Characterization of Diamond Samples
CHESS run - Spring 2011
4/28-5/3/2011

Thursday, 4/28

The following documentation was provided to us by Ken Finkelstein. It explains how to set up and focus the CCD camera, call the “Finger Lakes Lens-Coupled Detector” , or “fli” for short.

FINGER LAKES LENS-COUPLED DETECTOR USERS MANUAL

CONFIGURATION

- The detector is currently set up with a 35mm Nikon adjustable-aperture lens and a 76mm extension tube to provide 4x magnification of the scintillator onto the chip. The lens is currently set to f/4; it can be set to f/1.4 or f/2.8 for better light throughput at the cost of slightly worse resolution. The lens is mounted backwards to get the flattest possible focal plane at the chip.

- The scintillator is 11.7 microns of single-crystal GGG:Eu (Europium-doped gadolinium gallium garnet), grown on top of an layer of undoped GGG ~0.5mm thick (this protects the optical lens against x-rays that are not stopped in the doped layer).

FOCUSING

- The lens is in a fixed position relative to the chip, so focusing involves adjusting the position of the scintillator to put it in the focal plane of the lens.

- Gross adjustments can be made by moving the entire brass baffle. This should not be necessary at the moment unless the detector is disassembled to change the lens configuration.

- Fine adjustments are made by means of a micrometer mounted to the baffle rails.

- The basic idea is to image a knife edge and adjust the micrometer until the profile is optimal. You can do this according to your own beliefs or use the procedure outlined below (inspired by dynamic regridding in ODE solvers...)
- Gently tape a knife edge to the front of the baffle. Do not press directly on the scintillator. I orient the knife edge vertically so the beam profile will be uniform perpendicular to the edge, since F3 has a wide beam but not a very tall one; whether you orient it vertically or horizontally at C1 is up to you.

- Take a series of exposures at several micrometer positions to get a rough idea of the location of the focal plane. For large adjustments (0.010" - 0.050" per step,

say), this can be done by eye. For finer adjustments, use your program of choice or follow the instructions below to use tvx to check the knife edge profile.

- Determine which two points in your rough check are closest to the focal plane on either side. Take another series of exposures between those two points, using a smaller step (0.001", 0.002", or 0.005", say). Repeat until satisfied with the focus.

- I generally use the following series of step sizes:
  0.050" -> 0.010" -> 0.002" -> 0.0005"
  Focusing to +/- 0.002" is fairly quick, but it is possible to do better if you are willing to put in the time to do +/- 0.001" or even +/- 0.0005".

- Down to about 0.002" the tvx-based method below is probably sufficient to check the focus. Below that point I use a python script to differentiate the knife edge profile and fit the line spread function to determine the FWHM.

Checking the focus with tvx

- Open an image in tvx. In the middle dropdown menu (below the sliders), select "butterfly."

- Click the wings of the butterfly and drag them down to parallel, or set "splay = 0.0".

- Click the directional ray of the butterfly and drag it until it is perpendicular to the knife edge. A trick for doing this: click the image to move the center of the butterfly to a point on the knife edge. Then it is easy to rotate the butterfly to lie parallel to the edge, subtract 90 to find the perpendicular and use this to set "dir".

- Mouse over the center area of the butterfly until you get a two-headed arrow. Click and drag to pull the two wings together so they span a narrower section of the image, or set "sep" to something small. I sometimes use 0; use a nonzero number to average over more than one line of pixels, but I wouldn't go bigger than 9 or so.

- Now type "integrate" in the tvx window to get a 1D plot of the knife edge profile. Use the zoom feature to get a close look at the profile to see how many pixels it spans.

- Sometimes after a few iterations of this, tvx will begin to complain that it "can't open dens0.graf for writing" or something similar. Quit tvx and restart.

FINGER LAKES LENS-COUPLED DETECTOR USERS MANUAL

In case of questions or problems this document does not address,
page or call Robin Baur (607-205-8727).

BASIC USAGE

- Run spec from the directory to which you would like your images to be saved.

- Useful commands in spec:
  - ccd_on: this establishes a connection to the detector laptop. You should
    - see the message "Connection has full control of camera" in the laptop camserver terminal after issuing ccd_on. Once you have set ccd_on, the detector will automatically take an image every time spec does a count.
  - ccd_off: this disconnects spec from the detector laptop. The camserver terminal should say "Returning process token to main program" after issuing ccd_off. You should use ccd_off if you want to do any spec scans for which an image is not required.
  - newfile: changes the file in which spec is storing its data.
    - Use only alphanumeric characters and underscores. No hyphens, periods, or other symbols. Start the filename with a letter.
    - Your images will be named according to the pattern /path/to/current/directory/filename_scannumber_scanpoint.tif
      e.g. for spec running in ~/Data/baur using newfile "miscut", the first image in scan #1 will be
      /home/specuser/Data/baur/miscut_001_000.tif
  - dark: dark <time> takes a pair of shutter-closed exposures
    - for use in background subtraction. Take a dark set for every exposure time used during the experiment.

- Check the water level in the chiller daily. If it is below the topmost narrow slit, top it off with DI water from the chem room.

- A spec/FLI interaction quirk: if you want a single exposure, do
  tseries 1 <time> rather than ct. ct doesn't increment scan or image numbers, so spec appends the image to the end of the previous scan. Possibly if you do more than one ct in a row it will even overwrite the previous ct; I have not tried it.

THINGS TO KEEP IN MIND

- The single-crystal scintillator is underneath the black plastic taped over the front

of the brass light baffle on the detector. DO NOT press on, crash equipment into, or otherwise fold, spindle, or mutilate the front of the light baffle.

- The detector readout time is ~30 seconds.
- The detector image is vertically inverted. Left-right orientation is correct.
- Calibrating exposure times: the detector has 16 bits, so a pixel is saturated after 65535 counts (including a 10400-count pedestal).
- I (RMB) have found the camera's internal shutter unreliable below about 0.1sec exposure time.
- The vacuum pump and chiller generate a fair amount of heat. It is best to keep the hutch closed as much as possible to keep the hutch temperature uniform and minimize experimental problems arising from differential thermal expansion.

VIEWING/MANIPULATING IMAGES IN TVX

- Opening tvx: open a terminal on the detector laptop and type
  - cd tvx
  - ./tvx
- Displaying images: the syntax is
  - disp [full path to image] [min counts] [max counts] [# of grey values] where [min counts] and [max counts] determine the black-to-white range of the displayed image; they and the # of greyscale values can be adjusted with sliders after tvx brings up the image.
  - Example: disp /misc/f3/Data/baur/miscut_001_000.tif 10400 65535 5
- Background subtraction: assuming you opt not to do this in your data postprocessing program of choice, the syntax is
  - move [newfilename]=[full path to image]-[full path to background]
- Dezingering: the one time I (RMB) tried to do this, it hung tvx. That said, if you want to do this from within tvx, consult the "Instructions for FLI Lens Coupled_RevC" on the detector laptop's desktop.

TROUBLESHOOTING

- Problem: No connection to detector after typing ./camserver
- Solution: camserver intermittently fails to recognize the detector for no particularly good reason. Try the following:

        - make sure the detector is on before booting the laptop
        - make sure the parallel adapter is firmly seated
        - quit camserver and try ./camserver again
        - reboot the computer and try ./camserver again
        - shut down the computer, power cycle the detector,
        - reboot the computer and try again

    - Problem: spec hangs after printing a line similar to
      Image: /home/specuser/Data/baur/miscut_3p2_014_000.tif
      but never seems to actually take an image
    - Solution: spec is probably waiting because mostab is inhibited. Make sure:
        - CESR is not injecting
        - The hutch shutters are open

    - Problem: camserver continuously spewing lines of information about its shutter status
    - Solution: this happens sometimes, but not always, when a spec scan is aborted while the camera is exposing. One option is to wait until the exposure finishes naturally; sometimes the issue resolves itself. If it does not, or if the exposure is too long to wait for:
        - quit and restart camserver
        - issue ccd_on in spec
        - check the status of any equipment connected to the detector TTL breakout box; sometimes this problem can cause the shutter status TTL output to remain high even when the shutter closes itself. If this has happened,
        - taking a "sacrificial" short exposure generally corrects the TTL signal.

    - Problem: camserver complains it cannot open target file for writing
    - Solution: Generally this means the station computer was not mounted correctly. Open a terminal on the laptop and type
        - cd /misc
        - ./connect c1
        - When prompted, enter the specuser password (possibly more than once).
        - Make sure you can then browse /misc/c1 from the detector computer and see that it contains the directory structure from /home/specuser on the station computer.

    - Problem: Readout time is unusually short and no image seems to be saved; no error messages
    - Solution: This usually means the detector _thinks_ it can write to its desired save location, but actually can't. Make sure the permissions are set correctly for the directory from which you are running spec.

FINGER LAKES LENS-COUPLED DETECTOR USERS MANUAL

HARDWARE SETUP

1. Hook up the vacuum hose, the water lines, the power supply for the heating element, and the pressure relay.

2. Plug the heating element and the detector into the labeled outlets (TE Power and CCD Power) on the back of the interlock unit.

3. Plug the parallel cable into the downstream port on the detector body and run the other end through the cable labyrinth.

4. Open the vacuum valve on the detector and turn on the vacuum pump. The detector will pump down to around 30 mTorr. Continue pumping on the detector for the duration of the experiment.

5. Once the detector has pumped down, turn on the chiller. Check the connectors for leaks. The interlock unit should now have a green light indicating "Flow OK."

6. Hit the "TE Power Reset" button to power on the heating element.

7. Power on the detector.

8. Screw the parallel adapter firmly into the laptop parallel port and plug in the parallel cable.

9. Connect the computer to the internet via the USB-to-ethernet adapter. Do not use the built-in ethernet port.

LAPTOP SETUP

1. Power on the computer and log into the specuser account using the usual password.

2. Open a terminal and type

   cd camserver

   ./camserver

   Camserver should then display many lines of information about the camera configuration (number of pixels, chip temperature, etc). In the not-unlikely case

that camserver displays only two lines of information, consult the Troubleshooting section of this guide before continuing.

3. In camserver, issue the command

   flicommand setccdtemp -35

   to start cooling the chip to -35C. The command

   flicommand getccdtemp

   can be used to check the current temperature.

4. Open a second terminal and type

   cd /misc

   ./connect c1

   When prompted, enter the specuser password (possibly twice). This mounts the station computer as a writeable location for collected images.

STATION COMPUTER SETUP

1. In a terminal on the detector laptop, run /sbin/ifconfig and note the IP address.

2. Open ~/Macros/FL_c1/fliccd_for_c1.mac and change HOSTIP_HOSTPORT to the IP address of the detector laptop. The port should be left as 41234. Also check that SPECUSER_PATH is "/misc/c1/".

3. Restart spec or type
   udo fli_ccd_mode.mac
   to load the FLI macros.

Initial setup with Ken

Monochromator setting:

- beam energy: 15 keV, vertical expansion factor ~8
- beam dimensions: 1cm x 1cm, reasonably uniform
- monochromator reflection: silicon (3,3,1)

Camera configuration:

- camera magnification factor is now x4, relative to previous runs where it was x1.

The first thing that Ken did was to try to find a rocking curve peak using a high-quality polished

silicon wafer. The wafer is 50 microns thick and about 4 inches in diameter. The wafer has a surface normal to the (0,0,1) direction, and a flat edge that defines the (2,2,0) direction. We mounted it inside our large mylar hoop holder, roughly centered on the hoop. We want to find the (3,3,1) reflection in transmission geometry, using the beam from the (3,3,1) mono. We calculated the Bragg angle and the goniometer theta offset as follows.

- θ Bragg = 19.3722 degrees
- 2θ = 38.7444 degrees
- θ shift = 13.2627degrees
- θ goni = 6.1095 degrees
- χ when (2,2,0) is horizontal = -136 degrees
- χ when (3,3,1) is in vertical plane = -136 degrees

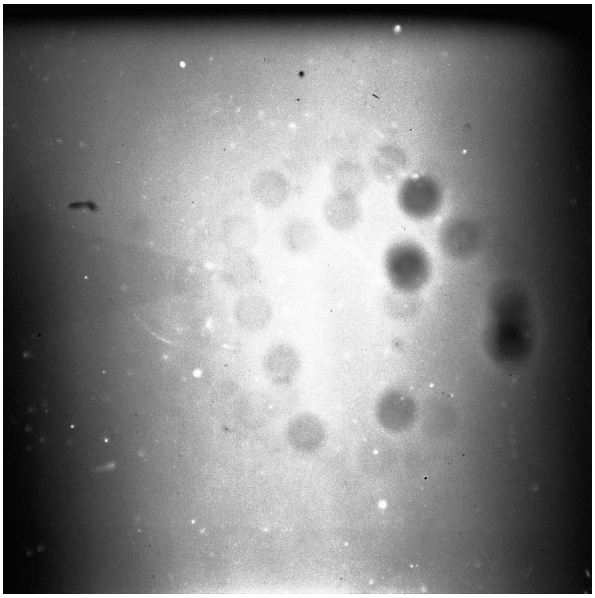
We did find a (3,3,1) reflection at approximately the above angle settings, but the shape of the image was very non-uniform. At a given theta, the intensity image looked like a worm. It walked across the field of view of the camera as theta was swept through the region of the rocking curve. The orientation of the stripe was vertical in images, horizontal in real life. We interpreted this to mean that the silicon wafer is far from planar at the angular resolution of the setup. We expect that the angular resolution of the setup is very narrow now, approaching the intrinsic rocking curve width of the (3,3,1) reflection in silicon of 1 arc second FWHM (5 μr). We may come back to this later. For the moment, we would like to see a diffraction image that shows the whole beam at once. This will be useful for normalizing images recorded later on, by taking out the intrinsic response of the camera together with any artifacts imprinted on the intensity profile of the beam by non-uniformities in the monochromator crystals.

To see a fuller diffraction image, we removed the thin 50 micron silicon wafer and the mylar hoop mount from the 4-circle, and installed a high-quality polished silicon target. This target is very thick, and has a high-quality polished surface with a (1,1,1) normal. It is mounted in reflected geometry. We decided to look at the (3,3,3) reflection. Here are the orientation coordinates for that measurement.

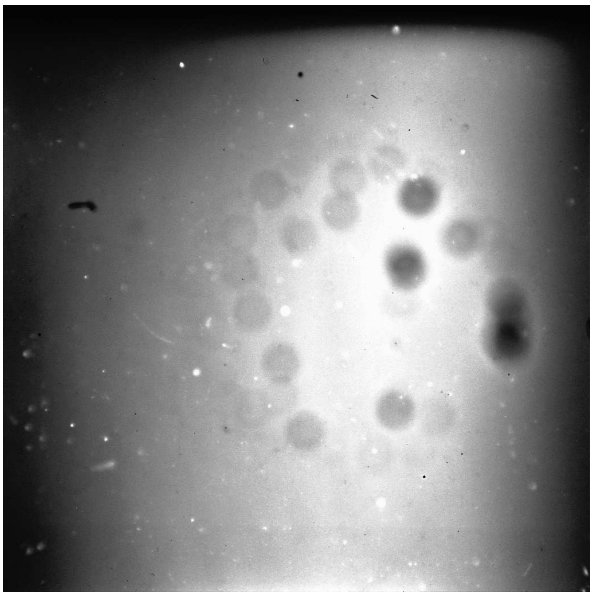
- θ Bragg = 23.2920 degrees
- 2 θ = 46.5840 degrees
- χ = -90 degrees

Here are some runs we took in this geometry.

number of steps	step size (degrees)	exposure (s)	name of scan
5	0.002	10	jones1_005
10	0.0008	10	jones1_006
20	0.0002	10	jones1_008
20	0.0002	30	jones1_009



logical OR of images in jones1_008



logical OR of images in jones1_009

Notice in particular the wedge-shaped shadow entering from the left upper part of the image. The wedge is much clearer in the left image, but it is present in both. There is nothing visible on the outside of the camera that would explain what is causing this artifact.

The second thing that Ken did was to adjust the focus on the CCD camera. To do this, he taped a slide-mounted foil of Molybdenum diagonally across the input face of the camera converter crystal. The foil does not absorb all of the X-rays, but enough of them to make a large intensity contrast between regions shadowed by the foil and not. He then took the following images with the camera, as he adjusted the micrometer that controls the focus on the camera.

step number	micrometer setting (mm)	file saved to image directory	improvement
1	0.155	c1setup042711_029_00.tif	reference
2	0.145	c1setup042711_030_00.tif	wrong way
3	0.150	c1setup042711_031_00.tif	better

4	0.155	c1setup042711_032_00.tif	pretty good
5	0.1575	c1setup042711_033_00.tif	better
6	0.160	c1setup042711_034_00.tif	better
7	0.162	c1setup042711_035_00.tif	better
8	0.164	c1setup042711_036_00.tif	no better
9	0.162	c1setup042711_037_00.tif	LOCK HERE

The tip of the pointed steel pin, that shows up as a shadow in the above images, points vertically upward in the hutch. This shows that the ccd images are rotated by 90 degrees, with physical "up" pointing to the right in the images. The working directory for this run is JONES/april2011 under the specuser account on machine c1.chess.cornell.edu (or perhaps other chess machines). Ken shared the password for this account with us, but it is not recorded here for security reasons.

The minimum step size in theta with this 4-circle goniometer is 1/20,000 of a degree.

Suggestion from Ken: There is a utility for looking at diffraction images that has a bunch of useful features. The name of the image viewer is called "imagej", that comes from NIH for allowing biologists to analyze diffraction images.

Study of the silicon (3,3,1) planes

Having seen that the beam is more or less uniform over the aperture of the camera, we decided to go back to the thin 50 micron silicon wafer we looked at earlier, and study the characteristics of the beam by measuring the rocking curves of the (3,3,1) planes in transmission geometry. It turns out that it was not possible to get access to the (3,3,1) planes at 15 keV using the thick highly-polished silicon crystal that we used in the previous measurements. The reason is because that crystal has the direction (1,1,1) normal to the surface, and the (3,3,1) plane the closest to the normal is 22 degrees away from it, too large to work with the Bragg angle of 19.4 degrees for silicon (3,3,1) in reflection geometry.

We now go back to the thin silicon wafer, with the surface normal direction (0,0,1). The alignment parameters to see (3,3,1) reflections in transmission geometry are the same ones as listed above, when this configuration was first looked at. The purpose of this scan is to determine the instrumental resolution of the beam line, using a crystal with as good a dispersion match to the monochromator as possible. Being thin, the crystal will no doubt show some bending effects, but the individual-pixel rocking curves should be as narrow as we have ever seen.

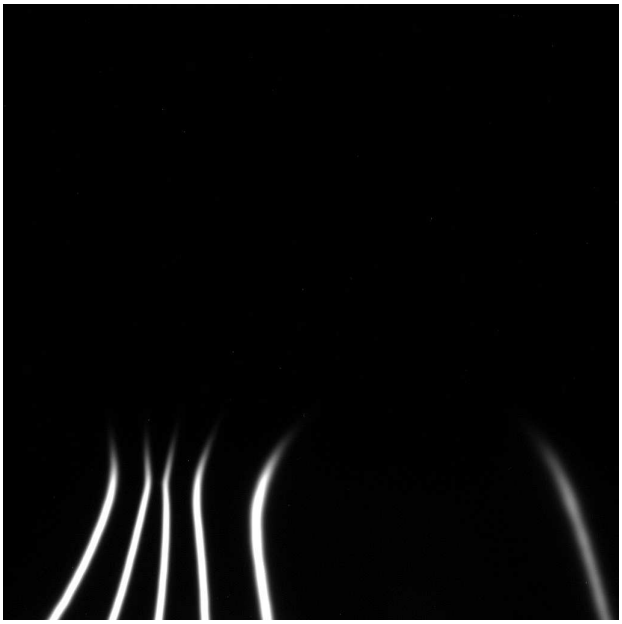
We found the peak again, and reproduced the earlier observation that the whole-beamspot

rocking curve spans the region of 0.07 degrees in theta, about 1 mr.

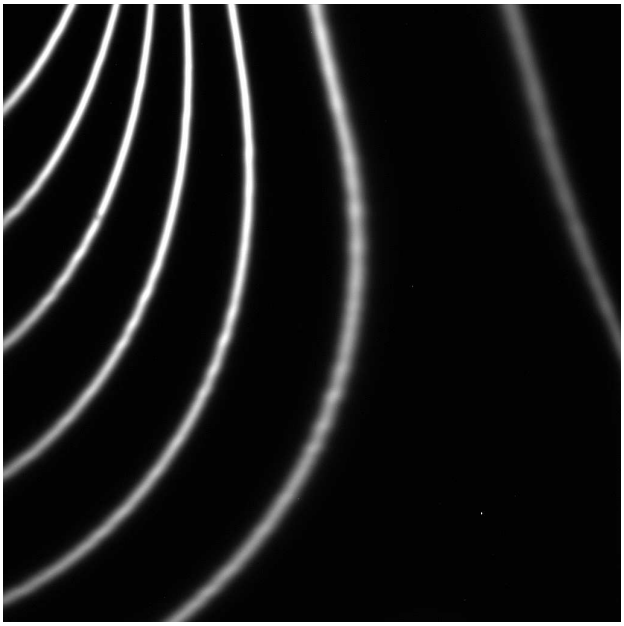
- 2θ = 38.740 degrees
- θ goni = 5.96 - 6.03 degrees
- χ = -136 degrees

We took a quick scan of this full range, using a 15 second exposure, to see that the chi angle is set to center the image in the viewport of the camera.

χ angle	number of steps	scan range (deg)	exposure time (s)	name of scan
-136	7	5.960 - 6.030	15	Si331_003
-138	7	5.949 - 6.012	20	Si331_008
-138	315	5.949 - 6.012	20	Si331_009



overlay of images from scan Si331_003



overlay of images from scan Si331_008

From the above images, one sees that the beam spot is larger than the viewport of the camera, so one cannot see the entire beam spot at one setting in $(2\theta, \chi)$. It is sufficient at this point that the beam has good intensity over the entire region of the viewport. Later on when we mount the diamonds, we will want to center them inside the viewport, because they are smaller than the viewport.

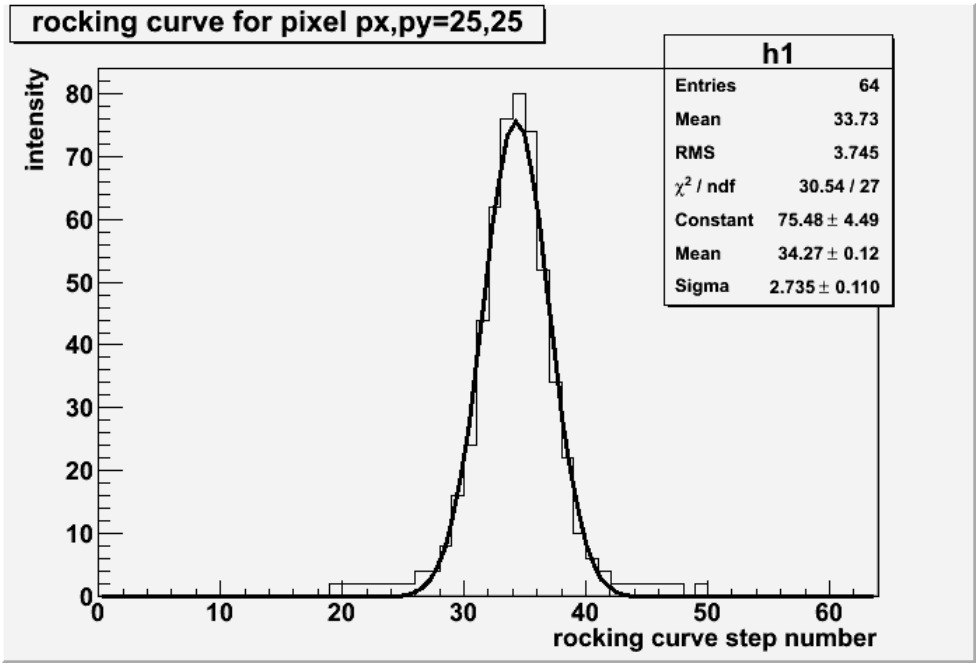
Next we will do an extended scan of the silicon (3,3,1) planes and map out the beam + camera

intensity profile, with a much finer step size of 3.5 μr.

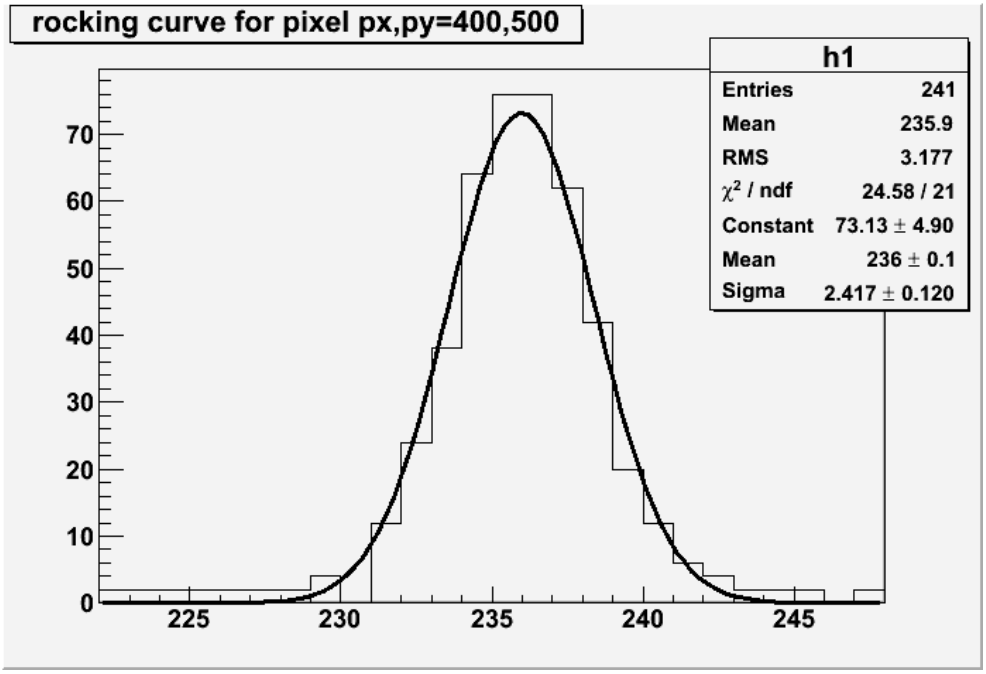
Friday 3/29

3:05am

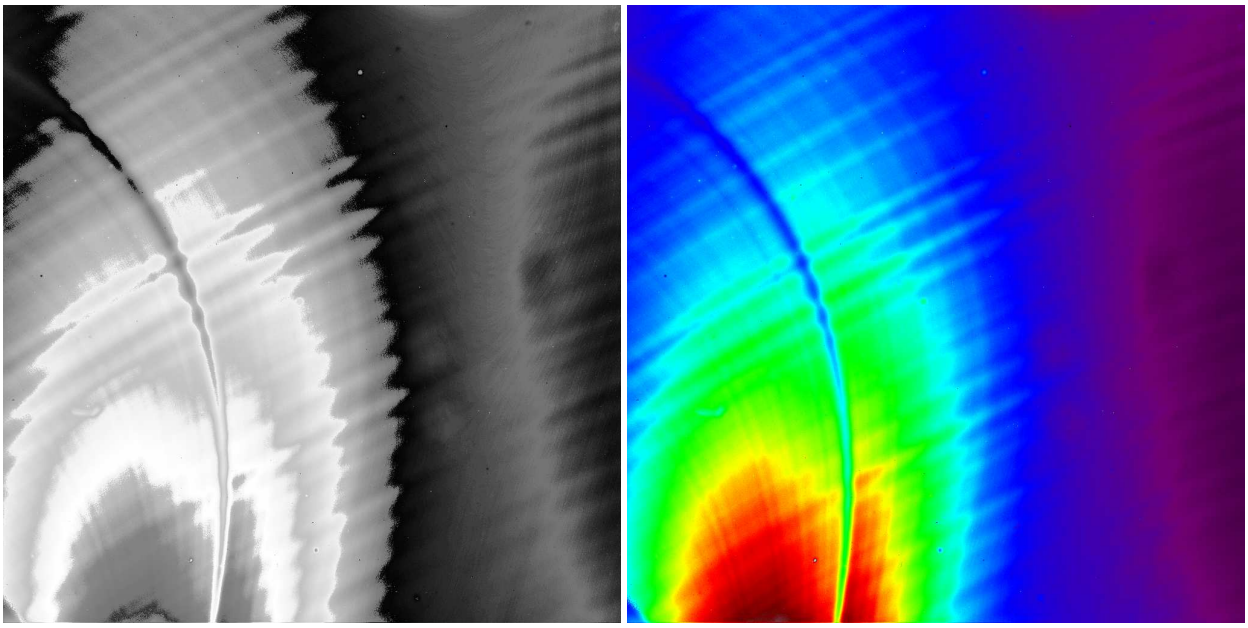
Fine scan Si331_009 was started with steps of 0.0002 deg (~3.5 μr). Here is a rocking curve that is taken from the upper left corner of the image, near where the peaks appeared early in the scan. The images are taken from run Si331_009. Here is a rocking curve for one of the pixels. A portion of the measured width of the line comes from its Darwin width at 15 keV (around 5 μr). Another contribution comes from the finite spatial resolution which, when coupled with the curvature of the crystal, increases the observed width because of walk in the centroid value across the width of the pixel.



Rocking curve for pixel 25,25 from scan Si331_009, less than 10 μr RMS

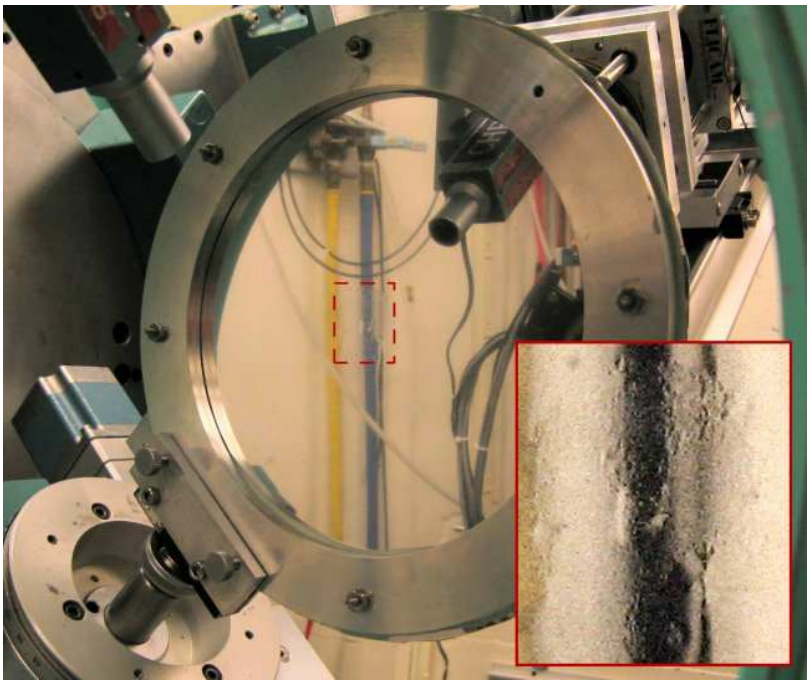


Rocking curve for pixel 400,500 from scan Si331_009, about 8 μr RMS



Summed images from scan 009 in grayscale and color contour maps. The apparent discontinuity in the grayscale image is an artifact of wrapping the grayscale more than once through black. The arc through the middle of the image corresponds to a moment during the scan when CHESS went down for a refill, and there was no beam for a few minutes.

Measurements with the Sinmat diamond



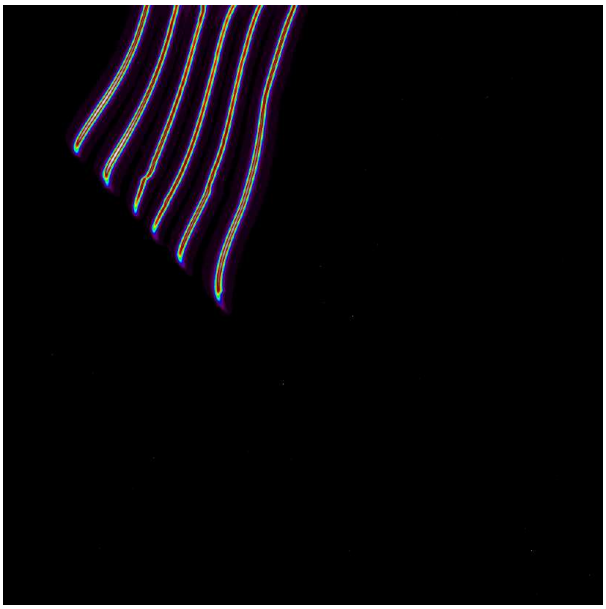
We mounted the Sinmat diamond in the large mylar hoop and centered it on the axis of the 4-circle (shown in adjacent photo). According to the manufacturer, this diamond has a surface normal of (0,0,1). We do not know the orientation of the edges, so we have to carry out a scan in both θ and χ to orient it. The nominal scattering angles for this diamond are:

- θ = 19.2 degrees
- 2θ = 38.3 degrees

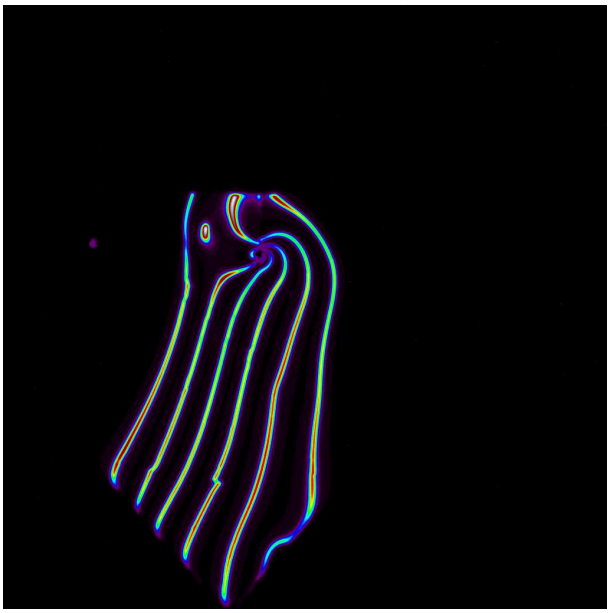
We did a scan around θ $[18.7, 19.7]$ degrees, and in χ $[135, 45]$ degrees. We used the ion chamber to look for the peak. Step sizes were 0.01 degrees in θ, 5 degrees in χ. We did not find anything. After this, we decided to concentrate on the symmetry axes in χ, that is -135, -90, -45 degrees, and enlarge the search zone in θ. This worked! We found a diffraction peak at:

- θ = 20.4 degrees
- 2θ = 38.3 degrees
- χ = -90 degrees

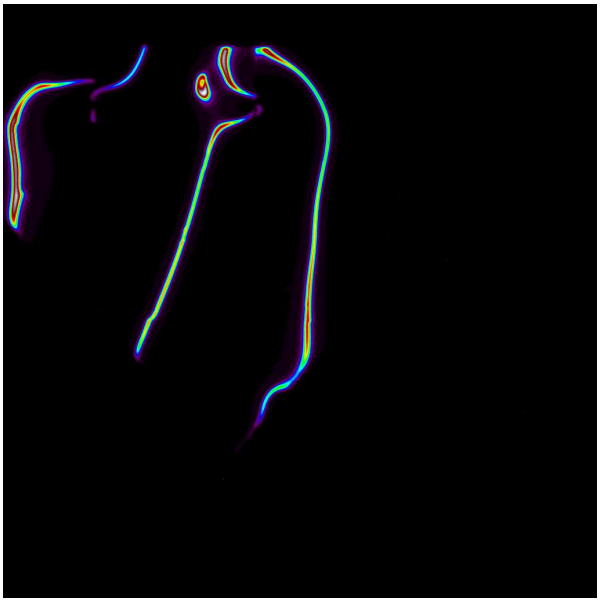
The rocking curve peak looks quite wide. To cover it we need to scan over 20.25 - 20.60 degrees, which is almost 6 mr !! We put in the viewing screen and found the diffraction spot quite a few degrees away from the vertical. We tweaked θ and χ to center the spot vertically and took a short scan to see what it looks like in the camera.al



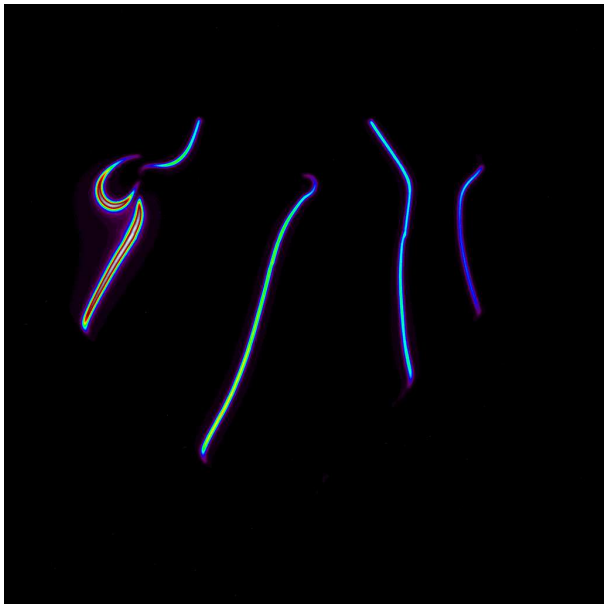
scan 025, step size 0.01 degrees, Sinmat stone



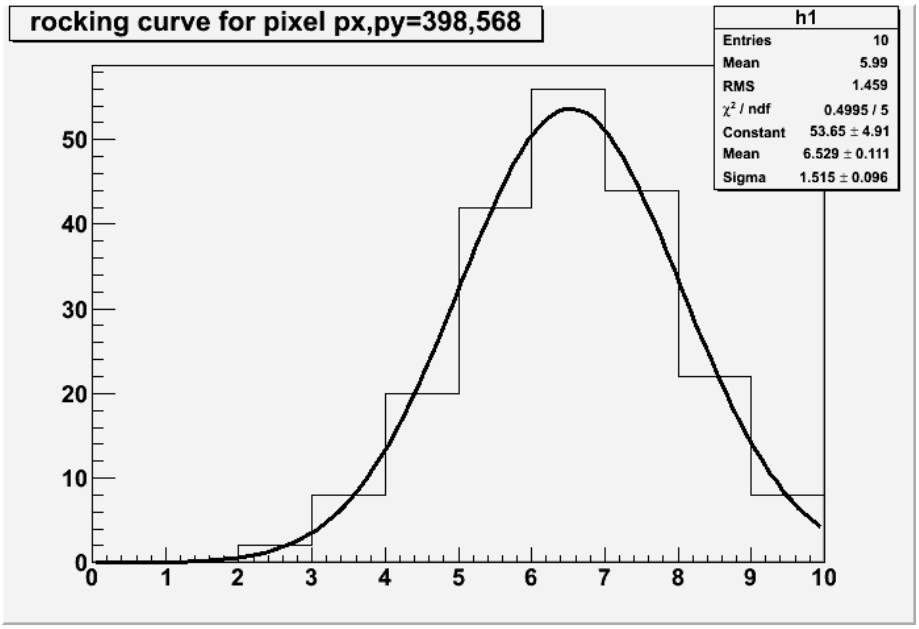
scan 029, small adjustment in χ



scan 033, still finding the sweet spot



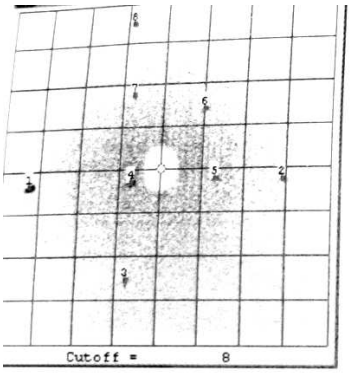
scan 035, now nicely centered

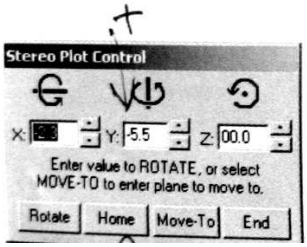


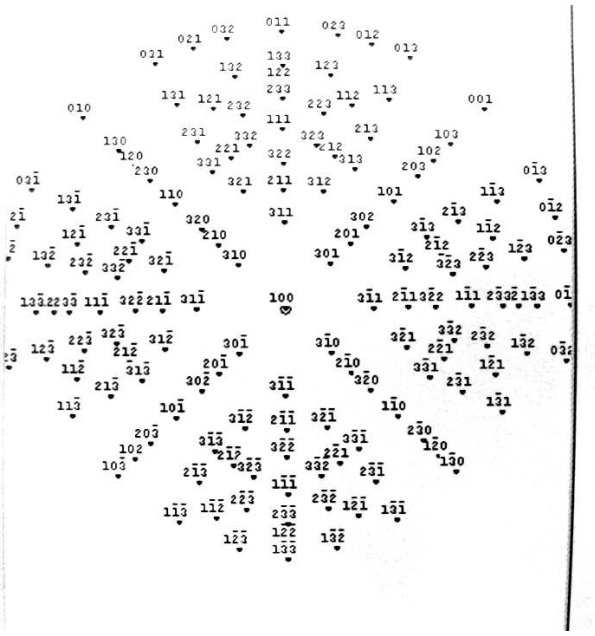
Single-pixel rocking curve from scan 038, RMS width 12 ur

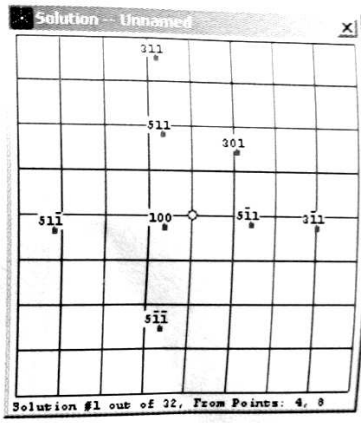
Laue Pattern Scan for the Element-6 10µm diamond

To avoid the difficulty of the search for the <220> in the Element-6 thin diamond, we took Ken's offer to have the wafer scanned in a wire-chamber/Laue scanner. The thin diamond produced few pronounced spots distinct from noise, but a plausible solution was constructed (shown below). The closest plane to surface normal turned out to be <100>, if we allow a 5.5° correction (in what would be φ)

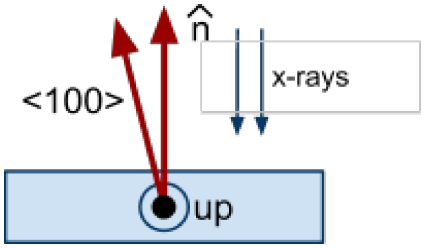








Ken explained the convention of vector direction (-5.5° shown in screen shot is actually a bug) with the diagram on the right, which shown the direction of deviation of the <100> from surface normal.



~11pm
Fine scan complete (Scan# sinmat_039). Looking for <220> relative 90° in χ shifted (in order to be able to map the curvature in the other dimensions to understand the diamond surface) It turns out that the diamond is mounted in an awkward middle of a fairly narrow permitted range for the χ motors. There was no choice but to re-mount the hoop in a 90°-shifted position and re-center the diamond.

After a short search in the peak was found with the following corrections:

χ = -103°, θ_{goni} = 18.60°

(compare to previous orientation's:

χ = -99.75°, θ_{goni} = 20.08°)

After some adjustments using the florescent paper and then ion chamber, a coarse scan was started: sinmat_043

Error: exposure time parameter was incorrectly left at 1s (vestige of ion scan). Performing "equalization" of produced photos: note suffice "_eq" before extension in files.

Saturday, 4/30

Horizontal Scan of Sinmat diamond

1:50 AM

The coarse scans to determine the rocking curve range are done, the parameters are selected as following

2θ = 38.7°

X = -102.75°

Φ = 155.6040°

Θ scan: 18.28° ↔ 18.43°

The beam will be off briefly for a new injection, will start fine scan afterwards.

2:08 AM

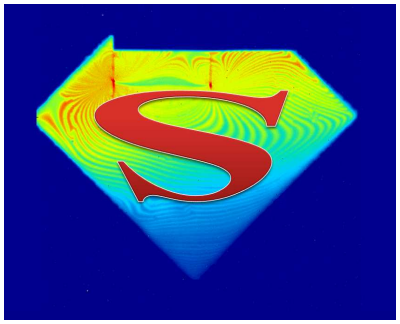
Fine scan started with scan sinmat_052, the scan step size is 0.0005 degree, 300 steps.

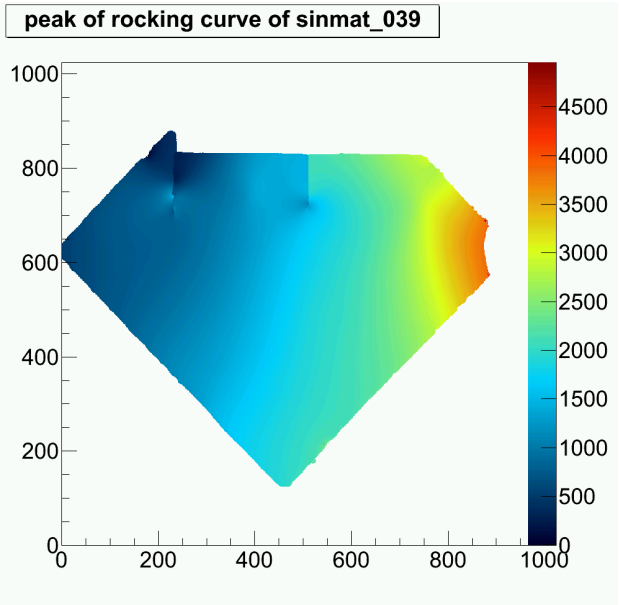
3:20 AM

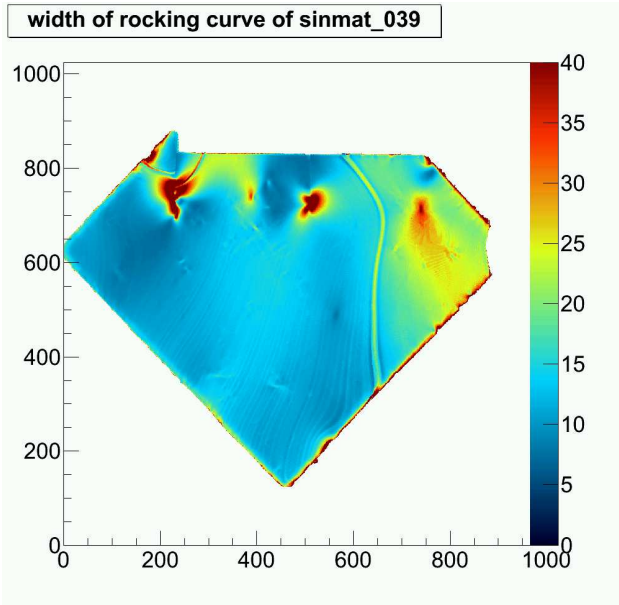
Noticed that the reflection from the diamond lattice disappeared since *sinmat_052_039*, and the readings of ion chamber 0 was also dropped by 2 magnitude since then. We lost monochromated photon source! Asked operator to troubleshoot the problem and eventually we called Ken. It turned out to be that the monochrometer needs readjustment. After that was done the readings of ion chamber came back to normal and we have X-ray in the hutch again!

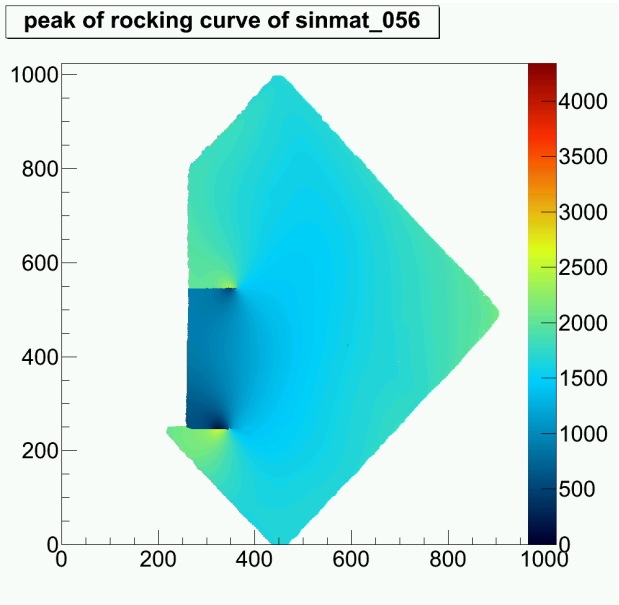
4:15 AM

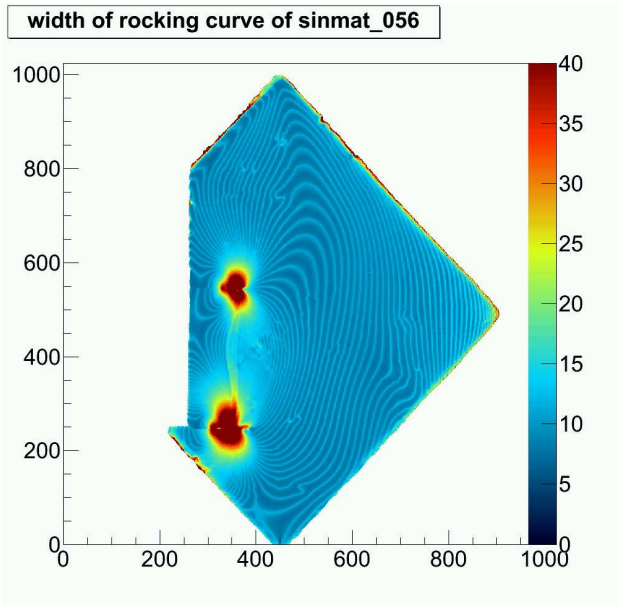
After a coarse scan to verify the beam quality, a new fine scan was started: sinmat_056 with step size 0.001 degree, 150 steps.











6:45 AM

Horizontal fine scan is completed. Now start to tune for the little corner piece. Coarse scan started from sinmat_057.

8:15 AM

Fine scan of the corner piece started : sinmat_061, step size is 0.001°, 100 steps

2θ = 38.7°

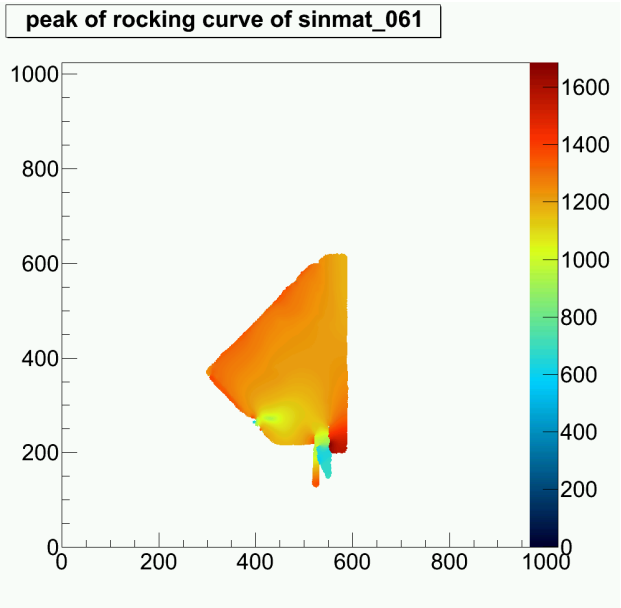
χ = -110.21°

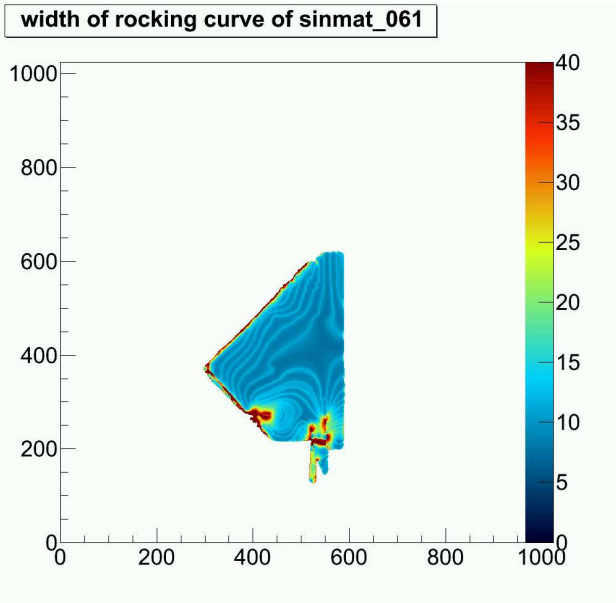
φ = 155.6440°

θ scan: 18.2185° ↔ 18.3185°

10:00 AM

Fine scan finished.





Measurements with the 10μm Element 6 diamond

Run file prefix: e6

First crude scan of the <022> peak:

Run: 013
θ scan: 23.26° ↔ 23.46° 4 steps

We performed form crude runs to tune our χ and 2θ and then started another crude scan:

Run: 017
θ scan: 23.38° ↔ 23.48° 3 steps

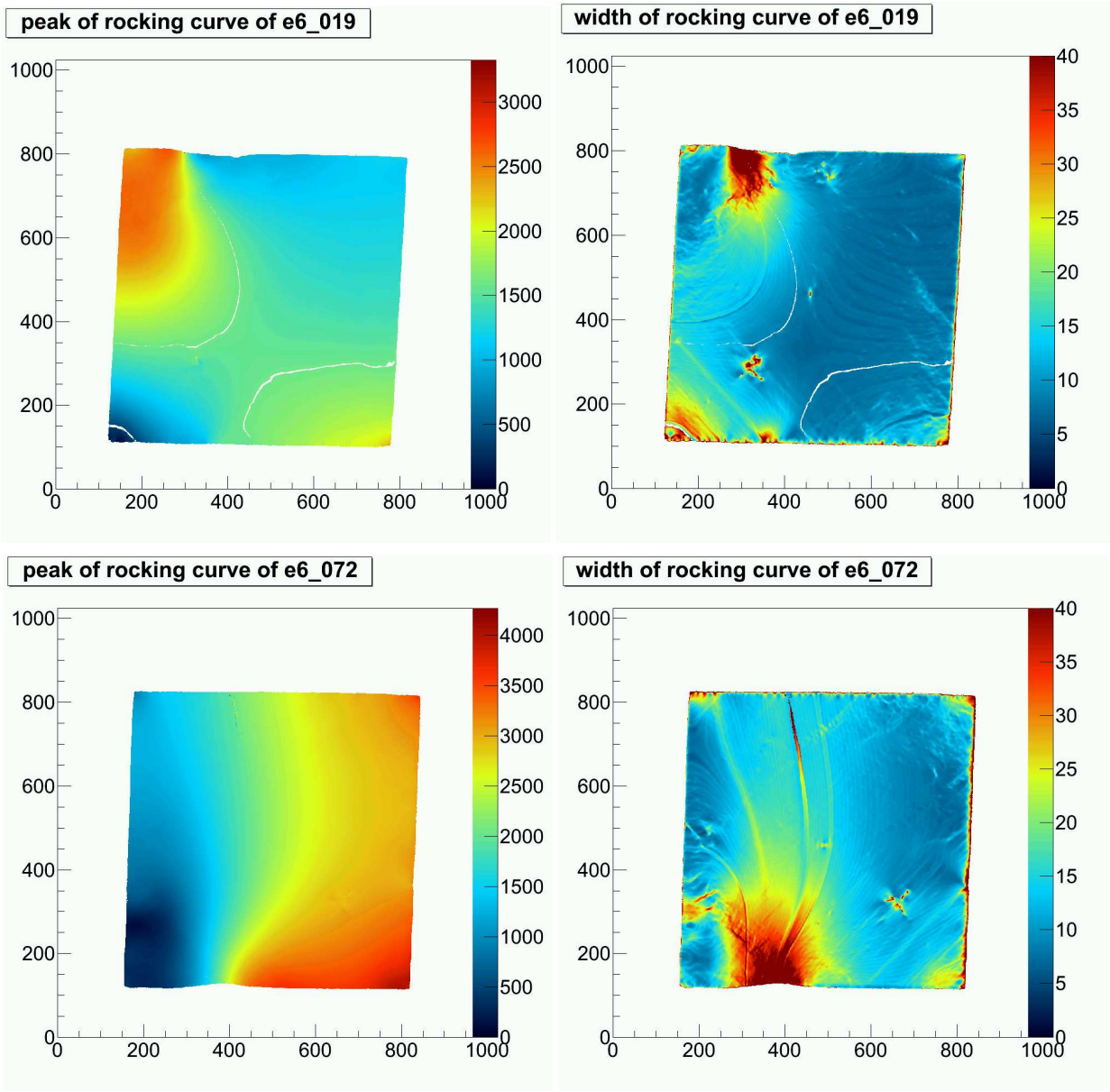
Satisfied that we have proper bounds set and that we had fairly good resolution for reconstructing narrow local rocking curves with 0.0005° steps in an earlier run, the following fine scan was set up:

Run: 019
θ scan: 23.27° ↔ 23.47°; 500 steps (0.0004° ~ 7μr step size)
φ: 149.265°
χ : -78.5°
2θ : 38.5°

Horizontal Scan

Due to the limitation of χ angle, the sample was taken out from the holder and rotated 90 degrees before put back. After some coarse scans, a fine scan was done

Run: 072
θ scan: 24.195° ↔ 24.431°; 590 steps (0.0004° ~ 7μr step size)
φ: 152.969°
χ : -91.7°
2θ : 38.3°



Sunday, 5/1

Reflection-mode scan of 10µm Element 6 diamond

We wanted to understand how much of the diamond curvature is forced by the mylar backing. A reflection-mode rocking curve measurement on member of the <022> family would be ideal, and

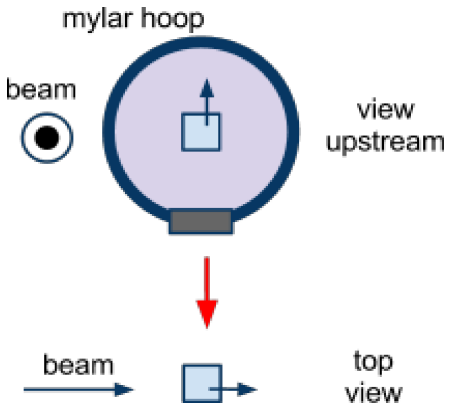
this can be done if we pick one that is not orthogonal to <100> like <202> or <220>.

Run 95

θ scan: 15.55° ↔ 15.93°; 100 steps

Diamond re-mounting record:

The diamond was remounted in such a way that the face seen looking upstream in transmission-mode scans now lies flat, seen from above, with its “up” orientation now pointing toward the camera. The adjacent figure shows this transformation.



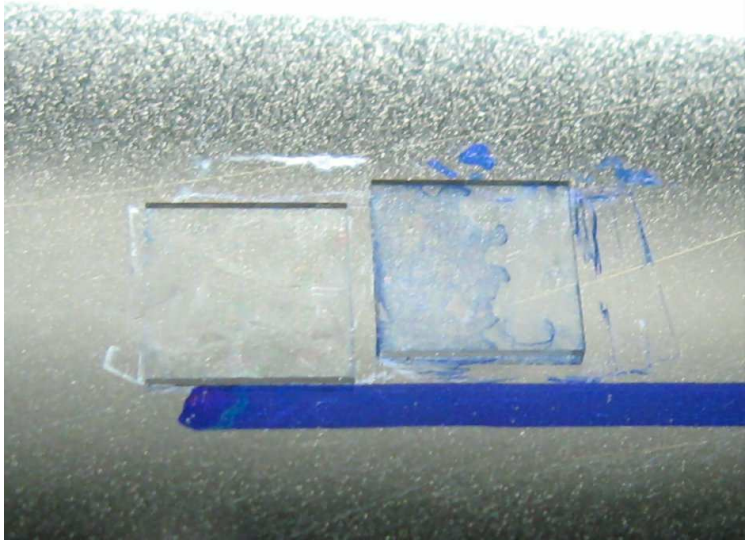
C1 computer crash

Attempting to do animate apparently too many files, the computer running fourc crashed. A restart was necessary, as well as some instruction from Ken as to how to get the proper settings back. There was also a problem of fourc see showing a proper scan and indicating the path to the capture file names but not such files were found there. Re-running `connect c1` on the ccd-controlling laptop to reconnect the directories of the two did the trick - apprantly the files just couldn’t be written over to this shared space.

300µm diamond scans

Run file prefix: `pristine`

Un-thinned, 300µm diamonds were inspected by Ken in the Laue chamber. The orientation of planes were exactly those of the previous diamond (i.e. see Laue spot projection above). The diamonds were difficult to pick up using the fiber/water technique, but seemed very thick and robust. Sweeping them from plastic backing of from their storage container onto



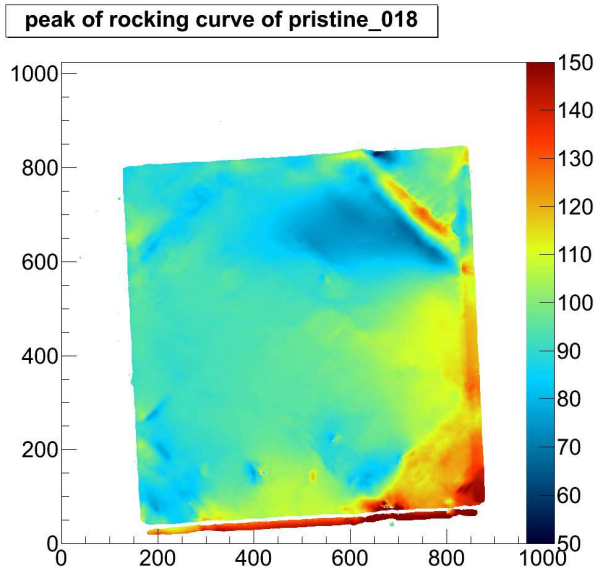
the mylar and adjusting them by pushing the edge did not seem to be a problem. Being so easy to mount and worrying about the time remaining in the run, both pristine samples were mounted together (shown in adjacent figure) in the hope that both can be spanned by the large beam and that their <220> spots will appear nearby (and not overlapping) for quick successive scans of each.

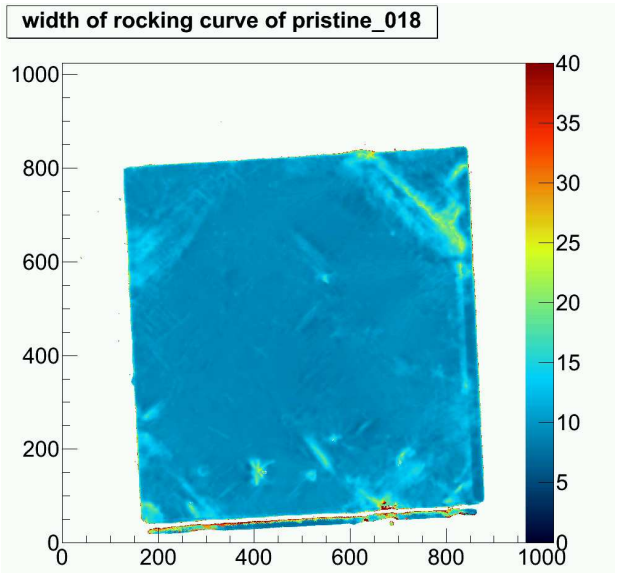
The following are coarse locations of their peaks (two values are given corresponding to peaks of each sample)

θ = 18.428, 17.895
χ = -85.66, -90.56
φ = 155.169
crude θ scan radius: 0.004°

Fine scan of pristine, 300μm diamond A

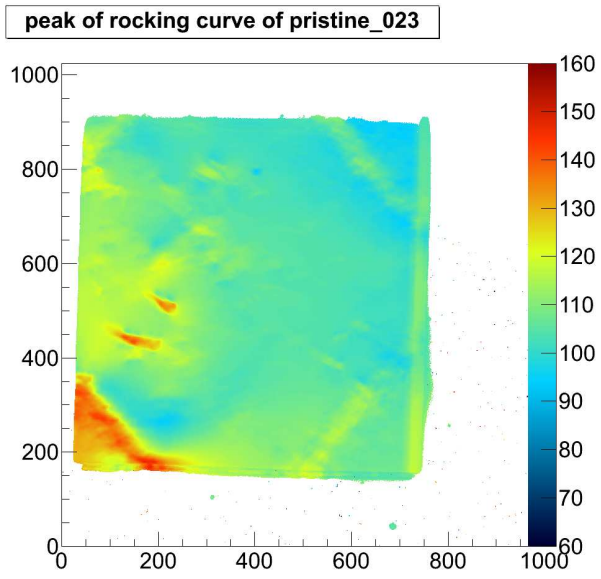
Run: 018
θ scan: 18.422° ↔ 18.432°; 50 steps (0.0002°~3.5μr steps)
χ = -86.26
φ = 155.169

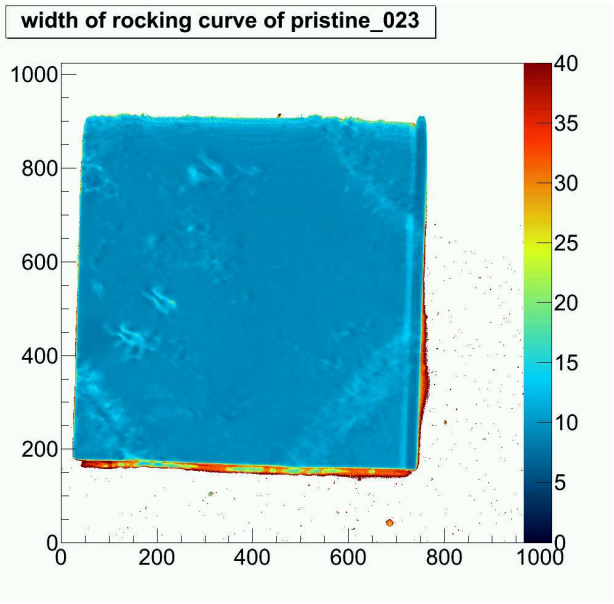




Fine scan of pristine, 300μm diamond B

Run: 023
θ scan: 17.8901° ↔ 17.9001°; 50 steps (0.0002°~3.5μr steps)
χ = -90.66
φ = 155.169





rotated the holder clock-wisely (along beam direction) by 90 degrees, continue with horizontal scan

Fine scan of pristine, 300μm diamond A (on bottom)

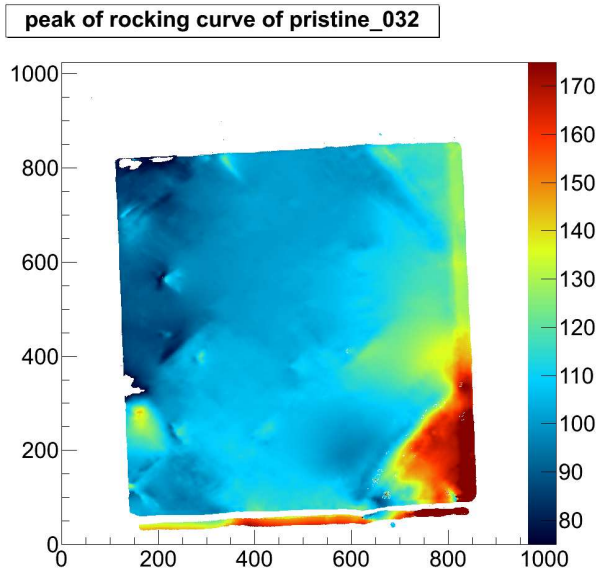
Run: 032

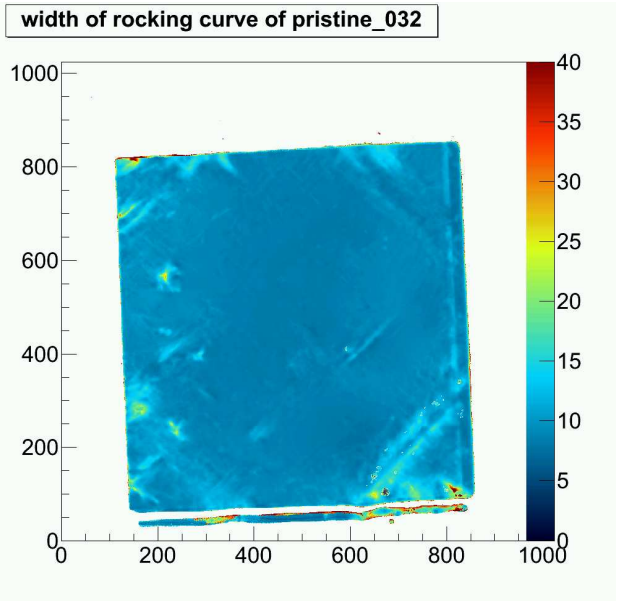
θ scan: 18.4886° ↔ 18.5016°; 65 steps (0.0002°~3.5μr steps)

2θ = 37.7°

χ = -104.1890°

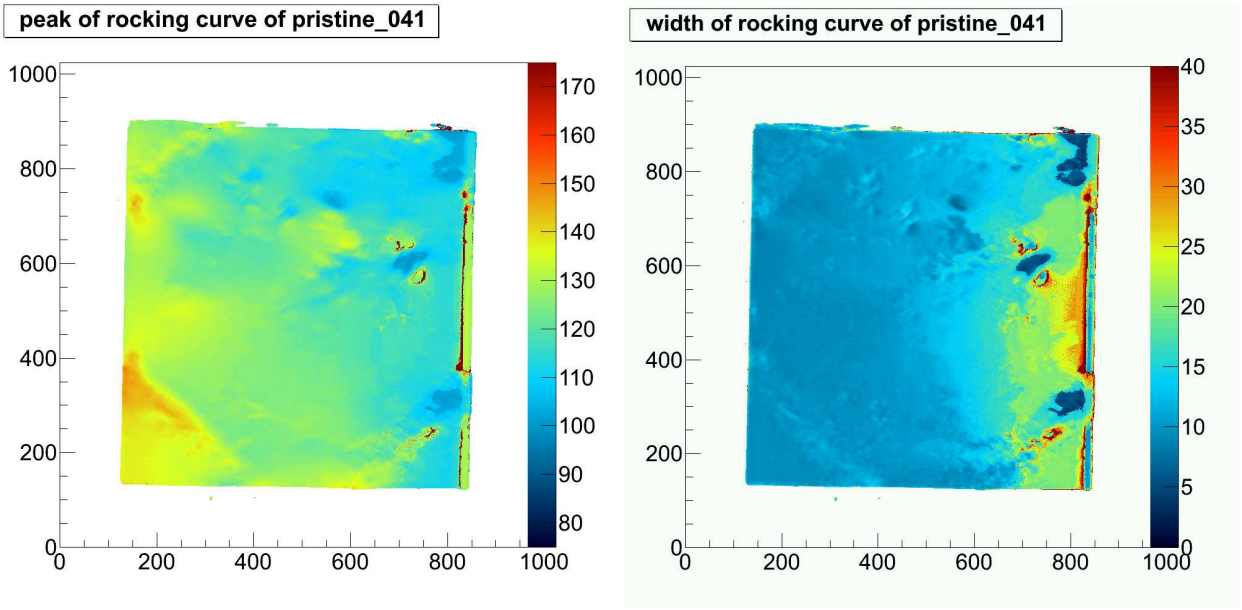
φ = 154.5690°





Fine scan of pristine, 300µm diamond B (on top)

Run: 041
θ scan: 19.9275° ↔ 19.9425°; 75 steps (0.0002°~3.5µr steps)
2θ = 38.72°
χ = -107.8890°
φ = 155.3590°



We removed the pristine diamonds from the sample holder and returned them to the box they were shipped in. The box has numbered slots in it. The slots were filled as follows.

- diamond A in slot #2
- diamond B in slot #3

SC Plate CVD Scan

Run prefix: `plate`

These SC plate CVDs have <100> normal to the surface. And we found out that that the edge of these diamonds has direction of <001>.
So the direction of <022> is in the plane and along the diagonal lines. The four samples identified as the location in the package

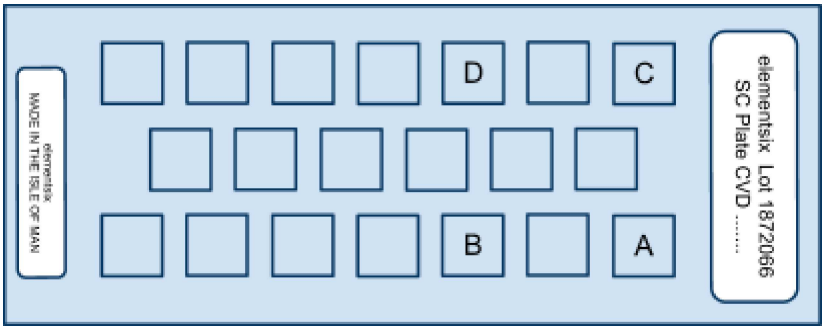
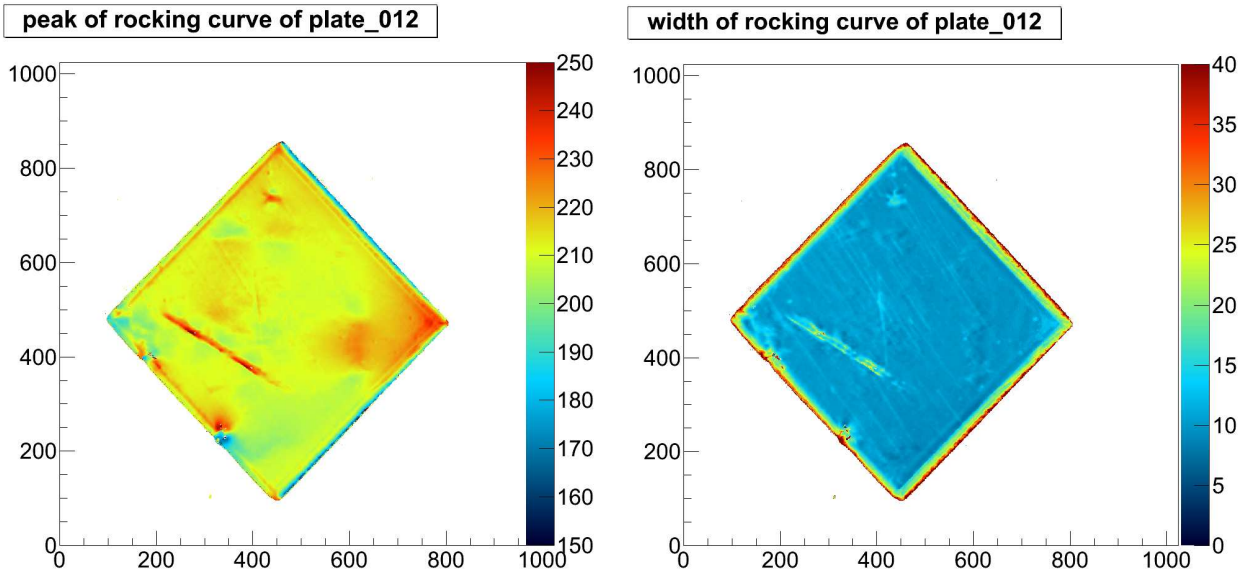


Plate A

Fine scan of plate A, vertical

Run: 012
θ scan: 19.387° ↔ 19.407°; 100 steps (0.0002°~3.5µr steps)

2θ = 38.52°
χ = -101.5°
φ = 155.359°



Fine scan of plate A, horizontal

Run: 017
θ scan: 19.3084° ↔ 19.3334°; 125 steps (0.0002°~3.5μr steps)
2θ = 38.02°
χ = -191.4°
φ = 155.359°

Plate B

Fine scan of plate B, vertical

Run: 027
θ scan: 18.632° ↔ ~~18.665°~~ 18.6524°; ~~165 steps~~ (0.0002°~3.5μr steps)
2θ = 38.42°
χ = -102.1°
φ = 155.359°

Beam lost on step 102

Run: 028
θ scan: 18.6524° ↔ 18.6650°; 63 steps (0.0002°~3.5μr steps)

Note: normalization change at step 26: monochromator was micro-adjusted - “machine warmup” changed after long down time.

Fine scan of plate B, horizontal

Run: 033
θ scan: 17.688° ↔ 17.721°; 165 steps (0.0002°~3.5μr steps)

2θ = 38.22°
χ = -191.6°
φ = 155.359°

Plate D

Fine scan of plate D, vertical

Run: 041
θ scan: 18.0726° ↔ 18.1026°; 150 steps (0.0002°~3.5μr steps)
2θ = 38.72°
χ = -93.90°
φ = 155.359°

Fine scan of plate D, vertical

Run: 049
θ scan: 18.5248° ↔ 18.5458°; 70 steps (0.0003°~5.2μr steps)
2θ = 38.52°
χ = -182.90°
φ = 155.359°

Plate C

Fine scan of plate C, vertical

Run: 057
θ scan: 17.6141° ↔ 17.6341°; 50 steps (0.0004°~7.0μr steps)
2θ = 38.42°
χ = -88.60°
φ = 155.059°

Fine scan of plate C, horizontal

Run: 079
θ scan: 16.289° ↔ 16.295°; ~~15~~ 11? steps (0.0004°~7.0μr steps)
2θ = 38.42°
χ = -88.60°
φ = 155.059°