



*nanoPi*<sup>™</sup>

Argonne CNM Instrument  
User Manual

20 July 2007



## **User Manual for the Argonne Nanoprobe Instrument**

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## **1. Introduction**

The nanoPi is a combined scanning and full-field x-ray microscope system. In scanning mode, it employs a high-resolution Fresnel zone plate (ZP) optic to produce a fine x-ray probe. The probe is scanned relative to the specimen under control of a laser-interferometer system. At each scan pixel, fluorescence, transmission and other signals can be recorded for image acquisition.

In full-field imaging mode, a condenser optic and a zone plate are used to produce a magnified image of the specimen on a 2-D detector, with the options of Zernike phase contrast for improved contrast of weakly absorbing specimens, and tomographic data acquisition for 3-D imaging.

For more background information on the operational principles of the system, the reader is referred to the Argonne Preliminary Design Report or other related documents.



## **2. Potential hazards and safety precautions**

The nanoPi is designed to be safe for the operator, maintenance and service personnel. However, potential hazards to the operator and the instrument do exist. This section describes the potential hazards, such as ionizing radiation and high voltage, their isolation and control methods. Please read and understand this chapter and follow the instructions during operation and maintenance. Always exercise caution and use common sense. Severe or catastrophic damage to the operator, the equipment or the facility can result if the prescribed procedures are not followed.

### **2.1 Ionizing Radiation**

The nanoPi uses x-rays provided by the Advanced Photon Source (APS) for imaging. Prolonged exposure to x-ray radiation can cause moderate to severe illness to the personnel mostly in form of soft tissue damage or cancer in severe cases. The x-ray source and the associated radiation shielding and interlock systems are not provided by Xradia. Users must complete all training required by the APS before operating the nanoPi and must follow procedures established by the APS before opening any x-ray shutter.

### **2.2 Electric shock**

The nanoPi system requires standard 120 V supply voltages for operation. Maintenance of electrical systems is to be performed by qualified Xradia personnel only. The risk from electrical systems is low. Severe or catastrophic injuries from electric shock are unlikely and minor to moderate injuries are rare.

### **2.3 Lasers**

The nanoPi uses eight HeNe lasers for the interferometer-based positioning and vibration-control system. Each laser emits 1 mW of 633 nm light making it a Class 2 laser. No interlock is needed but care must be taken not to look directly into it.

There is high voltage in the laser head modules. When the cover of this module is removed, the operator is exposed to high voltage. Make sure that all cables are firmly connected to both modules before turning on the laser power switch.

### **2.4 Pinch Hazards**

The nanoPi uses motorized translation stages. Placing body parts in their path while motor power is enabled can cause bodily injury. Minor injuries may occur in rare occasions. Serious injuries are extremely unlikely. The risk from the pinch hazards is slight.

### **2.5 Collision of Moving Parts**

Motorized components may collide when they are moved out of their normal operating ranges. Damage will be minor in most cases. Severe damage will be rare. The risk from collision is low. Collisions may cause misalignment and in more severe cases, damage to sub-components. Use caution when moving motors. Avoid moving a motor position outside its normal operating range.



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## 2.6 Hazardous Materials

The nanoPi contains the following materials that may be hazardous. They are not accessible by the operator or personnel performing routine maintenance. Please be aware of their presence and use caution when decommissioning the instrument.

**Only qualified Xradia personnel may alter any of these components!**

**Beryllium** - A beryllium windows is used on the CCD flight tube. The total quantity of beryllium is approximately 4 g.

**Thallium doped Caesium Iodide** - Thallium doped Caesium Iodide is used as scintillator and is integrated into the detector system. The quantity is approximately 2.5 mg. The Thallium content is approximately 0.2% by weight. Xradia will replace this scintillator every 12 months during scheduled maintenance. Xradia will be responsible for its disposal.

## 2.7 Magnetic Field

The nanoPi contains a number of motors that generate low levels of magnetic field. Minor damage to personnel or equipment may occur, but this is extremely unlikely. The risk from magnetic fields is low.

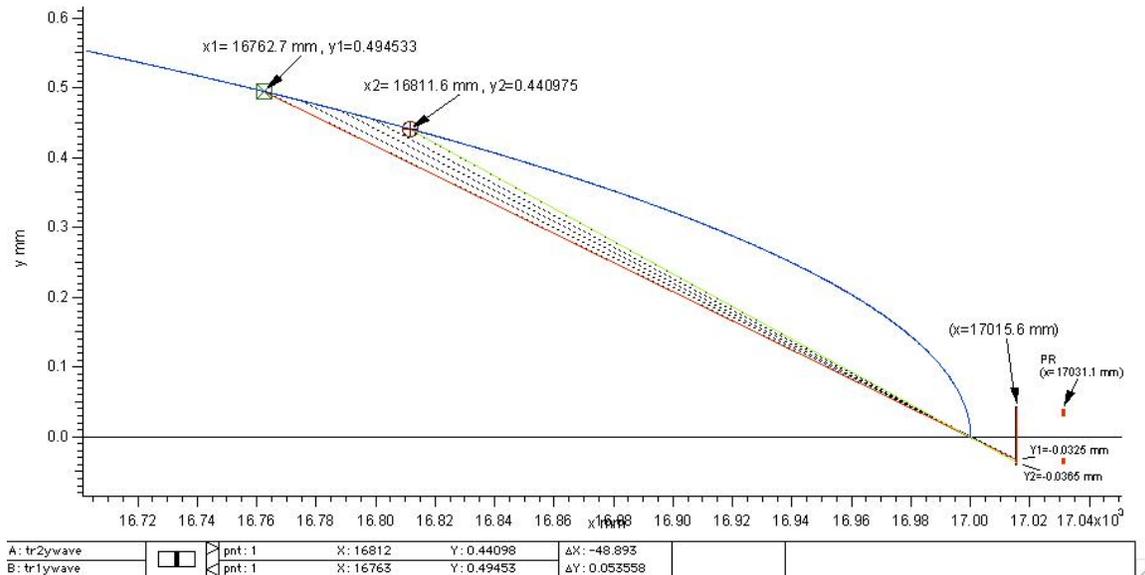
### 3. System specifications

#### 3.1 Full-field imaging mode

##### 3.1.1 Condenser

The nanoPi makes use of a capillary condenser with the following design parameters.

energy (keV)   10	ZP(D) um   80	PR(R) um   34.5	PR area/ZP area (%)   17.25
wavelength (Å)   1.23984	ZP (dF) nm   24	PR(I) um   4	PR material   Au
PR thick (um)   3.1586	PR phase (deg)   270	PR density   19.3	
ellipse semi-major axis (a) (mm)   17000	ZP Magnification   140	ZP focus (mm)   15.4858	ray1 angle (deg)   0.0601162
ellipse semi-minor axis (b) (mm)   2.97	Sample to ZP distance (mm)   15.5965	ZP NA (mrad)   2.583	ray1 mag   0.0070291
	ZP (#zones)   833.333	ray2 angle (deg)   0.0674174	ray2 mag   0.0055729
		Si orritical angle (deg)   0.178675	diverge ray1 (mrad)   0.0146422
			diverge ray2 (mrad)   0.0130364
<input type="button" value="Calculate"/>			



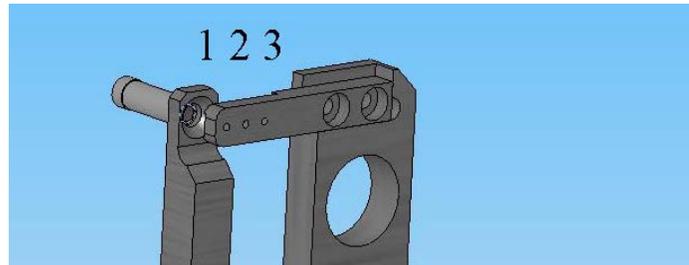
Type	Phase
Part #	CD-990-50-188
Serial #	CD_021407_2
Type	Ellipsoid
X-ray energy [keV]	8-10
Working distance [mm]	188
Length [mm]	50
Inner diameter – start [μm]	880
Inner diameter – end [μm]	900
Outer diameter [mm]	3.0
Central stop material	Au
Central stop diameter [μm]	600

### 3.1.2 Zone plate

The zone plate holder can accommodate one zone plate at a time. Please refer to the data sheet included with your zone plate for specific parameters.

### 3.1.3 Phase rings

The phase ring holder has space for three different phase rings to match the condenser illumination at 8-10 keV.



10 keV	Position	1
	ZP radial filling [ $\mu\text{m}$ ]	32.5-36.5 (17.25 % area filling)
	ZP to Sample distance [mm] (M=140)	15.6
	Optimum phase ring radius [ $\mu\text{m}$ ]	34.5
	Optimum phase ring width [ $\mu\text{m}$ ]	4.0
	Optimum phase ring thickness	3.2
9 keV	Position	2
	ZP radial filling [ $\mu\text{m}$ ]	29.3-32.8 (13.9 % area filling)
	ZP to Sample distance [mm] (M=140)	14.0
	Optimum phase ring radius [ $\mu\text{m}$ ]	31
	Optimum phase ring width [ $\mu\text{m}$ ]	3.6
	Optimum phase ring thickness	2.8
8 keV	Position	3
	ZP radial filling [ $\mu\text{m}$ ]	26.0.3-29.2 (11.0 % area filling)
	ZP to Sample distance [mm] (M=140)	12.5
	Optimum phase ring radius [ $\mu\text{m}$ ]	27.6
	Optimum phase ring width [ $\mu\text{m}$ ]	3.2
	Optimum phase ring thickness	2.5

### 3.1.4 Detector

In full-field imaging mode, the nanoPi uses scintillator screens with optical coupling to a CCD detector. Two objectives with an optical magnification of 2 and 20, respectively, can be interchanged manually. The specifications of the detector are listed in the following table:

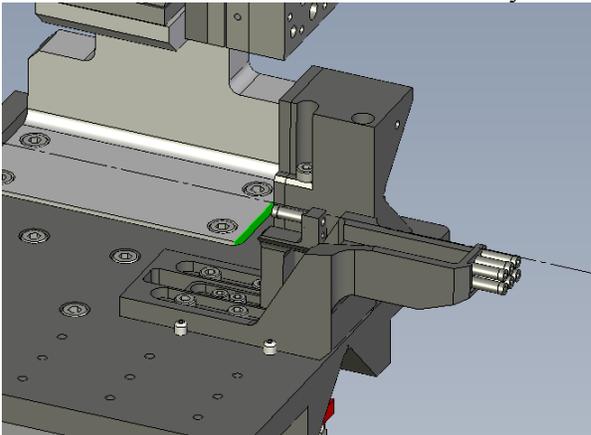
Slow-scan CCD camera	1Kx1K pixel array, back-illuminated, cooled to -70 deg
Pixel size on scintillator	0.65 $\mu\text{m}$ (20x objective) 6.5 $\mu\text{m}$ (2x objective)

X-ray imaging resolution	~1.3 $\mu\text{m}$ @8keV
Field of view on scintillator	0.65 mm
Quantum detection efficiency for complete system at 8 keV	> 30%
Electron yield in CCD per x-ray	~ 5 electrons per 10-keV x-ray photon
Readout noise (as function of ADC speed)	3.6 electrons per pixel @ 100kHz 9 electrons per pixel @ 2 MHz
CCD bit depth	16 bit
Readout rate at 1x1 binning 2x2 binning	10 sec per frame @ 100 kHz 0.58 sec per frame @ 2MHz 2.8 sec per frame @ 100 kHz 0.28 sec per frame @ 2MHz

## 3.2 Scanning mode

### 3.2.1 Zone plates

The holder for the scanning zone plates can accommodate up to six zone plates (see figure). Please refer to the data sheet included with your zone plate for zone plate specific parameters.



### 3.2.2 Detectors

Detectors specific for scanning mode are not included in the Xradia-provided nanoPi system.



## **4. Components**

The nanoPi system consists of the following components:

### **4.1 Vacuum chamber**

The vacuum chamber holds the specimen and all microscope components except the detector / camera assembly.

### **4.2 Translation stages**

The translation stages which hold the optical components and the specimen are grouped into various modules:

#### **4.2.1 Condenser module (CM)**

The condenser module provides space for up to three condensers. A high flux, low divergence (HFLD) zone plate with long focal length can also be accommodated. The module can be translated in X, Y and Z, and the condenser tip and tilt angles can be adjusted. Agitators can wobble the condenser to reduce coherence if desired.

#### **4.2.2 Focusing optics module (FOM)**

The focusing optics module provides space for up to six high resolution focusing zone plates as well as an additional high flux, low divergence zone plate with longer focal length. The module can be translated in X, Y and Z. The order sorting aperture (OSA) is mounted on a separate translation stage on top of the FOM with separate X, Y and Z adjustments. The OSA is also used as a filtering pinhole in full-field imaging mode.

#### **4.2.3 Sample module (SM)**

The sample module holds the specimen and can be translated in X, Y and Z. The sample holder can be exchanged for different experiments (rotation stage for tomography, cryo holder, etc.).

#### **4.2.4 Imaging optics module (IOM)**

The imaging optics module holds the zone plate optic for full-field imaging mode and can be translated in X, Y and Z. The phase ring holder (which can hold up to three phase rings) is mounted on a separate translation stage on top of the IOM with separate X, Y and Z adjustments.

#### **4.2.5 Detector / camera assembly**

The imaging detector is mounted on a long range Z translation stage to adjust the distance from the imaging optics. Manual adjustments are provided in the X and Y directions. A flight tube, which can be filled with Helium to reduce x-ray absorption, can be separately adjusted in the Z direction with two motorized translation stages mounted on top of each other.



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The flight tube translation stage can also hold an intermediate (Bertrand) lens for alignment of the phase rings, which has a separate motorized X and manual Y adjustment.

As part of the optical magnification system inside the camera assembly, the “tube lens” is mounted on a motorized translation stage to focus the CCD camera onto the scintillator screen. The tube lens focus has to be adjusted when switching between the two magnifications (2x and 20x).

### **4.3 Laser interferometer system**

The laser encoder system measures the relative position of the zone plate with respect to the sample. It is used for active vibration control and positioning. It consists of seven laser Doppler interferometers, an invar reference frame and the prism transport. The lasers provide position sensitivity better than 1 nm over a range of several mm. Two lasers are used to encode the Sample Module’s X and Y position. The other five are used to encode the Focusing Module as follows: two for X positioning, two for Y positioning and one for Z. This allows full characterization of position and angle for the zone plate. The feedback motion is executed by moving the FOM’s vertical and horizontal weaklinks. There is no angular correction.

The seven lasers are outside of the vacuum chamber and enter the chamber through thin viewports. The laser platform rests on the same platform as the vacuum chamber. However, the lasers are not directly attached to it. Instead, they are pre-aligned onto another plate that corrects each individual laser’s angular tilt. Every laser has to be tested and aligned after it arrives from factory. One additional aligned laser is provided.

Each laser emits 1 mW of 633 nm light making it a Class 2 laser. No interlock is needed but care must be taken not to look directly into it.

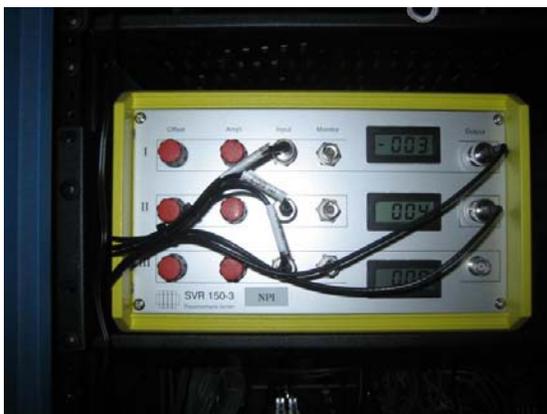
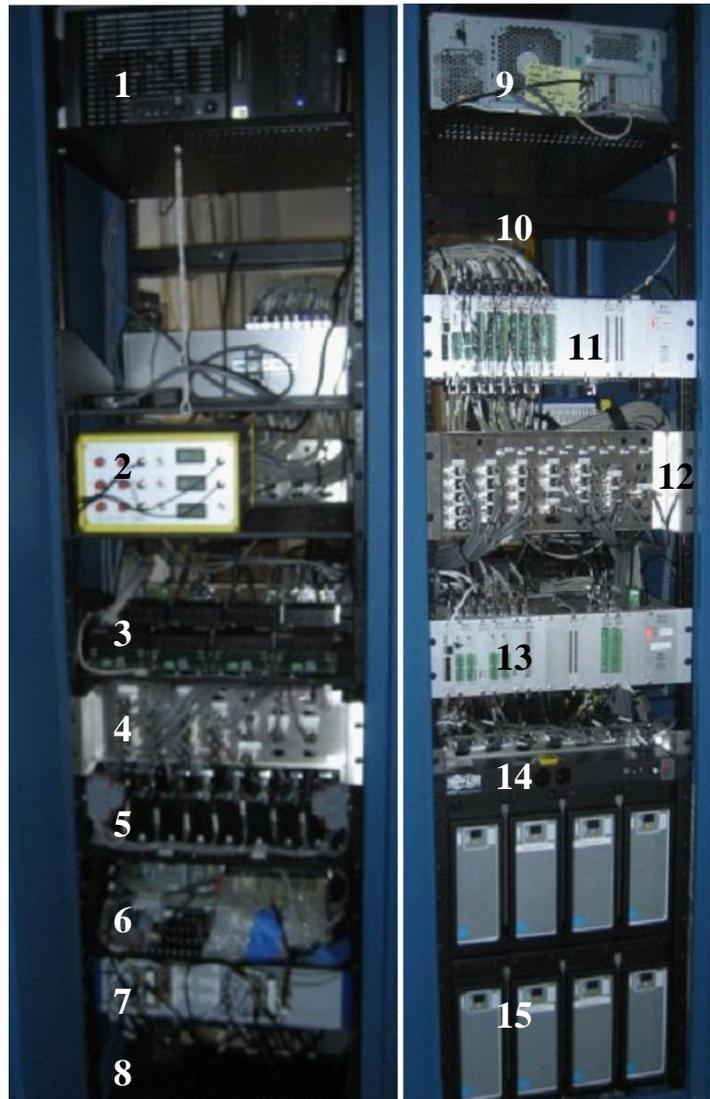
### **4.4 Computer Workstation**

A computer workstation interfaces with the data acquisition electronics (see below) and runs the nanoPi control software.

### **4.5 Electronics rack**

The electronics rack with all the devices installed is shown in the following figure.

1. Computer
2. Piezomechanik Amplifier
3. New Focus Amplifiers
4. Motor Connection Panel
5. E-DC & Nanomotion Amplifiers
6. E-DC & Nanomotion Switches
7. PI Controller
8. UPS battery backup
  
9. Computer back
10. Fan bank
11. Delta Tau 1 (PMAC 0)
12. Encoder Connection Panel
13. Delta Tau 2 (PMAC 1)
14. Tripp-lite Power Bar
15. Laser Rack (LDDM)



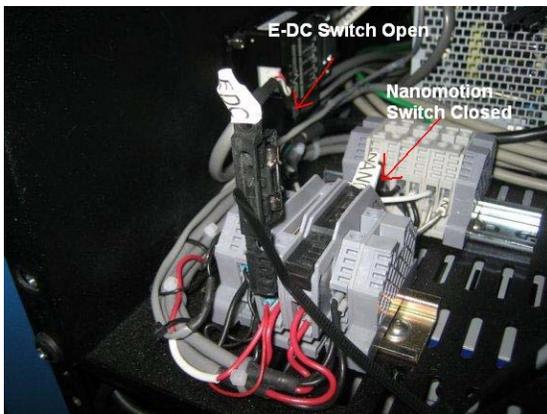
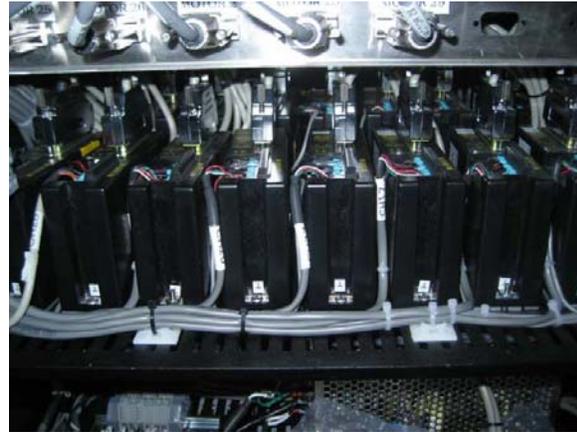
Piezomechanik – Amplifies Piezo input of 0-10V(0-32768) to 0-100V



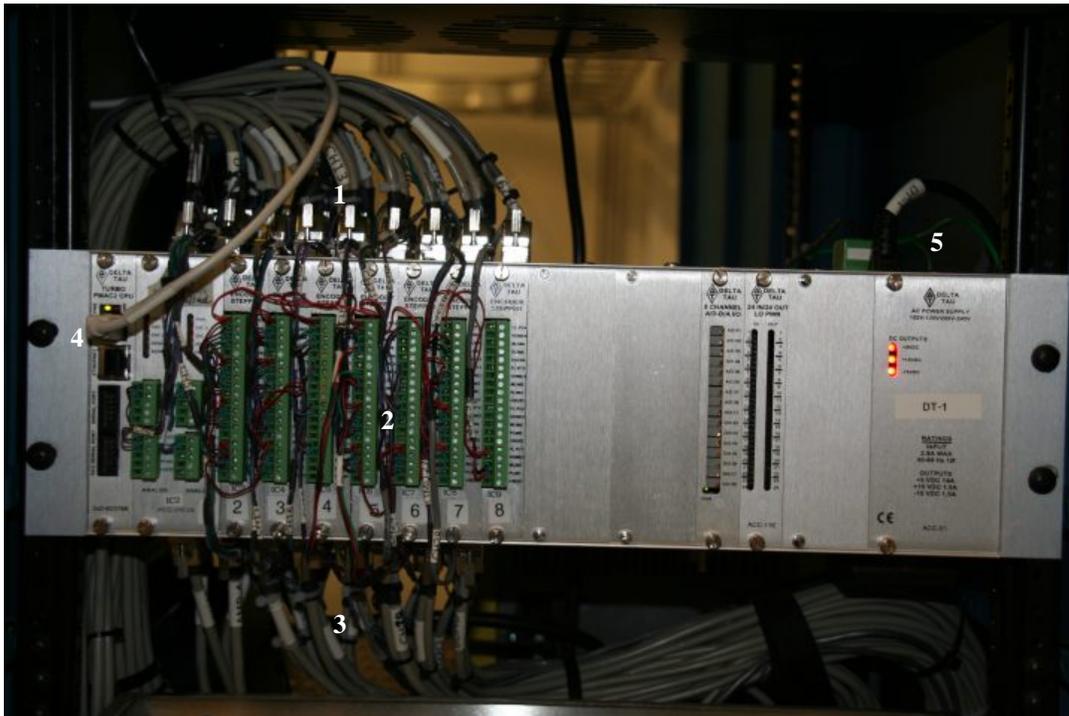
### New Focus Amplifiers

To enable or disable individual amplifiers push white power button.

### E-DC and Nanomotion Amplifiers

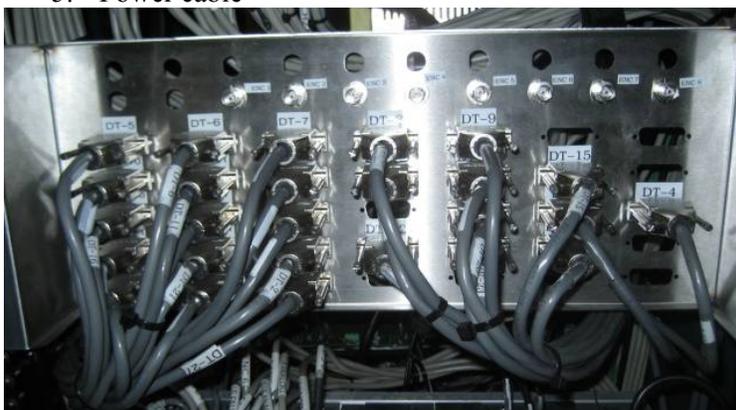


E-DC and Nanomotion switches. Lifting up on the switch removes the fuse from the circuit.

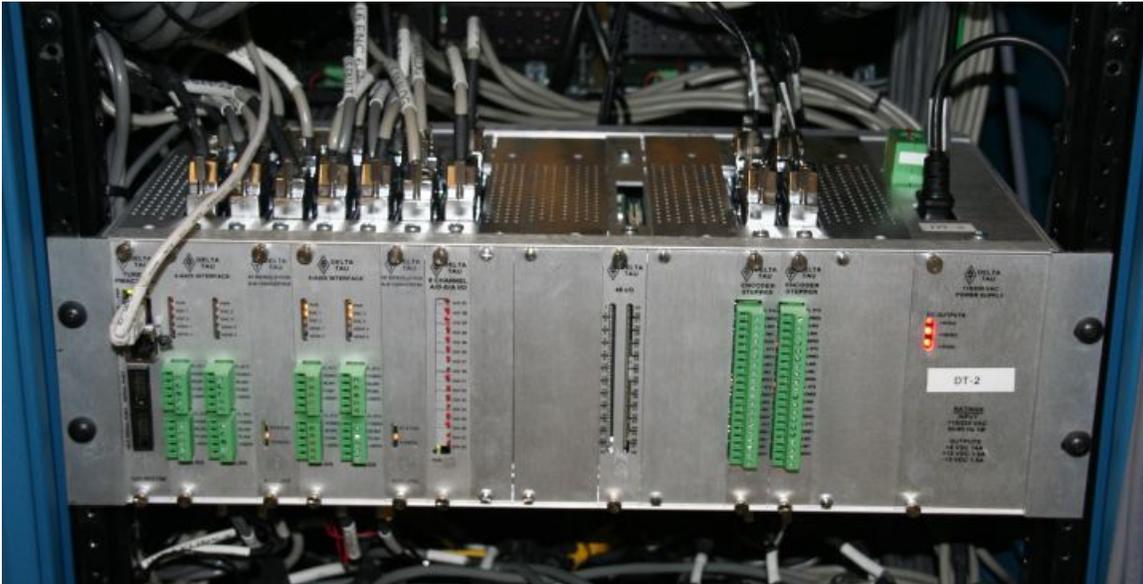


Delta Tau 1 (PMAC0) – controls output signals to stage motors and actuators

1. Encoder cables linked to Encoder connector panel
2. Limit switch and home mark connectors
  - a. Axis are identified by their IC and channel numbers
  - b. Numbering system is as follows IC-2:Ch 1, 2, 3, 4; IC-3:Ch 1, 2, 3, 4; etc. (i.e. Axis 9 would be IC-4: Ch 1)
3. Amplifier cables linked to Amplifier connector panel
4. USB to computer
5. Power cable



Encoder Connector Panel – Junction for encoder cables between motors and Delta Tau



Delta Tau 2 (PMAC1) – Controls LDDM system including Piezo actuators.

LDDM - Laser Doppler Displacement Meter

1. Power switch for lasers 1 (FOM Z) and 2 (FOM X1)
2. Power switch for lasers 3 (FOM Y1) and 4 (SAMP X)
3. Power switch for lasers 5 (FOM X2) and 6 (SAMP Y)
4. Power switch for lasers 7 (FOM Y2) and 8 (SPARE)
5. Main power for lasers





## **5. System power**

### **5.1 Shutting down the instrument**

Critical parts of the nanoPi electronics rack are connected to an uninterruptible power supply (UPS) and can stay powered up for about 5 minutes. If power is lost for longer times, the following shutdown sequence has to be performed.

- close all programs on the computer
- shutdown computer
- power off all laser power supplies
- power off PI controller
- power off ADE controller
- power off cooling fan
- power off TrippLite power bar (switch underneath plastic cover)
- power off UPS

### **5.2 Powering up the instrument**

To power up the instrument after a shutdown, please follow the procedure in Section 5.1 in reverse order. It is important to turn on the fan tray assembly to avoid overheating of the DeltaTau motion controllers.

## 6. Software Operation

The nanoPi system includes the TXMController program which controls motor motion and data acquisition. When the microscope is properly set up, all of its features may be controlled through TXMController. This application includes motion control, image acquisition, data analysis, and image manipulation functions. Many of these features will be described later, but, for now, we will focus on the motion controller functions and image acquisition. See the figure below for the main toolbar.



**Figure 1: Main toolbar of TXMController**

### 1. Open file



Selecting this option will launch a standard “Open file...” prompt, which allows the user to open any file with an XRM (single image) or TXRM (set of images) file extension.

### 2. Save file

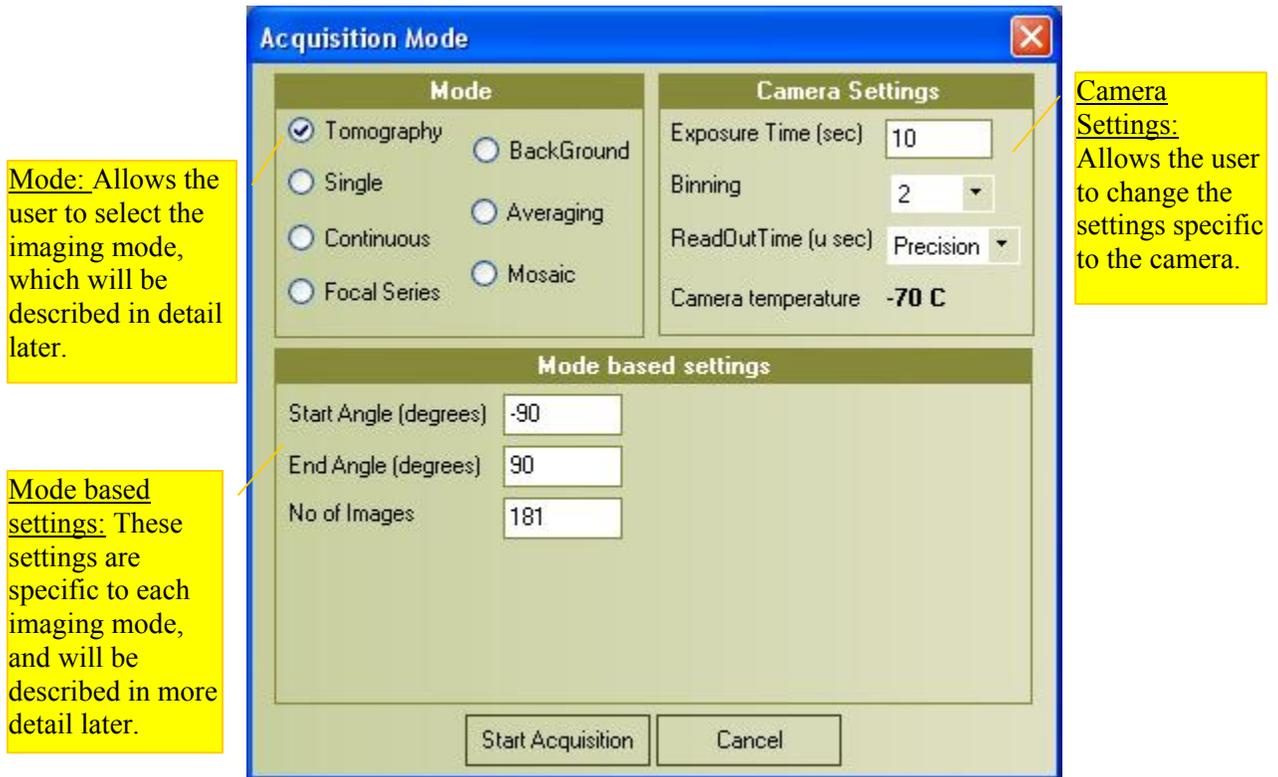


Selecting this option will save the current image to disk. If the image has already been saved, it will overwrite the previous file; otherwise, it will prompt the user for the location to use.

### 3. Change settings



Selecting this option will open the imaging settings window, as pictured below. It allows the user to select an imaging mode (e.g., Single, Averaging, Tomography, etc.) and set all of the necessary parameters contained therein.



Camera Settings: The main camera has 3 settings, which must be adjusted for optimal results:

1. Exposure time

Similar to any photo camera, this value sets the length of time for which the shutter should be opened during the acquisition of each image. A longer exposure time will naturally lead to a greater number of photon counts in the image, but will also lengthen the time of acquisition. It is also important to consider that the camera is **16-bit**, meaning that it will **saturate after 65,535 counts** (and potentially damage the CCD). So, exposure time must be chosen wisely to obtain an optimal image.

2. Binning

For situations in which either counts are low or resolution is not important, the software is equipped with the ability to *bin* the pixels on the CCD, essentially counting every  $2n$  pixels for every  $n$  (where  $2n$  is the binning number specified in the *Binning* field). This is a very useful feature, but the following considerations must be made:

- a) Changing the binning to a value higher than 1 will reduce the resolution of the camera by the same factor. At binning 1, the camera has a resolution of 1024 x 1024 px. If the binning is set to 2, the camera's resolution will change to 512 x 512 px, and the effective pixel size will increase by a factor of 2. Binning 2 can usually be used without significantly altering the resolution of the microscope, but a careful balance must be reached between resolution of the

camera and resolution of the zoneplate for effective imaging.

- b) The binning also has a drastic effect on the number of counts per effective pixel. By changing the binning by a factor of  $m$ , the number of counts per effective pixel will change by  $m^2$ . This can be very useful for low-light situations or quick exposures, but can also very easily lead to saturating the detector. When adjusting the binning, the exposure time should generally be adjusted as well to maintain a balance.

### 3. Readout mode

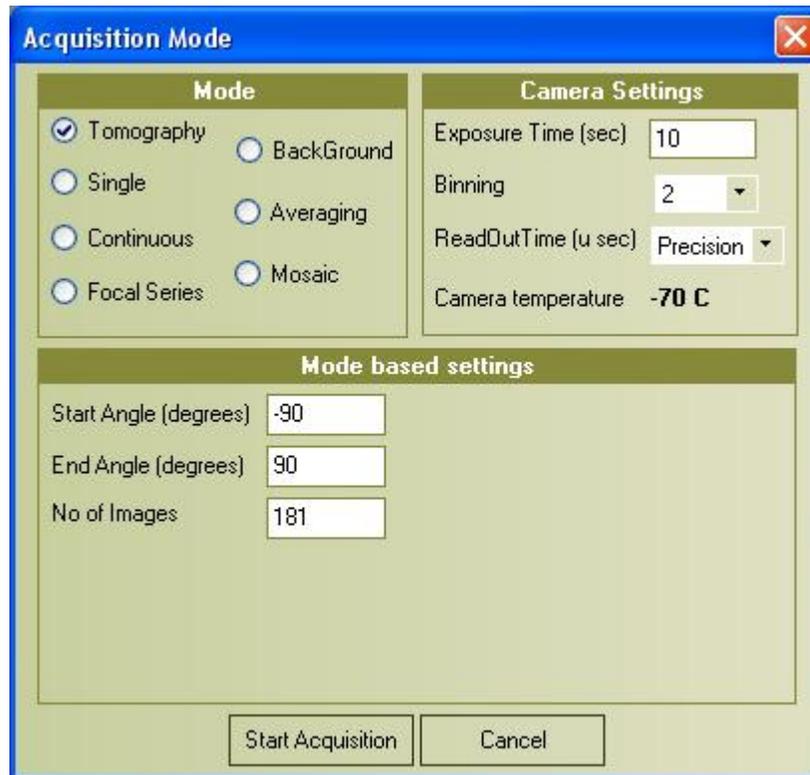
Once the acquisition process has completed, the camera must read out every row of charges on the CCD and transfer it to the computer. This process can be done in two modes, corresponding to different speeds: precision mode (100 Hz), and fast mode (2 MHz). With each of these *readout modes* comes some corresponding *readout time*. The tradeoff here is that, while precision mode takes considerably more time than fast mode, it also comes with slightly less noise in the resultant image. However, there are some cases where the increase in noise is not a problem, so it must be up to the user to decide which readout mode to use for each acquisition. More information on the specifics of each readout mode is listed in the camera specifications presented above.

In addition to the user parameters, this window also displays the current temperature of the camera. It is important to keep an eye on this number, as any changes in the camera temperature will impact the resulting images. **This temperature should always match the value set in TXMConfigure in order to achieve a quality result (typically -70° C).**

#### Mode & Mode based settings:

NanoXCT is capable of several different modes of acquisition, each with its own specific application. In what follows, a general survey of each setting and its associated parameters is presented, though the application of each setting is up to the user to decide.

#### 1. Tomography



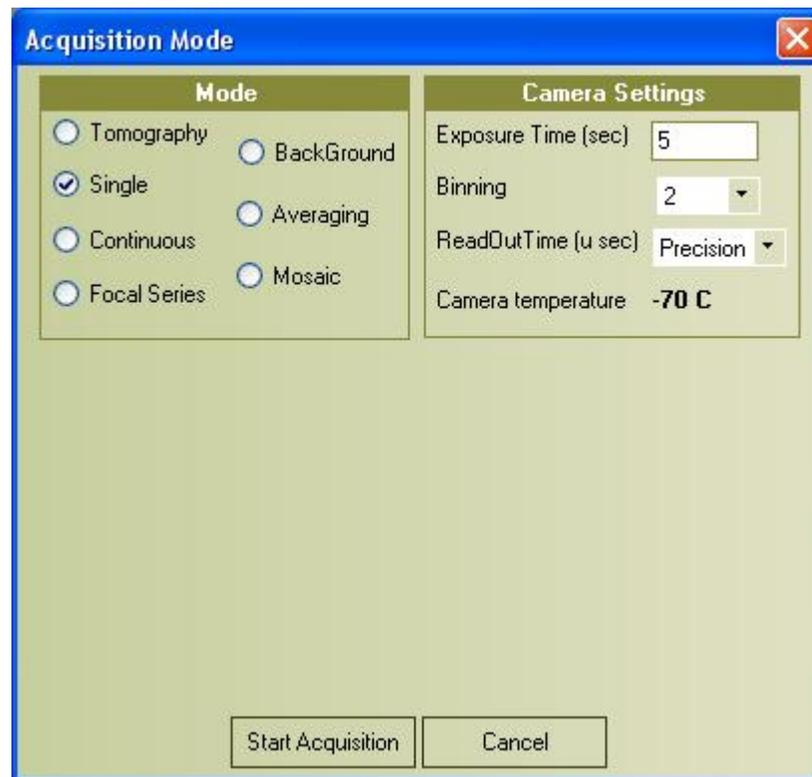
In tomography mode, the system will scan the sample through a specified angular range, stopping at discrete intervals to acquire an image. The following settings are available:

- a) Start Angle (degrees)  
This setting specifies the angle with respect to zero, in degrees, at which the software will acquire its first image.
- b) End Angle (degrees)  
This setting specifies the angle with respect to zero, in degrees, at which the software will acquire its first image.
- c) No of Images  
This setting allows the user to specify the total number of images that should be acquired during the tomography set.

The size of the angular steps is automatically calculated as below:

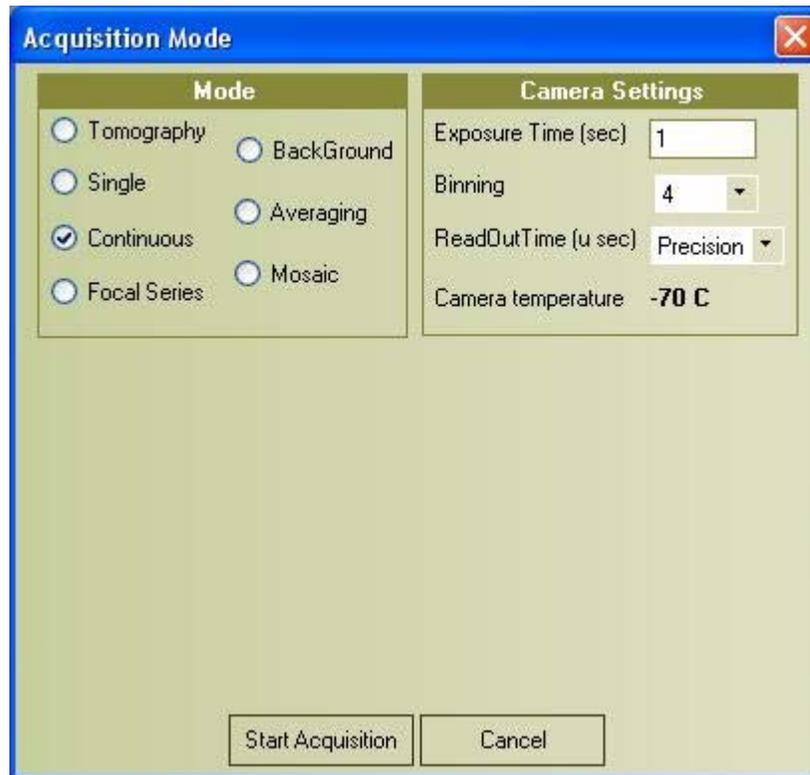
$$\theta_{step} = \frac{\theta_{end} - \theta_{start}}{n - 1} \text{ (where } n = \text{number of images)}$$

## 2. Single



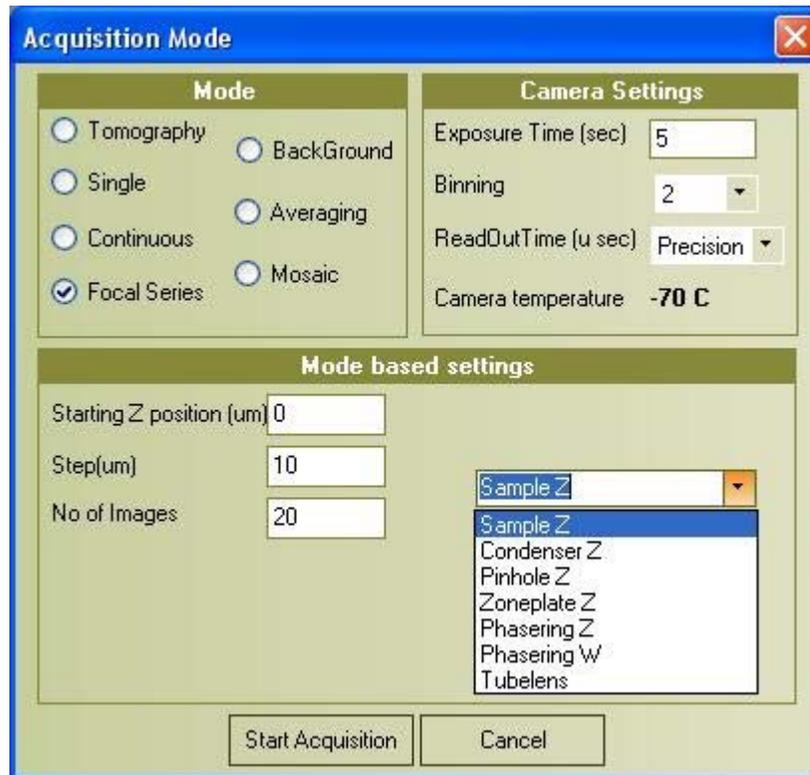
In single mode, only one image is taken, according to the camera settings. The image is stored in memory, and displayed on the screen for the user to manipulate. There are no mode based settings specific to single mode.

3. Continuous



In continuous mode, images are acquired and displayed on the screen continuously. Every new image overwrites the previous one, however, so it is important to stop acquisition as soon as an image arrives that is worthy of keeping. There are no mode based settings specific to continuous mode.

#### 4. Focal Series



In focal series mode, the selected axis is moved along the z-axis, stopping at discrete intervals for image acquisition. The result is a multi-image stack, which can be played as a movie or viewed as individual images one-by-one. It is a very useful mode for focusing the sample or one of the x-ray optics. The mode based settings are outlined below.

a) Starting Z position (um)

This sets the position, in microns, at which the first image should be taken.

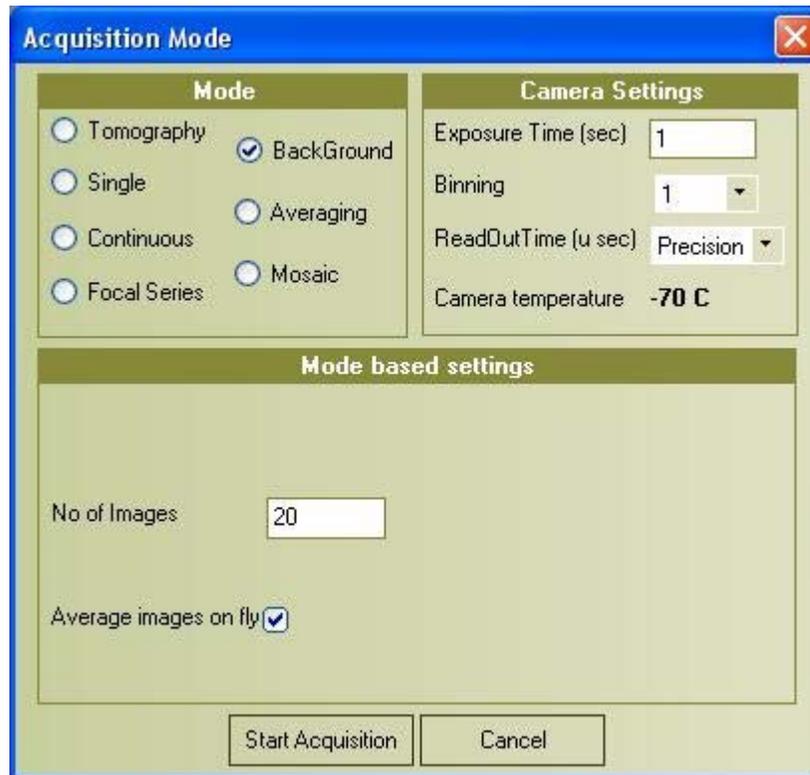
b) Step(um)

This sets the step size, in microns, of the discrete intervals at which images are acquired.

c) No of Images

This sets the total number of images that will be acquired during the focal series.

5. Background



The CCD used in the nanoPi detector has a certain amount of background charge, which will have an effect on the final number of counts in the form of background noise. To eliminate this as a problem, the software automatically adjusts each image by a background image (i.e., image taken with the shutter closed at the same binning and readout mode). These background images are usually collected and stored by Xradia personnel during installation, but, if something changes on the camera (e.g., temperature, camera orientation) or one of the files becomes corrupt, it may be necessary to recollect the background image. In that case, the background imaging mode should be used to collect the necessary image(s). This mode comes with two mode based settings, which are outlined below.

a) No of Images

This specifies the number of images to collect for the background. In the end, the images will be averaged to give a general idea of the background, but a greater number of images naturally improves the statistics of the result.

b) Average images on fly

With this option checked, the images will be averaged as they are read in. For background collection, it is not usually necessary to uncheck this box.

6. Averaging

7. Mosaic

4. Acquire single image



Selecting this option will instantaneously acquire a single image, using the current settings for “Single” in the Settings window. This acquisition may be performed at any time, including in the middle of another acquisition.

5. Acquire images continuously



Selecting this option will immediately begin continuous image acquisition, using the current settings for “Continuous” in the Settings window. The image screen will be continuously updated with a new image, overwriting the previous image with every new acquisition.

6. Stop acquisition



Selecting this option will immediately close the shutter and abort the current acquisition. If continuous imaging is currently in progress, then the most recent image will remain on screen (stored in memory).

7. Motion controller

8. Image calculator

9. Image tools and information

10. Adjust contrast

## 7. Alignment

### 7.1 Initial alignment procedure

#### 7.1.1 Alignment of chamber

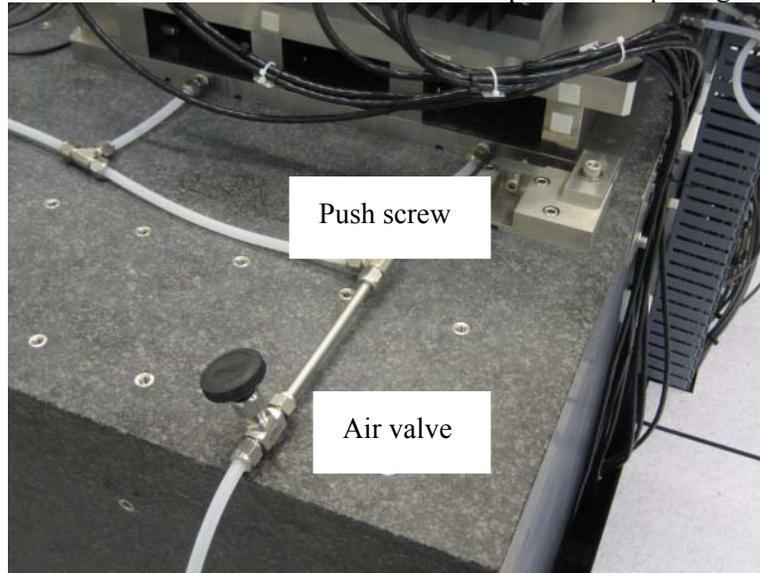


**Figure 2: The telescope used for alignment**

This assumes that there is no beam pipe installed between the hutch beam entrance and the chamber.

1. Make sure the beam is in the nominal position (this has to be determined by the APS beamline personnel)
2. Set all nanoprobe motors to their nominal position (center of travel range).
3. Align the telescope (see Figure 2) to the x-ray beam.
  - a. Attach burn paper to the beam exit and several points of the chamber (e.g., beam entrance and ZP mount) to get reference points. **With the telescope out of the beam path**, open the x-ray shutter to create burn marks on the paper.
  - b. Set the telescope to the center between exit window and chamber (make sure it is far enough from each that it can focus).
  - c. Focus the telescope on one burn mark, turn it by 180 degrees and check the location of the opposite burn mark. Adjust the telescope position and iterate the procedure until the telescope is aligned with the beam.

4. Float the table by opening the pressure valve to the air bearings (see Figure 3).
5. Move the chamber using the push-screws (see Figure 3). You can use the parallels (gauge blocks) to pre-set certain distances. Make sure all optical components are on axis.
6. [Could also check that Be window for camera flight tube is on axis, but should coordinate that with camera alignment section]
7. Doublecheck with another set of burns that all components are prealigned properly.



**Figure 3: Air bearing valve and chamber push-screws.**

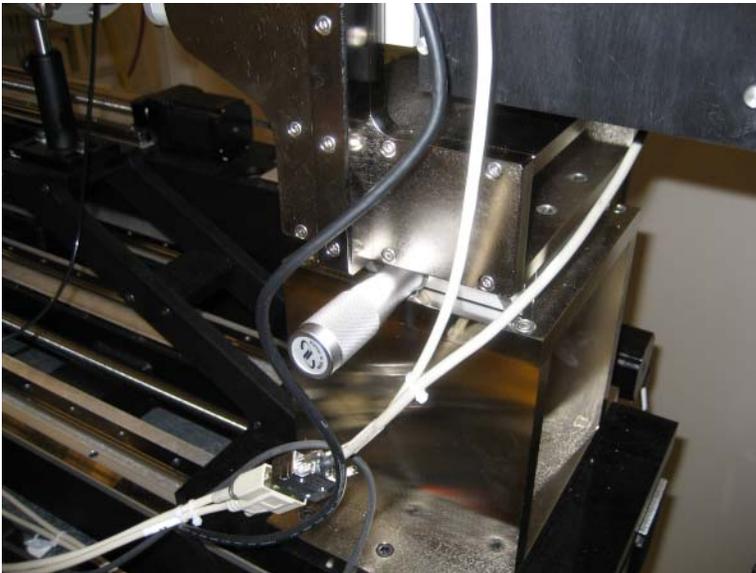
### 7.1.2 Alignment of full-field detector

Note: The field of view is 6.35 mm for the 2x objective and 0.635 mm for the 20x objective. The CCD pixel size is 13.5 microns.

1. Get the camera coarsely aligned. Use the telescope and/or burn paper to get beam onto the 2x scintillator (you can put burn paper directly onto the objective; show a figure of the camera box with cover open?)
2. Make sure the camera image is set such that the image displayed on the screen has the same orientation as if you were looking along the beam axis in the upstream direction. This can be set in TXMConfigure and can be checked by moving the camera *or* the beam and observing the effect on the screen.
3. Move the camera using the X/Y micrometer screws to center the beam on the 2x objective (see Figure 5 and Figure 4).
  - Camera X: Turning micrometer CCW (towards positive readings) moves the CCD outboard
  - Camera Y: Turning micrometer CW (towards negative readings) moves the camera up.
4. Now insert the 20x objective. The beam should be within the field of view. Move the camera again to center beam on the 20x (this will slightly off-center it on the 2x objective).



**Figure 4: The micrometer screw for camera Y adjustment.**



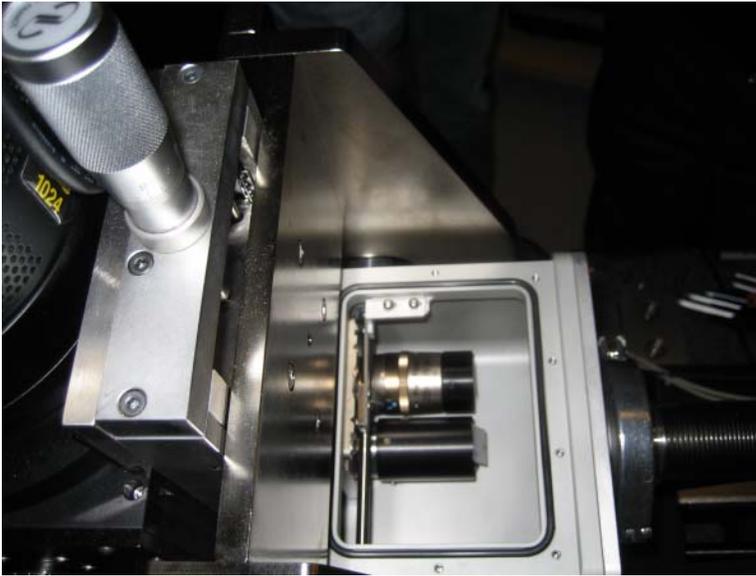
**Figure 5: The micrometer screw for camera X adjustment**

To focus the camera onto the scintillator screens, use the following procedure for each of the objective lenses (2x and 20x). Note that the focal position will be different for each of the objectives, so that the tubelens has to be moved each time the objective is switched.

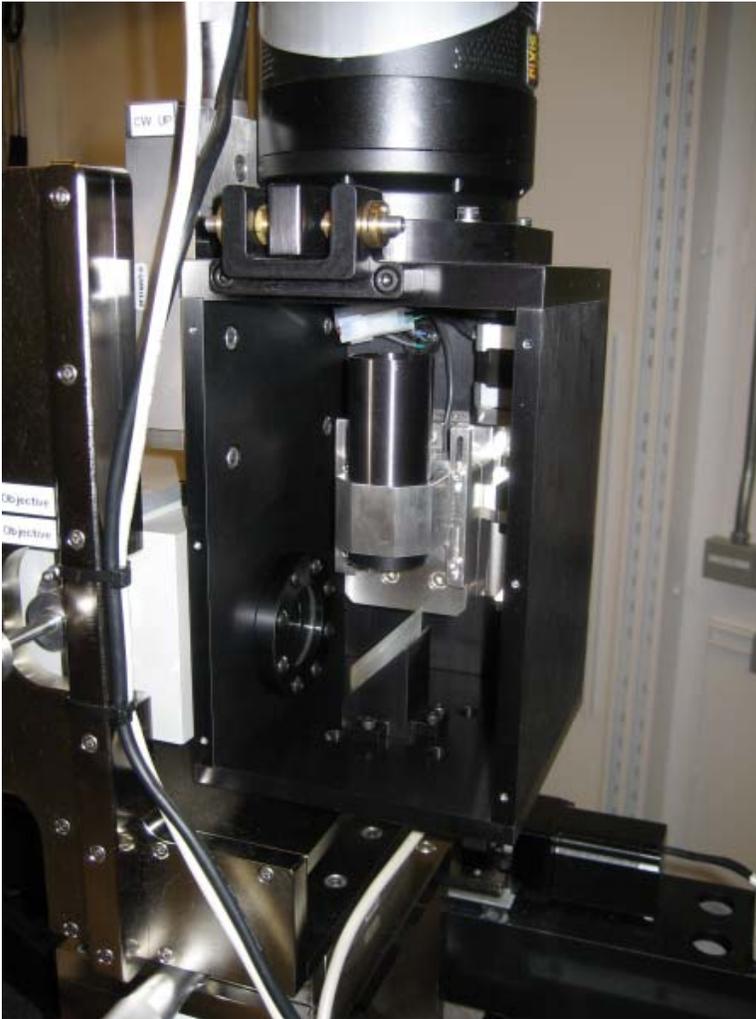
1. Stick a gold grid (e.g., with a 25 micron pitch) to a piece of Scotch tape. It makes sense to wrap the tape over to keep the sticky surface small (see Figure 6). Prepare one such grid for each of the objectives.
2. Open the camera housing to access the two visible light objectives and tape the grids in front of the scintillators (see Figure 7). [Note: We should soon have caps to hold the grids which can be slid over the objectives]
3. During the following procedures, place the lid loosely onto the objective housing to keep visible light out. You might also want to turn off the hutch light when turning on x-rays.
4. With x-rays on the scintillator, move the tubelens motor to see if you can get close to focus.
5. If the focal position is beyond the travel range of the tubelens, you have to manually move it on its stage inside the camera housing (see Figure 8). If this still doesn't provide enough range, you can move the scintillator on its sleeve with respect to the 2x objective lens. To do this, it might make sense to unscrew and remove the lens from the housing (see Figure 9). For the 20x lens, the scintillator is permanently attached to the objective and cannot be moved.
6. Once you are close to focus, take a focal series (moving the tubelens) to determine the best focal position. Again, the focal position will be different for each of the objectives. Figure 10 and Figure 11 show images of both objectives focused properly onto their scintillators.
7. Don't forget to remove the gold grids and close the camera housing properly.



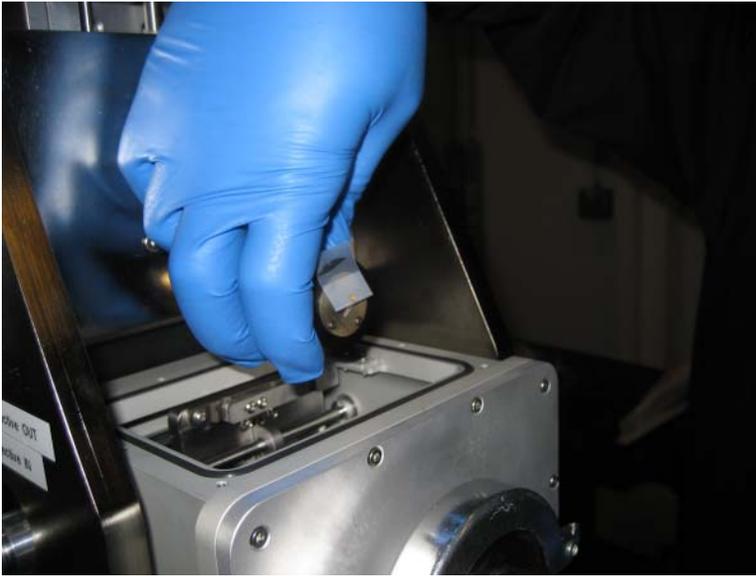
**Figure 6: Gold grid for scintillator focusing.**



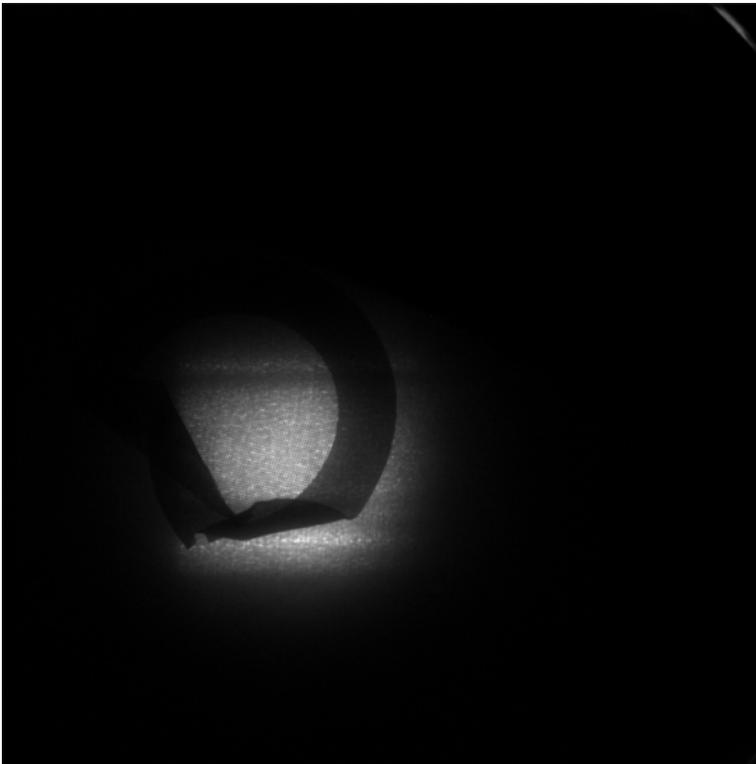
**Figure 7: Camera housing with visible light objectives exposed.**



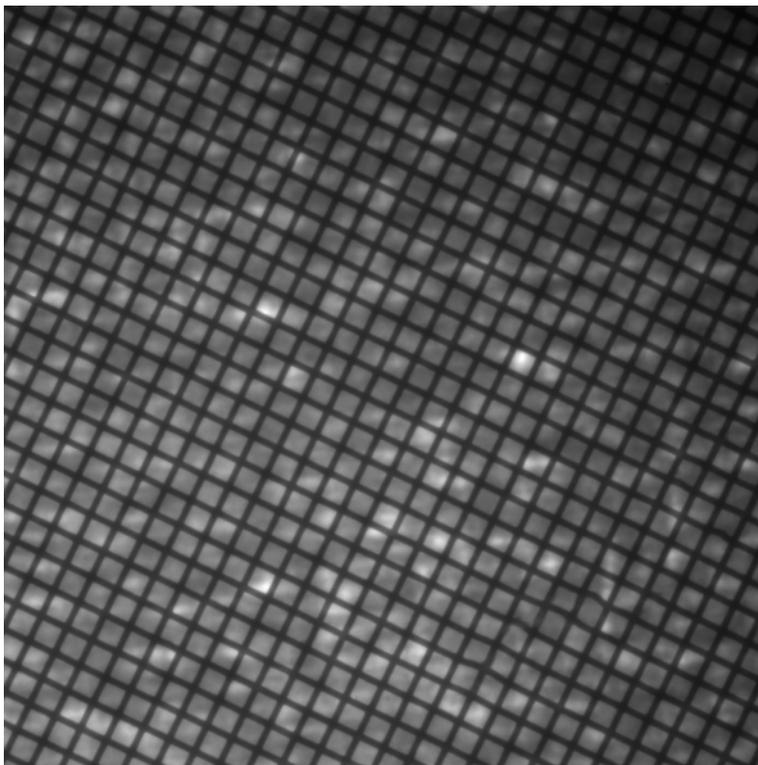
**Figure 8: Camera housing with tubelens exposed.**



**Figure 9: Removing the 2x objective lens from the camera housing.**



**Figure 10: CCD camera focused on the 2x scintillator, which has a gold grid mounted in front of it.**

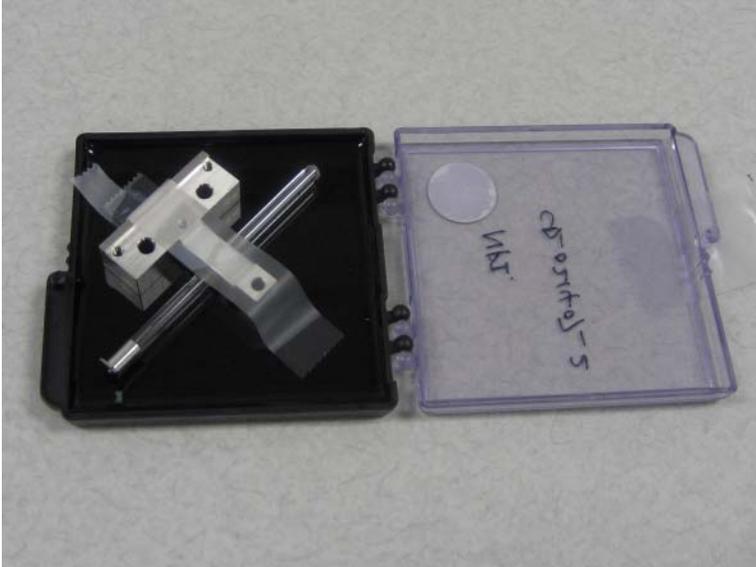


**Figure 11: CCD camera focused on the 20x scintillator, which has a gold grid mounted in front of it.**

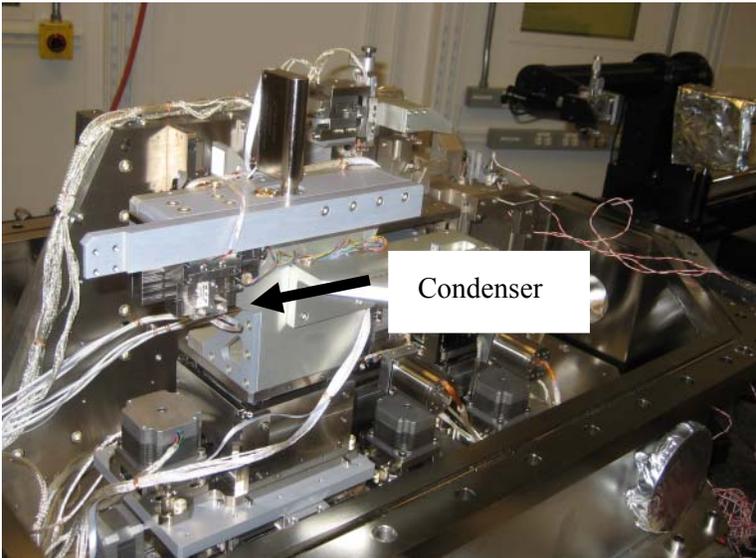
### **7.1.3 Installation and prealignment of condenser**

Caution – the condenser is very fragile. Handle with care.

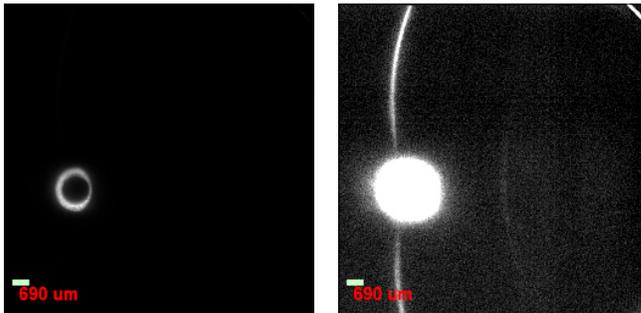
1. The condenser is first installed in a mounting piece (see Figure 12). Pay attention to the correct orientation. The central stop is located on the downstream side of the condenser.
2. To mount the condenser in the Nanoprobe, it makes sense to rotate the condenser platform on its stage stack by 180 degrees (see Figure 13). The condenser should be protected by a cover (not shown in the figure) after the initial alignment.
3. After the condenser is mounted, rotate the platform back to its correct position.
4. Make sure the distance from the condenser downstream end (central stop) to the nominal focal (sample) position is approximately correct (should be listed in condenser data sheet).
5. By eye, align the condenser approximately straight. You can bring the imaging optics holder onto the beam axis (determined above) and then use the telescope to visually align the condenser to the beam.
6. A spare condenser holder with a straight tube can also be used for preliminary alignment.
7. If you can see the direct beam with a condenser reflection, adjust the tip and tilt until the condenser ring is even and symmetric.



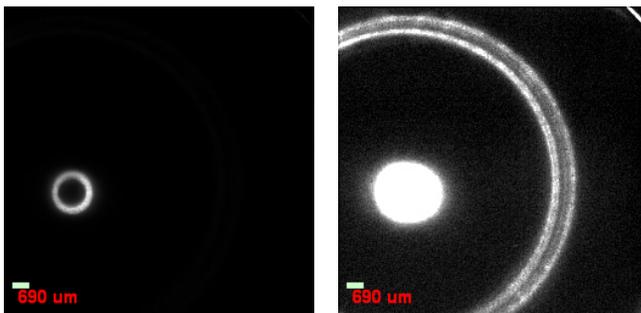
**Figure 12: Condenser in mounting bracket.**



**Figure 13: Condenser mounted in Nanoprobe. Notice that in this picture, the condenser platform is installed 180 degrees rotated to allow for easy access to the condenser mount.**



**Figure 14: Condenser misaligned.** *Both pictures show the same image with different brightness / contrast settings.* The condenser is the only optical element in the beam path, and the beam is observed on the CCD detector using the 2x objective. Left: Full contrast scale. The shadow of the central stop is visible in the center of the direct beam. Right: Contrast adjusted so that we can see a misaligned reflection of the condenser.



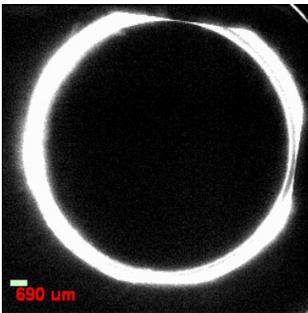
**Figure 15: Condenser aligned.** The optical setup is the same as in Figure 14, and again both pictures show the same image with different contrast adjustments. After adjusting condenser tip and tilt, we can see a nice and even ring on the camera (in this picture, the camera is not aligned perfectly to show the full ring). In the center, we can see the direct beam with the shadow of the central stop (left).

#### 7.1.4 Alignment of Pinhole

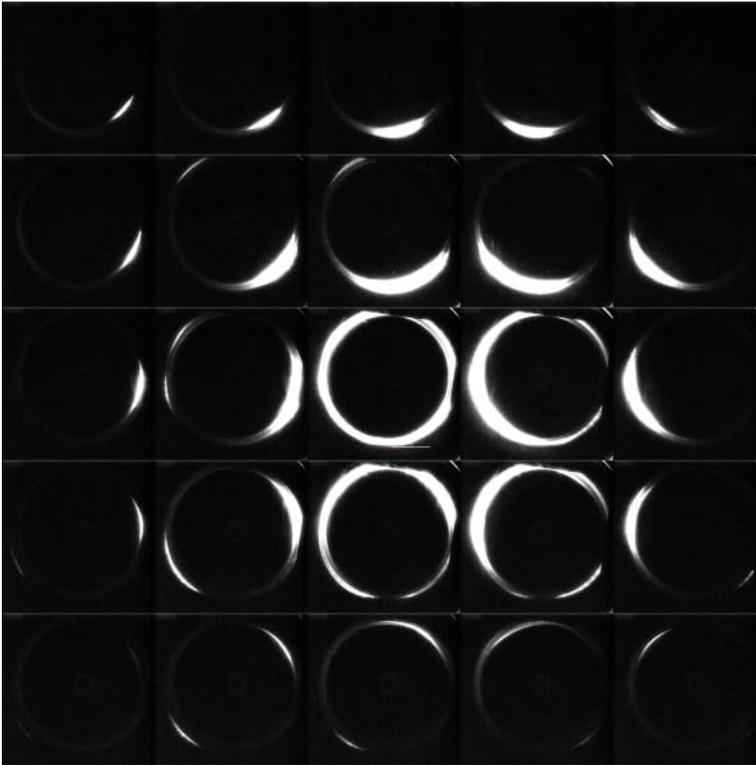
A pinhole is mounted between the condenser and the specimen stage to block the direct beam passing through the condenser and any stray rays. In principle the pinhole has to be only a little bit smaller than the central stop to pass all direct (unreflected) radiation [refer to a sketch of the optical setup]. However, since we are using the same pinhole which is used as OSA in scanning mode, which is located relatively close to the sample, we are using a rather small pinhole of about 35 microns diameter.

- Move the condenser all the way out.
- Position the telescope on the beam axis.
- Find a reference on the beam axis downstream of the pinhole location, for example the zone plate or its mounting hole on the imaging optics module. (Use the x-ray beam to make sure the reference is on the beam axis.)

- Possible find another reference, for example one of the zone plate mounting holes on the focusing optics module and make sure it is on the beam axis.
- Align the pinhole with the reference(s) using the telescope.
- Now the pinhole should be close to the beam axis. Make sure the beam passes through the pinhole.
- Move the condenser back in.
- Make sure the pinhole is close enough to the condenser focus not to cut off the beam [refer to a sketch of the optical setup]
- Align the pinhole in X and Y to optimize the condenser ring.
- Finally, you can take a mosaic moving the pinhole around the aligned position (see Figure 17). The pattern should be nicely symmetrical, and the pinhole should cut off the ring evenly in all directions.



**Figure 16: Pinhole aligned. We see the condenser ring as in Figure 15, but now the pinhole blocks the direct beam.**



**Figure 17: Mosaic of pinhole around aligned position. This image was recorded with a 35 micron pinhole and 20 micron steps.**

### 7.1.5 Using the alignment camera

An alignment camera is provided to monitor the beam at various positions along its axis. It consists of a scintillator screen, a mirror which deflects the scintillator image by 90 degrees, an optical microscope lens and a CCD camera, which is read out by a frame grabber card installed in the NanoPI control computer. The whole assembly is mounted on a rail on top of the microscope chamber (see Figure 18). The X positioning of the camera has to be done by moving the rail on the chamber. The Y positioning can be done by sliding the camera on the rail. The scintillator and mirror can be slid up and moved out of the way (see Figure 19).

The objective of the alignment microscope has manual zoom and focus dials. If the range of the focus wheel is not sufficient to focus the camera onto the scintillator, you have to mechanically move the camera assembly on its mount (the nominal distance between scintillator and the tube is 171 mm).

Note: To use the alignment camera, in TXMController go to Microscope → Switch Camera. Now all images will record the alignment camera instead of the main camera. [In the acquisition window, the camera options change, see later section about TXMController].



**Figure 18: The alignment camera mounted on its rail on the plexiglas cover.**



**Figure 19: The alignment camera lowered into the beam path. The scintillator is sitting right behind the specimen.**

### 7.1.6 Adjustment of condenser focus

To focus the condenser on the specimen, first define the nominal specimen position by moving the specimen stage to the center of its Z travel range (should also be the zero position). Now adjust the alignment microscope such that the scintillator screen is located at the same position. An adjustment “by eye” is sufficient because the condenser depth of focus is on the order of a millimeter. Record a condenser focal series in first in coarse steps (about 3 mm), then in finer steps (down to about 500 microns) to identify the best focal position of the condenser.



**Figure 20: Condenser focal spot observed with the alignment microscope. This is the ideal focal position extracted from a focal series.**

### 7.1.7 Aligning the imaging zone plate

- With the condenser removed, mark the pinhole location on the camera image using the annotation tools.
- Also remove the pinhole, then move the imaging zone plate into the beam. The silicon nitride window which holds the zone plate should be clearly visible on the camera; the zone plate will be located in the center of the window. Move the zone plate to the pinhole location marked in the previous step.
- After inserting the condenser and the pinhole, the zone plate focus should be visible on the camera image if the contrast is adjusted to show faint features (see figure)
  - If the zone plate is not visible, take a mosaic image (moving the imaging optics module) to locate it.
- Move the zone plate so that its image is in the center of the condenser ring.

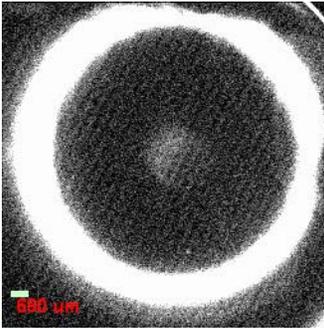


Figure 21: The zone plate focus visible on the CCD camera.

### 7.1.8 Alignment of stacked zone plates

To assure best performance using the stacked zone plate, the user has to assure proper alignment of the zone plates with respect to the X-ray beam. The following procedure shows how to achieve proper alignment of the zone plate.

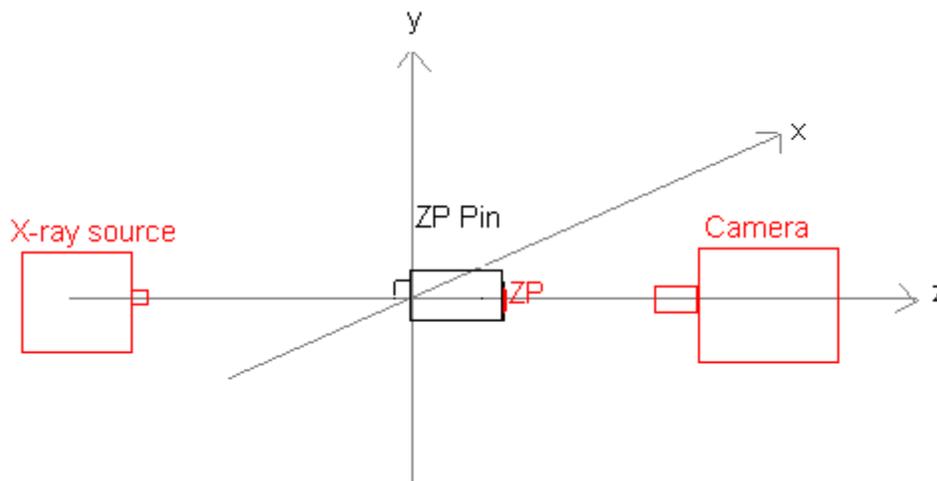
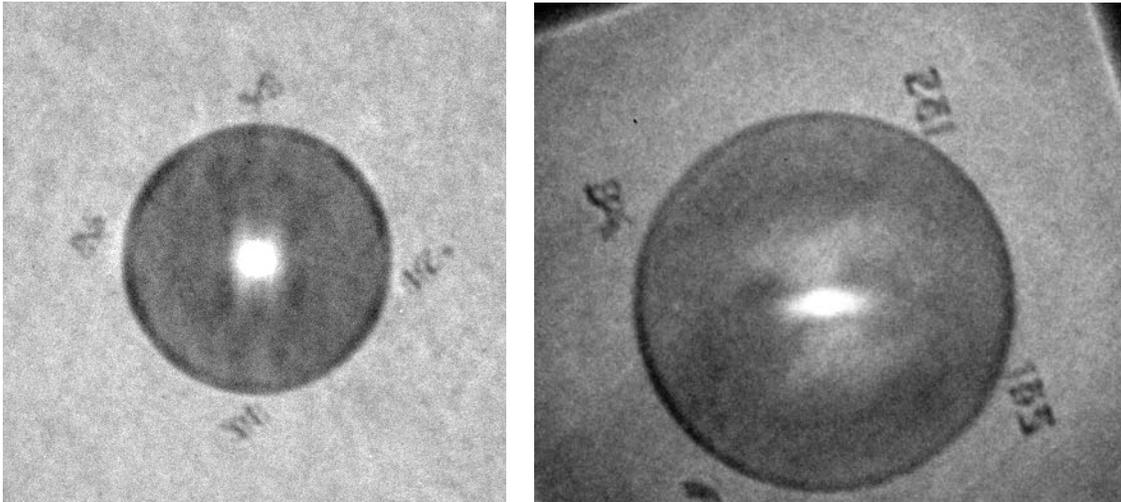


Figure 22) Schematic overview of elements used for alignment of a stacked zone plate.

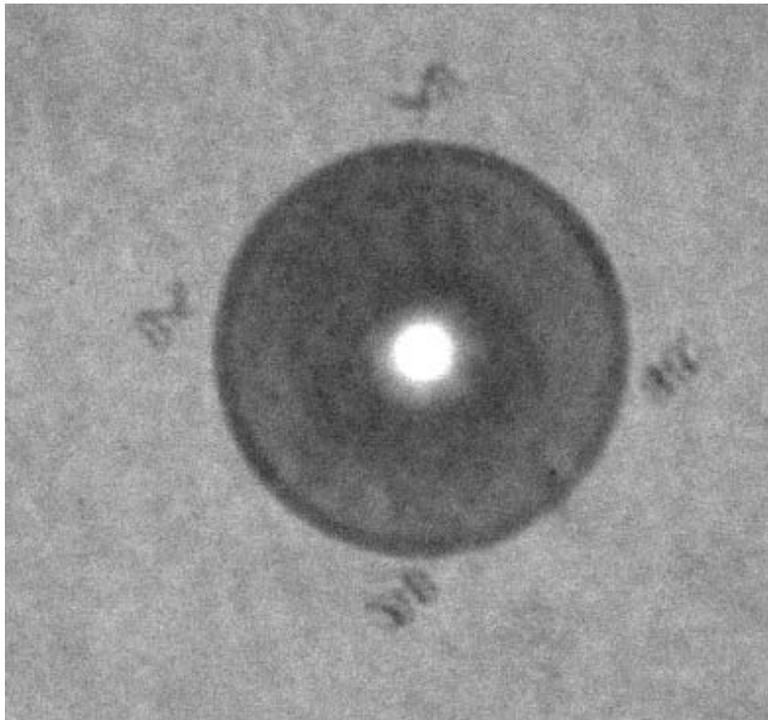
Using a configuration as shown in Figure 22, the alignment of the zone plate stack can be evaluated. Figure 23a) shows a zone plate that is misaligned in the X-direction and Figure 23b) shows a zone plate that is misaligned in the Y-direction. If the stacked zone plate is only tilted in x-axis but not y-axis, vertical fringes would be detected. The above image has fringes that are symmetric to the y-axis, indicating the stacked zone plates are well aligned in y but tilted in x. The image on the left has a tilting angle in x of approximately 0.5 degree.

Horizontal fringes imply tilting in y-axis (Figure 23b). The zone plate has a tilting angle in y of approximately 0.5 degree.



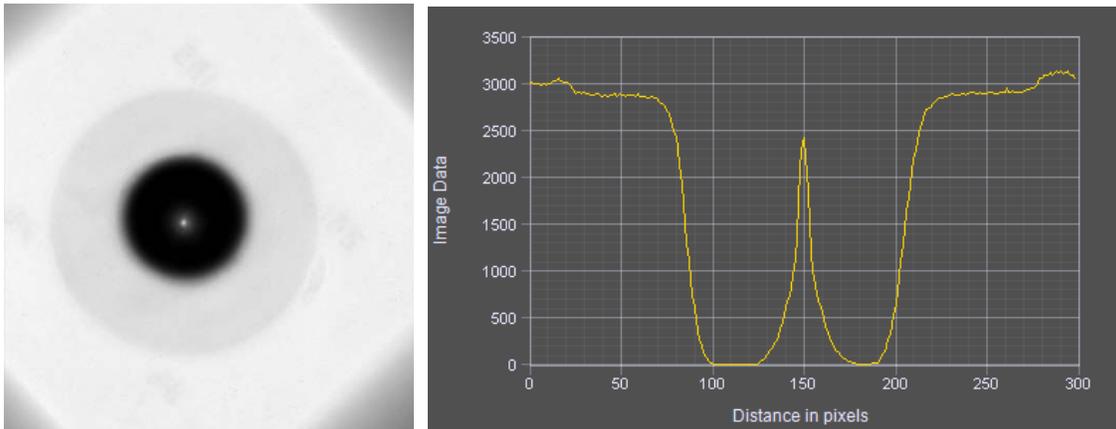
**Figure 23) Misalignment of stacked zone plate along a) X-axis and b) Y-axis.**

A perfectly aligned zone plate should have no fringes. It should have uniform intensity throughout the corrective ring and within the zone plate. The peak should have a bright and circular shape when the zone plate is aligned as shown in Figure 24).



**Figure 24) Perfectly aligned stacked zone plate.**

After tip-tilt correction using the fringe method, the stacked zone plate is well aligned to the X rays. If a more precise alignment is needed, a tip-tilt correction using the focus intensity can be performed. It is important to only adjust the tip or the tilt at a time and then compare the mean/peak intensity of the focus. By iterating this process the zone plate is aligned perfectly.



**Figure 25) a) Image of stacked zone plate with central stop. b) Extracted line profile across focus point.**

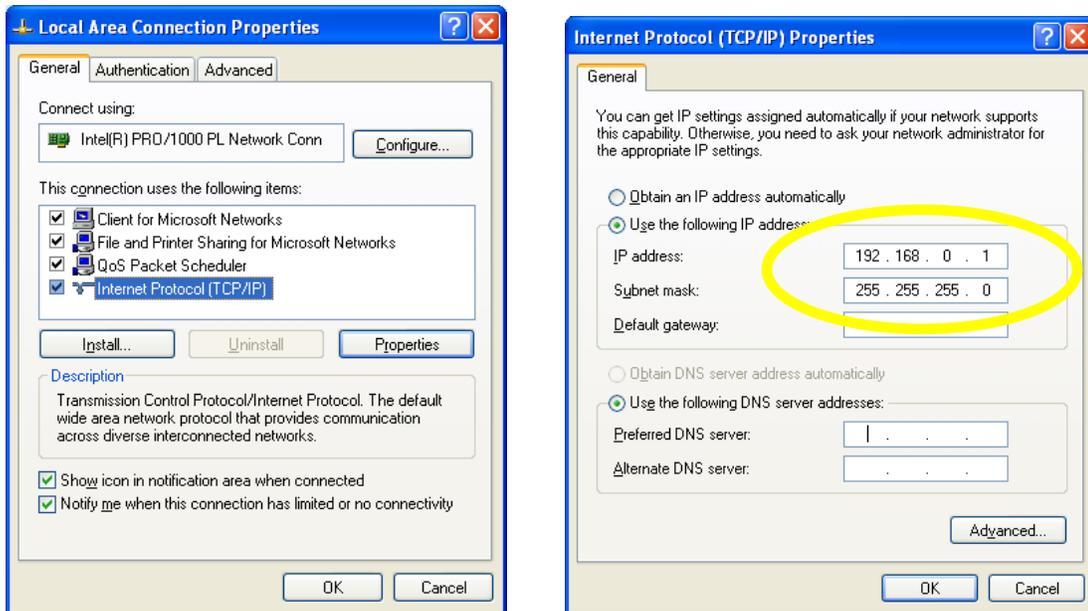
When a stacked zone plate with stop is in focus, there should be a focus point in the middle of the zone plate. Figure 25) shows a 133  $\mu\text{m}$  diameter zone plate with 24 nm outermost zone width and a 65  $\mu\text{m}$  stop. The focus point is visible on top of the blocked out region from the central stop. The intensity line plot of the focus is used to fine-adjust the zone plate.

## 8. Computer setup

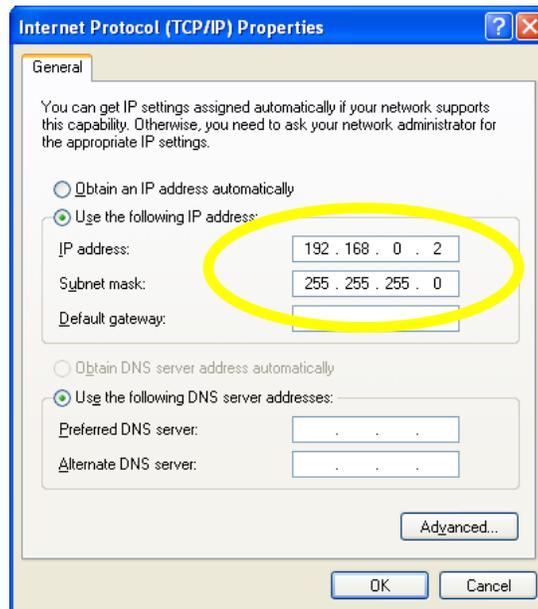
### 8.1 Exporting computer display to local client computer

It is possible to simultaneously display the microscope computer control screen on several client computers. This is helpful when adjusting laser encoder prisms or watching stage motion.

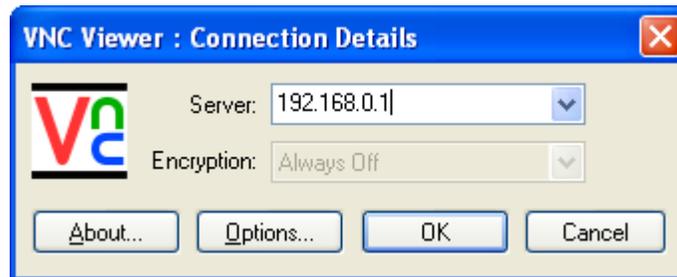
- use network cables and a network switch to connect the microscope computer and the client computers
- open the wired network configuration on the microscope computer and enter a private network address



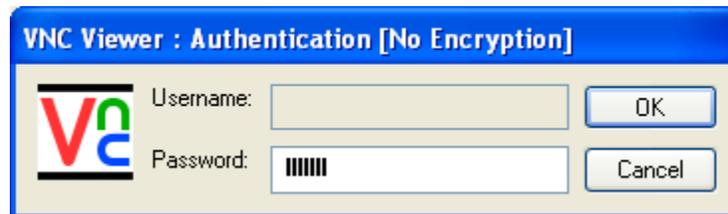
- click OK on both network configuration windows
- enter the same configuration on the client computers, but increment the last digit by 1 for each connected computer



- open RealVNC on the client computer
- enter the IP address for the microscope computer



- enter the password



- after pressing OK, you will be on the microscope computer

## 8.2 Adjusting the sharpness of the computer screen

If the Xradia monitors at ID26 in Argonne look blurry it could just be that the delay between the different colors is not good. To get to the menu where this can be changed press **Scroll Lock** twice quickly. Then use the \* (in the number lock keypad) to switch between the



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Concord, CA 94520  
Phone: (925) 288-1225  
FAX: (925) 288-1299

different colors and the + and - to increase and decrease the delay. Enter gets you out of that menu.



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## **9. Maintenance and Service**

### **9.1 Maintenance by the user**

The user must perform the following maintenance tasks to ensure optimal performance of the instrument.

Weekly: Check the resolution of the microscope using the supplied test pattern.

### **9.2 Maintenance by Xradia**

**Xradia personnel** will perform the following scheduled tasks.

Every six months - Service by Xradia personnel

Check the microscope alignment.

Check the microscope condition.

Verify that the safety components are in place and operable.

Every year - Service by Xradia personnel