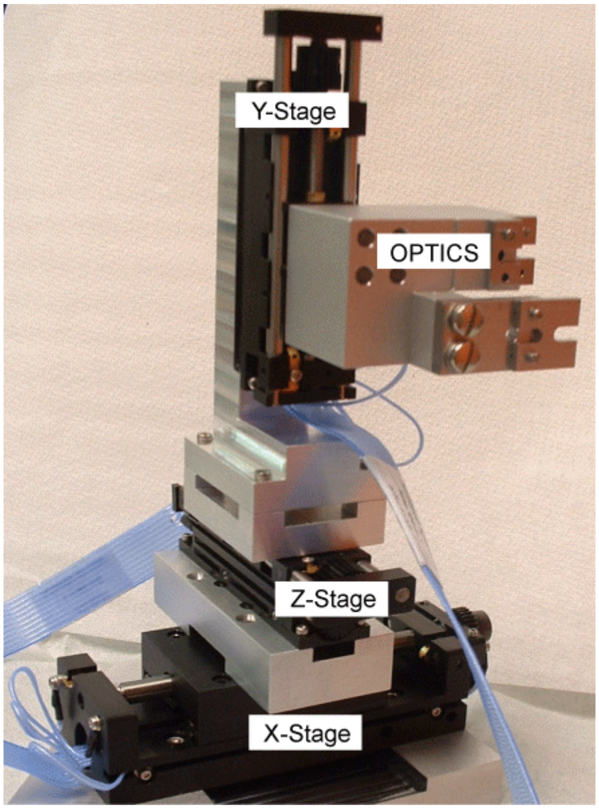

Figure 3: View of the motor stack with optics mount.

To quickly align various optical components like zone plates, beam defining apertures or phosphor screens, the new experimental chamber makes use of two in-vacuum xyz motorized translation stages with sub-micron precision [3]. One of the motor stacks is positioned upstream of the sample, the other downstream of the sample. The optics mounts are designed to hold several optical components to give great flexibility to change components without breaking vacuum. All stages can be scanned using the custom control and data acquistion software.

2.2 Detector system

The detector is a completely in-vacuum backside illuminated CCD camera from Roper Scientific (1340x1300 with 20 micron pixels) [4]. Modern CCD cameras can go to quite long exposure times with thermoelectric cooling, and this cooling method also allows for rapid camera warm-up time in case the chamber needs to be vented to atmospheric pressure. The detector system can be translated parallel to the beam axis inside the chamber over a distance of 17 cm. Its position closest to the specimen, 10 cm, allows for recording high resolution diffraction data and its furthest position from the specimen, ~1 m with the insertion of an extension tube, allows for high magnification zone plate imaging in diffraction tomography. A motorized stage underneath the camera makes it possible to translate the camera transverse to the beam by 5 cm to record off-axis zone plate images or higher resolution diffaction data. A motorized in-vacuum stage in front of the CCD camera carries a photo diode for alignment purposes. A choice of beamstops to block out the direct x-ray beam can be mounted on an xy motor stack.

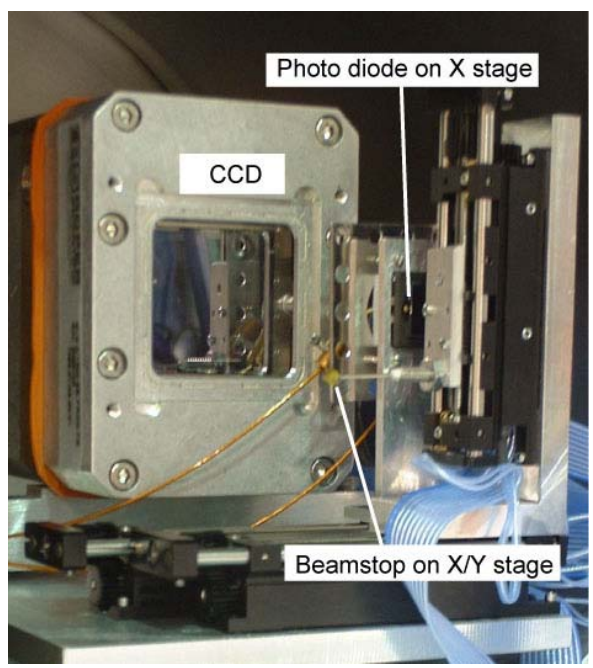

Figure 4: View of the in-vacuum CCD camera together with photo diode and beamstop stages.

2.3 Motion control and data acquisition

All stages and goniometer motors are controlled by two Galil motion controllers via a private network [5]. A custom built amplifier provides the necessary current to drive the motors. Custom control software allows to move or scan all motors and collect CCD images. A graphical user interface is in preparation.

2.4 Chamber overview

The whole experimental chamber is mounted on a moveable frame, equipped with precision actuators to allow for a fast alignment at different beamlines (see Figure 5). The chamber has a number of viewports and an access door, making alignment and adjustments straightforward so that the downtime is minimized.

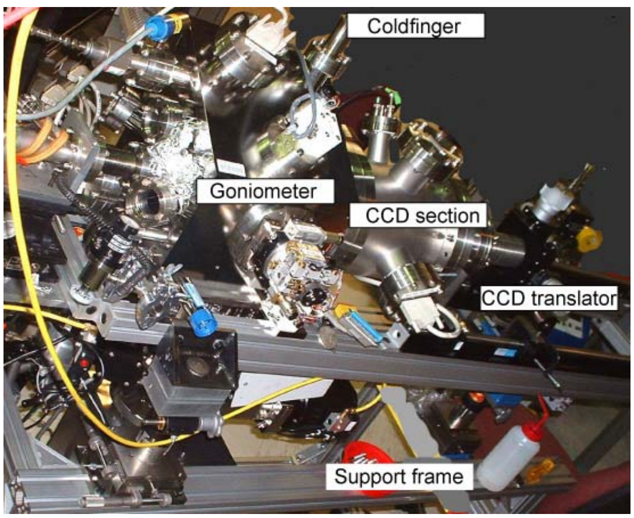

Figure 5 : Photograph of the experimental chamber including the CCD section and support frame.

The frame is also equipped with a linear slider underneath the CCD section which allows the CCD section to slide back and insert an extension tube needed for diffraction tomography (see Figure 6).

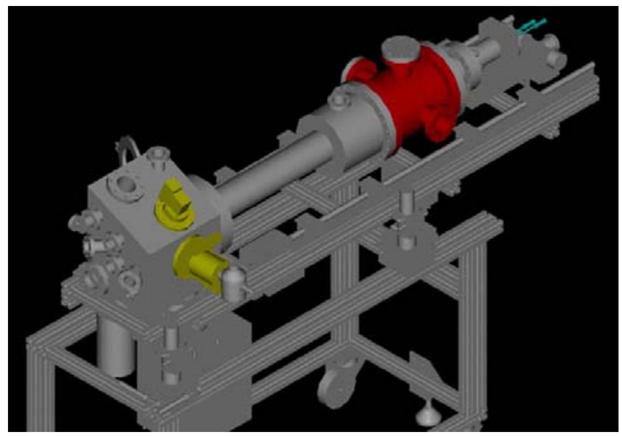

Figure 6 : Schematic view of the setup including the extension tube

3. APPLICATIONS

3.1 Diffraction imaging

The goal of this project is to obtain a high resolution, three dimensional map of the complex index of refraction of a single biological particle using the methods of x-ray diffraction. The specimen, an air dried or frozen, hydrated yeast cell, is illuminated with a highly coherent beam of soft x-ray photons (E=530 eV) from the X1B undulator of the NSLS at Brookhaven National Laboratory. The oversampled diffraction pattern of the specimen is recorded on a backside thinned CCD camera and subsequently fed into an iterative algorithm for phasing. The new apparatus allows for rotation of the specimen for 3D and high resolution 2D experiments and is designed to collect diffraction data to 10 nm resolution.

3.2 Diffraction tomography

In diffraction tomography the sample is illuminated with a coherent beam and the wavefield (hologram) after the object is recorded on a detector. The sample is then rotated and holograms are recorded at each rotation angle. To reconstruct the sample each hologram is backpropagated numericaly and the resulting wavefields are added up to yield a 3D reconstruction of the sample. For more details see the accompanying paper in this proceedings [6].

3.3 Magnetic speckle imaging

The enhancement of the X-ray magnetic cross section at certain resonances, like L-edges of the transition metals, allow us to probe the magnetic structure using coherent X-rays. The phase problem, which is inherent in diffraction experiments, is tackled by a direct method that relies on complementary information from measurements at multiple wavelengths around the resonance [7]. The setup can be changed from transmission to reflection geometry to accommodate samples with different easy-axis directions. Dynamic speckle measurements on magnetic samples is included in the future plans.

Acknowledgments

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References

[1] Gatan Inc., www.gatan.com
[2] JEOL USA, www.jeol.com
[3] National Aperture Inc., www.nationalaperture.com
[4] Roper Scientific, www.roperscientific.com
[5] Galil Motion Control, www.galilmc.com
[6] T. Beetz, et al., Soft X-ray diffraction tomography: simulations and first experimental results, this proceedings
[7] T. O. Mentes, et al., *J. Synchrot. Radiat.* **9** (2002) 90-95