# MECHANICAL ENGINEERING OF A CRYO STXM AT CLS*

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*Abstract*

A Scanning Transmission X-ray Microscope (STXM) is a useful imaging tool, but its application to certain types of samples is limited by significant rates of x-ray damage to the sample. Cooling samples to liquid nitrogen temperatures can delay radiation damage, but must be done in a vacuum environment to prevent rapid formation of ice on the sample. The Canadian Light Source (CLS) has constructed a Cryo-STXM, which can maintain sample temperatures at 100 K in an ultra-high vacuum environment and rotate the samples in the beam to collect tomographic data sets. This presentation will discuss the mechanical engineering aspects of the development of this Cryo-STXM including the finite element analysis (FEA) for stresses and vibrations, and present the performance parameters being achieved by the instrument.

## INTRODUCTION

The Canadian Light Source (CLS) is a third-generation synchrotron located in Saskatoon, Canada. One of the Phase 2 beamlines constructed at CLS was the spectromicroscopy (SM) beamline [1], an insertion device (ID) beamline with an ID in straight 10 of the CLS storage ring. The SM beamline has been operational since 2005, however, the EPU was not installed until May 2006. Before this the beamline used upstream bend magnet radiation to operate. Two end-stations were originally included on the SM beamline: a photon emission electron microscope (PEEM) and a scanning transmission x-ray microscope (STXM) which started operation in 2007.

The SM beamline is currently undergoing an upgrade. The first phase of this upgrade is the construction and installation of a cryo-STXM. The goal is to have a STXM with sub-30 nm resolution that is capable of cryogenically cooling the sample to near liquid nitrogen temperatures, and perform tomography measurements on the sample. There are currently very few cryo-STXMs in the world, and the one at CLS is the first soft x-ray STXM capable of tomography through a 90 degree rotation in both directions. As of August 2016 the cryo-STXM is just completing its final benchmark experiments, and is now operating at the CLS SM beamline.

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The mechanical design phase of the cryo-STXM project presented CLS with some interesting problems. This paper will briefly discuss the SM beamline, and will present an overview of the cryo-STXM. A few specific mechanical engineering issues related to the development of the instrument are examined, and finally the performance of the cryo-STXM to date is reviewed.

## THE CLS SM BEAMLINE

Figure 1 shows the layout of the SM beamline at CLS. The SM beamline uses an elliptically-polarizing undulator to supply synchrotron radiation. Downstream of the M1 mirror, a plane-grating monochromator (PGM) filters the desired energy out of the supplied spectrum. The range of energy that the SM beamline is designed to make use of is from a low end of roughly 130 eV to a high end of up to 2500 eV, placing it in the soft x-ray range.

From the PGM the beam proceeds through two further mirrors, M3PEEM and M3STXM, which split the beam and direct it down one of the branch lines. One ray proceeds through another mirror before reaching the PEEM, while the other goes directly into the existing STXM.

Table 1 shows specifications of the CLS SM beamline.

Table 1: CLS SM Beamline Specifications

| | |
|---|---|
| Spot size (STXM) | 30 nm |
| Spot size (PEEM) | 50 nm |
| Energy Resolution (E/ΔE) | 3000-10000 |
| Energy Range | 130-2500 eV |
| Wavelength | 95-4.5 Å |
| Photon Flux (STXM) | ~$10^8$ ph/sec in 30 nm spot |
| Photon Flux (PEEM) | ~$10^{12}$ ph/sec in 50 nm spot |

The regular STXM is capable of several techniques, including near-edge x-ray absorption fine structure (NEXAFS) in transmission mode, total electron yield (TEY) mode, and x-ray fluorescence (XRF) mode, soft x-ray tomography, x-ray linear dichroism (XLD), x-ray magnetic circular dichroism (XCMD), and ptychography. The goal of the new cryo-STXM would be to use some of these techniques, specifically TEY and ptychography, on a sample that can be kept at cryogenic temperatures to delay radiation damage and can be rotated +/- 70 degrees in the beam for tomography.

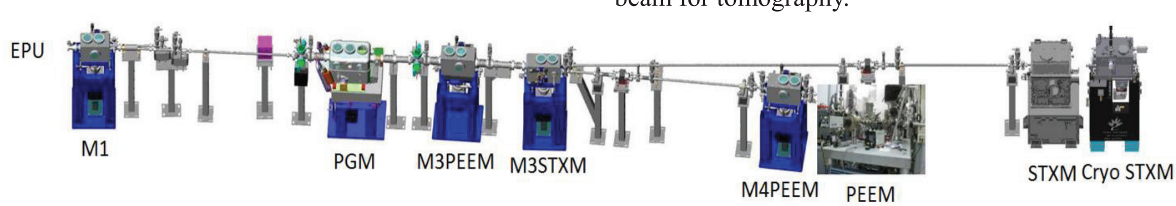


Figure 1: The layout of the SM beamline at CLS.

## STXM BASICS

There are three basic components to a STXM: the zone plate, the sample and associated sample holder, and detector. These components are shown in Fig. 2.

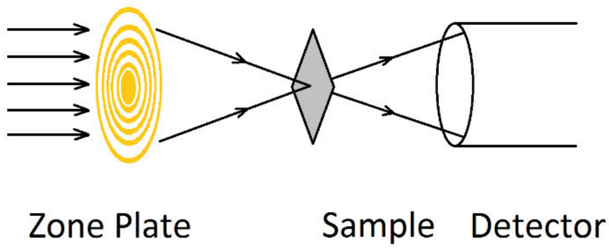


Figure 2: Fundamental components of a STXM.

The zone plate is a focusing device made of circular slits, using diffraction to create a focal point where, ideally, the sample is located. Zone plates at CLS are etched into a thin layer of gold coating a silicon nitride window, and the beam spot size is limited by the size of the etching on the zone plate. Several different types of detectors may be used. Photo-multiplier tubes (PMTs) are typically located behind the sample, while devices such silicon-drift detectors (SDDs) for x-ray fluorescence must be located to the side of the sample. The arrangement at CLS also uses an order-sorting aperture (OSA) [2] between the zone plate and sample to block zero- or higher-order radiation.

In order to place the sample in the focal point of the beam, the zone plate, OSA, sample, and detector can all be moved independently. Typically three of these components can be moved in x,y and z, while the fourth will only move in x and y, but in some cases all four can move in x,y and z. The motion of these components necessitates a significantly complex base of motion stages which, when considering that both coarse and very fine motions are desired, requires 3-axis coarse stages and 3-axis fine stages for each component.

The precision of the zone plate and the < 30 nm beam spot size means that the sample and/or zone plate must be positioned accurately. This requires feedback measurement of the sample position on the order of 3 nm or less. These types of measurements require a laser interferometer along with mirrors mounted on the sample or zone plate stage to reflect the laser signal.

The CLS STXM beam spot size of < 30 nm requires extreme stability to achieve. Vibrations with an amplitude of 15 nm will effectively move the beam spot to a completely new area of the sample between vibrational peaks and valleys. Any motion results in a "blurring" of the image or a distortion of the data collected. Vibration damping techniques are critical to realizing a clear image at small spot sizes.

## THE CRYO-STXM

The design of the cryo-STXM had several design requirements. These included:

- STXM with < 30 nm resolution
- Sample cooling to near liquid nitrogen temperatures
- Ability to introduce a cold sample through a load-lock
- Sample tomography (rotation) to +/- 70 degrees or more
- Near ultra-high vacuum (UHV) or full UHV chamber to prevent ice formation on the sample
- Can also function as a conventional STXM and at atmospheric pressure.

It should be noted that this combination of requirements is unique for any device in the world [3].

From these goals a set of engineering parameters were generated. The mechanical parameters of the cryo-STXM are listed in Table 2.

Table 2: Cryo-STXM Mechanical Design Specifications

| | |
|---|---|
| Vacuum Level | ≤$10^{-8}$ Torr |
| Sample Temperature | ≤ -175 C (98 K) |
| Vibration at Sample | ≤ 5 nm RMS from 0 to 200 Hz |
| Zone Plate Scanning Resolution (X,Y) | ≤ 1 nm |
| Conventional Sample Scanning Resolution (X,Y) | ≤ 5 nm |
| Detector Stage Resolution (X,Y) | ≤ 50 µm |
| Sample Rotation | ≥ +/- 70 deg |

## THE CRYO-TOMO UNIT

While UHV and motion requirements are typical for modern STXM units, the unique part of the CLS cryo-STXM is the cryo-tomography capability. In a general sense, tomography is imaging using data taken from some type of penetrating wave or mechanical method. In the case of the CLS cryo-STXM, cryo-tomography is the process of rotating a cryogenically-cooled sample to various angles in the beam, taking images at these angles, and then constructing a 3-D model of the sample based on this imaging.

Cryo-tomography work has been done in the field of electron microscopy, and options exist in this field for equipment capable of both rotating a sample and keeping it cooled to cryogenic temperatures. Through contacts in the field, CLS became aware of a JEOL goniometer available from Dr. Chris Jacobson at Northwestern University. The goniometer was not in use, but had previously been part of a successful imaging end station at the Advanced Light Source. Dr. Jacobson was contacted, and graciously agreed to allow CLS to use this goniometer in the cryo-STXM. Figure 3 [4] shows a model of this goniometer.

The JEOL goniometer is capable of 2.25 mm motion in the x-direction (inboard-outboard), and 0.8 mm and 1.0 mm travel in the y- (up and down) and z- (along the beam) directions respectively. This is sufficient for fine travel scanning, however, scanning can also be done with the zone plate, meaning the goniometer would only be required to rotate or to do coarse sample moves if required. This is the standard method that will be used for cryo-tomography in the cryo-STXM. Rotational motion was tested to be +/- 90 degrees, exceeding the CLS specification.

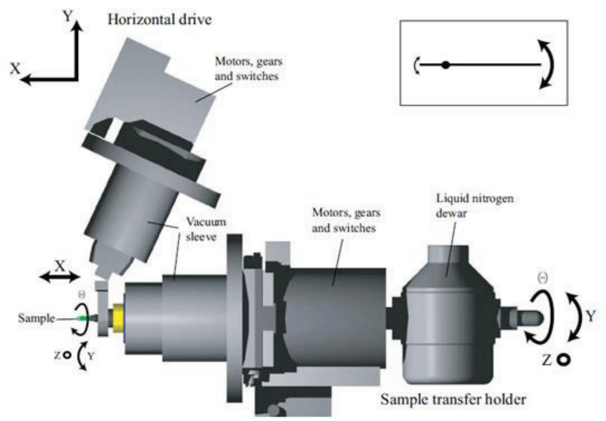


Figure 3: JEOL cryogenic goniometer.

The goniometer also contains a load-lock device, allowing introduction of cold samples into the STXM vacuum. This is done via a vacuum valve, differential pumping, and o-ring seals.

The size and shape of the JEOL goniometer dictated much about the size and shape of the vacuum chamber required. The chamber needed to accommodate the angular two-section goniometer design, and to have the appropriate space for the required stages, detectors and peripheral equipment and connections.

While the goniometer was complete, work was required to repair and upgrade the motion control. CLS undertook work to replace the motors, cables and drivers for the device before testing the motion.

## DESIGN PROBLEMS AND SOLUTIONS

### *Positioning*

The zone plate and sample motion resolutions are extremely fine, and require piezo flexure-based motion stages and laser interferometry feedback for accurate positioning. The Renishaw RLE20 was selected as the laser interferometer for sample and zone plate positioning. The RLE20 has a resolution as low as 0.39 nm, but the laser head is not vacuum-compatible so it was necessary to mount the head on the outside of the cryo-STXM vacuum chamber.

The mounting location outside of vacuum chamber makes laser pitch and yaw adjustments simple, since vacuum does not need to be broken to do these. However, there were design concerns regarding the flex of the chamber walls under vacuum relative to their positions at atmospheric pressure. This required careful analysis of the deflections of the vacuum chamber walls at vacuum pressures, and design of the chamber to ensure these deflections were not excessive. Geometric calculations showed that a deflection of 90 µm would be enough to cause problems with the interferometers, but it was agreed that a smaller deflection of less than 35 µm would be specified.

Also important were deflections of the chamber floor, to which all of the motion stages are mounted, and of the goniometer mounting locations. Large deflections here could result in alignment issues with the sample, and because the goniometer has two separate vacuum penetration points that connect inside the vacuum space misalignments could cause motion control issues and binding.

A basic ANSYS finite element analysis (FEA) was performed on the chamber to determine wall deflections. Initial results showed larger than tolerable deflections even with significant wall thicknesses, so ribbing was added to the outside of the chamber. After three or four ribbing design iterations a design was found that reduced deflections to less than 35 µm at the critical points: goniometer and interferometer mounting locations.

The ANSYS images for the cryo-STXM vacuum chamber deflection are shown in Fig. 4. Note that the red colour represents 50.6 µm of deflection, which is the maximum for the structure. Yellow-green represents deflections between 28 and 33 µm, and green represents deflections between 22 and 28 µm.

Once the cryo-STXM was constructed it was pumped down with dial-gauges in place as shown in Fig. 5 to measure deflections at the interferometer heads. The deflection at the x-head was measured at 29 µm, as compared to an ANSYS-predicted value of 28 µm, and at the y-head deflection was measured at 25 µm as compared to an ANSYS-predicted value of 15 µm. While the y-head deflection was 60% higher than predicted, this is a good demonstration of the design accuracy that can be achieved using FEA software. Both measured values are well below the required maximum, and to this point the interferometers have performed flawlessly.

### *Vacuum*

To prevent rapid ice formation on a cryogenically-cooled sample it is important to have the sample in a good vacuum. At liquid nitrogen temperatures carbon dioxide and water will freeze out of the air onto the sample, and even gases like oxygen will liquefy on the sample. These deposits will interfere with STXM measurements.

There are several ports on the vacuum chamber, and it was important to ensure a proper seal at each one. However, the importance of the seal had to be balanced with the necessity for ease of access to the chamber interior to change samples, adjust internal settings, or perform minor repairs. For this reason, o-rings were used for the majority of the seals on the chamber ports.

To facilitate access to the stages, it was decided that an o-ring would also be used to provide the vacuum seal between the chamber and the baseplate. However, because of the length of this seal it was decided to design it as a double o-ring with differential pumping between the o-rings. Some of the larger access ports were also designed in this fashion. Conflat® flanges were used wherever regular access was not required.

After baking at 60°C for eight days, vacuum levels in the chamber have been measured to be as low as $7.7 \times 10^{-9}$ Torr, better than the design goal of $2 \times 10^{-8}$ Torr.

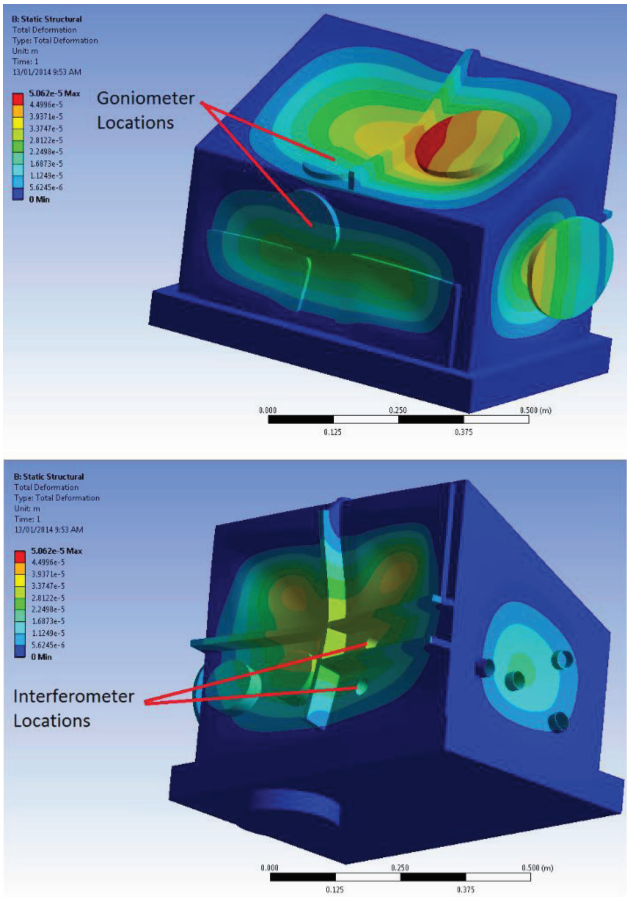


Figure 4: ANSYS results for cryo-STXM vacuum chamber deflections. The goniometer and interferometer mounting locations are indicated.

## *Vibration*

Analyzing vibration is, at the best of times, very difficult. The specification required vibrational amplitudes of less than 5 nm root-mean-square (RMS), but during the design phase calculation of these values is complicated by the complex shape of the model and the unknowns in, or changes to the input frequency spectrum. For this reason, the design focused more on minimizing calculated vibrations rather than achieving the parameter of < 5 nm RMS.

Analysis of vibrational spectra shows that almost all of the power in a vibrational signal is realized in the low frequencies, less than 100 Hz. Standard design practices for instruments that require high vibration isolation include mounting the instrument on a concrete or zanite (polymer concrete) block. Examining the unit as a dynamic system, we can say that the block and instrument will move according to Eq. (1).

$$m\ddot{x} + b\dot{x} + kx = F \tag{1}$$

In Eq. (1), $m$ is the mass of the block-instrument combination, $b$ is the natural vibrational damping provided by the block-instrument system, $k$ is the natural spring constant for the block-instrument system, $F$ represents the applied force, and $x$ represents the position in space of the sample point.

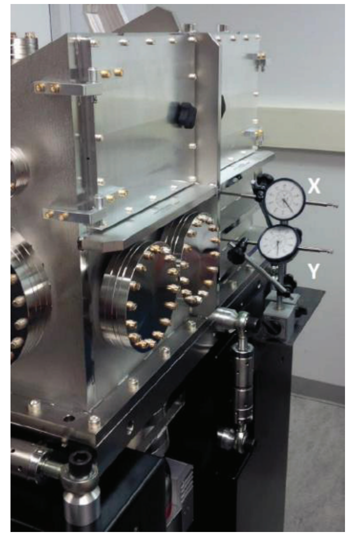


Figure 5: Dial gauges for measuring chamber deflection at the interferometer head locations.

The variables $\dot{x}$ and $\ddot{x}$ represent the velocity and acceleration of the sample point respectively. Basic dynamic analysis shows that Eq. (1) can be represented as

$$\ddot{x} + \frac{b}{m}\dot{x} + \frac{k}{m} = \ddot{x} + 2\xi\omega_n\dot{x} + \omega_n^2 \tag{2}$$

where $\xi$ is the damping constant for the block-instrument system and $\omega_n$ is the natural frequency of the block-instrument system. It can be inferred from Eq. (2) that

$$\omega_n = \sqrt{\frac{k}{m}} \tag{3}$$

meaning the natural frequency increases with and increase spring stiffness and decreases with an increase in mass.

A dynamic system will maximize its vibrational amplitude at its natural frequency, meaning that in order to prevent vibrations at low frequencies of 0-200 Hz it is desirable to design the block-instrument system's natural frequency to be as high as possible. From Eq. (3) it can be seen that to do this, the block should be very stiff but have relatively low mass. Zanite and concrete fit this description well, since these substances are extremely hard and therefore stiff, and at densities in the neighbourhood of 2600 kg/m$^3$ are relatively light compared to metals. Note that Styrofoam is another material that has a relatively high stiffness to weight ratio, largely due to its extremely low weight. Styrofoam feet are used on the cryo-STXM to support the Zanite block and provide some additional cushion.

The cryo-STXM itself requires its materials to be selected for properties other than high stiffness and low mass, so in order to avoid potential areas of lower frequency resonance a modal analysis of the unit was done with ANSYS.

The purpose of this modal analysis was not to calculate exact resonance frequencies but rather to look for areas with generally lower frequency resonance characteristics and determine how to design these to avoid vibration. Only one mode was found, on the DN160 vacuum valve separating the turbo-molecular pump from the main chamber, that was at any frequency close to 100 Hz. Figure 6 shows this mode. The natural frequency of this mode was increased by designing a clamp to hold the valve body firmly.

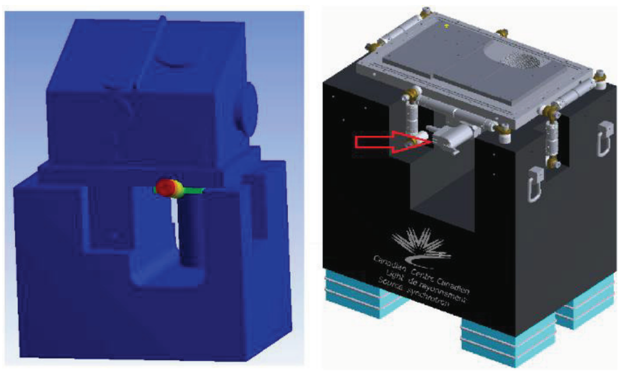

Figure 6: Lowest vibration mode on the cryo-STXM on the left. On the right is a picture of the Zanite block and STXM baseplate, with the offending valve body indicated by a red arrow.

Another aspect of vibrational design is to reduce or eliminate possible sources of vibration on the device itself. For the cryo-STXM the main sources of potential vibrations are the vacuum pumps. For scroll pumping vacuum conductance is generally not a concern, so the scroll pump was simply located at some distance from the cryo-STXM and placed off of the experimental floor. The turbo-molecular pump selected was a Pfeiffer HiPace 700M, which is a 700 L/s turbo that is magnetically levitated on five axes.

With all of these vibration considerations implemented, the Renishaw laser interferometers were used to measure the vibration of the sample stage. With the cryo-STXM on the experimental floor at CLS, the vacuum pumping in operation, and using active vibration damping via the stages, the interferometers measured an RMS vibration amplitude at the sample of less than 1 nm. This was better than the design goal of 5 nm by a significant margin.

## PERFORMANCE

Figure 7 shows a picture of the cryo-STXM at CLS.

As described, the steps taken to analyse vibration and chamber deflection resulted in a design that met its goals in these parameters. However, the true test of the cryo-STXM is the imaging that it is capable of performing. Initial tests have provided images with resolutions down to 30 nm, which is the design goal and near the limit of the zone plate.

## CONCLUSION

CLS has constructed a cryo-STXM capable of performing tomography on a cryogenically-cooled sample. This device is the first STXM in the world with this capability. During the design phase, careful consideration was paid to chamber wall deflections and vibration in the unit. This work resulted in a high-performance cryo-tomo-STXM that met or exceeded design goals.

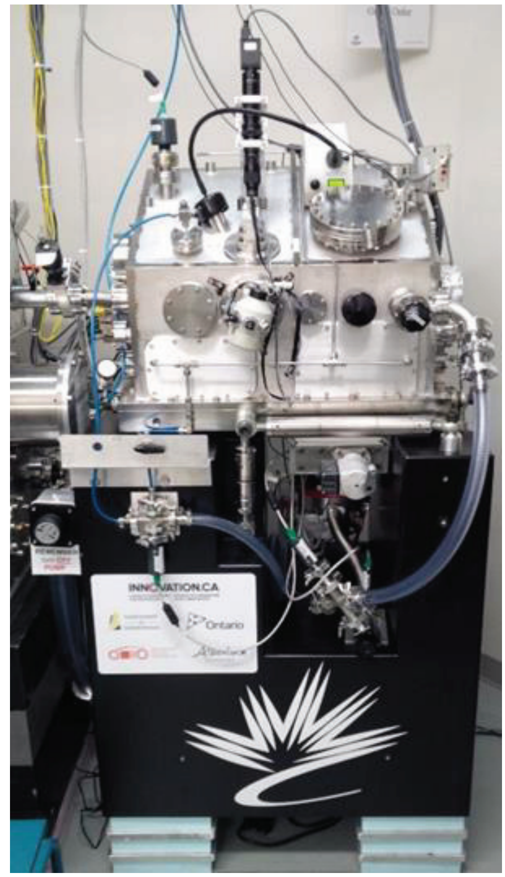


Figure 7: CLS cryo-STXM on the SM beamline.

## ACKNOWLEDGMENT

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## REFERENCES

[1] K. V. Kaznatcheev et al., "Soft X-ray Spectromicroscopy Beamline at the CLS: Commissioning Results," *Nucl. Instr. Meth.*, vol. 582, pp. 96-99, 2007.

[2] T. Warwick et al., "Development of Scanning X-ray Microscopes for Material Science Spectromicroscopy at the Advanced Light Source," *J. Synch. Rad.*, vol. 5, pp. 1090-1092, 1998.

[3] A. P. Hitchcock, "Soft X-ray Imaging and Spectromicroscopy," in Handbook of Nanoscopy, G. van Tendaloo, D. van Dyck, S. J. Pennycook, Eds. Weinheim, Germany: Wiley, 2012, vol. 2, pp. 745-792.

[4] T. Beetz, "Soft X-Ray Diffraction Imaging With and Without Lenses and Radiation Damage Studies," Ph.D. thesis, Phys. Dept., Stony Brook University, Stony Brook, NY, U.S.A., 2004.