nanoXCT-100

Laboratory Tool
User Manual

Version 1.0
Part Number: G000246

nanoXCT™ Laboratory Tool User Manual

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Chapter 1: Potential Hazards and Safety Precautions

The nanoXCT is designed to be safe for the operator as well as maintenance and service personnel. However, potential hazards to the operator and the instrument do exist. This section describes the potential hazards, their isolation, and control methods. Please read and understand this chapter before running or maintaining the instrument.

1.1 Personnel hazards

The nanoXCT uses x-rays up to 35-40 keV when imaging. Consequently, a high-voltage power supply is required to generate the high-energy radiation, and a shielding enclosure is required to contain the radiation. Potential hazards to the operator are described below.

1.1.1 High voltage

Severity: Major to hazardous
Likelihood of occurrence: Extremely remote

The x-ray source requires high voltage of up to 40 kV in normal operation. The high-voltage power is within the x-ray source in the enclosure.
Hazards from electrical sources of up to 250 V are also present. Severe or catastrophic injuries are unlikely and minor to moderate injuries are rare. The risk from electrical systems is low.

Potential damage

Severe to catastrophic injuries may result from electrocution by the high voltage source. Electric shock from hazardous voltage sources may result in minor to moderate injuries.

Hazard control

All high voltage sources are sealed and interlocked. Sources with voltages of up to 120 V are contained in locked cabinets and require a key to access. Operators and maintenance personnel should follow proper lockout/tagout procedures before attempting any service on the nanoXCT. Do not touch unknown conductor leads in the system or use conductive fluid near the x-ray source or its power supply. The optical table and all components mounted to the optical table are earth-grounded. Electrical faults will be shorted to ground. Depress the EMO button in an emergency.

1.1.2 Ionizing radiation

Severity: Major to hazardous
Likelihood of occurrence: Extremely improbable

The x-ray source generates x-rays up to 35-40 keV, with intense peaks at 5.4 or 8.0 keV used for imaging.

Potential damage

Prolonged exposure to x-ray radiation can cause moderate to severe illness to the personnel mostly in the form of soft tissue damage or cancer (in severe cases).

Hazard control

The radiation is isolated from the user by a protective enclosure with fail-safe interlock systems. The radiation level outside the enclosure is not measurable above background level during normal operation. Please do not attempt to defeat the interlock system. Press the EMO button in an emergency. Xradia

personnel will perform a radiation survey during a regularly scheduled service every 12 months. The radiation survey should also be performed if any part of the enclosure is modified.

1.1.3 Pinch hazards

Severity: Minor
Likelihood of occurrence: Remote

The radiation-shielding enclosure is made of steel plates with considerable weight. Placing body parts in their path when it is being closed can cause bodily injury. Minor injuries may occur in rare occasions. Serious injuries are extremely unlikely. The risk from the pinch hazards is minimal.

Potential damage

Physical pain or minor injuries can result from the pinch.

Hazard control

Exercise caution when opening or closing the door to the enclosure. If body parts are caught in a closing enclosure, simply reverse the motion of the enclosure to free them.

1.1.4 Magnetic field

Severity: Minor
Likelihood of occurrence: Extremely remote

The nanoXCT contains a number of motors that generate low levels of magnetic field, as well as magnetic latches to seal the enclosure doors. Minor damage to personnel or equipment may occur, but this is extremely unlikely. The risk from magnetic fields is slight.

Potential damage

The magnetic field strength is extremely low. Damage to personnel or equipment is extremely unlikely.

Hazard control

Be aware of the presence of the magnetic field around the motors and enclosure. Persons using a pacemaker or with objects that are sensitive to magnetic field should exercise caution.

1.1.5 Hazardous materials

Severity: Hazardous
Likelihood of occurrence: Extremely remote

The nanoXCT contains the following materials that may be hazardous. Please be aware of their presence and us caution when working inside of the instrument.

- **Beryllium** - Beryllium windows are used on the x-ray source, condenser chamber, and flight tube. The total quantity of beryllium is approximately 20 g. Windows on the condenser chamber and flight tube are exposed, and personnel should exercise caution when working around them.
- **Thallium doped Cesium Iodide** – Thallium doped Cesium Iodide is integrated into the detector system. The quantity is approximately 2.5 mg. The Thallium content is approximately 1% by weight. It is not exposed, and risk of exposure to the operator or service personnel is very low. Xradia will be responsible for disposal when necessary.

- **Lead** – The inside of the x-ray source contains a small lead shutter. These parts are not accessible to the operator or personnel performing routine maintenance.

Please refer to the appropriate MSDS for details on each material.

1.2 Equipment hazards

1.2.1 Collision of moving parts

Severity: No safety effect
Likelihood of occurrence: Remote

Motorized components may collide when they are moved out of their normal operating ranges. Collisions are most likely to occur when moving all components during homing, or when using the standard clip sample holder and rotating the sample theta stage to high angles. Damage will be minor in most cases. Severe damage will be rare. The risk from collision is low.

Potential damage

Collisions may cause misalignment and, in more severe cases, damage to sub-components.

Hazard control

Use caution when moving motors. Avoid moving a motor to a position outside its normal operating range. Make sure that the sample holder is correctly placed on the sample stage when loading a sample into the microscope. Watch the components during homing. Perform one rotation to the maximum angle and watch the motion before beginning tomography. Depress the MOTION STOP button in an emergency.

1.3 Explanation of safety labels

1.3.1 Safety labels

Symbol	Meaning
	This location is a ground point. All points bearing this indicator make electrical contact with each other.
	The equipment behind this indicator may only be serviced by qualified personnel. This extends to Xradia service personnel and others as authorized.
	The equipment produces ionizing radiation primarily in the x-ray spectrum. There should be no detectable radiation outside of the enclosure during normal operation, but radiation is present inside the enclosure.
	This cover is not to be removed except by

	authorized personnel employing appropriate safety precautions.

Table 1-1: Safety labels located throughout nanoXCT

Chapter 2: Overview of Components

The Xradia nanoXCT laboratory tool consists of a rotating anode x-ray source, train of x-ray optics, and a high-resolution detector. A brief overview of each component is outlined here, but a detailed explanation of the physics behind operation is presented elsewhere.

2.1 Overview of Components and Staging

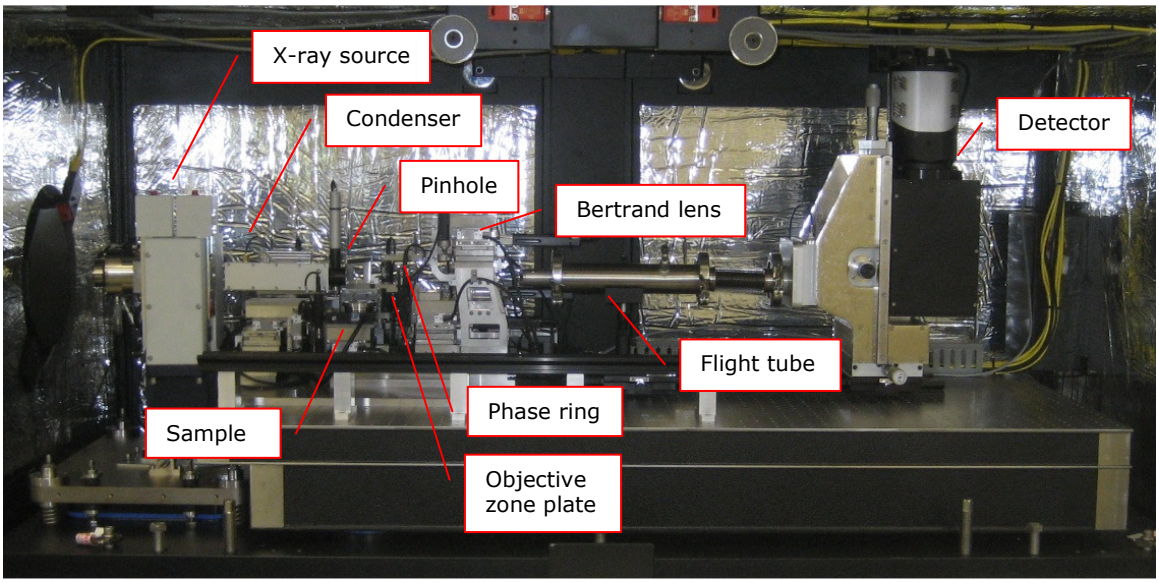


Fig. 2-1: Overview of the nanoXCT components.

2.2 X-ray source

Fig. 2-2: Rotating-anode x-ray source.

The nanoXCT laboratory tool uses a rotating anode x-ray source with either copper (8 keV) or chromium (5.4 keV) target material. Accelerated electrons from the filament (cathode) collide with the anode, temporarily dislodging electrons in a ~70 μm spot (FWHM) on the surface of the anode to excited states. When these electrons cascade back to their stable states, radiation of characteristic spectra is produced, depending on the target material. If a copper

anode is used, the peak of highest intensity is the K_α line, which corresponds to ~ 8.0 keV; for chromium, the most desirable peak is at ~5.4 keV.

2.3 Condenser

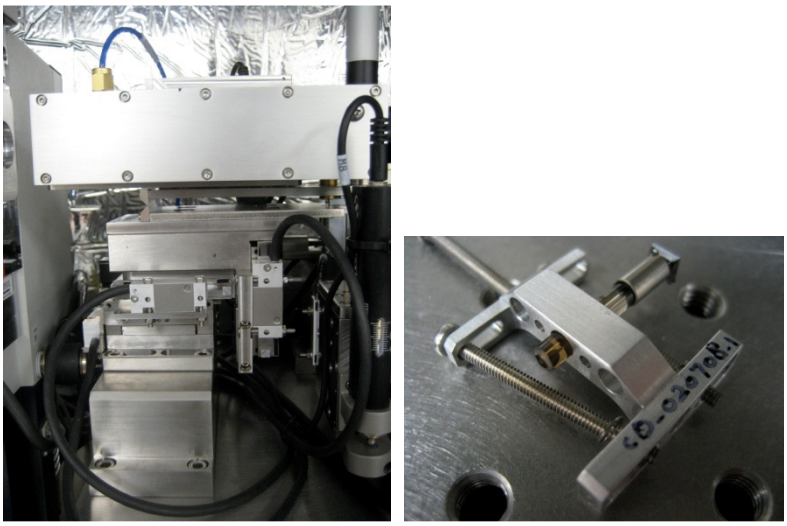


Fig. 2-3: Condenser chamber (left) and condenser mounted on ferrule (right).

Photons emerge from the x-ray source in all directions, and thus are subject to a fast decrease in projected flux-density as they move downstream. Roughly 150 mm from the source, an elliptical capillary lens is positioned to capture and reflect the light. This lens is called a condenser, and is designed with a 1:1 magnifying geometry such that the 70 μm spot of the x-ray source is "condensed" back to its original size ~300 mm from the source. Similar to a visible-light microscope or TEM, the position of focus is the optimal location for the sample. On the downstream edge of the lens, a thick Au stop is glued to block the beam from passing directly through the center of the tube.

2.4 Pinhole

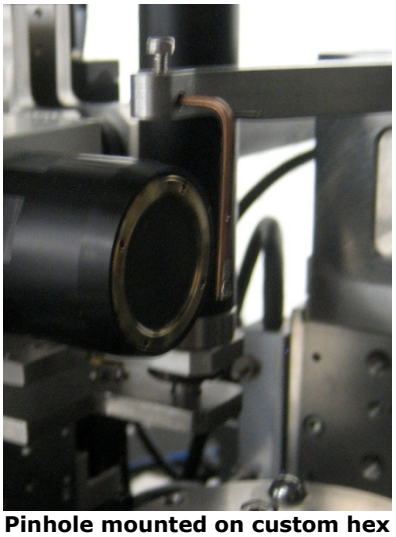


Fig. 2-4: Pinhole mounted on custom hex wrench.

The reflected light from the condenser is multi-ordered, but nanoXCT relies on a single focal spot in order to function with optimal performance. The brightest nonzero reflection is the first order, and higher orders are generally too weak for imaging with the table-top source. In

order to isolate the first order, a pinhole aperture is inserted ~140 mm downstream of the condenser, just before the sample. A 60 μm pinhole is used for the *high-resolution* configuration, and 150 μm for *large field of view*.

2.5 Objective Zone plate

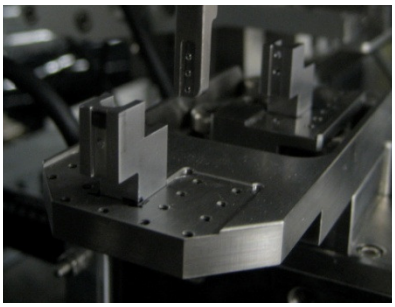


Fig. 2-5: Zone plate mounted on holder.

After the light passes through its focus (at the sample position), it begins to diverge. At a calculated position downstream from this location, another focusing optic is positioned, called a *Fresnel zone plate*. Zone plates are diffractive optics made with circular "zones" of material (Au, in this case), where the focal length, depth of focus, and imaging resolution are dependent on the size of the smallest outermost zones. The total magnification of the system is then dependent on the positions of the detector and sample relative to the objective zone plate.

2.6 Phase ring

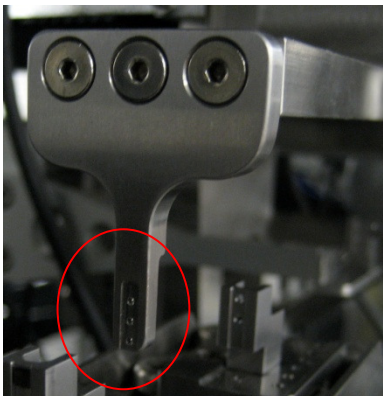


Fig. 2-6: Phase ring holder.

Some of the 1st order light from the condenser is not diffracted by the sample, but is still focused by the outer zones of the zone plate, contributing to the background and, thus, reducing contrast. To reduce the effective contribution of this illumination, Xradia has integrated a Zernike phase contrast mode of operation. In this mode, a metal ring with roughly the same numerical aperture as the zone plate, is inserted into the optical train at a distance such that it intercepts the focused, undiffracted light in the back focal plane of the zone plate. The annular ring, made from Au for the 8 keV system and Ni for 5.4 keV, is manufactured with a precise thickness such that it shifts the phase of the illumination by $3\pi/2$ ($\pm\pi/8$), so that when the undiffracted light recombines with that which was diffracted, the net effect is a phase cancellation of the background. This produces negative Zernike phase contrast, where features with net positive phase shift appear dark.

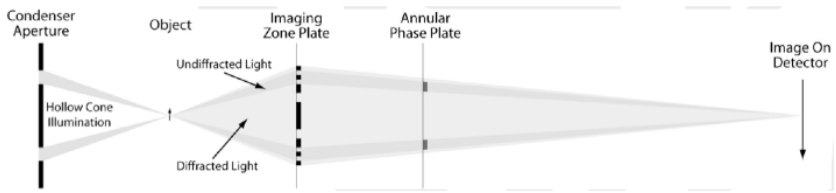


Fig. 2-7: Ray diagram of nanoXCT optical components.

2.7 Bertrand lens

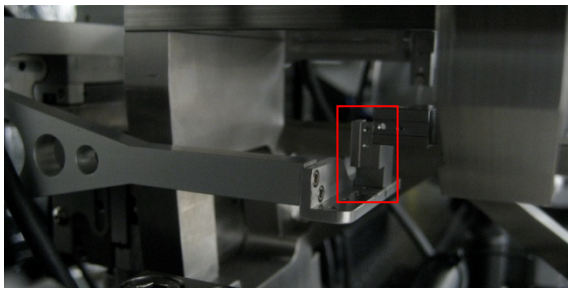


Fig. 2-8: Bertrand lens holder (boxed). A phase ring holder is also pictured (right).

In order to aid alignment of the phase ring, Xradia supplies another zone plate to match, which is configured to image the light in the back focal plane of the zone plate. This intermediate lens is called a *Bertrand lens*, and is only used during alignment of the phase ring.

2.8 Flight tube

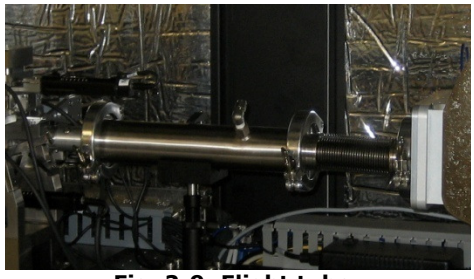


Fig. 2-9: Flight tube.

Normal air, consisting primarily of N gas, is significantly absorbent of 8 keV and 5.4 keV photons. At 8 keV over the distance between the optics and detector, this can lead to as much as a factor of 2 throughput loss, and is much more dramatic at 5.4 keV. To minimize this effect, a flight tube is installed to cover the majority of the path length, which is filled with ~ 3 PSI of He gas. The gas is sealed by a thin Be window at the entrance, which is very fragile and should be left alone. The flight tube is equipped with a pair of valves for venting the chamber when filling or servicing.

2.9 Transmission detector system (TDS)

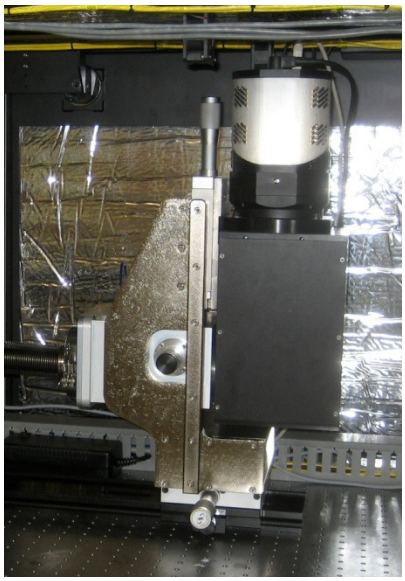


Fig 2-10: nanoXCT detection system.

The final component of the nanoXCT system is the *transmission detector system* (TDS). This detection system consists of two different magnifying, scintillating lenses (2X magnification for alignment and 20X for imaging), mounted on a motorized, software-controlled *objective shifter*. These lenses convert the x-ray photons to the visible-light spectrum, and are then focused by a non-magnifying *tubelens* onto the 16-bit, Peltier-cooled CCD detector.

2.10 Component positions (design)

Component	Position (mm)	
	HRES	**LFOV**
Source	-300	-300
Condenser Entrance	-159	-159
Condenser Center	-150	-150
Condenser Exit	-141	-141
Sample	0	0
Objective Zone Plate	18.52	67.61
Phase Ring	38.76	153.51
Bertrand Lens	163.85	223.12
Detector	754.4	754.4

Table 2-1: Designed positions (relative to the sample) for the x-ray optical components of nanoXCT.

2.11 Pre-alignment microscope (PAM)

The nanoXCT system comes with an optical microscope, called the *pre-alignment microscope*. When properly calibrated to the nanoXCT, this device greatly aids in region-of-interest location before using the x-ray system. Two objective lenses are available with 10X and 50X magnification, as well as transmission and reflection illumination modes.

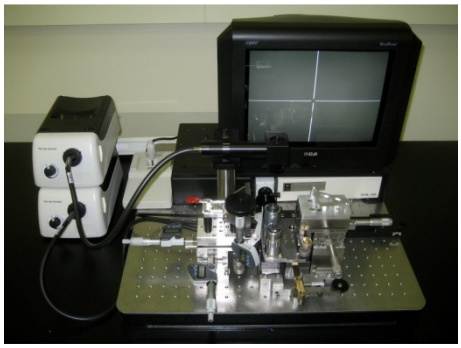


Fig. 2-11: Pre-alignment microscope

Chapter 3: System Alignment

In the following procedure, it is assumed that the microscope is completely misaligned. If only minor changes are necessary, skip the direct-beam method and proceed to the alignment of the x-ray optics. It is recommended to check the alignment of the x-ray optics at least once a week, as components may drift during normal operation.

3.1 Direct-beam method

The technique outlined in this section begins with aligning the condenser, and then uses the circle annotation tool to mark the center of the cone of reflected light. All other components are then individually aligned to the center of the circle, thus locating approximate aligned positions with respect to the beam).

a. Locate direct beam
   To begin, move all components out of the way so that the direct beam is visible in the 2X objective (in the picture here, the direct beam is shown through the aperture of the flight tube).

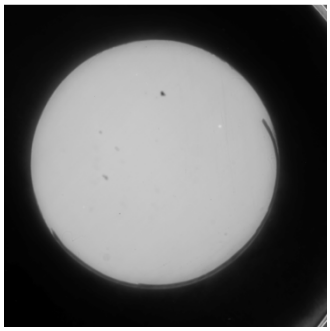


Fig. 3-1: Direct beam image, apertured by the flight tube. A small burr in the tube is visible on the right side of the image.

b. Condenser
   Using a combination of the condenser x/y and tip/tilt motors, adjust the condenser position so that the first-order reflected light evenly surrounds the projection of the stop (appearing as the "hole" in the middle of the "doughnut"). Then, using the circle annotation tool ○, draw an outline of the reflected light and save the image.

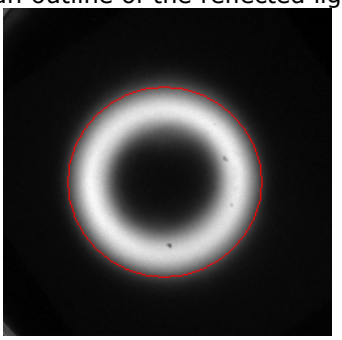


Fig. 3-2: Image of the aligned condenser.

c. Pinholes
   Move the condenser out of the way so that direct beam is again visible. Open the condenser image, and use the pointer tool from the annotation menu to select the circle drawn in the previous step. After selecting the circle with the left mouse button, click the right mouse button and select "copy." Begin a continuous image collection using high binning (matched to that used in part a) and short exposure time. On the new live image, right click and select "paste." Finally, use the pinhole x/y motors to steer each pinhole to the center of the circle.

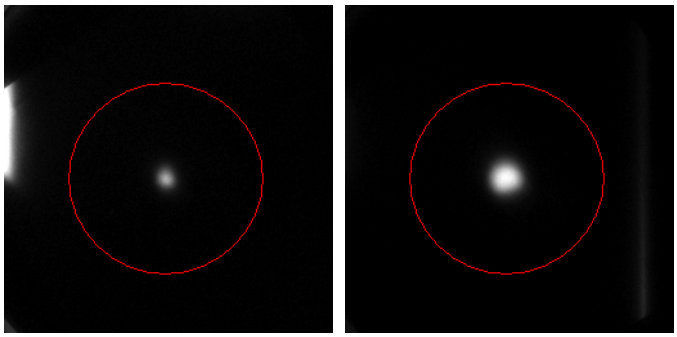


Fig. 3-3: High-resolution (60 μm, left) and large field-of-view (150 μm, right) pinhole projections.

Once alignment is complete, save each image with the annotation.

Note: The two different pinholes are located on the same holder, and are separated by ~ 3 mm vertically with minimal horizontal shift.

d. Zone plates
As in the previous step, move the pinhole out of the way so that direct beam is again visible. Begin another continuous acquisition, as before, and copy and paste the circle onto the new window. Use the zone plate x/y motors to position each zone plate to the center of the circle, saving each aligned image.

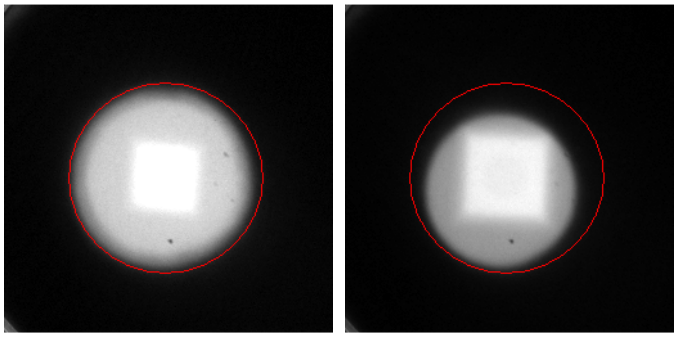


Fig. 3-3: High-resolution (ZP35-80-7, left) and large field-of-view (ZP30-320-4, right) zone plate projection images.

e. Phase rings
Repeat the same procedure as before to align the phase rings. The PR64 contains four phase rings in a 200 μm x 200 μm grid pattern, so it is best practice to align to a point near one corner of the membrane. In practice, the phase ring used on the PR64 membrane is generally in the upper right corner, but aligning to any corner will allow navigation to any of the other phase rings.

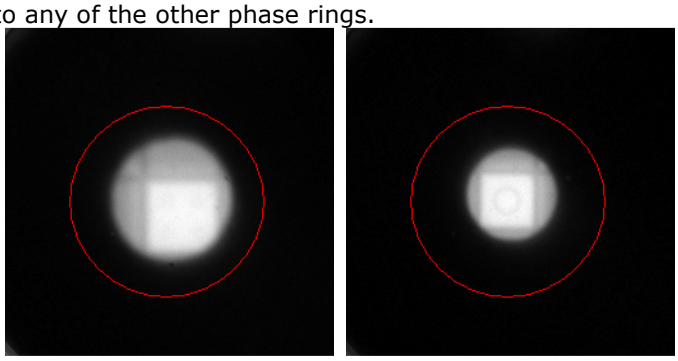


Fig. 3-4: High-resolution (PR64-4.75-2.7, left) and large field-of-view (PR215-13-2.7, right) phase ring projection images.

f. Bertrand lenses
Repeat the same procedure as before to align the Bertrand lenses.

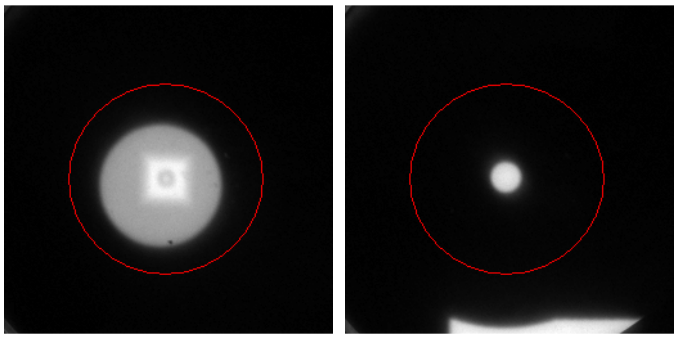


Fig. 3-5: High-resolution (ZP100-160-16, left) and large field-of-view (ZP30-320-4 w/ 300 μm OSA) Bertrand lens projection images.

3.2 X-ray optics alignment

In this procedure, it is assumed that approximate aligned positions of each component are known, either from the direct beam location method described before or from a previous alignment.

To find the old alignment images, open the Image Control set and scroll over to the tab labeled "axis positions." Locate the positions of the axis of interest, and use the motion controller to move the corresponding stage to the approximate aligned position. Then, perform minor changes to align the stage as pictured below.

3.2.1 High-resolution (HRES)

i. Condenser

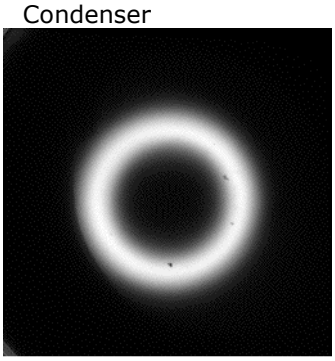


Fig. 3-6: Condenser aligned.

ii. Pinhole

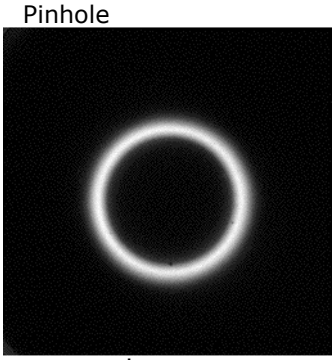


Fig. 3-7: 1st order reflection isolated by 60 μm pinhole.

iii. Zone plate

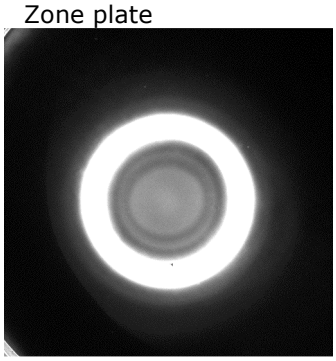


Fig. 3-8: High-resolution zone plate aligned.

iv. Bertrand lens

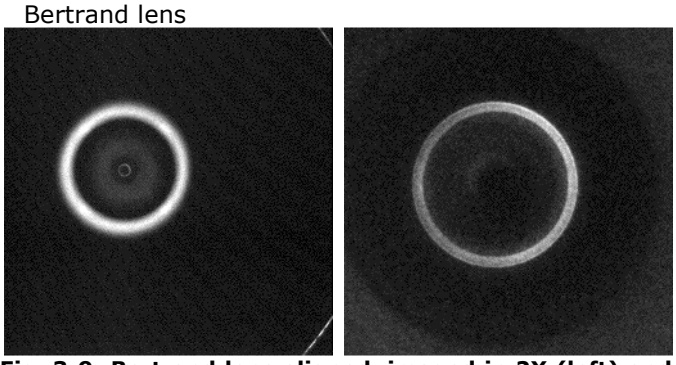


Fig. 3-9: Bertrand lens aligned, imaged in 2X (left) and 20X (right).

Record the coordinates for the Bertrand lens aligned position here:

Bertrand X	
Bertrand Y	
Bertrand Z	

v. Detector (20X)
With the light from the Bertrand lens focused on the 2X objective and centered in the ring, switch to the 20X objective. If the focused ring is not in the center of the field of view, adjust the manual micrometers on the detector until the 20X image appears as above.

Tip: The pixel size on the detector is 0.65 μm using the 2X objective at 1x1 binning. To calculate the amount the detector should be moved, multiply 0.65 μm by the binning, and then multiply the result by the number of pixels necessary to move the ring to the center. Then, shift the detector in x and y by the final calculated distance (each minor division on the micrometer represents 10 μm of motion).

vi. Phase ring

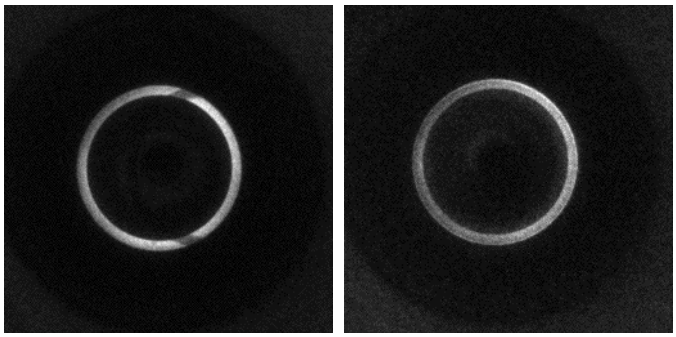


Fig. 3-10: Phase ring moving into the illumination (left) and aligned (right). Imaged with 20X objective.

The phase ring, when imaged with the Bertrand lens, will appear as a shadow in the focused illumination. The goal of alignment is to shift this shadow using the phase ring motors until it completely covers the bright ring. The phase ring membrane contains four rings in a 200 μm x 200 μm grid, each with a different size. If the first ring does not fit, shift by 200 μm in x and/or y until another is visible and try again.

When phase ring alignment is complete, move the Bertrand lens out of the optical path and, when it has completed motion, click the

button in the motion controller. Then, take one image of the flat field and save it for future reference.

vii. Collect an image of the X50-30-7 test pattern
In order to qualify the high-resolution alignment, collect an image of the X50-30-7 test pattern. Throughput should be around 140 cts/min at binning 1 (for a clean anode and new filament), and contrast at the frequency corresponding to the second region from the center should be ~14%. In the image shown below, reference correction has also been applied.

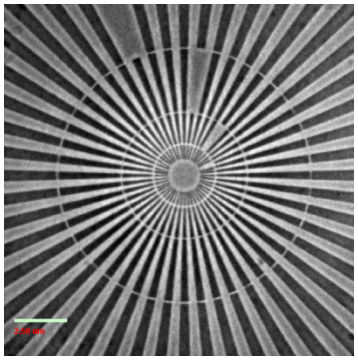


Fig. 3-11: Test pattern with 50 nm tips imaged in phase contrast.

3.2.2 Large field-of-view (LFOV)

i. Condenser (if misaligned)

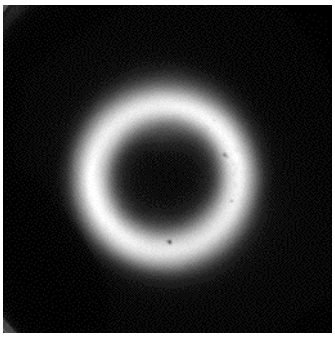


Fig. 3-12: Condenser aligned.

ii. Pinhole

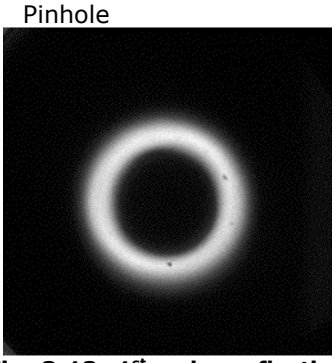


Fig. 3-13: 1st order reflection isolated by 150 μm pinhole.

iii. Zone plate

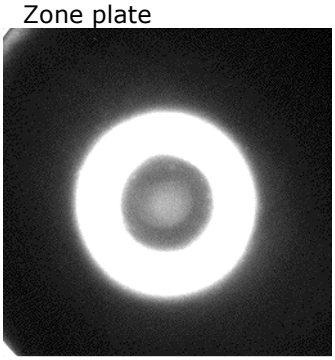


Fig. 3-14: Large field-of-view zone plate aligned.

iv. Bertrand lens

[example picture needed]

Record the coordinates for the Bertrand lens aligned position here:

Bertrand X	
Bertrand Y	
Bertrand Z	

v. Phase ring

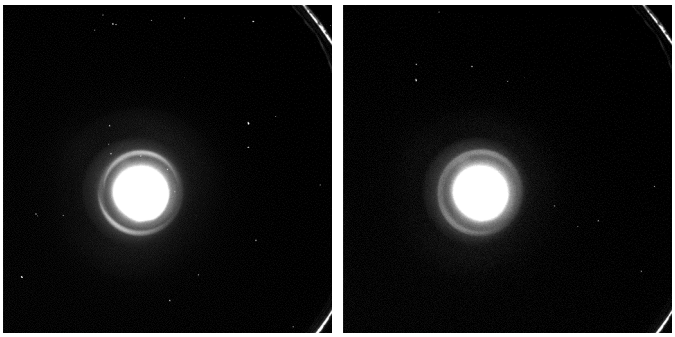


Fig. 3-15: Phase ring moving into the illumination (left) and aligned (right). Imaged with 2X objective.

vi. Collect an image of the X50-30-7 test pattern.
As before, collect an image of the X50-30-7 test pattern to qualify alignment. Throughput should be ~700 cts/min at binning 1 for a single zone plate, and contrast ~ 7% for the third region from the center.

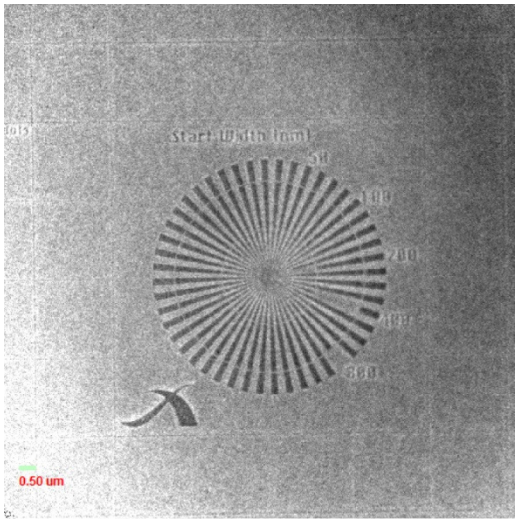


Fig. 3-16: Test pattern with 50 nm tips imaged in large field of view with Zernike phase contrast.

NOTE: A small z shift is necessary between HRES and LFOV zone plate configurations. Refer to Table 2-1 for the proper placement of these optics relative to the sample (for use during installation), and then refer to old images (after installation) for the proper positions relative to home.

3.3 Standard alignment correction procedure

During the course of normal operation, the system alignment may drift slightly, prompting a need for performing small corrections. The procedure for checking and correcting for these drifts is outlined here. To ensure optimal operation of nanoXCT, it is recommended that the alignment be checked at least once per week, and always before a long tomography set is started.

1. Switch to the 2X objective.

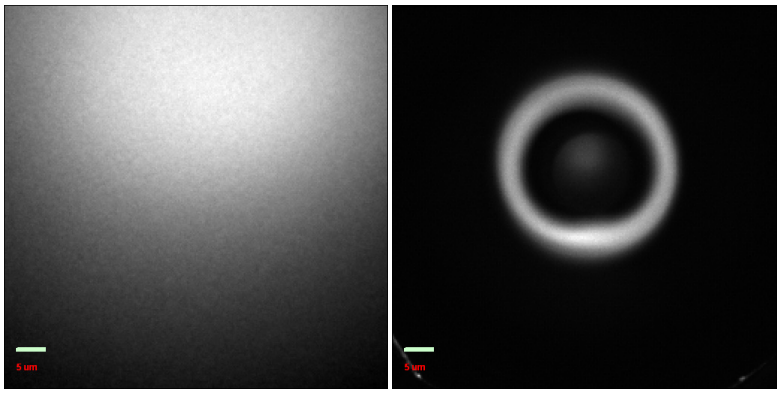


Fig. 3-17: Images from a system in which the alignment has drifted. On the left is an image taken with the 20X objective, and the right with 2X.

2. If the phase ring is inserted, use the incremental motion function to move it 2500 µm toward zero.
3. Remove the zone plate by using the incremental motion function to move it 5000 µm toward zero.
4. Remove the pinhole by using the incremental motion function to move it 3000 µm toward zero.
5. Check the alignment of the condenser. If it does not appear aligned per the figures in the previous section, perform small motions until it is realigned.

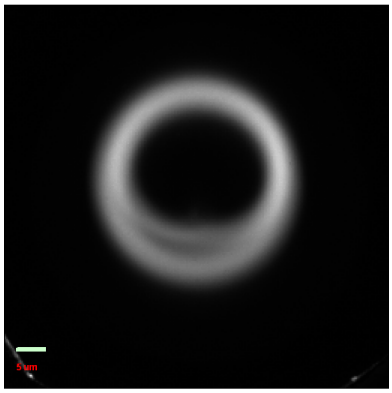


Fig. 3-18: Condenser is misaligned by approximately 30 µm in the Y direction.

6. Move the pinhole back in by using the incremental motion function to move it 3000 µm away from zero. Perform small motions until it is realigned per the figures in the previous section.
7. Move the zone plate back in by using the incremental motion function to move it 5000 µm away from zero. Perform small motions until it is realigned per the figures in the previous section.
8. If phase contrast is desired:
    a. Move the Bertrand lens back in by using the incremental motion function to move it to its aligned position. Perform small motions until it is realigned per the figures in the previous section.
    b. Move the phase ring back in by using the incremental motion function to move it 2500 µm away from zero. Perform small motions until it is realigned per the figures in the previous section.
9. Take one image of the flat field and verify that the FOV is detected as expected. If it is severely off-center, use the manual micrometers on the detector box to perform very small motions until it is recentered.
10. Open Microscope->Configure System, click on the Energy tab, and enter the appropriate configuration information into the fields (8 keV for high-resolution, 8.1 keV

for large field of view, selecting absorption or phase contrast as appropriate). Then, click "Store Current positions to energy table" and click OK. If asked to want to move motors, click "no."

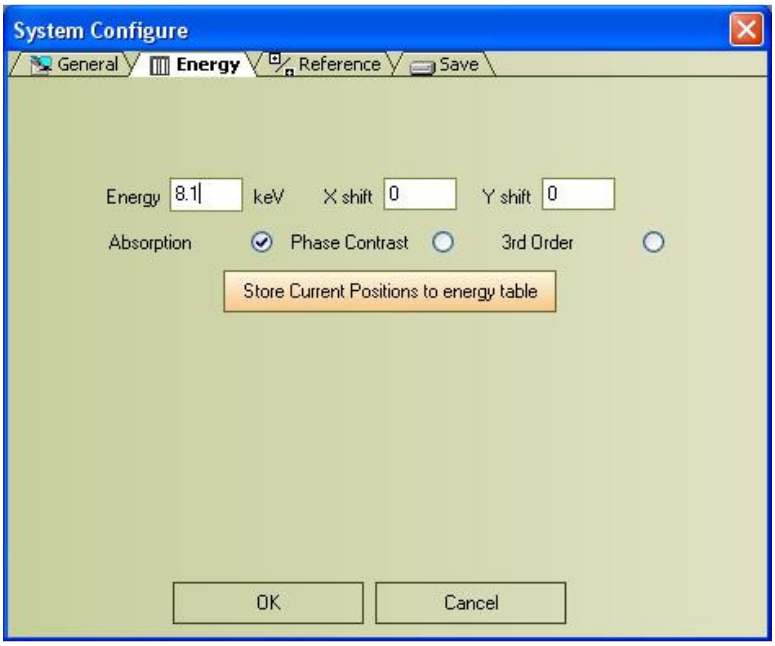


Fig. 3-19: Energy table settings window.

11. Switch back to the 20X objective.

Chapter 4: Motion Controller

4.1 Introduction

TXMController offers control over each motorized axis available in nanoXCT. Motion may be executed either to an absolute motion or incrementally, and the details of the controller are outlined below.

4.2 Motion Controller Functions

The motion controller may be launches by clicking the motion controller button . The functions are explained below.

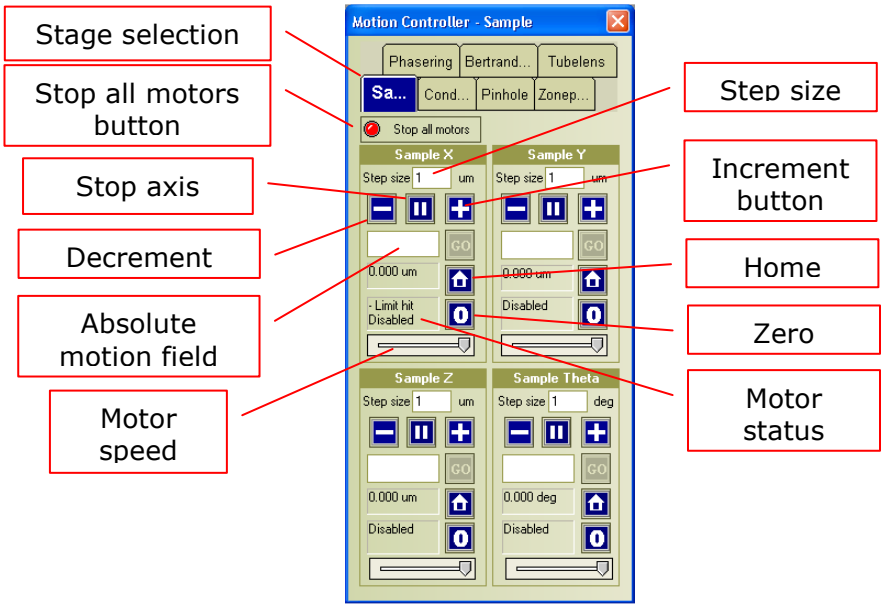


Fig. 4-1: Motion controller window.

1. Stage selection tabs
   At the top of the motion controller window, a variety of tabs are present, one for each stage. Select the appropriate tab to reveal controls for each of the available motors on the stage.

2. Stop all motors button
   By clicking the *stop all motors* button, all axes become disabled, meaning that power is no longer sent to any motor and all motion is halted. It is recommended, as general practice, to keep all motors disabled when not in use.

3. Stop axis button
   The *stop axis* button has similar functionality to the *stop all motors* button, but only applies to a single axis. If, for example, many axes are moving but only one needs to stop, clicking this button will allow the others to continue moving.

4. Absolute motion field
   The *absolute motion* field allows the user to move the selected axis to a specified position in microns. To perform this type of motion, type the desired position in the field to the left and click *GO*.

5. Motor speed selection
   The *motor speed* selection slider allows control over the speed at which the specified motor will move. With the slider all the way to the right, motion will execute at the

maximum rate, and setting the slider all the way to the left will not perform any motion. Reducing the speed will increase the precision of motion, but will also increase the wait time for the motor to reach its position. For general purposes, it is recommended to keep this set at 100%.

6. Step size field

The *step size* field allows the user to specify the distance, in microns, to move the specified axis during incremental motion.

7. Increment/decrement buttons
The *increment*/*decrement* buttons execute relative motion by the amount specified in the *step size* field.

8. Home button
The *home* button executes the homing routine for the specified axis along with all of its dependencies. See the section on motor homing (Appendix A) for more information on this feature.

9. Zero button
The *zero* button sets the current position of the specified axis to zero, and stores the relative distance from home in a separate location. When motors are re-homed, they will return to the zero position by default.

10. Motor status window
The *motor status* window provides updated information on the status of the specified motor. It will report if the motor is enabled or disabled, as well as any travel limits encountered.

Chapter 5: Pre-alignment microscope

5.1 Introduction

To ease the location of samples and regions of interest (ROIs), the nanoXCT is equipped with an auxillary visible-light microscope, called the pre-alignment microscope (PAM). The microscope has two illuminators, two objective lenses, and a sample stage that is calibrated during setup to the nanoXCT (see Appendix A for calibration procedure).

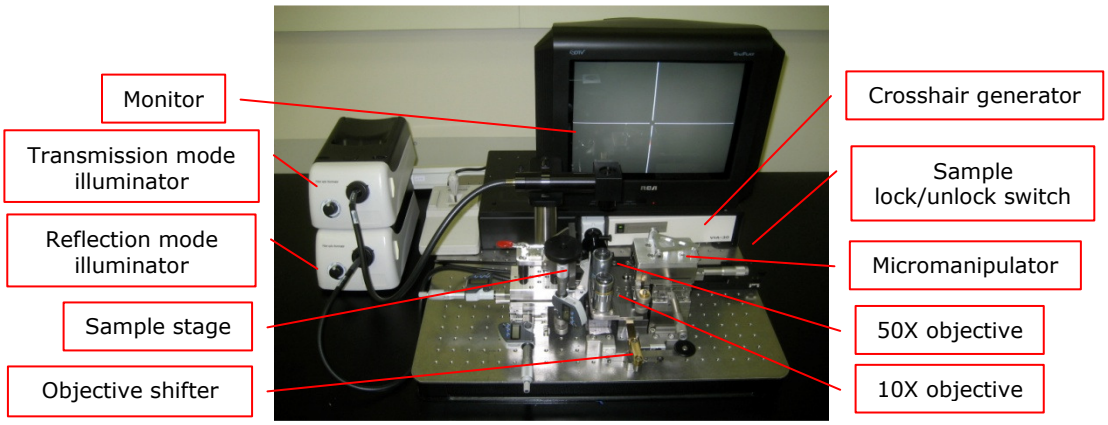


Fig. 5-1: Pre-alignment microscope.

5.2 Illumination

The pre-alignment microscope includes two fiber-optic illuminators, to provide both transmission- and reflection-mode illumination.

5.3 Objective lenses

Two objective lenses are included with the pre-alignment microscope, with 10X and 50X magnification. An objective shifter is also installed to easily switch between the two objective lenses.

5.4 Sample lock/unlock & micromanipulators

On the corner closest to the monitor, a black switch is positioned to unlock and re-lock the clip on the chip sample holder. With the chip unlocked, the Si-tip micromanipulator may be used to physically reposition the sample on the sample holder.

5.5 Monitor, crosshair generator, & CCD camera

The pre-alignment microscope includes a TV monitor and crosshair generator for viewing the output from the CCD camera. The crosshair generator must be turned on in order to view the image.

5.6 Operation procedure

1. With the red handle pointing away from the sample stage, position the sample holder so that the three balls on the stage fit into the matching grooves on the sample holder. With the sample firmly supported, flip the lever so that it points toward the sample. The magnet in the sample stage should now sufficiently support the sample holder.
2. Turn on the TV monitor and set it to VIDEO mode. Turn on the crosshair generator.
3. Turn on the appropriate illuminator and tune the illumination to a reasonable level.
4. Switch to the 10X objective. Using the sample stage micrometers, position the sample so that the ROI is clearly visible in the center of the crosshairs. Switch to the 50X objective, and perform fine tuning.

5. If the sample is in the clip-style holder and far from zero in the x-direction, unlock the clip using the black switch and use the micromanipulator to reposition the sample. Then, repeat step 4 until the ROI is again centered.
6. Write down the coordinates from the digital micrometers and reverse step 1 to remove the sample holder. Place in nanoXCT and move the sample stage to the noted positions.

Chapter 6: Collecting 2D Images

6.1 Introduction

Xradia's TXMController software package enables several different modes of 2D image collection. A brief description of each mode is given in this section. To launch the *Acquisition Mode* dialog, click the corresponding icon in the toolbar.

6.2 Camera Settings

In the acquisition mode, camera settings must be specified for each imaging mode. The available parameters are as follows:

1. Exposure time
   The *exposure time* is the length of time that the CCD will be exposed during the collection of each image. The total number of counts has a linear dependence on exposure time, meaning that a longer exposure time will increase the signal-to-noise ratio of the resulting image.
2. Binning
   The *binning* value controls the effective CCD size in pixels. A binning value of *n* will sum the reading of n^2 adjacent pixels, increasing the total number of counts by the same factor. While this quickly increases signal-to-noise and decreases file size, it also limits the resolution of the system, and must be used with caution.
3. Readout time
   The *readout time* of the CCD represents the amount of time the camera spends reading data from the chip. The *precision* readout mode produces very clean images, but may take as long as 10 seconds for images collected at binning 1; *fast* mode readout, by contrast, takes only a few seconds, but comes with a slight increase in noise level. For images with >2000 counts on average or where image quality is not critical, fast mode is usually sufficient.

6.3 Typical imaging times

Imaging times are highly sample dependent. The camera used in nanoXCT has a 16-bit depth, however, so a general minimum, in practice, is ~2000 counts for acceptable statistics. For low-density samples (i.e., minimal x-ray absorption), this translates to:

~300s at binning 2 for the high-resolution configuration
~180s at binning 1 for large field-of-view

Naturally, these times are simply general guidelines, and will change depending on the properties of the material being imaged. For expected x-ray absorption of known materials, it is often useful to consult the *X-ray Interactions with Matter* calculator from Lawrence Berkeley National Laboratory at:

http://henke.lbl.gov/optical_constants/filter2.html

Be sure to reference the photon energy specific for the nanoXCT laboratory tool in use (8000 eV for a copper target, 5400 eV for chromium).

6.4 Aborting image acquisition

To halt the image acquisition at any time, click the stop icon on the main toolbar.

6.5 Acquisition modes

6.5.1 Single

In single image mode, one image only is collected according to the camera settings. The image is stored in memory and displayed on the screen for the user to manipulate.

Single acquisition may be started directly by clicking on the still camera button in the main toolbar. The settings used will be identical to the previous single image collection.

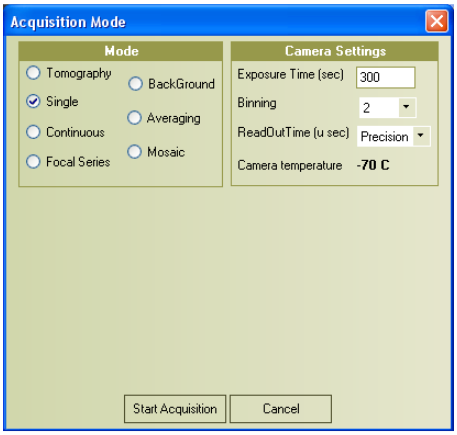


Fig. 6-1: Single acquisition mode.

6.5.2 Continuous

In continuous mode, images are collected according to the camera settings and continuously updated on the screen. This action takes place inside a single display window, meaning that every new image overwrites the previous.

Continuous acquisition may be started directly by clicking on the video camera button in the main toolbar. The settings used will be identical to the previous continuous image collection.

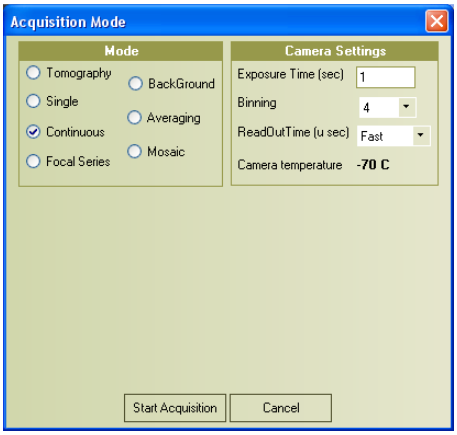


Fig. 6-2: Continuous acquisition mode.

6.5.3 Mosaic

Mosaic imaging mode allows the collection of multiple images at different locations, which are automatically stitched together into one single image. This provides the ability to see a large region of the sample, in order to easily locate a region of interest. Mode-based settings for mosaic imaging are as follows:

1. Fast axis
   The *fast axis* is moved most frequently in a consecutive manner, and is represented on the final mosaic image as the horizontal axis.
2. Slow axis

The *slow axis* is moved only after each complete row has been collected, and is represented on the final mosaic image as the vertical axis.

3. Fast axis center (um)
   The *fast axis center* is the position, in microns, about which the fast axis will move.
4. Slow axis center (um)
   The *slow axis center* is the position, in microns, about which the slow axis will move.
5. Fast axis step (um)
   The *fast axis step* specifies the amount of motion to be performed by the fast axis between each successive tile.
6. Slow axis step (um)
   The *slow axis step* specifies the amount of motion to be performed by the slow axis after each complete row of tiles has been collected.
7. No of Mosaic Rows
   The *no of mosaic rows* specifies the total number of images to collect along the slow axis direction.
8. No of Mosaic Columns
   The *no of mosaic columns* specifies the total number of images to collect along the fast axis direction.

NOTE: For mosaics greater than 3x3, it is recommended to use binning 4 or 8, as memory consumption at binning 1 or 2 can become large enough to impact general performance of the computer workstation.

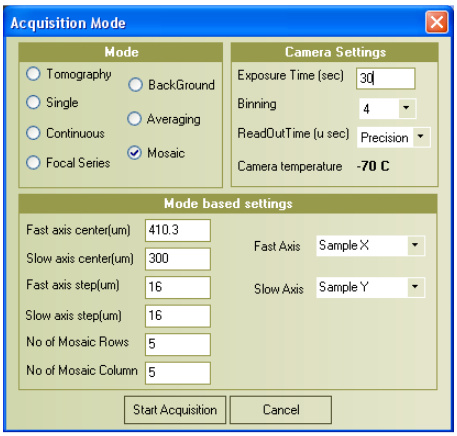


Fig. 6-3: Mosaic acquisition mode.

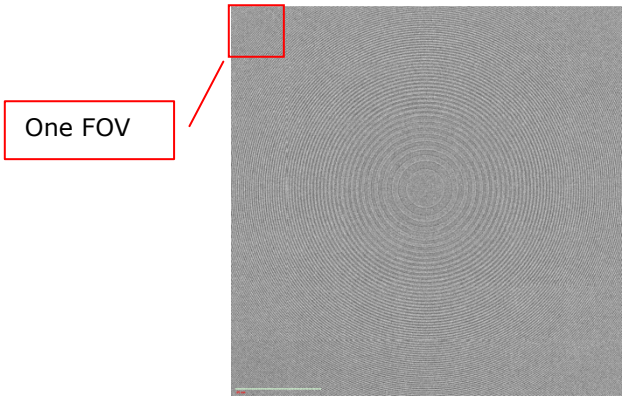


Fig. 6-4: 7x7 mosaic of a plastic zone plate imaged in phase contrast.

6.5.4 Focal Series

In focal series mode, the motor selected in the mode-based settings is moved along its axis, stopping at discrete intervals for image collection, resulting in a file containing a series of images. The typical application for this mode is to move a z-axis motor, such as sample z, across a small image to determine the position of best focus. Mode-based settings for focal series collection are as follows:

1. Motor
   One the right side of the mode-based settings window, the motor to be used in the focal series must be chosen. The most typical axis used in focal series acquisition is sample z.
2. Center Z position (um)
   The *center z position* specifies the location about which the specified axis will be moved.
3. Range (um)
   The *range* is the total amount of motion that will be executed during collection. Motion is executed from center-range/2 to center+range/2.
4. No of Images
   The *no of images* specifies the total number of images that will be collected during the focal series. The step size of the motor for each image is the quotient of range and number of images.

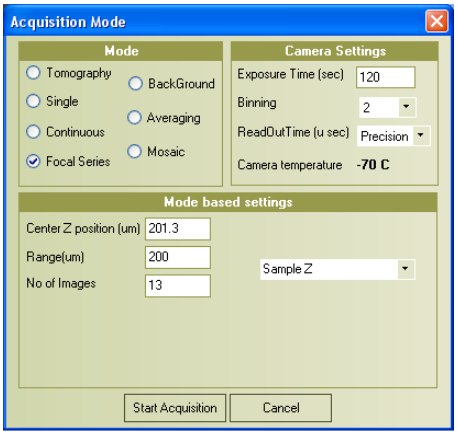


Fig. 6-5: Focal series acquisition mode.

6.5.5 Averaging

To increase the overall signal-to-noise ratio of the final image, many images may be collected and then averaged together. While the signal remains roughly constant, the random noise is reduced, leading to a cleaner, clearer image. The software may do the averaging while doing the acquisition, or the task of averaging may be left to the user to perform after the fact. This feature is useful for collecting clear images of a single angular projection as well as collecting a flat-field image for reference correction.

The mode-based settings are as follows:

1. No of Images
2. Average images on fly
   The *average images on fly* option, when checked, averages images as they are collected, resulting in a single, averaged data file. If it is left unchecked, the resulting file is a file containing a set of all the individual images, where averaging is left to the user to perform using the *alignment* tab in the *image control set* window.

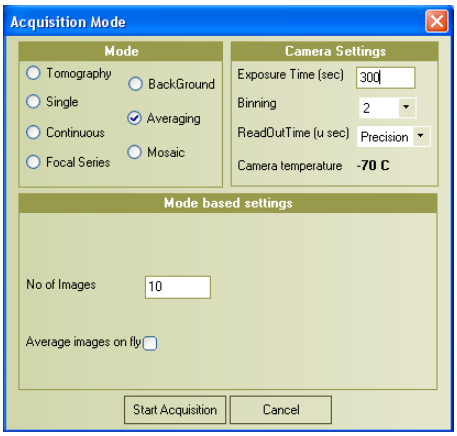


Fig. 6-6: Averaging acquisition mode.

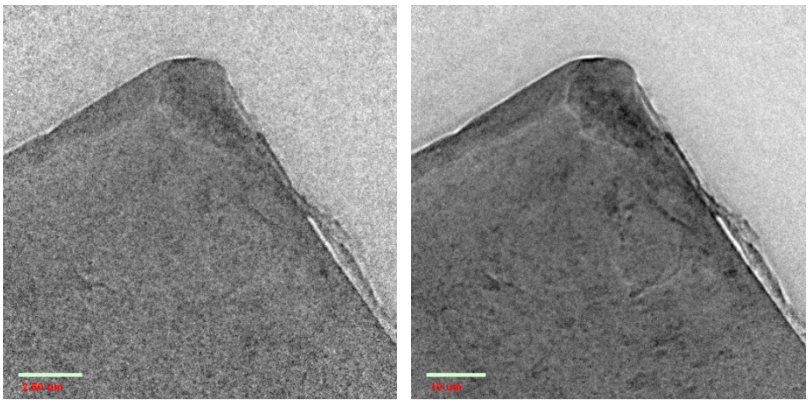


Fig. 6-7: Single image (left) and 10 images averaged (right).

For collecting averaged data, the recommended procedure is as follows:

i. Collect the averaging series, **not** averaging on the fly.
ii. Collect an averaged reference with ~50% more counts than the data set (averaging on the fly is ok).
iii. Apply the reference to the averaging series.
iv. Average the data.

Chapter 7: Collecting 3D Tomography Data

In this section, a procedure is outlined for collecting a set of tomography data. High-contrast metal foam is used as the example, and data is collected up to ± 90° (i.e., full 180° rotation) in the large field-of-view configuration. It is assumed that the rotation axis aligned has already been completed and the nanoXCT and pre-alignment microscope are properly aligned to each other and the rotation axis.

7.1 Preparing the sample

1. Load the sample into the holder and place into the pre-alignment microscope (PAM).
2. Center the feature of interest in the PAM crosshairs using the 50X objective lens and note the X/Y/Z positions of the micrometers.
3. Remove the sample from the PAM and place it into the nanoXCT (NCT).

7.2 Initial alignment

1. Position the NCT sample X/Y/Z motors to match the location found using the PAM.
2. Collect a single image.
3. If the image collected does not match the feature found using the PAM, perform a small mosaic (e.g., 3x3 for large field-of-view and 5x5 for high-resolution configuration). Once the feature of interest has been located, use the *drive to* feature of TXMController to move the sample stage to that location. Once the feature has been located, collect a single image for record keeping.

7.3 Preparing for tomography with software stage correction

1. Navigate to Microscope->Configure System to open the System Configure window.

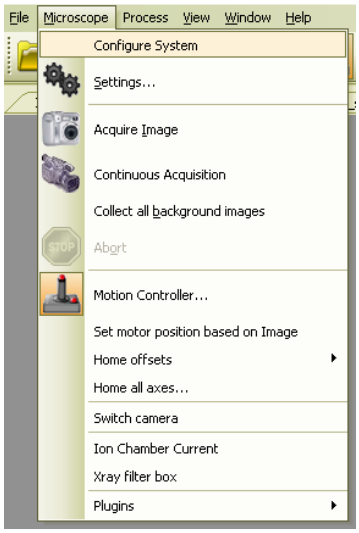


Fig. 7-1: Location of Configure System option under the Microscope menu.

2. Check the box next to *Apply stage correction*. Then, set *X Correction* to the current sample X position and *Z Correction* to -1 * current sample Z position. When finished, click OK to close the window.

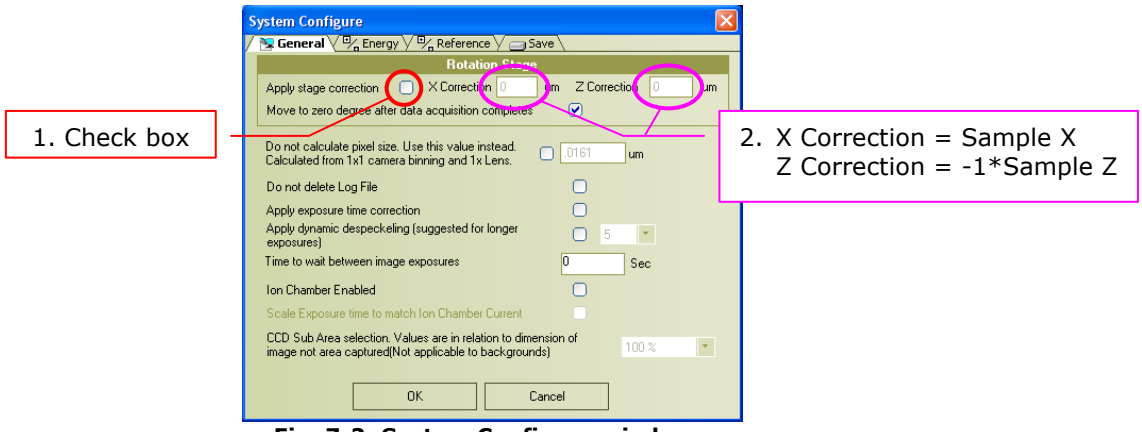


Fig. 7-2: System Configure window.

3. Open the *Acquisition Mode* window and select *Tomography*. Set the start angle to something small, such as -20° for large field-of-view imaging, and set the end angle to be symmetric about 0° (e.g., +20°). Set the number of steps to 7, and the imaging time and binning to a quick, coarse value. Then, begin acquisition.

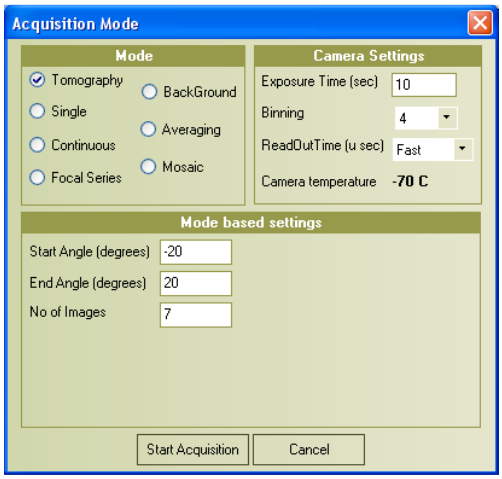


Fig. 7-3: Tomography mode window.

4. When the data set has finished collection, open the *Image control set* menu and navigate to the *Alignment* tab. Click *Manual* mode, and then select image #4, corresponding to 0°. In the data set, double-click on the feature that is being aligned to the rotation axis. Repeat for image #3, 2, and 1 to track this feature through the projection images. Then, move to image #5 and repeat through #7. When finished, click *Stage Calibration* and then select *OK* in the resulting prompt window.

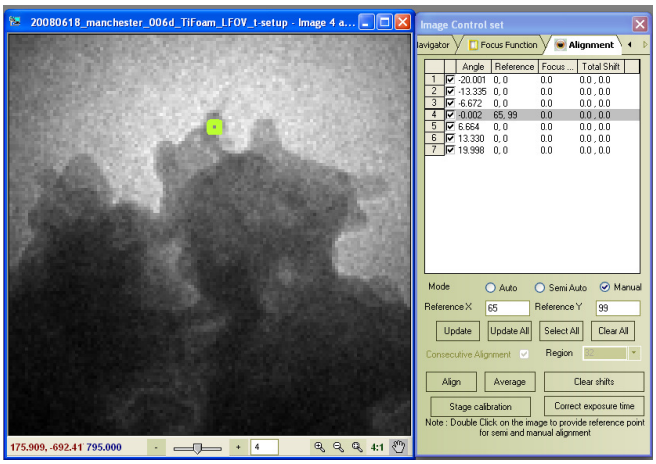


Fig. 7-4: Aligning the data set.

5. Iterate steps 3 and 4 with increasing angular range and number of images until the apparent image shifts are almost zero and the range covers all angles that will be used for tomography in 10° steps.

7.4 Collect the tomography data

Begin collecting a final tomography set, collecting over as large of an angular range as possible with 1° steps. Set the exposure time and camera binning such that >2000 counts are observed and the necessary resolution is achieved (increase binning by a factor of two increases effective throughput by a factor of four, but decreases resolution by the same amount).

In this example, data is collected from -90° to +90°, with 181 total images.

7.5 Collect a reference & correct the tomography data set

Move the sample out of the x-ray beam by either using the motors on the sample stage or physically removing the sample holder from NCT. The collection mode should be *Averaging* of at least 5 images (10 is recommended), exposure time and binning should match the tomography data set. For ease of use, *Average images on fly* may be selected.

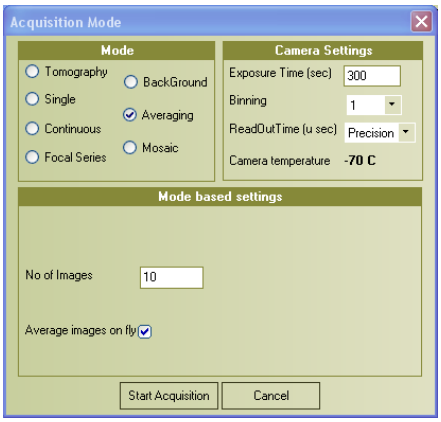


Fig. 7-5: Averaging mode window.

When collection of the reference is complete, open the image calculator and multiply it by 1.5. Then, save the new multiplied image as a new file. Select the tomography data set, open the *Image Control Set* window, and navigate to the *Reference* tab. Browse

for the averaged, multiplied reference image, then select *Apply background adjustment*. Finally, save the tomography data set.

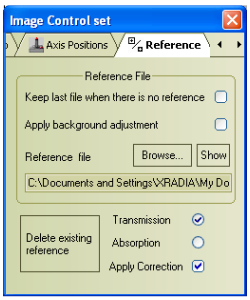


Fig. 7-6: Reference correction dialog box.

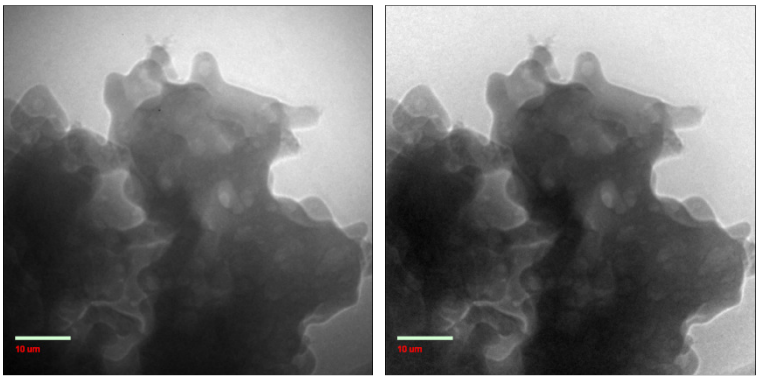


Fig. 7-7: Tomography data with no reference correction (left) and reference correction applied (right).

7.6 Align the tomography data

Follow the same procedure as in step 4 of section 7.3 to track the alignment feature through each of the projections. When finished, save the data set.

Chapter 8: Reconstructing 3D CT Data

After collecting a tomography data set with TXMController, TXMReconstructor may be used to generate the 3D reconstructed data set. The procedure is outlined below.

8.1 Reconstruction Procedure

1. Close the tomography data file in TXMController if it is open.
2. Open TXMReconstructor, and use it to open the file containing the collected data set. The bright box around the data represents the region of the data that will be reconstructed, corresponding to the 2D region present in all projections (the rest is cropped off due to alignment).
3. Open the *Reconstruction Setting* window .

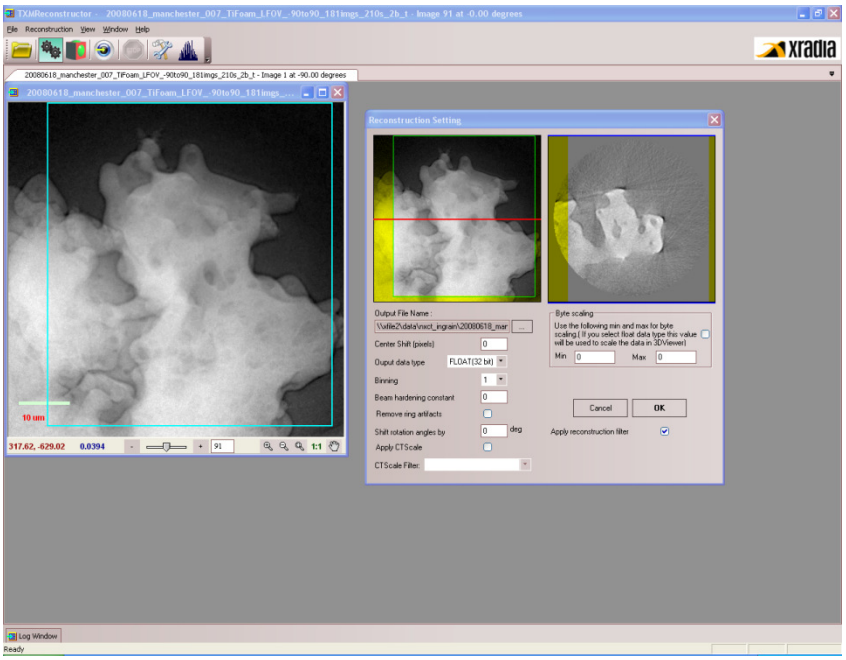


Fig. 8-1: TXMReconstructor

4. Browse to select the output file name.
5. For best results, the following settings are recommended:

Center Shift (pixels)	0
Output data type	FLOAT (32 bit)
Binning	1
Beam hardening constant	0
Remove ring artifacts	No
Shift rotation angles by	0 deg
Apply CTScale	No
Byte scaling	No (auto)
Apply reconstruction filter	Yes

6. Click OK to close the *Reconstruction Setting* window.
7. Begin the reconstruction . Reconstruction will begin immediately, and usually takes about 5 minutes to complete.

Chapter 9: Viewing and Exporting Reconstructed Data

9.1 Viewing data

9.1.1 Opening a reconstructed volume

3D reconstructed data is generally viewed in TXM3DViewer, available via the Microsoft Windows Start menu. Launch the program and open the reconstructed volume using the *File->Open* dialog.

Some large files (those reconstructed at binning 1, for example) are too large to fit in the computer's memory. In this case, a prompt is shown asking about the method to be used for opening the file. It is recommended to use *out-of-core* mode here, which writes another file to disk storing all of the temporary information. These files are quite large, and may be deleted or omitted from backup routines after closing the program.

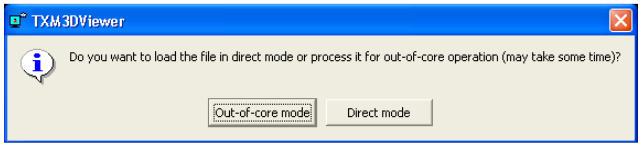


Fig. 9-1: Large TXM file load prompt.

9.1.2 Layouts

Many view options are available in the main window. There are four standard view modes, shown below.

Fig. 9-2: Layout toolbox.

2D slice layout

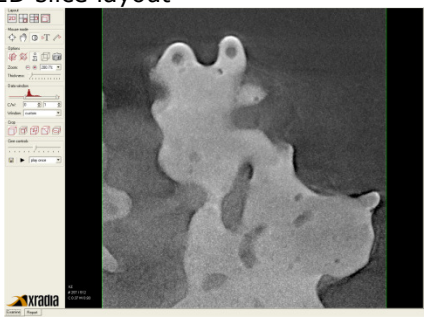


Fig. 9-3: Example of 2D slice layout.

Simultaneous slice & volume layout 1 (default)

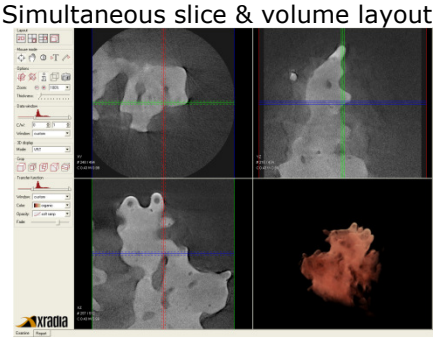


Fig. 9-4: Example of simultaneous slice & volume layout 1.

Simultaneous slice & volume layout 2

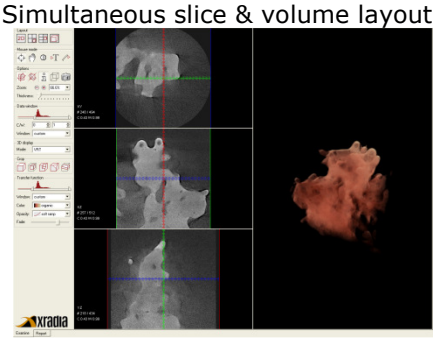


Fig. 9-5: Example of simultaneous slice & volume layout 2.

3D volume layout

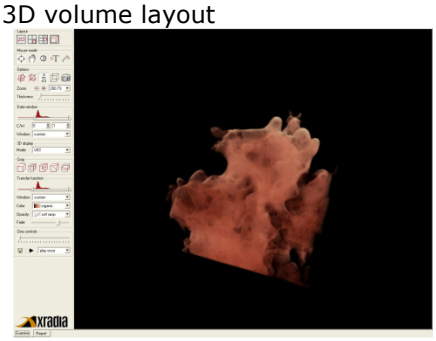


Fig. 9-6: Example of 3D volume layout.

9.1.3 Mouse mode

The mouse mode options allow selection of several different mouse effects.

Fig. 9-7: Mouse mode toolbox.

Navigation – Clicking and dragging navigates through slices or moves the volume rendering

Pan – Clicking and dragging pans the selected view

Contrast – Clicking and dragging dynamically changes contrast

Annotation – Clicking on a point in one view applies an annotation

Measurement – Clicking on two points in a view measures the distance between the points

9.1.4 Options

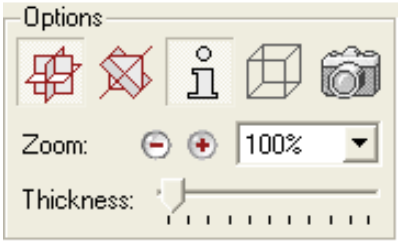


Fig. 9-8: Options toolbox.

Navigation modes

Navigate through the slices in orthogonal (XYZ) mode
Navigate through the slices in oblique (arbitrary angle) mode

Extra information

Display axis & slice number information on the slices
Display a 3D box on the volume rendering for orientation information

Capture

Capture the current view to be exported into the report (see section on exporting)

Zoom

Use the increment/decrement buttons and/or percentage control to zoom the current view by a specified amount.

Thickness

Adjust the thickness slider to integrate several slices into each view. For noisy reconstructions, it is often helpful to increase this slider by a small amount to reduce the noise and smooth out the images.

9.1.5 Cropping

Fig. 9-9: Crop toolbox.

By cropping the reconstruction in slice view, it is possible to eliminate regions of pure noise to keep them from contributing to the volume rendering. Several different cropping modes are available:

No cropping
Orthogonal (XYZ) cropping
Orthogonal (XYZ) corner cropping
Oblique (arbitrary angle) corner cropping
Oblique (arbitrary angle) cropping

9.1.6 3D display modes

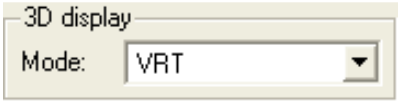


Fig. 9-10: 3D display mode toolbox.

Several different modes of 3D display are available:

MPR – Multi planar rendering. This mode displays the 2D slices as planes in 3D space.

MIP – Maximum intensity projection. Using a technique similar to the thick slice rendering, the projection chooses the brightest voxel along each viewing ray in the 3D window. As a result, the 3D object appears ghostlike, but three-dimensional

VRT – Volume rendering technique. Similar to MIP, this mode computes a 2D projection from all the data values in 3D. A color and transparency mapping, called transfer function, is applied to each voxel, determining the amount that is visible "behind" a voxel.

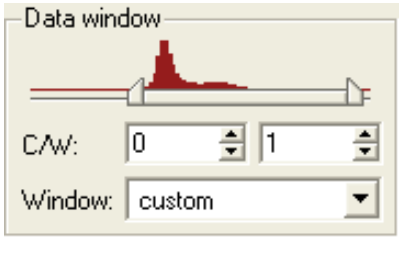
SVRT – Shaded volume rendering technique. This mode is similar to VRT mode, but adds virtual "shading" from a virtual light source. This shading adds highlights, such that curvature differences or small "bumps" on smooth surfaces can be easily detected

SSD – Shaded surface display (iso-surface). This rendering mode is useful for extracting structures that are mapped to a specific range of intensity values in the data. SSD does not use a color/transparency mapping as VRT and SVRT do, but a single threshold value that is used to define a virtual surface that represents that value in the data.

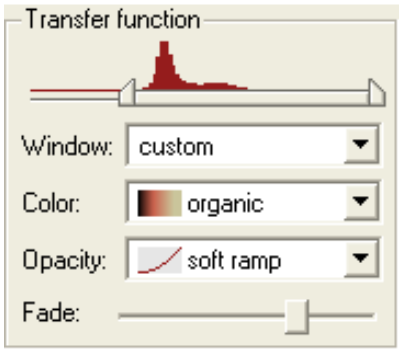
VRT is typically used for most cases, but SSD can be useful for low-noise, high-contrast images where surface rendering is important.

9.1.7 Data window, transfer function, fade, and color

Changing the *transfer function* and *fade* parameters for the 3D volume or the *data window* parameters for the slices will alter the appearance of the data. To change a parameter, click the appropriate slider and drag it to the desired position. The appearance image will change in real time.



(a) *Data window* threshold parameters (for slices)



(b) *Transfer function* and *fade* parameters (for volume)

Fig. 9-11: Data window (a) and transfer function(b) toolboxes.

The *transfer function* window allows some additional control, over both the color and the opacity function governing the effect of the *fade* slider.

Data window

Changing the *data window* sliders will alter the viewed dynamic range of the slices.

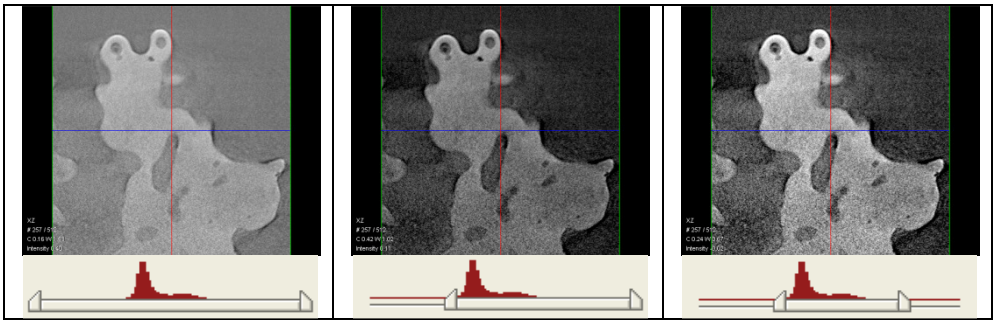


Fig. 9-12: 2D slice effects of adjusting the data window sliders.

Transfer function

Changing the *transfer function* sliders will alter the viewed dynamic range in the 3D volume rendering.

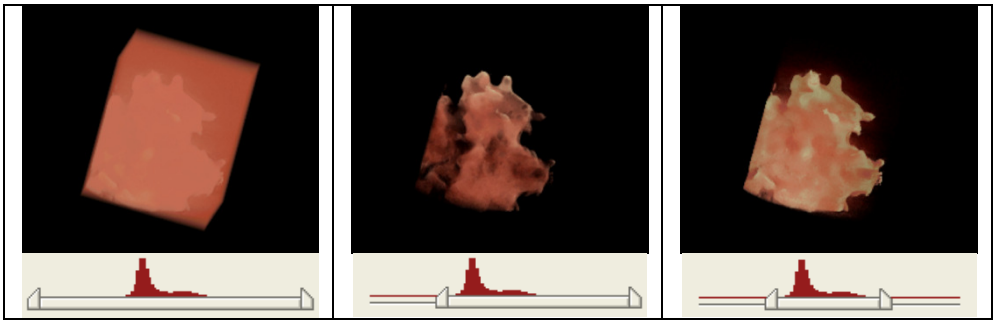


Fig. 9-13: 3D volume effects of adjusting the transfer function sliders.

Fade

Changing the *fade* slider will alter the threshold of the coloring.

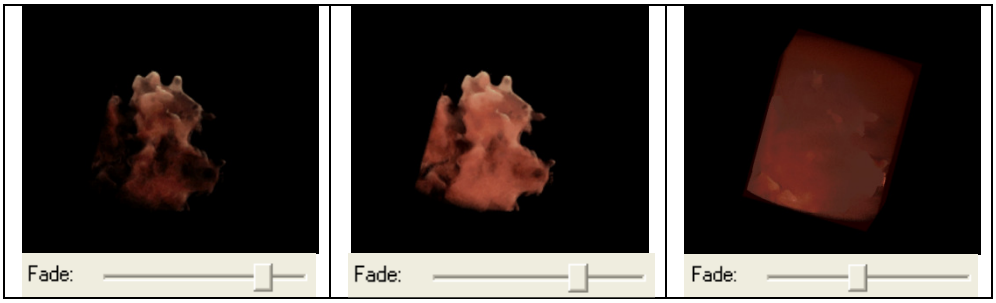


Fig. 9-14: 3D volume effects of adjusting the fade slider.

Color

Changing the *color* parameter will change the color spectrum used to fill in the 3D volume rendering.

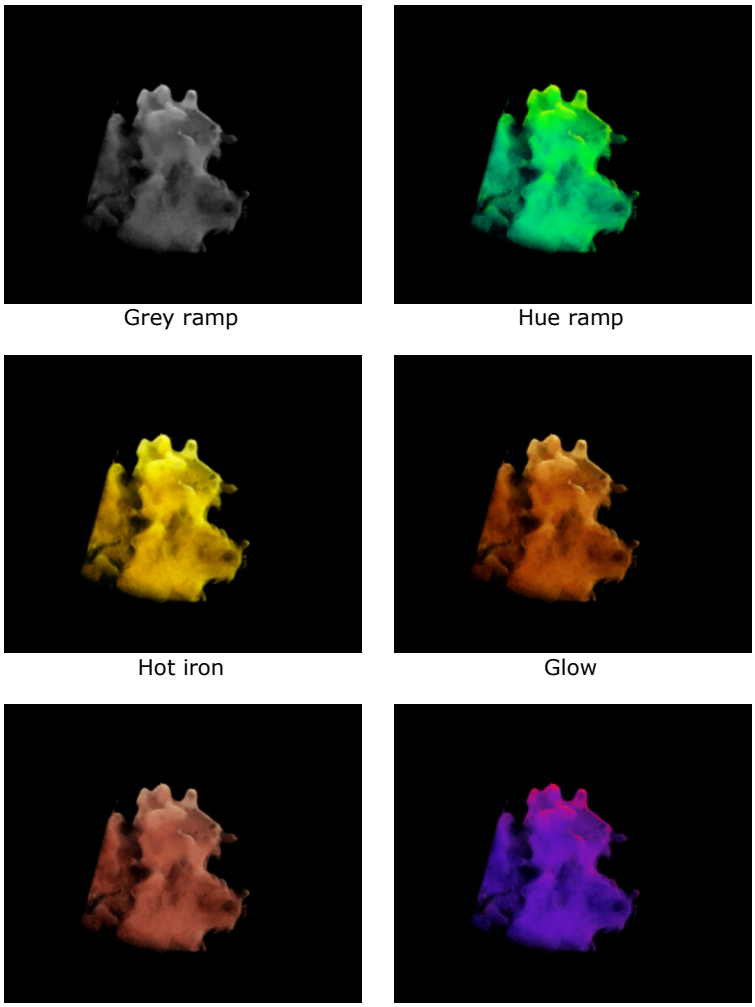


Fig. 9-15: 3D volume effects of changing the color scheme.

9.2 Exporting data

There are three techniques to exporting data. The first involves using TXM3DViewer to export an animated video file, the second to export a report of single slices to Microsoft Word, and the third uses TXMController to manipulate and export.

9.2.1 MPEG video files from TXM3DViewer

In the 2D slice or 3D volume layout mode, the *cine options* toolbox is enabled, allowing the preview and export of slice and volume animations as MPEG (*.mpg) files.

Fig. 9-16: Cine controls toolbox (active only in 2D or 3D view).

Clicking the play button ▶ will begin the animation, and window settings may be adjusted on the fly. To export the animation, click the save icon in the bottom left corner, launching the *cine options* window.

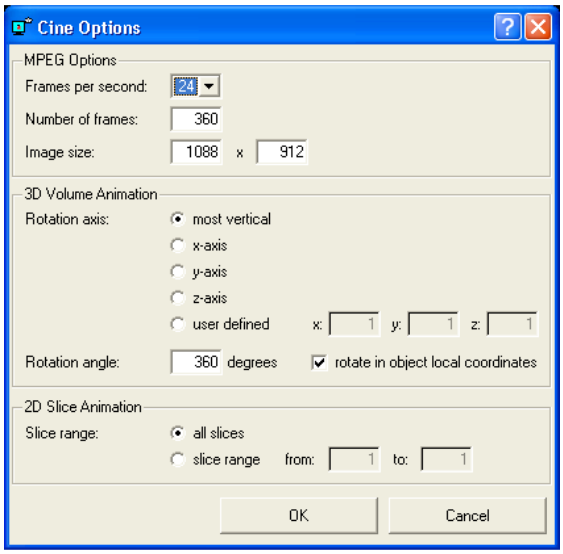


Fig. 9-17: MPEG export options.

The options available are as follows:

MPEG Options

Frames per second – Specifies the frame rate for the resulting MPEG file.
Number of frames – Controls the total number of frames for the resulting MPEG file. The time length of the video will be computed as the number of frames divided by the frame rate.
Image size – Specifies the size, in pixels, of the exported images.

3D Volume Animation

Rotation axis – Specifies the axis to be used for rotation (3D volume export only). The software may be allowed to calculate the most vertical axis for rotation, the user may specify one axis in particular, or an arbitrary vector may be defined.

2D Slice Animation

Slice range - Specifies whether to include all slices in the export or to restrict to a certain range.

Accepting all of the default values is typically sufficient.

When finished selecting options, click OK and select the location in which to save the file. Then, click OK again to begin the export.

NOTE: *The workstation must be left alone during the export process!* The export routine works through a system of screenshots, and opening any other windows will cause empty frames to be inserted in the exported file.

9.2.2 DOC reports from TXM3DViewer

If single slices are desired for export, TXM3DViewer offers an automatic report generation routine, which will insert them into a Microsoft Word (*.doc) document. To capture single images, follow the procedure below:

1. Select the camera icon from the *options* toolbox.
2. Click on the desired slice or volume view. Note that annotations (such as measurements, text notes, scale bar, etc.) will be captured as well.
3. Repeat steps 1 & 2 for each additional view.
4. Click on the *report* tab at the bottom of the viewer window. All of the captured images should be in the *snapshots* toolbox at the left of the screen.

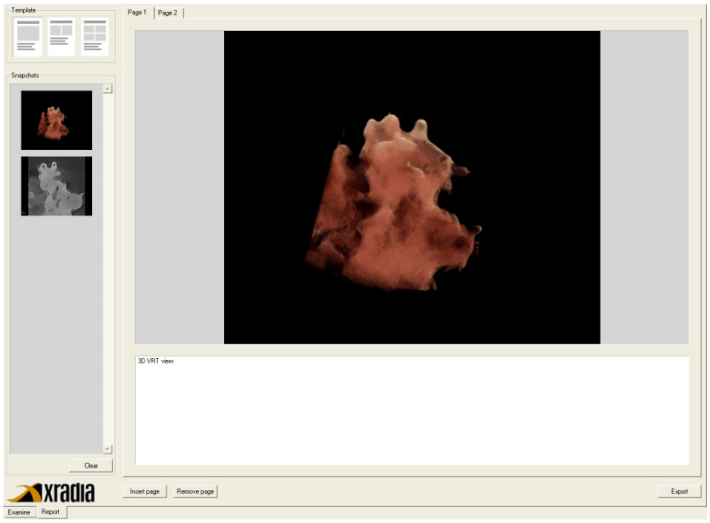


Fig. 9-18: Report generation utility.

5. Select the desired output template from the *templates* toolbox at the upper left corner of the *report* window.

Fig 9-19: Report template toolbox.

6. Click on the first image from the *snapshots* toolbox and drag it into the main window. Add notes, if desired, using the text box at the bottom of the screen.
7. To add an additional page (e.g., for another image), click the *insert page* Insert page button at the bottom of the screen, and select the tab corresponding to the new page (at the top of the window).
8. Iterate steps 6 and 7 until all images desired for export have been included. To clear images from the snapshots toolbox, click the *clear* button Clear; to delete a page, click the *remove page* button Remove page.
9. When finished, click the *export* button Export to launch Microsoft Word with the new report imported. The images may be moved to other applications using standard copy-and-paste techniques.

9.2.3 AVI & Image files from TXMController

The reconstructed files (*.txm) may also be opened in TXMController, which will display the XY slice view.

Writing XZ & YZ planes

Navigate to *Process->Write two other planes*.

Single slices

Use the functionality in *File->Save as…* (described in detail in the next chapter).

Animated video file

Navigate to *File->Create AVI…* and use the on-screen dialog. The available options are similar to those in the TXM3DViewer MPEG creation routine.

Chapter 10: Extended Functions of TXMController

In this section, several of the most commonly used "extended functions" of TXMController are described.

10.1 Changing objective lens magnification

As mentioned earlier, the detector uses two objectives: 2X magnification for alignment and 20X for imaging. These objectives are on a motorized shifting mechanism, and may be changed by selecting the proper lens from the magnification change icon in the main toolbar:

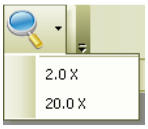


Fig. 10-1: Magnification change dialog.

10.2 Reference correction

After collecting an image or image series, it is generally beneficial to apply a reference image to normalize the data for effects contributed by the beam and detector. Reference images must be of the same or lower binning as the set to be normalized, and the count rate of the reference image should be greater. To minimize the chance of injecting noise into the normalized data, it is recommended to take at least five reference images and average.

To apply reference correction, open the *image control* set toolbox and navigate to the *Reference* tab. Click the *Browse…* button, and select the single (preferably averaged) reference image. Then, click *Apply Correction* to perform normalization. If more images will be collected with the same parameters in the same configuration, select the *Keep last file when there is no reference* checkbox, and the reference file will be associated with all future images as well.

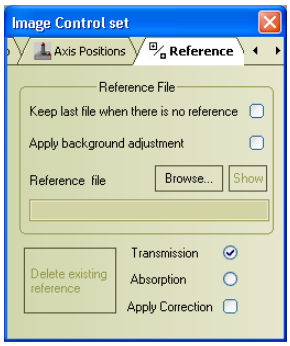


Fig. 10-2: Reference correction window.

Specific options are as follows:

1. Keep last file when there is no reference
   Enabling the *keep last file when there is no reference* checkbox makes the current reference file persistent, and stores it with all future image collections. Reference correction is not automatically applied, however, and the *apply correction* checkbox must still be selected in future images.

2. Apply background adjustment
   The *apply background adjustment* scales the amount of CCD dark current normalization to adjust for changes based on the value specified in TXMConfigure. It is generally not used, and should be left unchecked.

3. Reference file

a. Browse…
   The *browse* button opens a file browser window to select the reference file.
b. Show
   The *show* button displays the stored reference image on the screen in a new data window.

4. Transmission/Absorption
   By selecting the default *transmission* mode normalization, the current image is divided by the reference image. In *absorption* mode, the logarithm of the *transmission* mode normalization is taken, inverted, and then multiplied by -1. The result of *transmission* mode is a normalized image where x-ray absorption is visible as dark regions, and the opposite is the case in *absorption* mode.

5. Apply Correction
   Selecting the *apply correction* checkbox applies the normalization to the current image.

6. Delete existing reference
   The *delete existing reference* button will remove the current reference data file from the image.

10.3 Navigation through multiple-image data sets

When a multiple-image data set is collected (e.g., tomography, averaging, focal series), there are two main options for navigating through the data.

1. Data window navigation
   At the bottom of the data window, there is a slider with increment/decrement buttons allow quick manual navigation through the images. Drag the slider, click the +/- buttons, or type in an image number (and press enter) to cycle.

Fig. 10-3: Data window navigator.

2. Image control set navigation
   For automatic cycling of images, open the image control set and navigate to the *Navigator* tab. Using this feature, the data set may be cycled automatically, playing like a movie through each collected image. To keep the data cycling, select the *play continuously* checkbox.

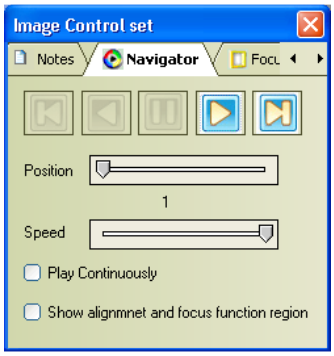


Fig. 10-4: Image control set navigator.

10.4 Image alignment and manual averaging

If an averaging series is not taken with *average on fly* selected, the resulting set must be averaged using the *alignment* feature of TXMController. Additionally, if the sample drifts during the collection of an averaging series, the individual images may be manually aligned first, minimizing any blurring effects in the final, averaged image.

To manually align a data set, open the image control set toolbox and navigate to the *alignment* tab. Under *mode*, select *manual*, and then double-click on a common point in each of the images in the data set. When finished, click *align*. Save the image if it is satisfactory or, otherwise, click *clear shifts* and repeat the alignment.

To average the data (after alignment, if necessary), click the *average* button. If bright spots are observed randomly distributed throughout the data set (from high-energy x-rays colliding directly with the CCD), the *dynamic despeckle* checkbox may be selected before averaging to remove them.

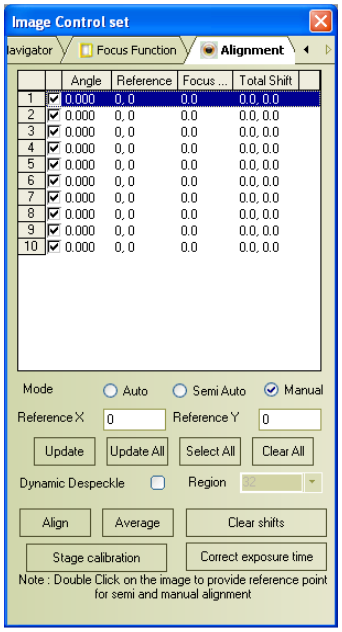


Fig. 10-5: Image alignment & manual averaging tool.

10.5 Retrieving old motor positions

TXMController stores current motor positions with each collected image. To retrieve the motor positions for any image, open the image control set toolbox and navigate to the *axis positions* tab. Here, axis positions relative to zero at the time of image collection are displayed. By selecting the *show distance from home position* checkbox, absolute positions of each axis relative to home are displayed. If the data file contains more than one image, the menu at the bottom may be used to plot a graph of the position of any axis through the entire set.

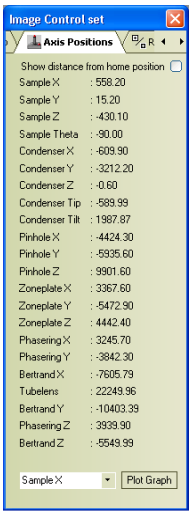


Fig. 10-6: Axis positions information.

10.6 Retrieving image and exposure information

All relevant machine and imaging parameters are stored along with each image, including exposure time, binning, stage correction values, and etc. To display this information, open the image control set toolbox and select *info*.

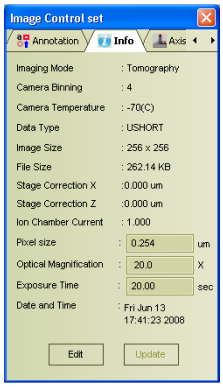


Fig. 10-7: Image information.

To manually change the pixel size, optical magnification, or exposure time, click the *edit* button, type in a new value, and then click *update*.

10.7 Exporting data

If it is desired to open a collected image in an external program for analysis or presentation, TXMController offers a wide variety of exportable formats for both single- and multiple-image files. The available formats include BMP, JPEG, RAW TIFF, Standard TIFF, BIN, and, for multiple-image files, AVI.

1. Exporting a single-image file (*.xrm)
   To convert a single-image file to another format, navigate to *File->Save As* and select the desired file type from the drop down list at the bottom of the browser window.

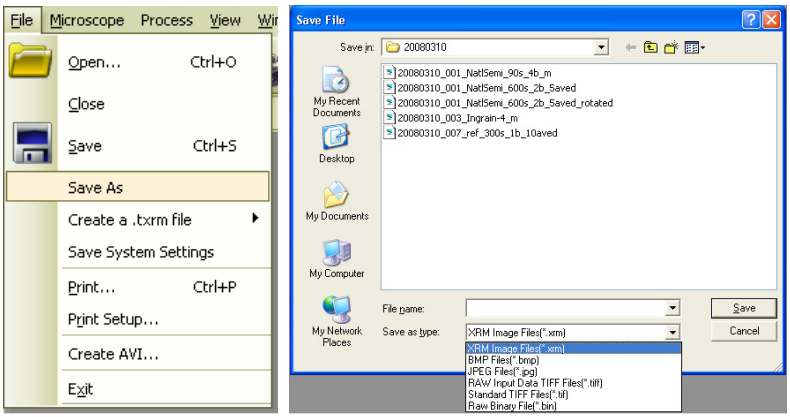


***Fig. 10-8: Save as* dialog for exporting a single-image file.**

2. Exporting a multiple-image file (*.txrm)
   If the current data file contains more than one image, it is possible to export one single image or the entire series. To begin, navigate to *File->Save As*, as in the single-image case above. Select the desired output format from the *save file as* list, and then select whether to output the *whole series* or only the *current image*. To include annotations (such as the *micron bar* or *scale bar*), select the *include annotations* checkbox. In the *location* field, specify the directory for output and a name for the image series. Images will be exported in accordance with the naming convention given, appended with a number indicating the image number in the series.

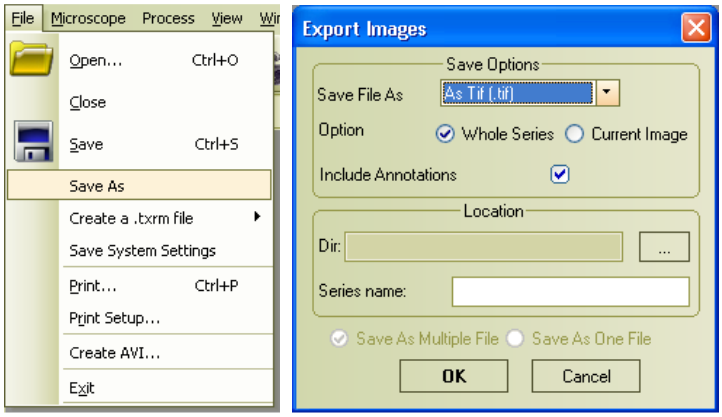


Fig. 10-9: *Save as* dialog for exporting a multiple-image file.

10.8 Focus Function

After collecting a focus function data set, TXMController offers an automated routine to determine the position of best focus. Open the image control set toolbox and select the *focus function* tab. Select the check box corresponding to the sample type (*chip* for high-contrast materials and *general* for most other cases), and then choose the region to examine (*full image* to examine the entire image or *selected region* to choose a small region only). If a *selected region* is chosen, select the linear pixel size for the region of interest and then double-click in the center of this region on the image. When finished, click *plot focus function* to view the calculated results.

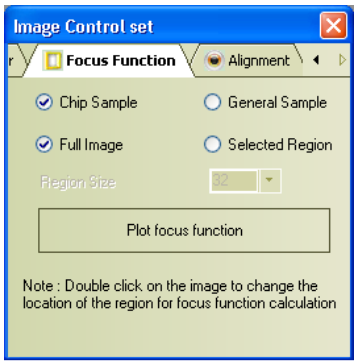
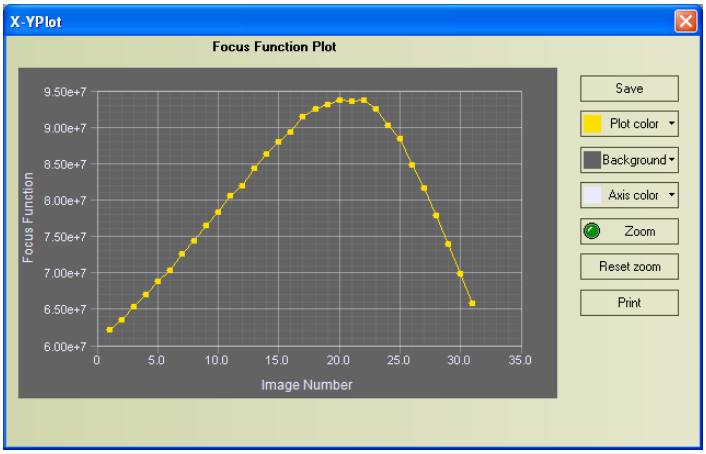


Fig. 10-10: Focus function window.



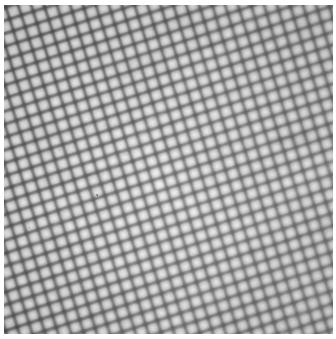


Image 1

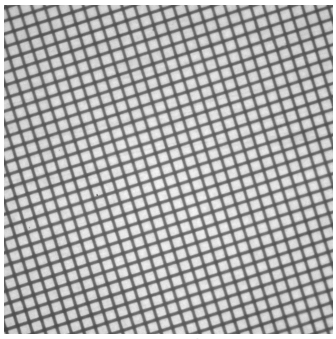


Image 21

Fig. 10-11: Resulting plot of calculated focus function.

Chapter 11: Troubleshooting

Periodic errors may be encountered when using the nanoXCT. In this section, the most common problems are outlined, along with typical solutions. If the problems are not solved using these methods, please contact Xradia for support (see Appendix E).

11.1 Motion controller communication failure

Symptom

TXMController complains, "Unable to communicate with motion controller."

Problem

USB communication with the motion controller has been lost.

Solution

There are several possible fixes, which are outlined here in the order in which they should be tried. As soon as the problem is fixed, proceed no further.

1. Shut down the computer and unplug the *USB cable only* from the front of the motion controller (do not turn off the power). Wait 10 seconds, and then plug the USB cable back in. Start the computer workstation back up and reopen TXMController.

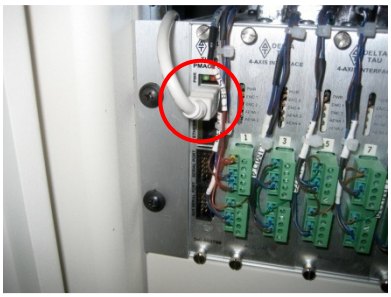


Fig. 11-1: Location of the USB-B connector on the motion controller.

2. The device may be "removed" and "reinserted" through the use of a software program. To do this, open PEWIN32PRO by navigating to Start -> All Programs -> PEWIN32PRO -> PEWIN32PRO. It may take up to 30 seconds for the program to open, so patience is the key if it appears to hang.

   Navigate to Setup -> General Setup and Options.

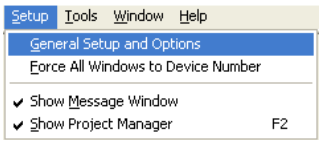


Fig. 11-2: Navigating to *General Setup and Options* menu.

   Under the *Default Device* tab, click the box labeled *Select*.

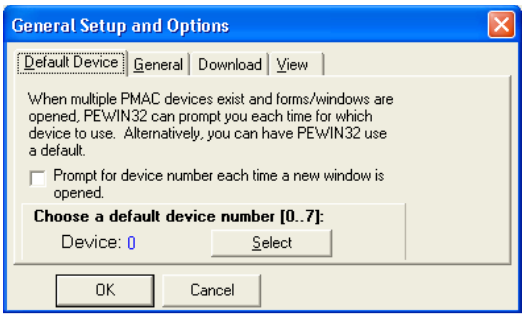


Fig. 11-3: *General Setup and Options* menu.

If a USB device (e.g., "USB0 – Plug and play") is listed, click it once to highlight and then click *Remove*. This will perform a soft-removal of the USB device from the system.

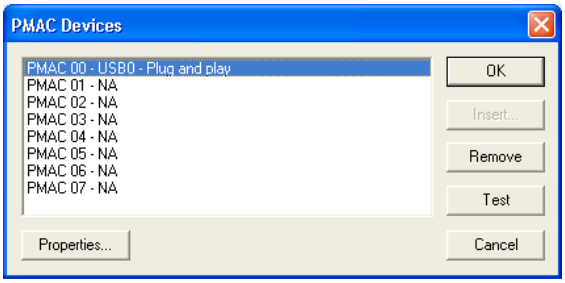


Fig. 11-4: *PMAC Devices* menu

If an error is encountered in which the software asks for applications to be closed, click *OK* on the message window, close PEWIN32PRO, unplug the USB cable from the motion controller, and restart PEWIN32PRO. Then attempt device removal again. When removal is successful, plug the USB cable back in.

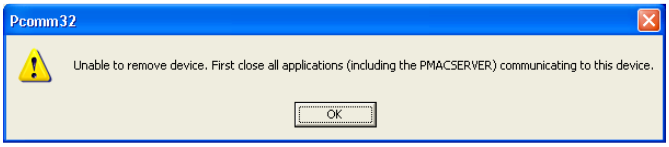


Fig. 11-5: *Pcomm32* error message.

Click once on *PMAC 00* to select it and then click *Insert*. In the *Available PMAC Devices* list, select *Pmac USB0* (*USB1* on some systems) and click *OK*. If no *PMAC USBx* devices are listed, restart the computer and try again. When the device is successfully reinserted, click *OK* to close the *PMAC Devices* window.

Close all active windows. Navigate to *View* -> *Terminal* to open a new terminal window. If the device has been correctly initialized, the terminal should say *Press Enter/Return to send command to PMAC*. If, instead, it says *Unable to communicate*, reboot the workstation and attempt the procedure again.

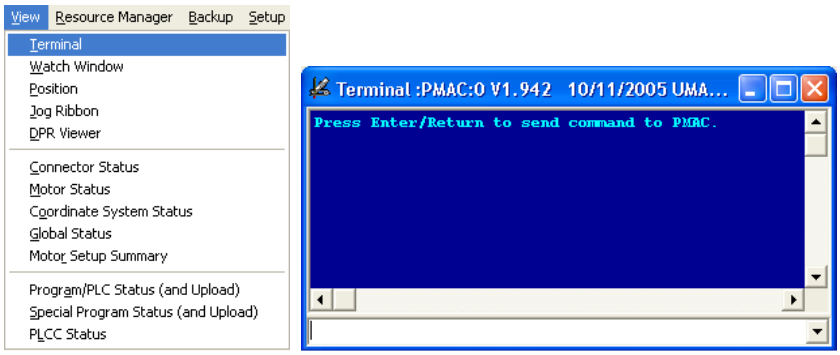


Fig. 11-6: Launching the terminal.

Alternatively, from the main screen, select *Setup -> Force All Windows to Device Number*. Click once on the device that was just reinserted to select it, and then click *OK*. This will cause all open windows to try to communicate with the motion controller, and any open terminal windows should display the message shown above.

Once the device is recognized by PEWIN32PRO, close the program and reopen TXMController.

3. As a last resort, the power to the motion controller may be reset. Note, however, that this will most likely result in needing to perform a complete system realignment.

   Shut down the computer workstation. Unplug the power and USB cables from the motion controller. Wait 10 seconds. Plug the USB cable back in and then plug the power cable back in. Restart the computer workstation and reopen TXMController. If communication has been re-established, TXMController will ask to re-home the motors. Remove the sample holder (if it is in) and click *Yes*.

Fig. 11-7: Location of the power connector on the motion controller.

If the motion controller is not recognized, try to reinsert it via PEWIN32PRO as described in the latter half of step 2.

The system will now most likely be misaligned, so it will be necessary to perform a complete alignment. It may be helpful to open a recent aligned image and move the motors to that position by navigating to *Microscope->Set motor position based on Image*. This will return the system close to its aligned state, but it is recommended to then remove each component one by one and perform minor alignment adjustments per the procedure outlined in chapter 3.

11.2 Camera communication failure

Symptom

TXMController complains, "Main Camera initialization failed."

Fig. 11-8: TXMController error message when camera communication is lost.

Problem

USB communication with the CCD camera has been lost.

Solution

Follow this procedure:

1. Close all programs that have potential communication with the camera (e.g., TXMController).
2. Inside the NCT, turn off the power switch on the transformer box.

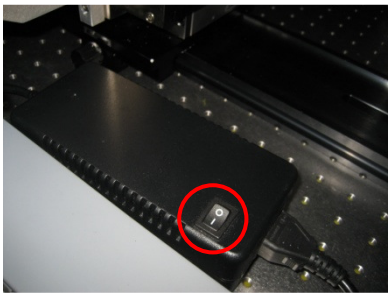


Fig. 11-: Location of the power switch on the CCD power transformer.

3. Unplug the USB cable from the CCD camera.

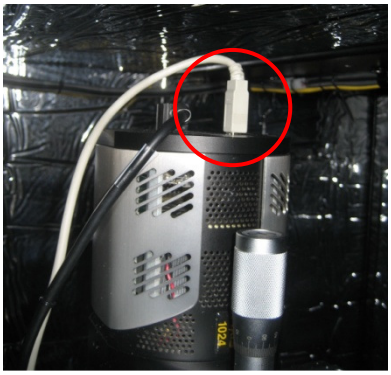


Fig. 11-: Location of the USB-B connector on top of the CCD camera.

4. Wait 10 seconds.
5. Plug the USB cable back into the CCD camera.
6. Turn the power back on.

7. Navigate to Start -> All Programs -> Roper Scientific -> RSConfig. Make sure that *Camera1* registers in the *Camera 1* field and click *Done*. If it does not, close RSConfig and reopen. When the camera registers, reopen TXMController.

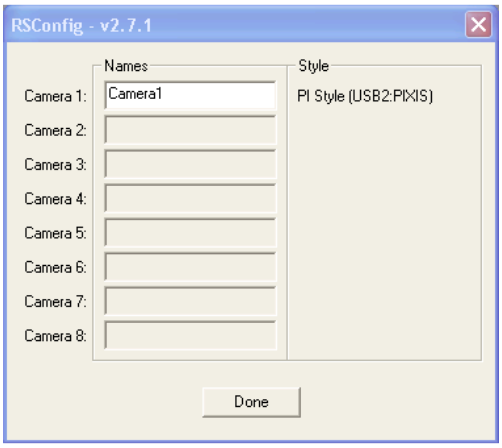


Fig. 11-9: RSConfig window.

In the rare event that this does not fix the problem, try restarting the computer. If communication problems persist, try switching the camera to a different USB port on the computer.

11.3 Poor/uneven image contrast

Symptom

The image contrast drops to low levels (e.g., <10% for the X50-30-7 test pattern) in some or all locations.

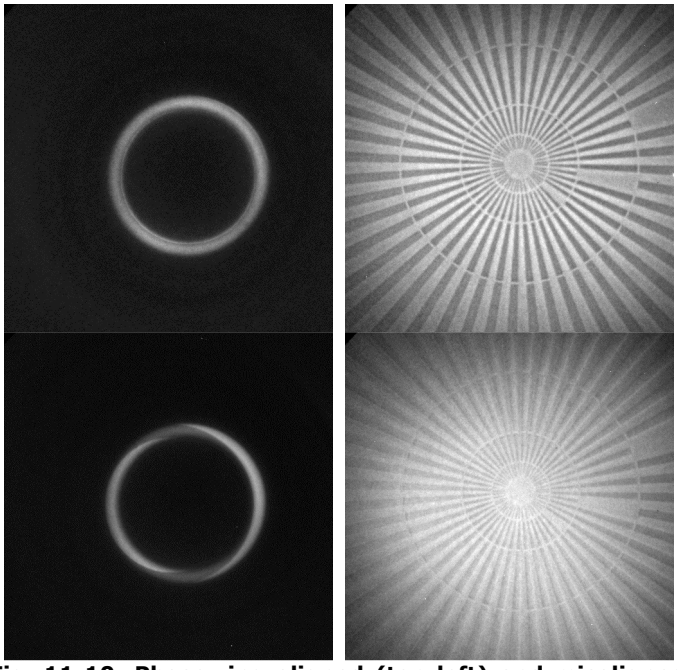


Fig. 11-10: Phase ring aligned (top left) and misaligned (bottom right) with corresponding test pattern images.

Problem

The phase ring has drifted out of its aligned position.

Solution

Move in the Bertrand lens and check the alignment of the phase ring. Reposition the phase ring so that it completely blocks the illumination ring.

11.4 Poor/uneven illumination profile

Symptom

The illumination profile is uneven, or parts of profile are missing.

Problem

The system has become misaligned. Usually, this is due to a drift in the condenser (either due to staging or source drift). Occasionally, this is due to a misalignment of the flight tube (due to mechanical collision with a user).

Solution

Switch to the 2X objective and remove each component one by one until the culprit is revealed (using the images in chapter 3 as reference). Realign the system according to the procedure in chapter 3.

If the condenser was misaligned, try to make adjustments so that the pinhole position will remain unchanged (usually, this means adjusting x/y more than tip/tilt). Align the condenser and then insert the pinhole. Then, iterate adjusting the condenser and removing/inserting the pinhole until the 1^{st} order reflected beam passes through the pinhole without tweaking the pinhole position.

If the flight tube was misaligned, remove all components and then adjust the flight tube saddles on the detector rail until the beam passes through as in fig. 3-1. Then, reinsert all optical components.

Chapter 12: Additional Information

12.1 Disposal and decommissioning

Xradia will assume responsibility for machine disposal and decommissioning. Please ship all components back to Xradia for proper disposal.

12.2 Maintenance

Please refer to your maintenance contract for the appropriate maintenance schedule.

Appendix A: Advanced Alignment Procedures

A.1 Motor Homing

Due to the dynamic nature of the axis positions, the current positions are not automatically stored in permanent memory. As a result, whenever power to the motion control hardware is lost, all of the axis positions go with it and must be reset. In order to minimize fonusion and ralte past positions to those after a power outage, Xradia has implemented a system of homing routines, which align all of the axes to hardware reference points with high precision.

When homing is necessary, the user is prompted with a dialog when launching TXMController, as pictured below. If this message appears, remove the sample holder and click yes to begin execution of the homing routines.

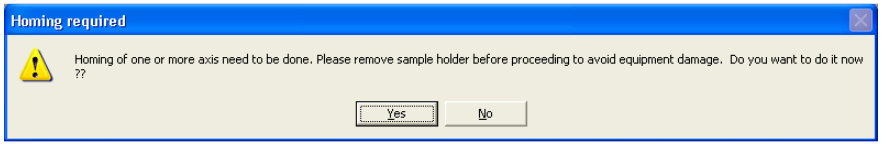


Fig. A-1: Automatic prompt if homing is necessary.

If homing cannot be performed immediately, it is also possible to start the sequence manually through TXMController. In this case, remove the sample holder as before and navigate to *Microscope->Home all axes…*

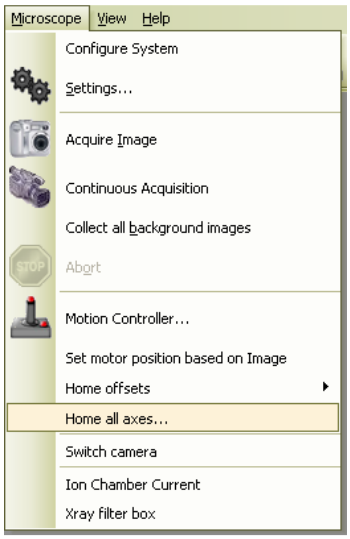


Fig. A-2: Manual homing.

Once the homing sequence is complete, all axes will be at the positions last marked as zero. In any case, system alignment should be checked, as minor corrections are typically necessary.

A.2 Optical axis definition

The optical axis is defined by the detector rail, which is registered to the optical train plate and breadboard. To define the optical axis, use the following procedure:

1. Open the flight tube. then mark the current position of the detector along the rail.

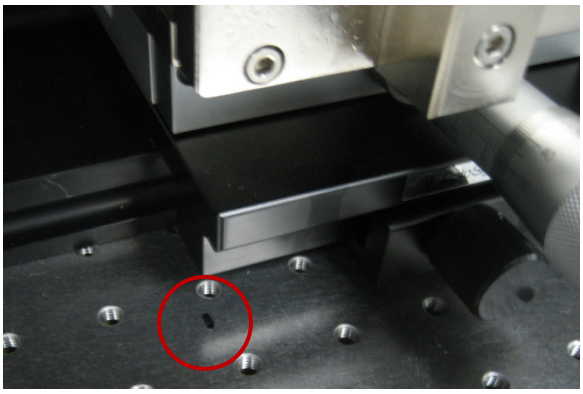


Fig. A-3: Marking the detector position.

2. Move all components out of the beam path except for the condenser. Make sure the condenser is well-aligned.
3. Move the detector as far downstream (away from the source) as possible and take one image. Mark its position on the breadboard. Do not realign the condenser, we will just use the projection of the beam stop for measurement.
4. Move the detector as far upstream (toward the source) as possible and take one image. Mark its position on the breadboard. Do not realign the condenser, we will just use the projection of the beam stop for measurement.

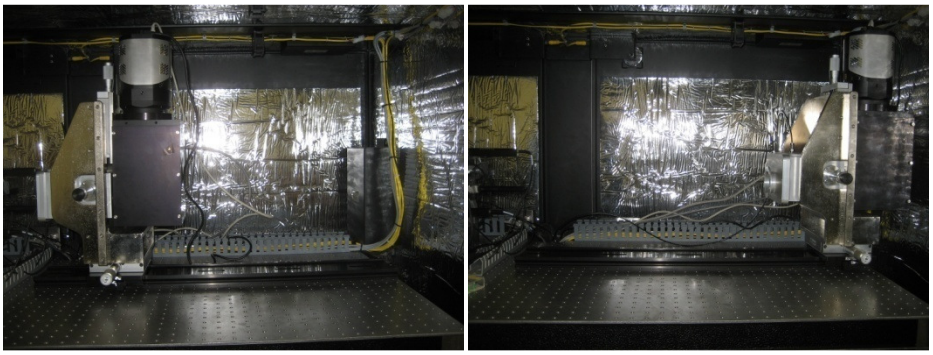


Fig. A-4: Detector moved to downstream (left) and upstream (right) limits.

5. Measure the distance between the detector downstream and upstream positions, in mm, and call the distance Δz.
6. Use the circle annotation tool on each image to find the center of the beam stop (dark circle observed in the center of the condenser image). Take (x_1, y_1) to be the center of the image with the detector downstream and (x_2, y_2) upstream, with the measurements in pixels. Then, define:

$$\Delta x = x_2 - x_1$$
$$\Delta y = y_2 - y_1$$

7. Calculate the condenser pitch and yaw shift, (Δc_P, Δc_Y) respectively, via the following formulae where *b* is the binning of the images collected of the condenser in the two different detector positions:

$$\Delta c_P = \frac{124.1 * 6.5 * \Delta y * b}{\Delta z}$$

$$\Delta c_Y = \frac{92.3 * 6.5 * \Delta x * b}{\Delta z}$$

8. Move the condenser pitch by Δc_P and yaw by Δc_Y. Use the condenser x and y motors to realign the 1st order reflected light to be concentric with the 0th order. Move the detector if the condenser goes out of the field of view as a result of translation.
9. Iterate steps 3-8 until the following conditions are satisfied:

$$\frac{6.5 * \Delta x * b}{\Delta z} \ll 1$$

$$\frac{6.5 * \Delta y * b}{\Delta z} \ll 1$$

10. The optical axis should now be properly defined.

A.3 Stacked zone plate alignment

For nanoXCT-100 systems using aligned ("stacked") zone plates, an additional, manual pitch/yaw alignment must be performed to ensure proper operation of the lenses.

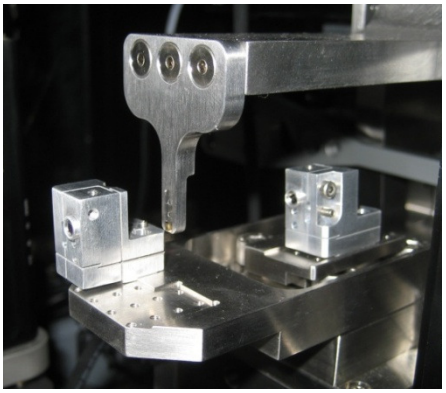


Fig. A-5: Stacked zone plate holders installed for HRES (left) and LFOV (right).

The ZP30-320 (used for large field of view) may be aligned without the use of any additional lenses, but the ZP35-80 uses the Bertrand lens to image the zone plate in order to enable alignment.

The following procedure outlines the necessary steps to align the stacked zone plates to the optical axis.

A.3.1 Large field of view (ZP30-320)

1. Align the condenser, pinhole, and zone plate as described in section 3.2.2. Take one image in the 20X objective with high statistics, then switch back to 2X.
2. Take one image with high statistics in the 2X objective (10 seconds at binning 4 generally works well).
3. If the zone plates are misaligned, a Moiré fringe pattern will be visible around the outside of the condenser ring. Vertical fringes indicate a yaw misalignment and horizontal fringes indicate a pitch misalignment. Diagonal fringes indicate a combination of pitch and yaw misalignment.
4. Use an extra-small (0.035") hex key to make a small adjustment to the appropriate set screw. If turning the screw does make a difference, it is possible that the set screw is at a travel limit; in that case, use a small 1.5mm hex key to adjust the preload screw as appropriate.

   NOTE: It is best to first correct any yaw misalignment and then finish with pitch.

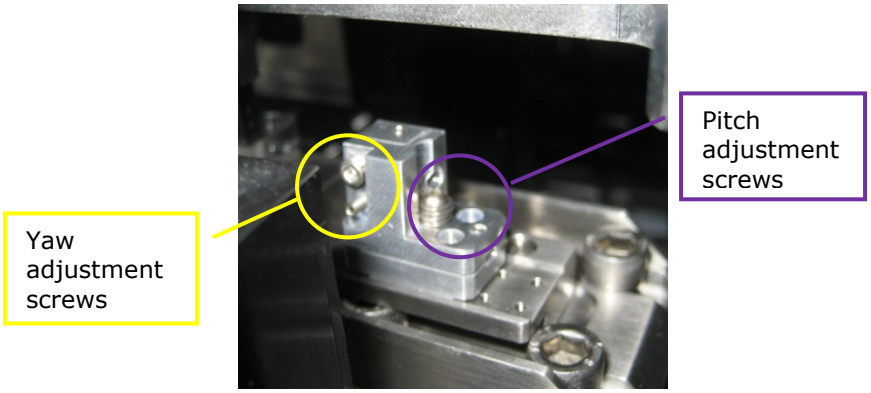


Fig. A-6: Stacked zone plate holder.

5. Image in continuous mode (e.g., 1 second at binning 4) and use TXMController to reposition the zone plate in the center of the condenser ring, as shown in section 3.2.2.
6. Iterate steps 2-5 until the number of fringes diminishes to zero. In the final few moves, the fringes will be visible as blockages on the outer edge of the condenser ring; small, precise motions with the 0.035" hex key should remove these and make the ring uniform.

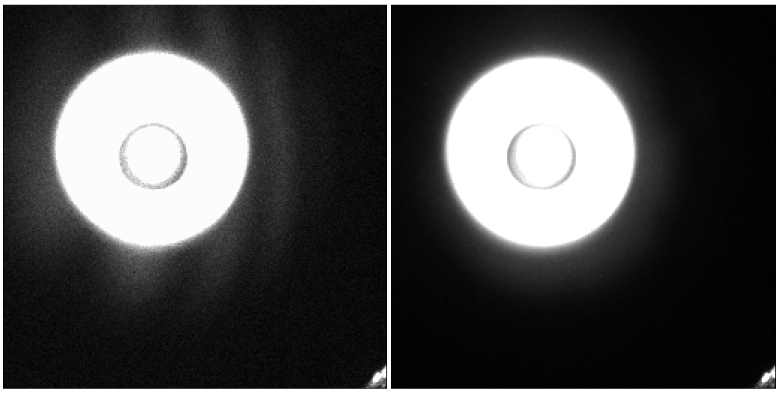


Fig. A-7: Stacked ZP30-320 misaligned in yaw (left) and aligned (right), imaged with the 2X objective.

7. Switch back to the 20X objective and take another image with the same parameters as in step 1. If the zone plates were misaligned to begin with and are now aligned, then mean count rate should increase by approximately a factor of 2.

A.3.2 High resolution (ZP35-80)

1. Align the condenser, pinhole, and zone plate as described in section 3.2.1. Take one image in the 20X objective with high statistics, then switch back to 2X.
2. Use the motion controller to move the phase ring X to its negative limit and phase ring Z to its positive limit.
3. Remove the arm that supports the HRES Bertrand lens holder and physically shift the Bertrand lens by 20 mm toward the source from its aligned position. This generally requires moving the Bertrand lens holder to the very last hole, so that it is supported by the rear screw only (the front screw hole hangs over the side of the support arm). Then, reinstall the Bertrand lens support arm into nanoXCT.

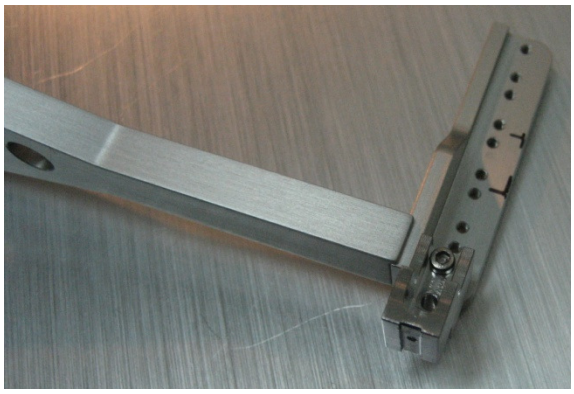


Fig. A-8: HRES Bertrand lens (ZP100-160) physically shifted by 20 mm from its designed position.

4. Realign the HRES Bertrand lens (ZP100-160) and center it in the FOV using the 2X objective. Switch to the 20X objective and take one image with high statistics (e.g., 30 seconds at binning 2).
5. If a Moiré fringe pattern is visible in the image from the Bertrand lens, then the zone plates are misaligned. As in the previous section, vertical fringes correspond to yaw misalignment, horizontal to pitch, and diagonal to a combination of yaw and pitch.
6. As in the previous section, adjust the yaw and then pitch using the small hex wrenches as appropriate.
7. Switch back to the 2X objective and translate the zone plate in x and y until the illumination is centered inside the condenser ring and around the image from the Bertrand lens. Then, switch again to the 20X objective and take one image with the same parameters as in step 4.
8. Iterate steps 5-7 until no fringes are visible.

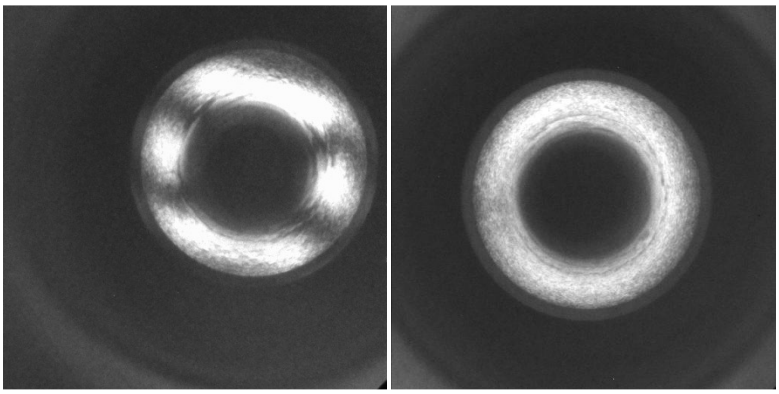


Fig. A-9: Stacked ZP35-80 misaligned in both pitch and yaw (left) and aligned (right), imaged with the 20X objective.

9. When the zone plates are finally aligned, move the Bertrand lens by 7 mm to remove it from the optical train and take one image with the same parameters as in step 1. If the zone plates were misaligned to begin with and are now aligned, then the mean count rate should increase by approximately a factor of 2.
10. Shift the Bertrand lens back into its phase ring alignment position by removing the support arm from nanoXCT, physically moving the holder back to where it started, and then reinstalling the support arm.

A.4 Rotation axis alignment

As part of the setup procedure, particularly for the collection of RSD data, it is necessary to align a tungsten STM tip to the physical axis of rotation of the rotation stage. This technique is also useful for collection of tomography data when any sample is grossly misaligned from the rotation axis (e.g. > 1mm), to minimize the effects of stage pitch as the software stage correction translates the sample.

The following procedure outlines the technique for aligning a sample on the flexure-style holder to the axis of rotation, using a tungsten STM tip mounted in the pin vise as the example. The large field of view configuration works best for this technique, but the high-resolution configuration is demonstrated here. By the end of this procedure, the nanoXCT and pre-alignment microscope (PAM) will be aligned to each other, with x=0 and z=0 referring to the axis of rotation.

Hardware rotation axis alignment

1. Install and focus a test sample.
   Mount a sample in the holder and center it in the field of view. Adjust the sample Z position until the image is in sharp focus at $\theta = 0°$.

2. Note the X position of the sample at $\theta = 0°$, 90°, 180°, and 270°.
   Write down the sample X position at $\theta = 0°$. Use the sample theta motor to adjust the angle to 90°, 180°, and 270°, noting the X position at each. If the sample is far from the rotation axis, it may be helpful to perform a small mosaic to ease the process.

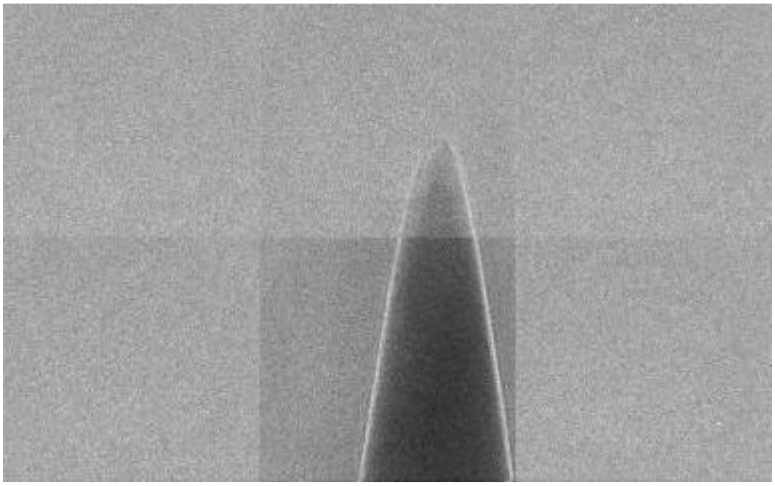


Fig. A-10: Small mosaic performed to locate sample.

3. Calculate the location of the center of rotation.
   By virtue of this projection-location method, it is possible to calculate the displacements necessary along the X and Z axes to bring the sample close to the physical axis of rotation. First, use the $\theta = 0°$, $\theta = 180°$, and $\theta = 270°$ projections to the sample X axis to calculate the position of the center of rotation through the following formula:

$$cr_x = \frac{x(0°) + x(180°)}{2}$$

$$cr_y = \frac{x(90°) + x(270°)}{2}$$

   where x(0°), x(180°), and x(270°) are the X positions of the sample at $\theta = 0°$, $\theta = 180°$, and $\theta = 270°$ respectively.

4. Calculate the displacement for X and Z.
   Using the value calculated above, the displacements necessary in X and Z may be calculated through the following formulae:

$$\Delta x = x(0°) - cr_x$$
$$\Delta z = x(90°) - cr_{yr}$$

   where x(90°) is the X position of the sample at θ = 0° and x(0°) is defined as before.

5. Center the sample on the pre-alignment microscope.
   Remove the sample from nanoXCT and place it into the pre-alignment microscope (PAM). Locate the sample first with the 10x objective, and then switch to the 50x objective. Use the PAM micrometers to center the sample in the crosshairs.

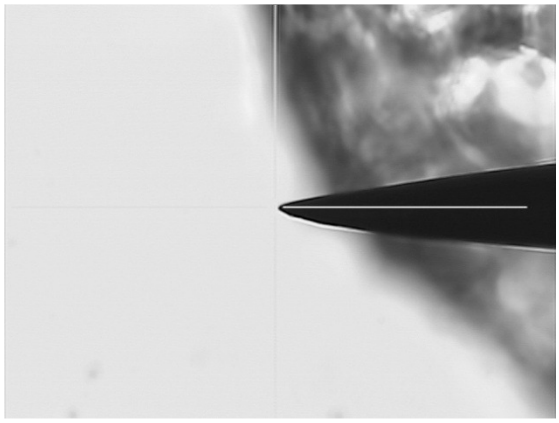


Fig. A-11: Tungsten STM tip centered in the crosshairs of the PAM.

6. Zero the PAM and physically reposition the sample to the calculated axis of rotation.
   With the tip centered in the crosshairs, press the "ZERO/ABS" button on the PAM micrometers. Move the micrometers to x=-Δx, z=-Δz, as calculated above. Using a small hex wrench, adjust the screws on the sides of the flexure base to push the sample back to the crosshairs. It is best to do this without switching objectives, if possible.

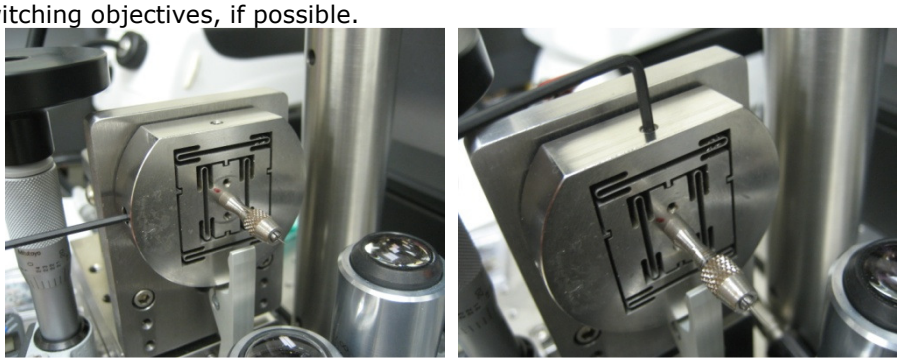
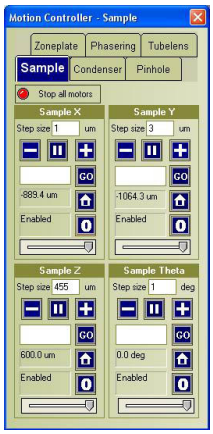


Fig. A-12: X (left) and Z (right) adjustment of flexure base hardware.

7. Iterate the procedure, if necessary.
   Put the sample back into nanoXCT, and recheck the X positions at the same angles as before. Iterate the procedure until all of the positions are within 1-2 μm of each other.

8. Zero the sample stage motors in TXMController and PAM micrometers.

With the sample centered in both nanoXCT and PAM, click the "0" button on the sample X, Y, and Z axes in TXMController. Press the "ZERO/ABS" button on the PAM micrometers as before. Both instruments are now aligned to each other as well as the axis of rotation of the sample stage.



a) Click the "0" button on each axis to zero the software.

b) Press the "ZERO/ABS" button to zero the micrometer.

Fig. A-13: Zeroing NCT (a) and PAM (b).

A.5 RSD calibration

In order to overcome systematic wobble of the rotation stage, TXMController version 5+ has an integrated correction routine to shift the sample in the x-direction during tomography, thus aligning the sample to a virtual rotation axis. This is called "rotation stage deviation" (RSD) correction, and involves the software reading shift values from a text file that resides in the path of the program.

The following procedure outlines the steps necessary to generate the text file and describes the process of enabling the correction routine.

A.5.1 Generating the RSD file

1. Follow the procedure to align a sample (e.g., tungsten STM tip) to the rotation axis using the hardware method. Iterate the procedure until variations between 0, 90, 180, and 270 degree sample theta positions are within 2 μm of each other.
2. Collect three tomographic data sets using the following parameters:
   Start angle: - 180 deg
   End angle: +180 deg
   Step: 1 deg
3. Align the first data set using TXMController's alignment feature. After clicking the "align" button, navigate to C:\Program Files\Xradia Inc\Micro-XCT, and locate the file called XYShifts.txt. Copy this file to a new location and rename it to *rsd1.txt*.
4. Repeat step 3 for the second and third data sets, calling them *rsd2.txt* and *rsd3.txt* respectively.
5. Import the XShifts list from each *rsdX.txt* file into your favorite data analysis program (e.g., Igor, MATLAB, Microsoft Excel, etc.), and create a new data set consisting of the mean values of each line.
6. Multiply each mean value by the pixel size, in microns, of the collected data sets.
7. Export this new data set, which should have 361 lines, to a text file, and call it RotationStageDeviation.txt. Open the file in a text editor and insert one line at the top exactly as below:

//////Rotation Stage Deviation -- DO NOT DELETE THIS LINE///////

8. Navigate back to C:\Program Files\Xradia Inc\Micro-XCT\, and open the directory called StageCorrection. If this directory does not exist, then create it.
9. With TXMController closed, copy the RotationStageDeviation.txt file into C:\Program Files\Xradia Inc\Micro-XCT\StageCorrection.

A.5.2 Enabling RSD correction

1. Open TXMController.
2. Navigate to Microscope->Configure System.
3. At the top of the system configure window, select the check box next to "Apply stage correction." Stage correction values in this window may both be set to zero.
4. Click OK in the system configure window. RSD is now enabled.

A.5.2 Example RotationStageDeviation.txt file

```
//////Rotation Stage Deviation -- DO NOT DELETE THIS LINE///////
-0.516
-0.43
-0.774
-1.204
-1.892
-1.29
-1.892
-1.978
-1.548
-1.032
-2.322
… etc …
```

A.6 CCD rotation correction

Whenever a new CCD camera is installed, its rotation relative to the axes of the other staging must be adjusted to within Xradia's specification of 1 mrad. In this section, a procedure for checking and correcting the CCD rotation is outlined.

1. Locate an appropriate sample for the correction procedure. The preferred sample is a test pattern mounted on a thin rod, but a test pattern mounted in a standard sample holder, when used with extreme caution when rotating at high angles where collision with the pinholes is possible, can work as well.

Fig. A-14: Star pattern mounted on a thin rod, thus allowing full 180° rotation.

2. Using the PAM, align the test pattern to the rotation axis as best as possible.
3. Insert the pattern into NCT and take a single image for reference.

4. In one corner of the test pattern, there is a 30 x 30 μm array of Au dots located ~ 45 μm in x and y from the center. Locate this region using binning no greater than 2.

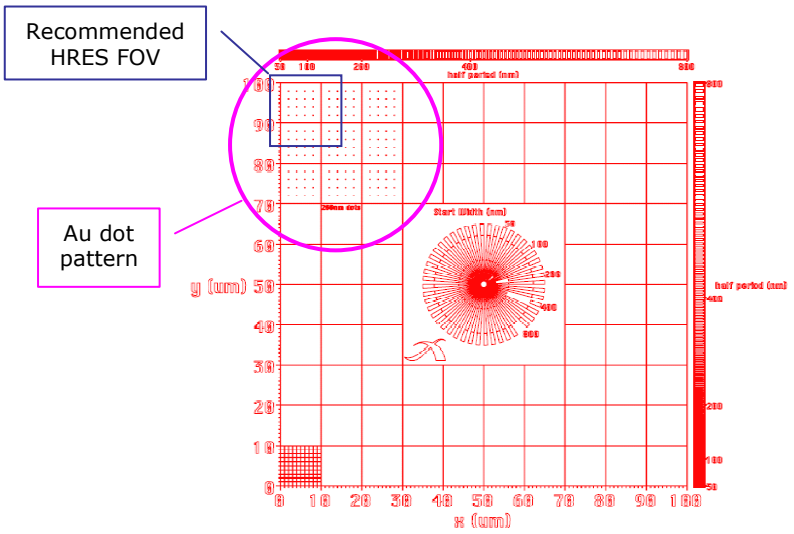


Fig. A-15: CAD model of the "star" test pattern. The pattern measures roughly 100 μm from edge to edge, and the Au dot pattern is pictured in the upper left corner.

5. Take one single, low-binning exposure (e.g. 300sec at binning 1). The best image to take will encompass as many of the dots as possible in both directions, but retain the outer edge of the test pattern in the FOV (see figure below).
6. Rotate sample theta to 180° and locate the same region as before. Take a single image with the same acquisition parameters as before.

   CAUTION: If the standard sample holder is used, make sure to remove the sample holder completely from the stage before rotating and immediately after image collection (the system will automatically return to 0° after collecting a single image).

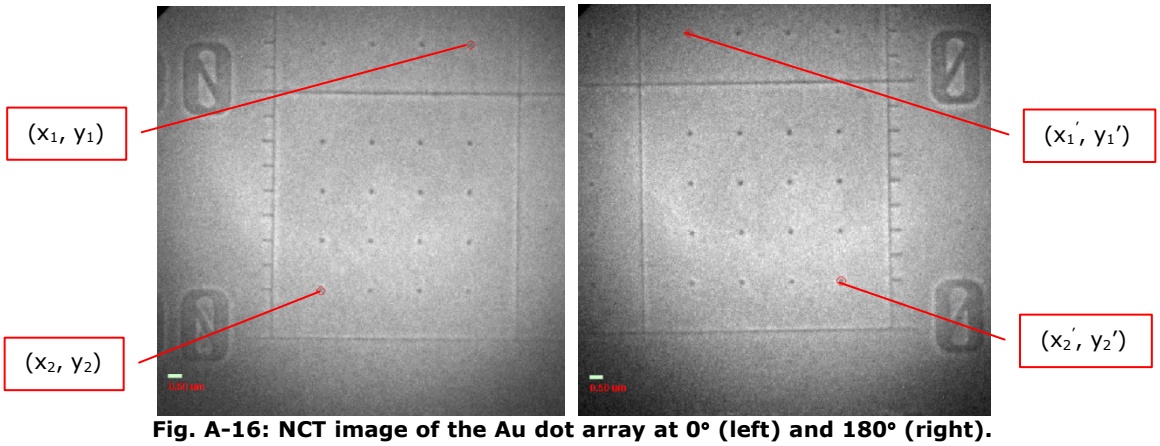


Fig. A-16: NCT image of the Au dot array at 0° (left) and 180° (right). In this example, the circled dots are used for measurement.

7. Use the circle annotation tool to draw a circle around a pair of dots visible in both images and separated by a reasonably large distance along the diagonal. Measure the distance between the centers of the dots and use the following formula to calculate the current CCD rotation.

$$\text{CCD rotation offset} = \frac{\left|(y_1 - y_2) - (y_1' - y_2')\right|}{\left|x_1 - x_2\right| + \left|x_1' - x_2'\right|} \text{ [radians]}$$

8. If the CCD rotation offset value is more than 0.001 rad, then it needs to be corrected. Assuming that TXMConfigure has the main camera orientation set correctly:

   $(y_2' - y_1') < (y_2 - y_1) \Rightarrow$ rotate CCD counterclockwise

   $(y_2' - y_1') > (y_2 - y_1) \Rightarrow$ rotate CCD clockwise

   Rotate the CCD is the appropriate direction by loosening the four screws around the base of the CCD, adjusting the manual micrometers with a hex key, and then retightening the four screws. **1 rotation of the micrometers corresponds to approximately 0.005 rad.**

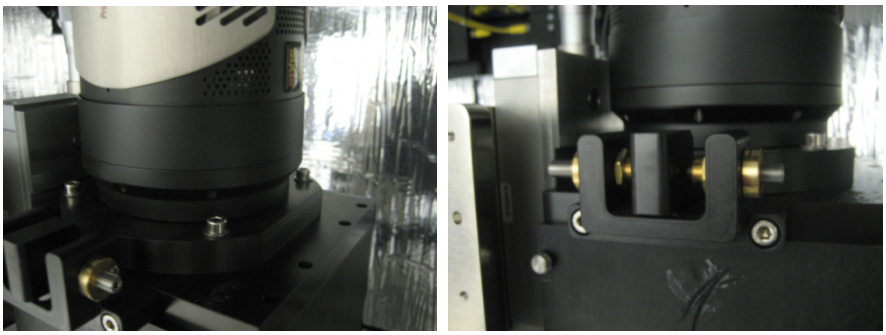


Fig. A-17: The four screws (left) around the base of the CCD must be loosened before rotation adjustment and then retightened after. The manual micrometers (right) work in a push-pull manner to rotate the CCD and are adjusted by using a small hex wrench.

9. Iterate steps 5-8 until the measured CCD rotation offset is less than 0.001 rad.

Appendix B: X-ray Generator Procedures

B.1 Powering down the x-ray generator

To bring the x-ray generator from a full operating state to a powered-off state, following the procedure outlined in this section. Please note that this procedure takes approximately 2 hours to fully complete.

1. Use the automated aging routine under the software source control advanced options to slowly age the source down to 20 kV x 10 mA.
   NOTE: If software source control is not available or not functioning properly, then use the buttons on the front of the x-ray generator panel. First, decrease the current to 10 mA in 5 mA increments, pausing for 1 minute between each step. Next, decrease the voltage to 20 kV in 5 kV increments also pausing for 1 minute between each step.

Fig. B-1: Relevant generator controls.

2. Allow the system to stabilize for 5 minutes at low power (20 kV x 10 mA).
3. Use the x-ray OFF button under the software source control advanced options to turn off x-rays. The red light on top of the enclosure should turn off along with the x-rays.
   NOTE: If software source control is not available, not functioning properly, or if the red light on top of the generator does not turn off, then use the button on the front of the x-ray generator panel. Locate the button labeled OFF under the "X-ray" strip and press it. Verify that the red light turns off as well.
4. Allow the system to sit in this state for 1 hour. While x-rays are not being produced, cooling water is still flowing to the anode allowing it to cool properly.
   CAUTION: Failure to follow this step may result in damage to the anode.
5. Use the vacuum stop button under the software source control advanced options to stop the vacuum pumps.
   NOTE: If software source control is not available or not functioning properly, then use the button on the front of the x-ray generator panel. Locate the button labeled STOP under the "vacuum" strip and press it. Verify that the roughing pump stops running.

Fig. B-2: Vacuum stop and power-off buttons.

6. Use the power OFF button under the software source control advanced options to turn off power to the x-ray generator control systems.
7. Allow the system to sit in this state for a minimum of 30 minutes.
   CAUTION: Failure to follow this step may result in damage to the turbo molecular pump (TMP).
8. Open the bottom access panel and located the "Main Power" strip. Press the button labeled OFF.

Fig. B-3: Main power off.

9. Open the rear access door and locate the circuit breaker. Rotate this to the OFF position and use a standard safety lock to secure it in place.

Fig. B-4: Circuit breaker

B.2 Powering up the x-ray generator

Taking the x-ray generator from its completely powered down state to its operating conditions is roughly the reverse of the previous procedure:

1. Rotate the circuit breaker on the rear of the generator to the ON position.
2. Press the main power ON button on the front of the generator and wait for 30 seconds.
3. Press the START button under the vacuum strip.
4. When the number on the STATE screen has decreased below 180 mV, press the power ON button in the software source control advanced options (or press the physical ON button under the "power" strip on the generator). Wait 30 seconds for the control system to stabilize.

5. Press the x-ray ON button in the software source control advanced options (or press the physical ON button under the x-ray strip on the generator).
6. Allow the system to settle until the value under the STATE screen has decreased back to below 180 mV. Depending on how long the system has been off, this may take anywhere from 10 minutes to 24 hours. If the value does not decrease below 180 mV in this time frame, please contact Xradia for support.
7. Use the automated aging up routine in the software source control advanced options to bring the source back up to full power. Alternatively, increase the voltage to 40 kV using the buttons on the front of the x-ray generator control panel in 5 kV steps pausing 5 minutes in between each step (or longer if the STATE does not drop below 180 mV in this time period), and then increase the current to 30 mA in 5 mA increments pausing 5 minutes in between each step (same condition as previous applies). If the system has been off for more than a couple of weeks or exposed to air for longer than a few hours, follow the procedure for "SHORT_TIME aging" in the Rigaku manual.
8. If the generator has been off for more than a few minutes, allow the source to settle for a minimum of 3 hours before beginning system alignment.

B.3 Filament service

Approximately every 3 months or 3000 x-ray hours, the x-ray source will require a minor anode/cathode service (filament change). This is usually indicated by x-rays turning on briefly and then abruptly turning off with an alarm on the x-ray generator.

Filaments are user-serviceable parts by following the procedure outlined in this section. If there are any questions that arise before, during, or after the service, please contact Xradia for support.

There are two components to the filament service procedure. The first involves changing the filament itself, and the second polishing the anode. Servicing the anode is not strictly necessary in order to bring the machine back to operation, however it is recommended in order to maintain maximum x-ray flux from the source.

CAUTION: Laboratory gloves must be worn while performing this procedure and changed as soon as they become significantly unclean. Any skin oils, if deposited inside the rotating head or on one of the sensitive components, could seriously damage the system leading to costly vacuum repairs. Nitrile is the preferred material, but latex or vinyl will work as well. One pair of gloves is provided in the Rigaku toolbox.

B.3.1: Changing the filament

1. Power down the x-ray generator by following the procedure in section B-1.
2. Loosen the vacuum bleed valve on the far side of the generator and allow the head assembly to vent for a few minutes.

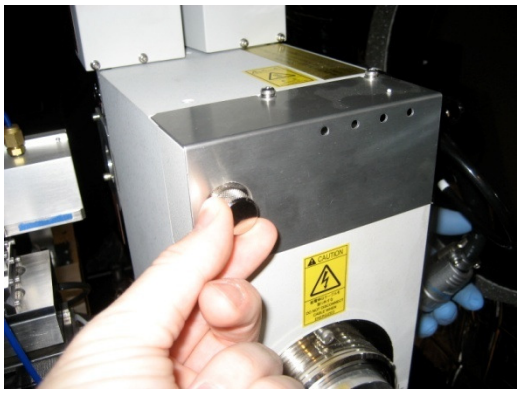


Fig. B-5: Vent the vacuum chamber with the screw on the far side of the head assembly.

3. Remove the six screws around the outside of the side filament access panel on the anode. Make sure to support the panel while removing the final two screws.

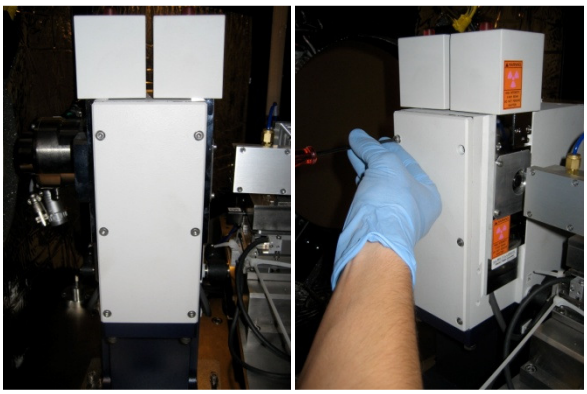


Fig. B-6: Gain access to the filament by removing the side panel.

4. Remove the panel and set it and the o-ring aside on a clean surface (clean room wipes work well for this purpose).

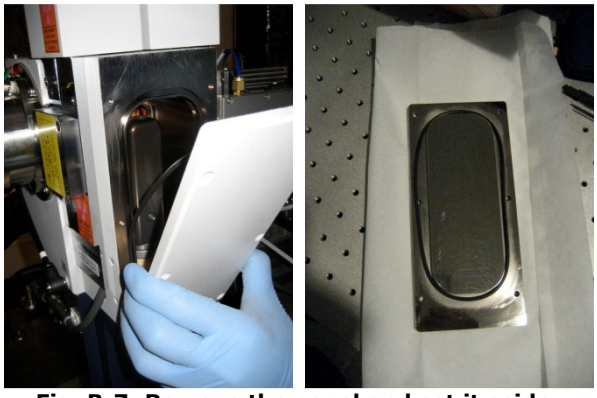


Fig. B-7: Remove the panel and set it aside.

5. Using a hex wrench cleaned with methanol, carefully unscrew the cover to the filament housing while supporting it with another hand, and set it aside on another clean surface. Note that the retaining screw will not come out of the housing cover.

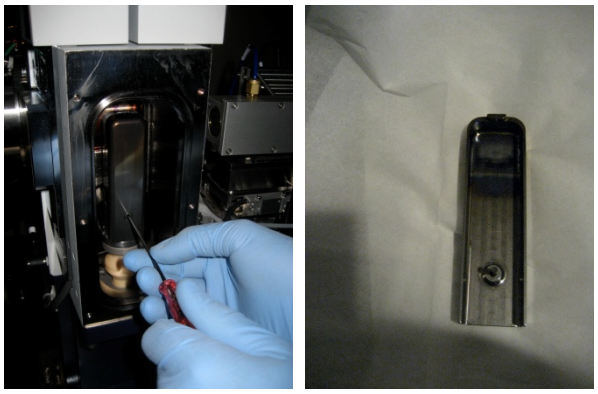


Fig. B-8: Remove the filament housing cover.

6. Using another hex wrench cleaned with methanol, carefully unscrew the two wire leads connecting the power supply to the filament. Allow them to dangle. Note that these screws also will not come out of the wire leads.

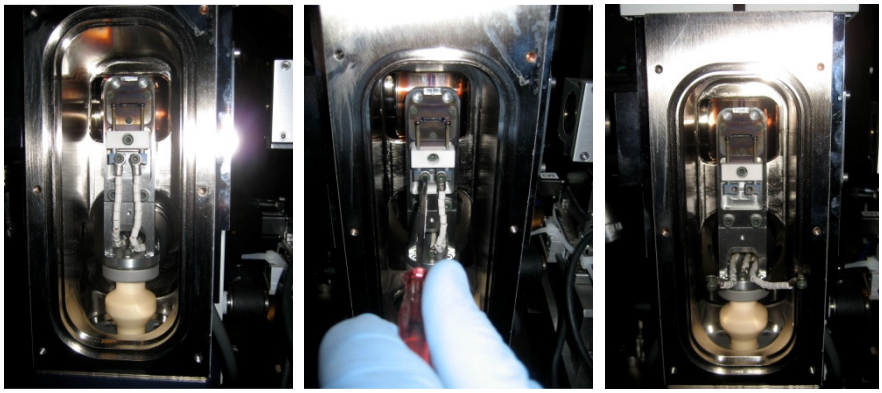


Fig. B-9: Disconnect the wire leads from the filament.

7. While supporting the filament housing with one hand, slowly begin to loosen the two screws at the bottom of the filament housing. The housing has slots at the bottom, so the screws need not be completely removed. Once it is free, slide it up and out of the housing, and place it on a clean surface.

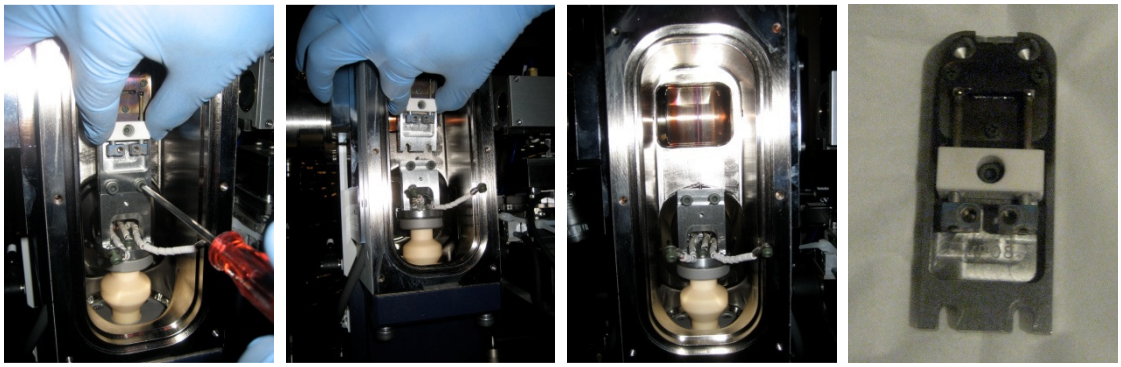


Fig. B-10: Remove the filament housing.

8. Remove the center screw on the filament and remove the filament from the housing. Set the filament aside. Be sure to save the screw because it will be necessary for installation of the new filament.

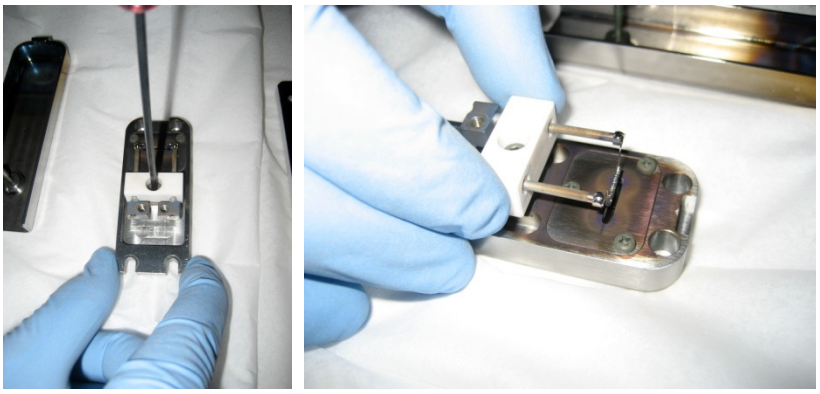


Fig. B-11: Remove the old filament.

9. Remove the three screws around the window of the housing and separate the components of the housing on the clean room wipe.

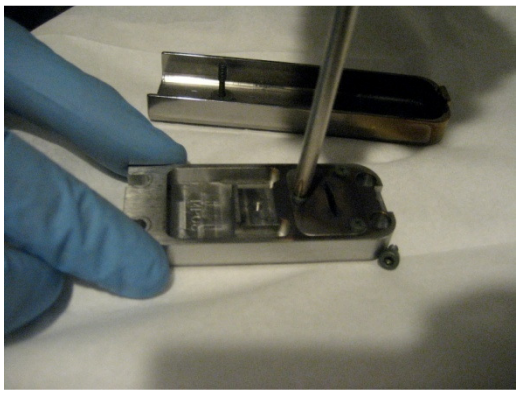


Fig. B-12: Disassemble the housing in preparation for cleaning.

10. Using fine-grade abrasive scrubbers (provided with the system in the Rigaku toolbox, e.g. fine grade Scotch-Brite pads) and methanol, clean the housing, window, and housing cover, paying close attention to the slot in the window. Alternate between moistening the part with methanol, scrubbing with the abrasive pad, and repeating. When the gloves become dirty, change them. Ocassionally, wipe the item with a lint-free delicate wipe (e.g., clean room wipe) and observe what comes off. When the parts wipe clean, stop scrubbing and put the window back on the housing – the parts are ready for reinstallation.

Fig. B-13: Clean the window as well as the rest of the housing.

11. Locate the new filament. Carefully remove it from the packaging and set the screw aside. Without touching the filament, place it in the appropriate groove of the housing so that it is well-centered in the window. Then, secure it in place using the original screw (removed with the old filament).

    CAUTION:

    1) Failure to properly center the device will result in both reduced x-ray flux and a reduced lifespan for the filament. Placing the window against a white background often helps to ensure adequate centering.
    2) Always use the old screw (slightly green in color) to secure the filament in place. The stainless steel screws that come with the new filaments are only to hold them in place during shipping and are not able to handle the high temperatures present inside the anode. Using the wrong screw may result in serious damage to the unit.

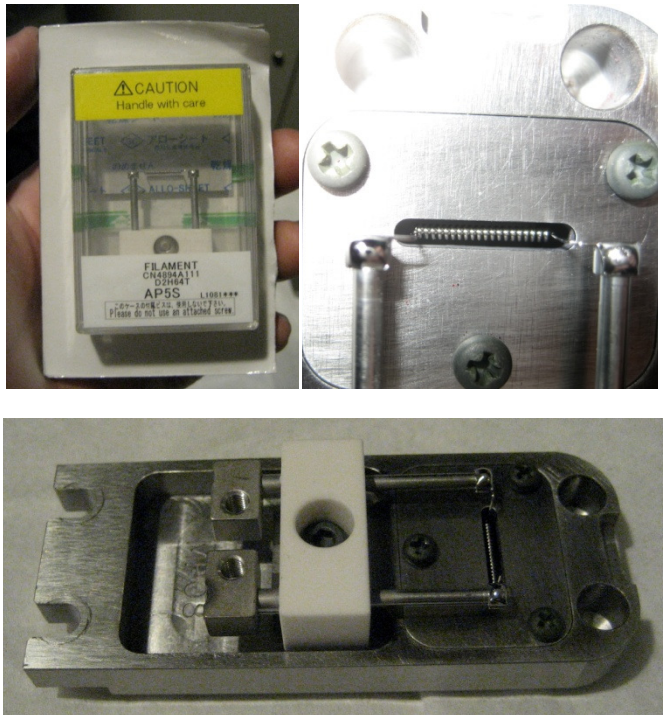


Fig. B-14: Install the new filament.

12. With clean gloves, carefully place the housing assembly back inside the head unit and secure it with the same two screws from which it was originally removed.

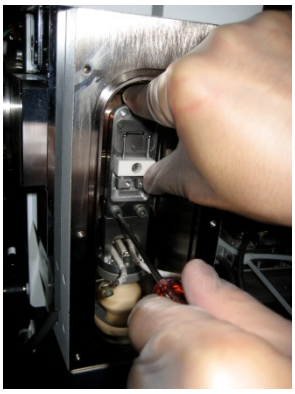
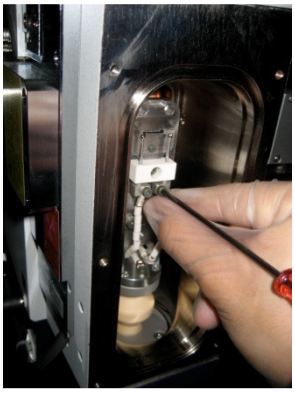


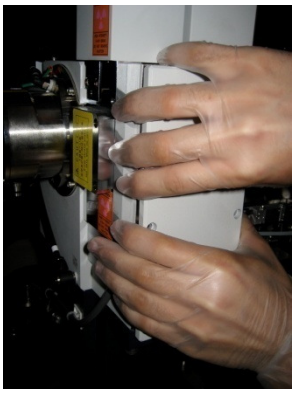
Fig. B-15: Reinstall the housing into the head assembly.

13. Connect the two wire leads to the new filament.



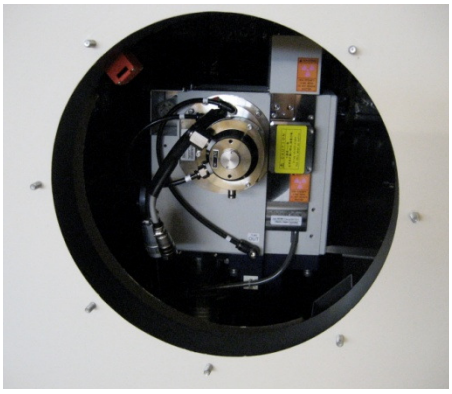
14. Reinstall the housing cover, putting just enough torque on the screw to hold it in place. This screw is easy to break, so do not over tighten. If any resistance is felt while installing it, stop, back the screw out, and run a standard M3 tap through the hole before trying again.

15. Reinstall the cathode chamber cover, again using just enough torque to keep it in place (the vacuum will do the rest of the work).

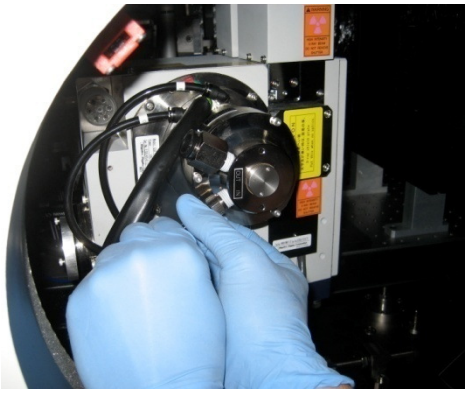


B.3.2: Polishing the anode

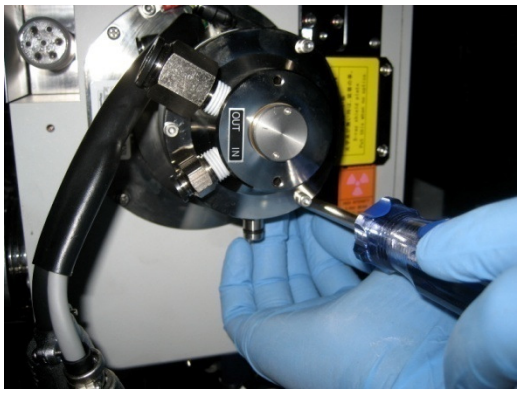
1. With the source and vacuum still off, remove the acorn nuts around the anode access panel. Before removing the final nuts, make sure to support the panel in case it should fall. When all nuts have been removed, hold the panel by its two handles and slowly pull it away from the enclosure. It is quite heavy, so be prepared to support about 10 kg of weight.



2. Take note of the position of all hoses going into the anode and then disconnect them. Have a small cup and towels nearby to catch any water that may leak out. Water hoses may be removed by pushing in on the outer collar of the connector and then pulling out the hose.

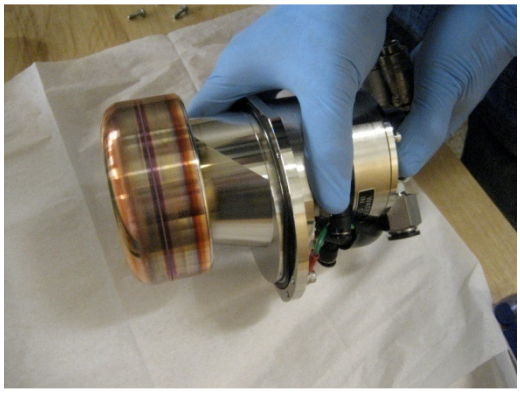


3. Using a Phillips-head screwdriver, remove the four screws around the edge of the anode while supporting it with a spare hand (or the help of a colleague).

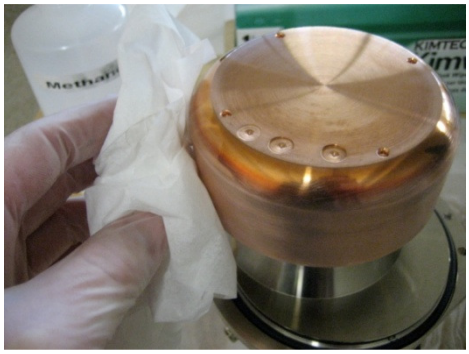


4. Pull the anode out until it stops. Then, slide it approximately 2 cm to the left and very gently pull it out of the assembly. The size of the anode is a very tight fit with respect to the generator but contact with the walls should be avoided.

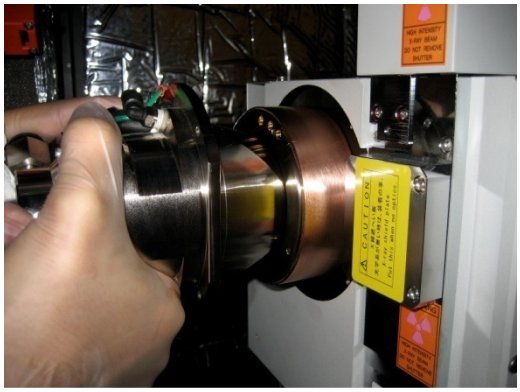
5. Place the anode on a clean, lint-free surface (e.g. clean room wipe).



6. Using methanol and abrasive pads, scrub the surface of the target until a clean copper surface has been revealed. Then, wipe it with the clean, lint-free wipes until they wipes come away with no discoloration or debris.

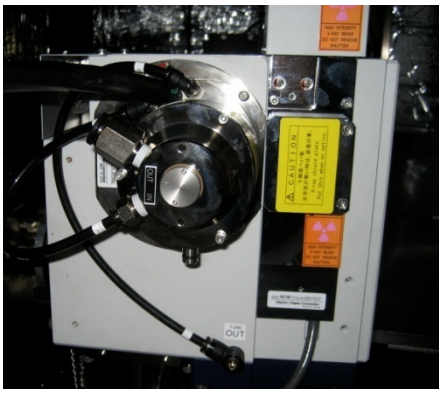


7. Carefully insert the polished target back into the head assembly with the flat portion facing to the left, avoiding any contact with the walls. Slide it in until it stops, then slide it over to the right until the outer mount is flush with the surface of the head assembly.

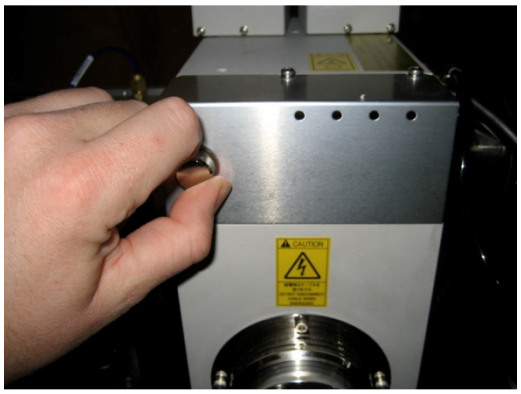


8. Secure the anode by reinstalling the four Phillips-head screws around its perimeter.

9. Reinsert the water hoses into their proper connectors.



10. Close the vacuum bleed valve.



11. Follow the procedure in the previous section for powering up the x-ray generator to bring the system back into its operational state. Allow at least 3 hours for the x-ray beam to stabilize on the fresh target surface, but be aware that the beam may continue to drift over the next few days (so frequent alignment checks will be necessary).

Appendix C: Technical Specifications

C.1 Facility requirements

1. Room size
   The room must be ~ 4 m x 5 m to house the nanoXCT, water chiller, computer workstation, pre-alignment microscope, and other items.

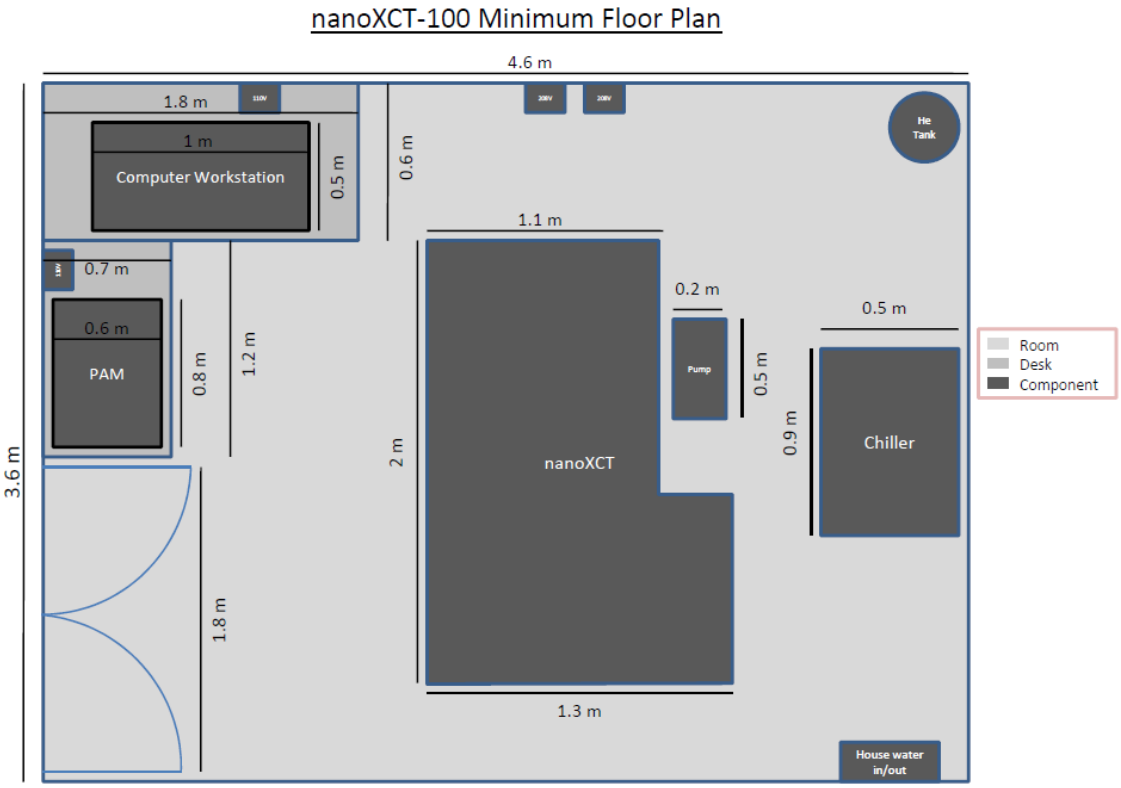


Fig. C-1: Minimum floor plan for NCT system.

2. Door entrance width and height
   The entrance width must be 1.6 m wide x 2.5 m high to allow passage of the nanoXCT unit.
3. Floor load capacity & vibration spectrum
   The floor must have a load capacity of at least 500 kg/m^2 and be free of sensible vibration. The figure below shows a typical floor vibration spectrum with < 100 nm integrated peak-to-peak amplitude.

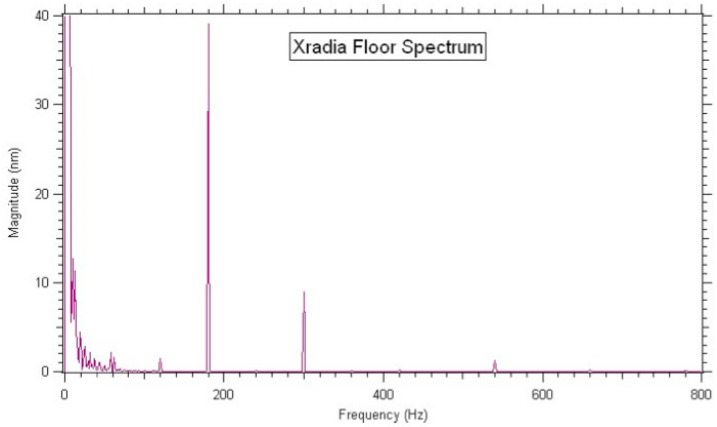


Fig. C-2: Floor spectrum as measured at Xradia laboratory.

4. Footprint of nanoXCT
   2.0 m length
   1.3 m depth
   2.0 m height
5. Water chiller
   A closed-loop water chiller is supplied by Xradia. The footprint is 0.5 x 0.9 x 1 m, and placement must be within 4 m from the generator. It is recommended to place the chiller in a separate room to reduce noise.
   The chiller requires 5 gallons of distilled water, which is kept in closed-loop circulation at ~ 1.1 gpm.
   The customer is expected to supply house water with 25-35 PSI pressure differential for heat rejection. Tap water may be used for this purpose with drainage to a sink.
6. Electricity
   a. nanoXCT unit
      200 VAC ±10%
      60 Hz
      20 A
      3 Φ
   b. Water chiller
      208/230 V
      60 Hz
      15 A
      1 Φ
7. Room temperature
   The room temperature should be kept between 65 to 75 °F (±1 °F) with humidity < 70%.
8. Other
   a. Electromagnetic compatibility
      There are no special requirements for electromagnetic compatibility.
   b. Helium gas
      An industrial-purity helium supply (bottle or central) is expected with a regulator set to <100 PSI.
   c. Desk space
      i. A computer desk of at least 30” width, 24” depth is expected for the computer workstation. Risers for the computer tower are strongly recommended.
      ii. A sturdy desk or laboratory bench with 1 x 1 m surface is expected, capable of supporting 200 lbs.

C.2 X-ray nano-tomography system

X-ray Energy [keV]	8.04 (Cu anode)
X-ray Source	Microfocus rotating anode
X-ray Condenser System	Reflective ellipsoidal mirror
High Resolution X-ray Objective Lens	Diffractive Fresnel zone plate
X-ray Detector Type	CCD sensor optically coupled to scintillating screen – 16 bit
Detector pixels	1024 x 1024
Large Field of View Optics (Standard)	150 nm spatial resolution (Rayleigh) 60 µm field of view 65 nm pixel size Absorption contrast Zernike phase contrast $(60\ \mu m)^3$ CT reconstruction volume 60 µm depth of focus 4 hr CT acquisition time for 5% contrast
High Resolution Optics (Optional)	50 nm spatial resolution (Rayleigh) 15 µm field of view 16 nm pixel size Absorption contrast Zernike phase contrast $(10\ \mu m)^3$ CT reconstruction volume 20 µm depth of focus 12 hr CT acquisition time for 5% contrast
Computed Tomography	Hardware accelerated parallel beam 1k x 1k x 1k reconstruction in 15 min
Working Distance [mm] (Distance Sample to Optics)	>10mm
Sample stage load capacity [kg] (Sample holder + sample)	2
Sample pre-alignment station	Optical microscope with 10x and 50x optical magnification, CCD camera, monitor
Computer	Two dual core Intel Xeon 2.66GHz processors 4GB Ram RAID5 HD array 146GB Microsoft Windows XP 30" flat panel display
Software	Xradia microscope control software Xradia hardware accelerated CT reconstruction software Xradia 3-D viewer and analysis software Microsoft Office Professional
Enclosure	Radiation safe 4-door enclosure with safety interlock and x-ray on indicator light
Weight	2600 kg
Warranty	12 months for full parts and labor, against manufacturing defects

C.3 Application-relevant specifications

X-ray Attenuation Length @ 8keV [µm] (Recommended max. sample thickness for solid sample with material indicated)	Carbon (C): 1000 H_2O: 1000 Silicon (Si): 70 Aluminum (Al): 80

	Iron (Fe): 5
Maximum recommended sample size [mm]	1 x 5 x 10
Space available for custom sample environment	The standard Xradia sample holder can be modified by the customer for additional sample manipulation (environment, stress/strain, temperature, etc.) Drawings are available upon request

C.4 X-ray source specifications

The Xradia nanoXCT x-ray microscope incorporates a proven, state-of-the-art, high-flux x-ray generator manufactured by Rigaku Corporation.

Manufacturer:	Rigaku
Type:	MicroMax™-007 HF Microfocus Rotating Anode
Focal Spot Size [µm]	70
Target Power [kW]	1.2 (Cu anode)
Tube Voltage [kV]	40 (Cu anode)
Anode Material	Cu
Characteristic X-ray Energy [keV]	8.04 (Cu anode)
Maintenance Intervals [months][1]	• Approx. every 3 months: Filament replacement • Approx. every 6 months: Anode inspection / polishing • Every 12 months: Refurbishment of Target anode
Extended Maintenance Options (after expiration of 1 year warranty)	• Included with Xradia extended maintenance package (recommended) • Directly from Rigaku USA
Consumables	X-ray source filament (2500 hrs life) Available directly from Rigaku USA (Part Number RAG-4894A111 for package of three) or from Xradia factory

[1]based on continuous usage of source

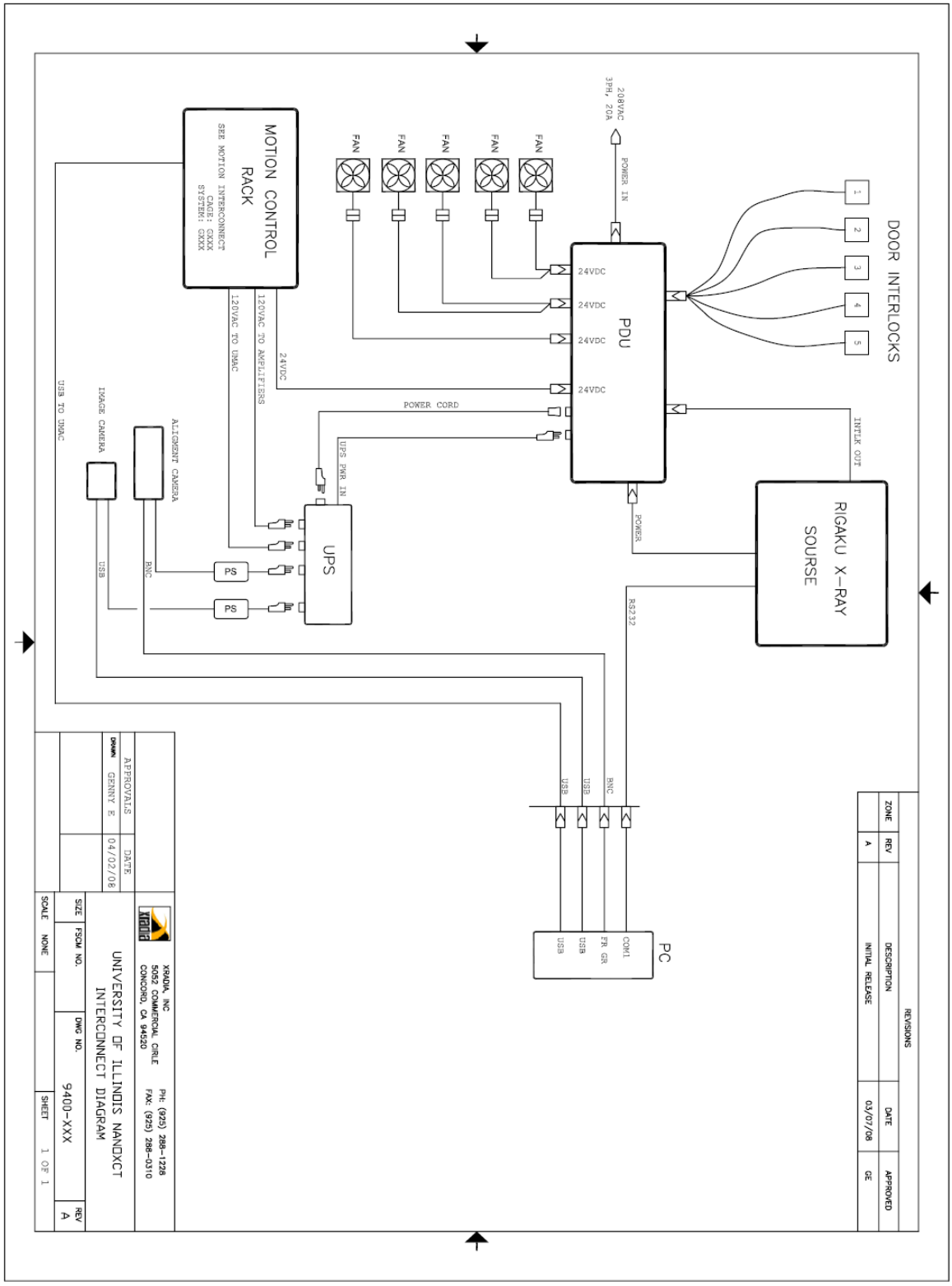
C. 5 Spare parts

Part No.	**Description**
9000050	Graphics card
9000079	Actuator
9000093	Copper anode, MicroMax-007HFM
9000131	Perc 5/E SAS RAID adapter battery
9000593	Gold powder spherical, APS 0.8-1.5 um 1g size
9000594	Gold powder spherical, APS 1.5-3.0 um 1g size
9000602	4 channel servo card, UMAC
9000603	Interpolation 1000, output frequency 1.8 MHz
9000680	Filament ST/3, AP5S point focus
9000685	De-I filter cartridge, NGDI-10
9000686	Nylon suction strainer
9000687	30- micron filter
9000869	Lamp, Eke, 150W, 21 V, halogen bulb
9000870	Battery, SR-44, silver oxide, 1.55V

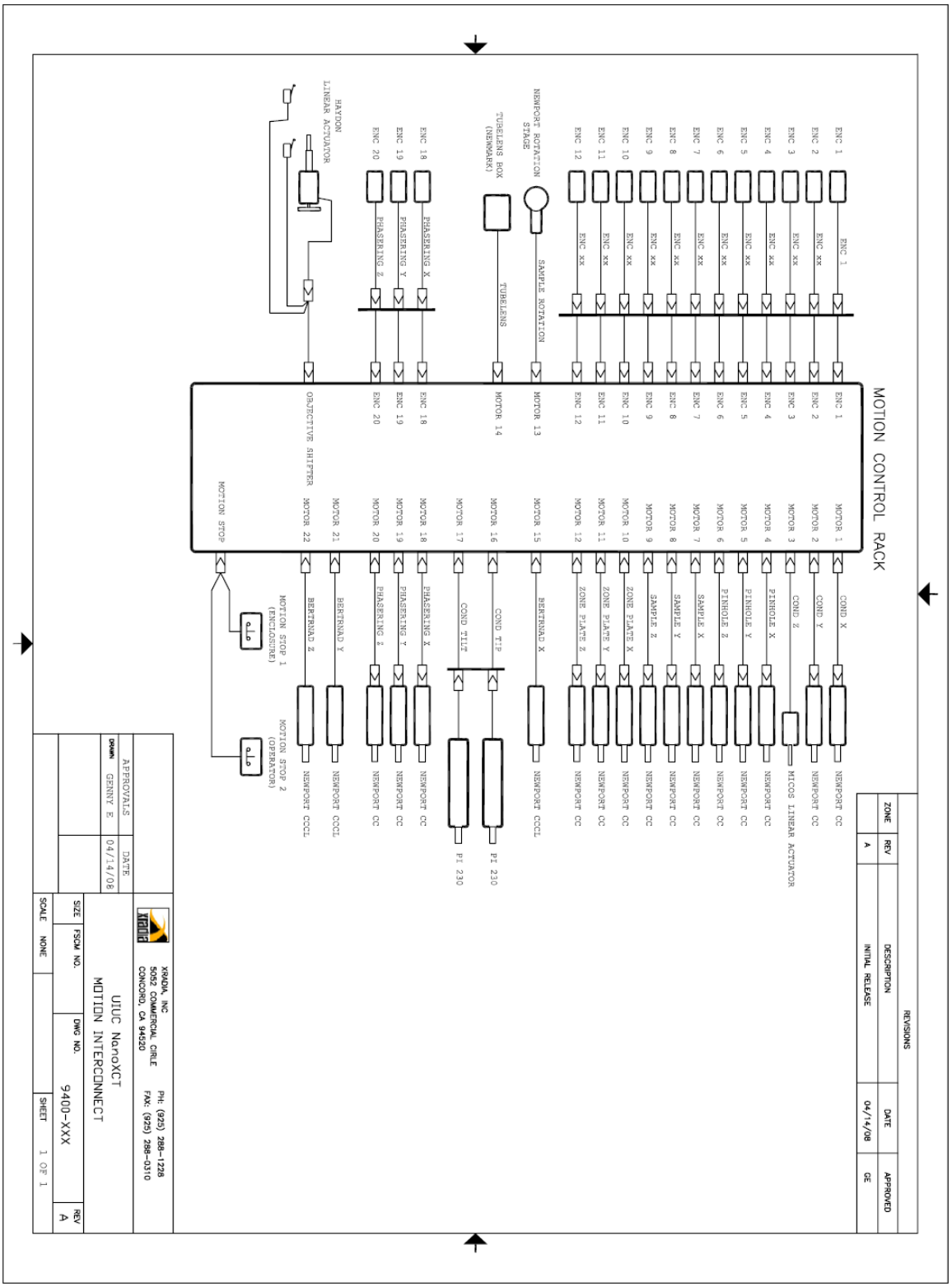
1005-064	Detector mirror mount
1005-310	Sample holder, notched, assy
1007-172	2X Nikon sleeve
1018-065	Zone plate holder
1018-080	Flexure hinge
1018-205	Scintillator + mirror ass.
4000-0052	Power supply for Pixis 1024B
9000-293	Vacuum pump, turbomolecular, 500 lps
9100-074	Adjustment screw
9100-137	Port window 2" dia
9100-138	Port o-ring
9100-146	Welded bellows
9100-294	20X objective, Nikon
9100-540	LIA encoder, 1m cable
9100-615	Mounting scale with adhesive
9100-722	.250 dia Be window buff both side's
9200-007	Rotation stage
9200-008	Micrometer not encoded
9200-156	Micrometer
9200-165	4 channel stepper encoder
9200-184	Digital CCD Pixis camera
9200-187	Turbo UMAC CPU board
9200-399	DC motor closed loop
9200-402	DC motor closed loop 12.5mm travel
9200-443	Dell, secondary workstation
9200-449	Stepper drive, 0-2A, 256 microstepping
9900-022	18-8 SS sealing pan head phil machine screw 6-32
9900-105	1/8 tube 1/8 NPT SS fitting
9900-113	Tungsten tip probe (box of 25)
A000067	Be window and flange assy
A000140	Pinhole assembly, 60 um
A000339	ZP2 + holder assembly labtool
A000341	ZP4 + holder assembly labtool
A000345	PR 1 assembly with holder
A000346	PR 2 assembly with holder
A000349	Bertrand lens 1 assem. Holder labtool
A000351	Bert. Lens assem. 2 holder labtool
A000370	ZP 1 assembly with tip tilt
A000371	ZP 3 LFOV with tip tilt
M000198	Condenser extension

Appendix D: Electrical Documentation

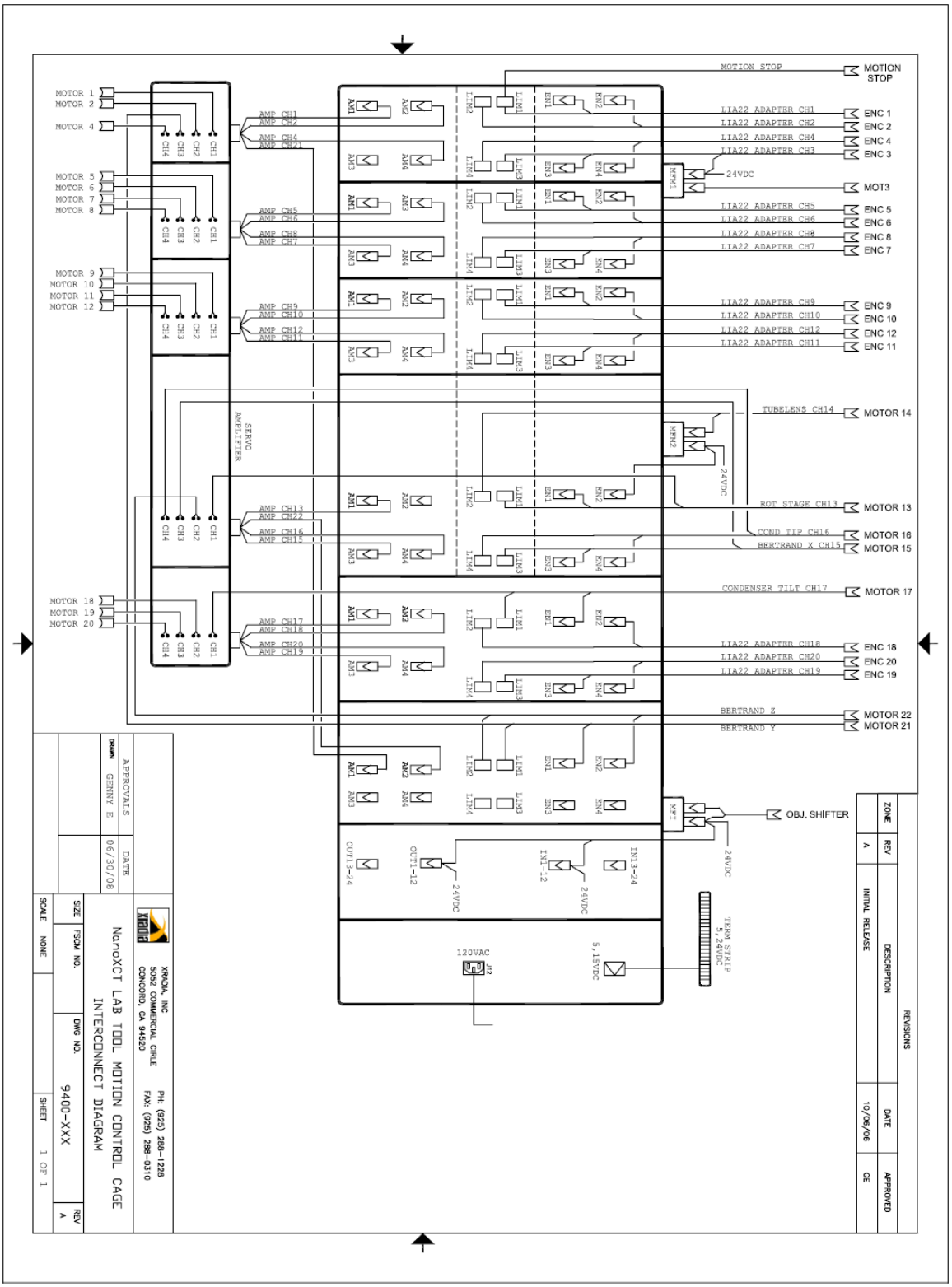
D. 1 Interconnect Diagram



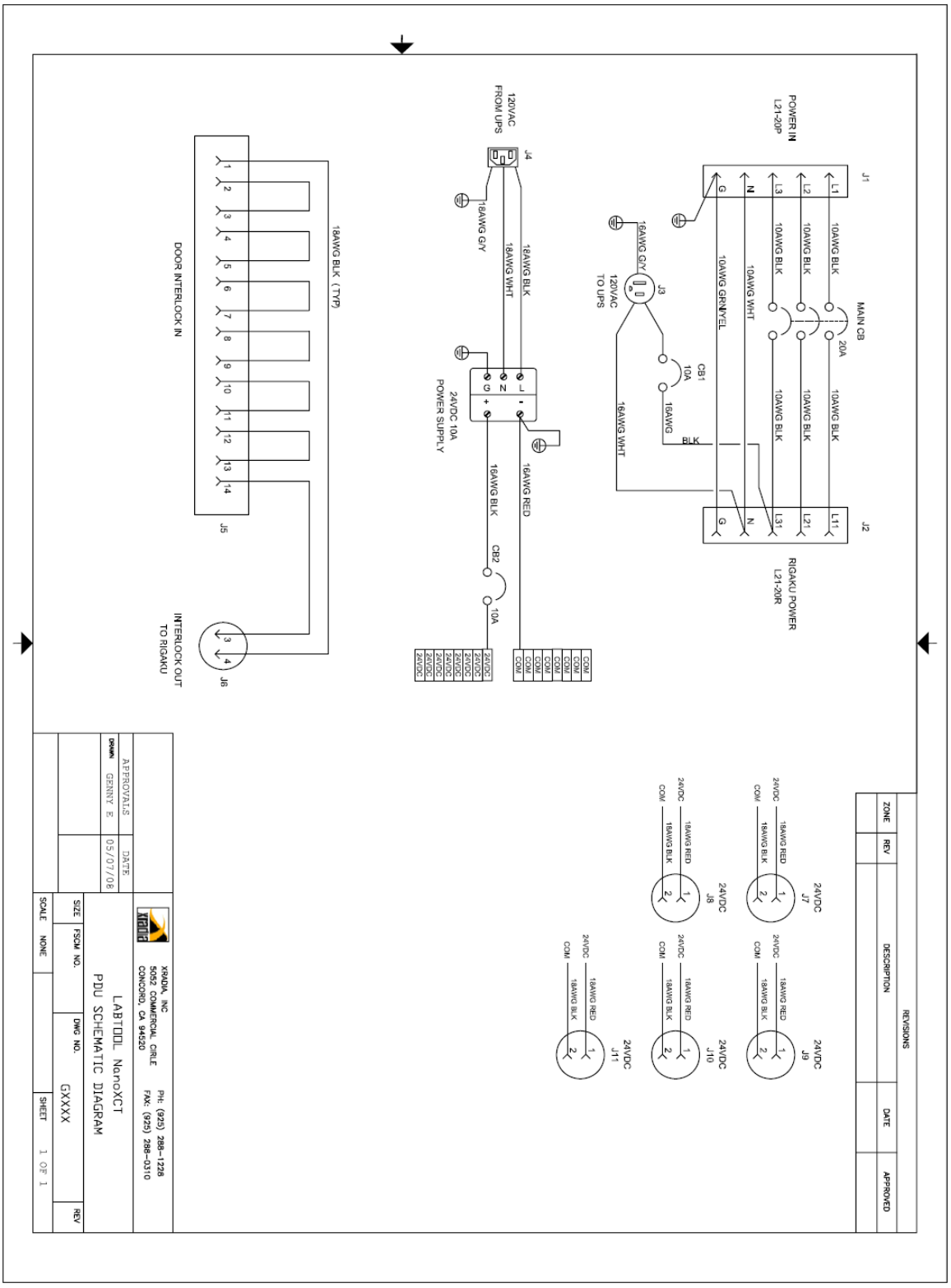
D. 2 Motion Control Rack



D. 3 Motion Control Interconnect



D. 4 Power Distribution Unit



Appendix E: Contact Xradia, Inc.

If assistance is needed on the nanoXCT instrument, please contact the Xradia support team using one of the following methods:

Internet

support@xradia.com

Telephone

+1 (925) 288-1228

Mail

Xradia, Inc.
5052 Commercial Cir.
Concord, CA 94520
USA