Characterization of Diamond Samples -- CHESS Run Fall 2012

11/7-11/12/2012

Goals for this run

1. Learn how to use the new X-ray camera installed in the C-line endstation.
2. Measure the resolution of the topograph images taken with the new mono and camera configuration, using as a reference samples that were measured back in June, 2012
3. Take new rocking curves of the following samples in both x and y orientations:
   - **U40** (“dave”)
   - **S90** - intermediate thickness sample from Sinmat, vintage June 2012
   - **S30** - thin sample from Sinmat, vintage June 2012
   - **S40** - masked sample from Sinmat, vintage September, 2012. This one has a hole in the middle of the thinned area, which makes it less useful as a radiator, but we want to look at it with X-rays anyway to see what it looks like.
   - several samples from the Hall B collection. Ken Finkelstein was looking around in his cabinet, and he found a whole box of several crystals that he says were left

behind back in 2009 by Franz Klein. Several of these are mounted in an aluminum frame which makes it difficult for us to scan them, but we will try to find a way, since they might come in handy during commissioning of the Hall D polarized photon source.

4. Do systematic studies of how much the shape of U40 might depend on where it sits in the mount. Similarly for S30.
5. As soon as Brendan arrives from UConn with new ablated samples, measure these. This is the most important goal of the run, to see how repeatable is the success that we had with U40, and to extend the measurements down from 40 microns to 20.

1. Checking out the new camera

Nov. 7, 2012, RTJ, AEB

We arrived at CHESS and checked in around 2:30pm today. We learned about several changes that have taken place since our last visit. The most important one is that new rules have been put in place for access to the machine shop. Only people who have received training and approval by the machine shop supervisor are allowed to work in the shop. As of now, none of us has the required training. For the present, we will be content to route our requests to the machinists during regular working hours. Another change is that the machine now operates in “top-up” mode for the positron beam, which means that the positron current is maintained at a more or less constant level during a spill. The electron current still decays exponentially like it did before, and so one still has interruptions every couple hours so that a new batch of electrons can be stored.

Before we arrived, Ken had already set up the Si(331) monochromator for us. The beam in the hutch is 15keV, as usual. The spot is approximately 6mm wide and 3-4 mm high. The width is set by the positions of a north and south slits. The vertical slits are pulled back so that the vertical height of the beam is set by the diffraction condition at the mono.

For as long as I can remember (RTJ) the second crystal in our asymmetric Si(331) monochromator was mounted on a stage with a piezoelectric crystal fine-adjust that we could tweak to optimize the beam intensity profile at the target. Ken changed it this time around to save time when he has to switch over from our mono to the configuration required for the next user. So now we no longer have the piezo voltage to tweak, and have to step the MONOU and MONOD stepping motors by discrete amounts to adjust the vertical position of the beam. The MONOD motor has a resolution of 1/10000 degrees, which is pretty good, but the MONOU stage has a finer resolution of 1/40000. I find it is more convenient to adjust the MONOU stage when small adjustments are needed because of the smaller step size that is available.

Instructions for operating the data acquisition system for the new camera

Here are some instructions that Ken gave us for how to operate the data acquisition setup for the new camera.

- During alignment and orientation of the sample:
    - `FOURC> andor_lineup_on 0.5`
    - `FOURC> tw th 0.01 <return> <return> … CTRL-C`
    - `FOUR-C> tw chi 0.1 <return> <return> … CTRL-C`
    - `FOUR-C> andor_lineup_off`

  This command sequence puts the camera into a loop taking pictures with an exposure of 0.5 seconds. If the “Image J” application is running on the main DAQ screen, a new camera image is displayed every 2 seconds. It continues taking pictures at regular intervals while the user scans through various orientations, looking for the diffraction maxima. The final command turns off the camera acquisition loop, in preparation for taking a rocking curve -- next step.

- To record a rocking curve scan, recording an image at each angle:
    - `FOUR-C> ad_on`
    - `FOUR-C> dscan th -0.2 +0.2 40 2`
    - `FOUR-C> ad_off`

  This command sequence puts the camera into a mode where it waits for a signal from the fourc sequencer before taking its images. The arguments to dscan are <motor> <relative start angle> <relative stop angle> <number of steps> <exposure time in seconds>.

- To view acquired images in Image J:
    - Use File->Open to pick a particular image for display.
    - Locate the B&C window and press Auto to adjust the dynamic range of the displayed images.
    - To project the intensity along a horizontal axis, drag a box across the image and press Ctrl-k. A histogram of intensity vs pixel number shows up in a separate window.

- To control where the images are stored by the camera controls
    - `FOUR-C> ad_setup`

  This command prompts for a number of control values for the camera, including the directory in which rocking curve images are saved. Defaults are offered for all of the parameters that result in them retaining their former values.

First images of the beam spot with alignment pin silhouette

Now I am going to start the first of a set of scans and scan the MONU1 motor to center the beam on the alignment pin. The pin is in the chi=90,theta=0 degrees position, pointing straight up. The camera is at 2theta=0 degrees, viewing the shape of the beam with the pin silhouette showing up near the center of the image. Note the lines that criss-cross at various slopes across the gray region of the image. Ken Finkelstein explained these as surface features of the monochromator crystals.

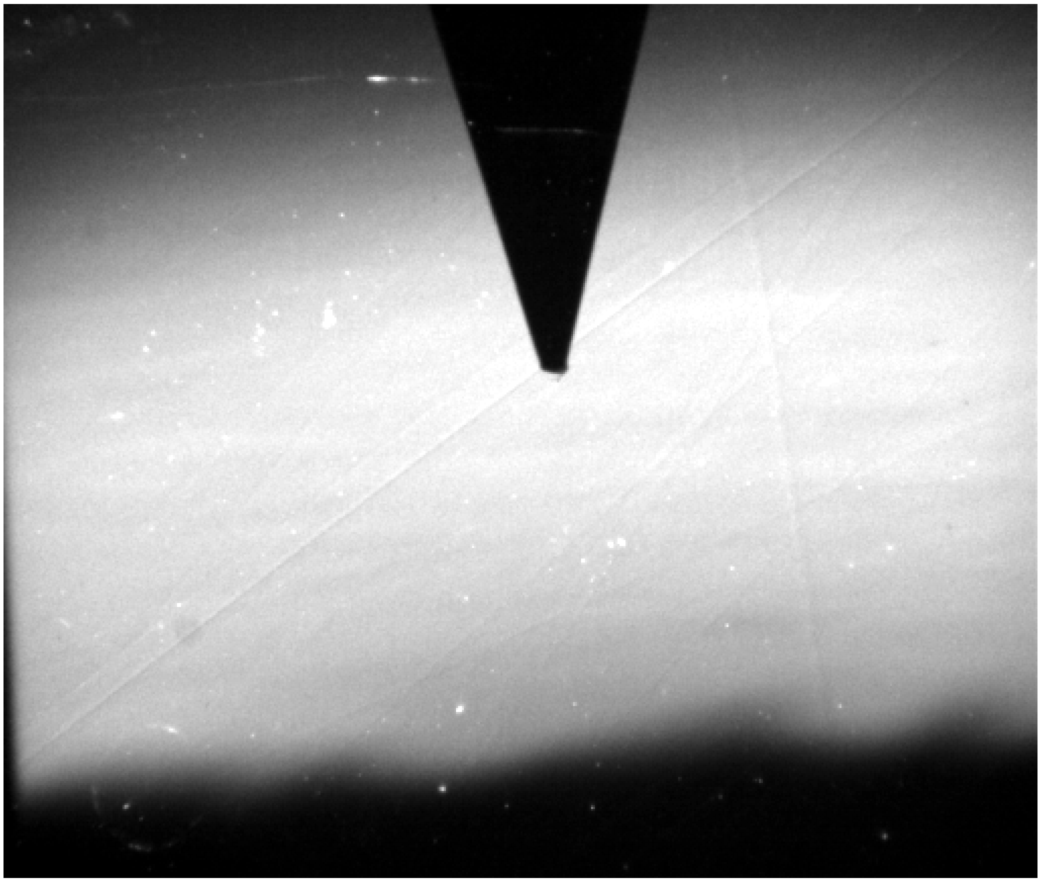
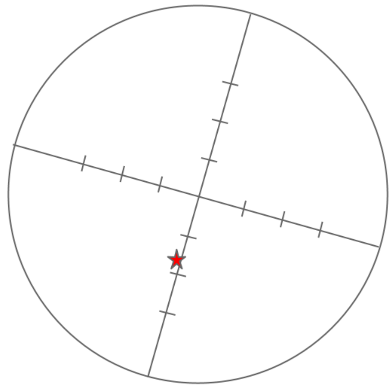


Image file setup110712_scan019_002.jpg: X-ray beam spot viewed directly by the camera at 0 degrees. The original image was 2560 x 2160 pixels, with each pixel being of dimension 6.5 x 6.5 microns, for a total image size of 16.6 x 14.0 mm^2.

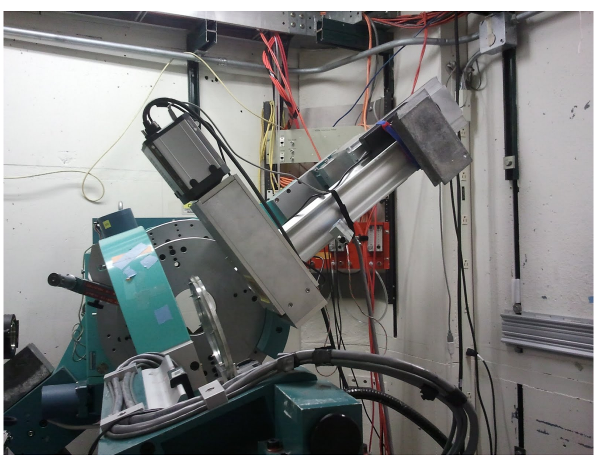


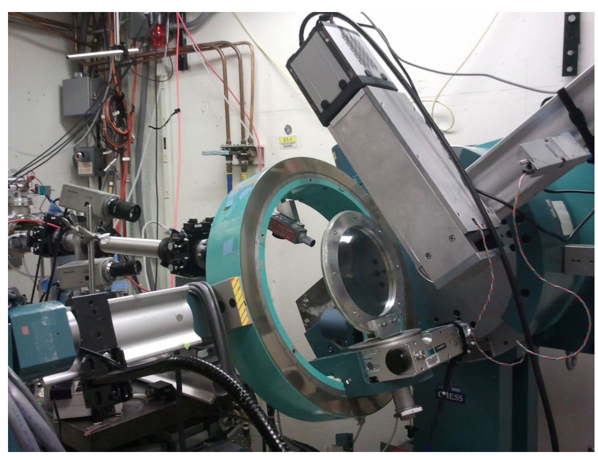
The position of the tip of the alignment pin, as viewed through the telescope mounted upstream of the four-circle axis. The pin appears inverted in the telescope eyepiece.

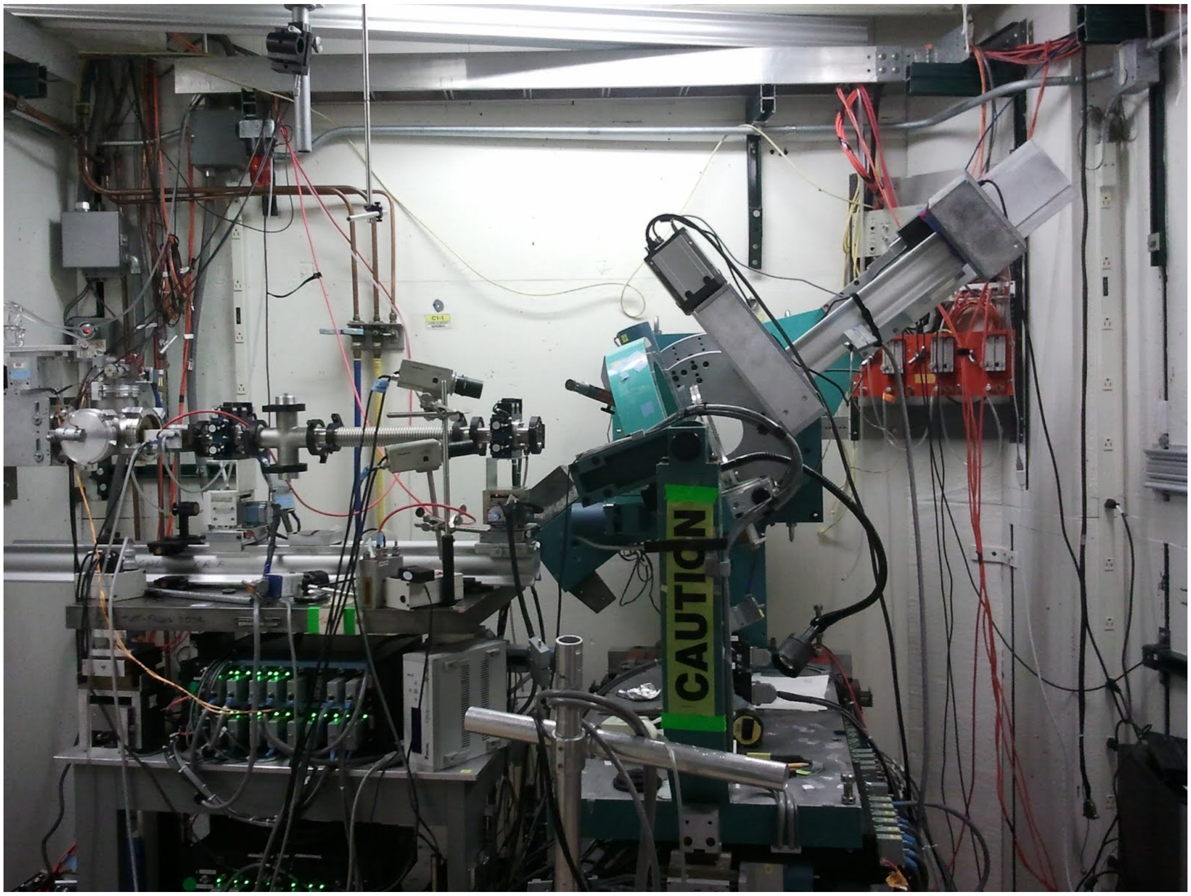
2. Scanning U40 (Dave)

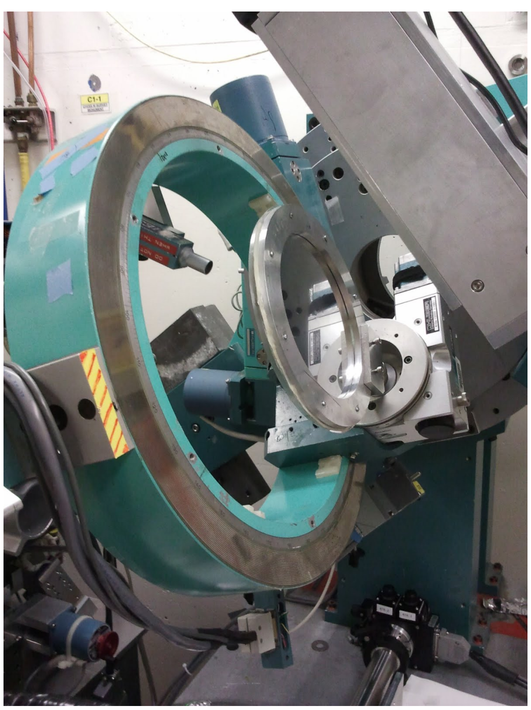
Nov. 8, 2012, RTJ, AEB

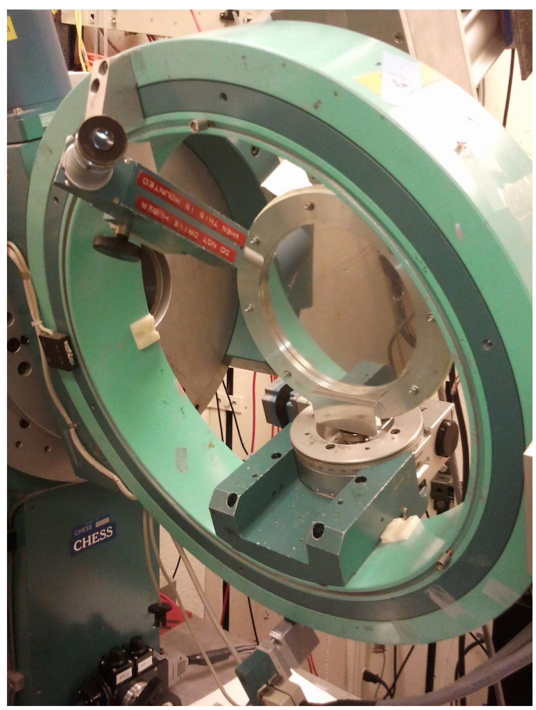
We mounted ‘Dave’ in the mylar and attached the drum as was done in June.

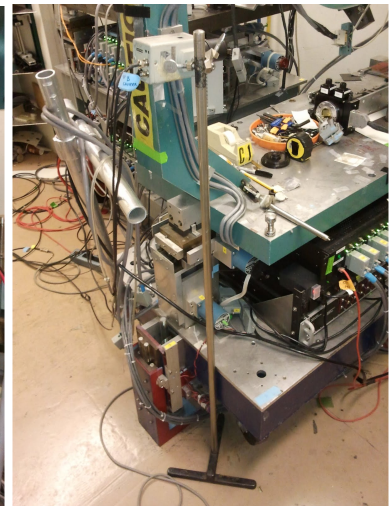




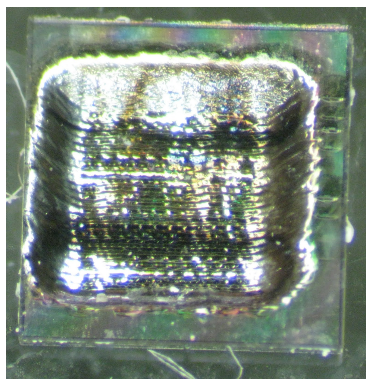

CAUTION




CHESS

Here is a picture of Dave taken with our optical microscope in lab 403.



Once ‘Dave’ was mounted, we attached fluorescent paper onto the camera window. Using a mirror we oriented a video camera to view the fluorescent paper in order to find our theta value. After finding a general theta value we removed the fluorescent paper and tweaked the chi and 2theta values to bring the diamond into the center of the image. The effective range of theta was found and a run was started.

- To make a new data file
    - FOUR-C> newfile <name>
    - Next Snapshot Number (1)? <return>

- ad_setup defaults
    - Detector EPICS PV prefix (ANDOR1)? <return>
    - Remote path for images (/home/specuser/Data/jones)? <return>
    - Next Snapshot Number (1)? <return>
    - Fast image-streaming mode (NO)? <return>
    - Enter maximum time for single exposure (28000)? <return>

Table of 'Dave' runs indicating the file name, crystal orientation, and face orientation

Filename	Crystal Orientation	Face orientation	Step size (degrees)
U40study01_scan001	2,2,0	front	0.0002
U40study02_scan001	2,2,0	front	0.0002
U40study03_scan001	2,2,0	front	0.0002
U40study04_scan001	2,2,0	front	0.0002
U40study05_scan001	2,-2,0	front	0.0002
U40study06_scan001	2,-2,0	front	0.0002
U40study06_scan002	2,-2,0	front	0.0002
U40study06_scan003	2,2,0	back	0.0002
U40study06_scan004	2,2,0	back	0.0002
U40study06_scan005	2,2,0	back	0.0002
U40study06_scan006	2,-2,0	back	0.0002
U40study06_scan007	2,-2,0	back	0.0002
U40study06_scan008	2,-2,0	back	0.0002

Using ad-setup we created a run U40study01 with a theta range of 18.725..18.745.

- To scan with absolute values:
  - ascan th 18.725 18.745 100 5

  The arguments to ascan are <motor> <absolute start angle> <absolute stop angle> <number of steps> <exposure time in seconds>.

The bottom half of the diamond did not light up so monu1 was adjusted. The beam intensity should be approximately 23,000.

AEB

A new file was created, U40study02, with a theta range 18.725..18.745. By the end of the scan the beam intensity had dropped by 2000. I readjusted monu1 and scanned 'Dave' again in this orientation into file U40study03. Each scan took approximately 11 minutes.

U40study03 started with a beam intensity of ~21000 and ended with ~154000. I'm (AEB) not sure why the intensity keeps dropping. After the last run I tweaked monu1 by 0.00005 (+) and had a beam intensity of ~20,600. In the course of 1 minute it has dropped to ~18,000. The current beam run is ending soon and may be the cause of this drop. I'm making one more scan of 'Dave' in this orientation but I'm going to tweak monu1 enough that instead of being at the max it will be slightly below and will increase during the run. This will be U40study04. The intensity started at ~21,700 and ended at ~22,200. This time the majority of the run had an intensity of ~22,400.

While the beams were down I reoriented 'Dave' in chi by 90 degrees. Following the procedure from before I used the fluorescent paper to help find a theta range. I then recorded U40study05 and U40study06.

Note: Ken informed me that I don't need to make a new file each time. Once a file is made it increments by adding filename_scanXXX. I made a new file each time because I thought it would overwrite otherwise.

The third scan of the 2,-2,0 front orientation of 'Dave' was recorded as U40study06_scan002.

The parameters before changing phi were: th = 19.557, chi = 48.72, phi = 91.00

I believe we have enough scans for this side of 'Dave' so I turned the drum 180 degrees by adjusting phi. I found a theta that would illuminate the fluorescent paper but once the paper was removed I could not see an image of the diamond. I looked through the scope and the diamond center was only slightly off from our reference point. I asked Ken for help and he explained that our 2,2,0 planes are not perpendicular to phi. This results in the reflection not going through to the camera but still illuminating the paper. To redirect the reflection into the camera, chi needs to be adjusted (moving the spot on the fluorescent paper to the right corresponds to moving chi in the negative direction). As chi is moved theta will also change. Adjust chi by 1 and then scanning theta by 0.002 until a spot can be seen. During this process beam was lost.

Beam is back up and we finished adjusting the diamond. On the backside the parameters are: tth = 38.0500, th = 18.6112, chi = 40.3200, and phi = 271.000

The first run on the backside of 'Dave' has been recorded as U40study06_scan003. The current was a little low so I adjusted monu1 but it didn't affect the intensity. I adjusted mond1 by 0.00005 and it brought the intensity back to 22,600. I then ran another scan. The intensity remained constant throughout the run. A third run was recorded for the back side 2,-2,0 orientation.

Chi has been adjusted to 130.3200 to look at the 2,2,0 back-side orientation.

RTJ, AEB

For the third run (U40study06_scan008) we lengthened the exposure time to 30 seconds.

3. Scans of S40

Nov. 9, 2012, AEB

I mounted Sinmat 40 (the one with the hole). Once again the beam intensity keeps dropping over time. I set chi = 135 and swept theta from 15 to 23 and didn't see anything. Initially theta was set to 0 for mounting and when I swept from 0 to 15 I noticed something but my assumption is that this is not a 2,2,0 reflection. I changed chi by -1 degree and swept through theta again. I continued this until chi = 130 and then tried going the other direction from 135. I adjusted chi in 1 degree increments to 140 while scanning theta and did not see a reflection. I moved chi = 40 and swept through theta. I couldn't find anything between chi = 40..50.

I went back to 135 and swept theta to 0 to find the one reflection I had seen. It occurs at chi = 135, th = 3.090, psi = 271.0000, tth = 38.0110. Due to the low theta value I believe this is not actually 2,2,0. When chi = 140 this reflection appears at theta = 3.660 but on the opposite side of the screen.

I continued to scan for a reflection and I found another at chi = 170 theta = 18.654. This theta corresponds to theta values we found for 'Dave' but the chi orientation is 40 degrees different from 'Dave'. I think this could be a 2,2,0 reflection but it might not be. Ken explained to me the different Bragg planes and orientations and said that this is most likely the 2,2,0 but that the diamond had been oriented differently than our other diamonds. We weren't able to understand where the reflection at chi = 135 theta = 3.090 comes from.

The reflection found at chi = 170 theta = 18.654 was too far to the right for the camera to view. To bring the reflection into the camera window chi had to be increased and theta had to be decreased. The parameters are now chi = 181.4 and theta = 18.237.

Due to our mirror setup we cannot view a large amount of chi angles below 130.

A new file was made called S40study01. The video camera was repositioned in order to have chi = 91.4. For S40study01_scan004 the parameters are tth = 38.021, th = 17.0727, chi = 91.4, phi = 271.

Phi was turned to 91 in order to see the backside of the diamond. The new parameter values are tth = 37.9610, th = 20.3181, chi = 87.8, phi = 91.

Once the first set of backsides images were taken the diamond was rotated in chi. The new values are: tth = 37.891, th = 18.2539, chi = 177.8, phi = 81. S40study01_scan012 had a dropping beam intensity. The run also froze on step 222 and I had to ctrl-c. Because Brendan will be arriving shortly with new diamonds I decided not to redo that run and instead move on to S90.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
S40study01_scan001	2,2,0	front	0.0002
S40study01_scan002	2,2,0	front	0.0002
S40study01_scan003	2,2,0	front	0.0002
S40study01_scan004	2,-2,0	front	0.0002
S40study01_scan005	2,-2,0	front	0.0002
S40study01_scan006	2,-2,0	front	0.0002
S40study01_scan007	2,-2,0	back	0.0002
S40study01_scan008	2,-2,0	back	0.0002
S40study01_scan009	2,-2,0	back	0.0002
S40study01_scan010	2,2,0	back	0.0002
S40study01_scan011	2,2,0	back	0.0002
S40study01_scan012	2,2,0	back	0.0002

4. Scans of S90

I exchanged sinmat40 for sinmat90. Once mounted I oriented the diamond such that the diagonal was pointing up. After scanning through theta the new parameter values are: tth = 40.591, th = 18.030, chi = 128.500, phi = 91.00. I changed chi to get the 2,-2,0 reflection. The new values are: tth = 40.591, th = 18.977, chi = 38.19, phi = 91.00. Adjusting to see the back the new values are: tth = 40.291, th = 19.327, chi = 50.19, phi = 271.00. For the final orientation the parameter values are: tth = 40.391, th = 18.4027, chi = 140.49, phi = 271.00

This diamond is too big for the camera, not all of it fits.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)

S90study01_scan001	2,2,0	front	0.0002
S90study01_scan002	2,2,0	front	0.0002
S90study01_scan003	2,2,0	front	0.0002
S90study01_scan004	2,-2,0	front	0.0002
S90study01_scan005	2,-2,0	front	0.0002
S90study01_scan006	2,-2,0	front	0.0002
S90study01_scan007	2,-2,0	back	0.0002
S90study01_scan008	2,-2,0	back	0.0002
S90study01_scan009	2,-2,0	back	0.0002
S90study01_scan010	2,2,0	back	0.0002
S90study01_scan011	2,2,0	back	0.0002
S90study01_scan012	2,2,0	back	0.0002

5. Scans of old Hall B diamonds

Exchanged sinmat90 for J2a100.

The JLab diamonds come in 3 different varieties and I have labeled them as such: **J2a100**, **J2a50**, **J1a50**. J stands for JLab, 1a and 2a are the different types, and 50 and 100 are their thickness in microns.

The parameter values for J2a100 are: tth = 40.3910, th = 17.5607, chi = 93.75, phi = 271. The first scan J2a100_scan001 was aborted due to a computer error. The images were not updating and I could not find the images it claimed to have taken. I shortened the range a little to save some time. J2a100_scan002 shows in the directory but not _scan001.

Brendan arrived with the new diamonds so we stopped scanning J2a100 and mounted Bob.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
J2a100_scan002	2,2,0	front	0.0002

6. Scans of U50 (Bob)

Nov. 10, 2012, BJP, AEB

Bob was mounted so the diagonal of the diamond pointed up. Parameters: tth = 40.7910, th = 19.4901, chi = 88.3, phi = 271. Bobstudy01_scan0002 is void because it overlapped into a beam injection. Scan006 froze at step 84. Do not understand why.

Rotated in chi to find 2,-2,0. New parameters are: tth = 40.741, th = 16.2967, chi = 178.15, phi = 271.

Rotated phi to look at the backside. New values are: tth = 40.741, th = 17.3273, chi = 180.45, phi = 90

Rotated in chi to get the last orientation. New values are: tth = 40.791, th = 17.6284, chi = 90.7, phi = 90.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
Bobstudy01_scan001	2,2,0	front	0.0002
~~Bobstudy01_scan002~~	~~2,2,0~~	~~front~~	~~0.0002~~
Bobstudy01_scan003	2,2,0	front	0.0002
Bobstudy01_scan004	2,2,0	front	0.0002
Bobstudy01_scan005	2,-2,0	front	0.0002
~~Bobstudy01_scan006~~	~~2,-2,0~~	~~front~~	~~0.0002~~
Bobstudy02_scan001	2,-2,0	front	0.0002
Bobstudy02_scan002	2,-2,0	front	0.0002
Bobstudy02_scan003	2,-2,0	back	0.0002
Bobstudy02_scan004	2,-2,0	back	0.0002
Bobstudy02_scan005	2,-2,0	back	0.0002
Bobstudy02_scan006	2,2,0	back	0.0002

Bobstudy02_scan007	2,2,0	back	0.0002
Bobstudy02_scan008	2,2,0	back	0.0002

7. Scans of U60 (Casey)

Exchanged Bob for Casey. New values: tth = 40.7910, th = 18.823, chi = 89.5, phi = 90. Rotated Casey by 90 in chi. New values: tth = 40.321, th = 19.4134, chi = 184, phi = 90. Rotated phi 180. New values: tth = 40.351, th = 19.409, chi = 184.3, phi = 270. Rotated chi by 90. New values: tth = 40.401, th = 18.6523, chi = 94.3, phi = 270.

Filename	**Crystal Orientation**	**Face Orientation**	**Step Size (degrees)**
Caseystudy01_scan001	2,2,0	front	0.0002
Caseystudy01_scan002	2,2,0	front	0.0002
Caseystudy01_scan003	2,2,0	front	0.0002
Caseystudy01_scan004	2,-2,0	front	0.0002
Caseystudy01_scan005	2,-2,0	front	0.0002
Caseystudy01_scan006	2,-2,0	front	0.0002
Caseystudy01_scan007	2,-2,0	back	0.0002
Caseystudy01_scan008	2,-2,0	back	0.0002
Caseystudy01_scan009	2,-2,0	back	0.0002
Caseystudy01_scan010	2,2,0	back	0.0002
Caseystudy01_scan011	2,2,0	back	0.0002
Caseystudy01_scan012	2,2,0	back	0.0002

8. Scans of Hall B inventory, take 2

Exchanged Casey for J2a100. I oriented the diamond so that the diagonal pointed up.

New values: tth = 40.751, th = 19.7403, chi = 94.95, phi = 270. To save time in case we need to do any rescanning I am only going to do 2 scans per orientation rather than 3.

Rotated chi by 90. New values: tth = 40.6310, th = 19.966, chi = 184.95, phi = 270.
This orientation has a very wide range in theta, 19.856 - 20.076. A scan will take 1:50 so I will only do one scan for this orientation.

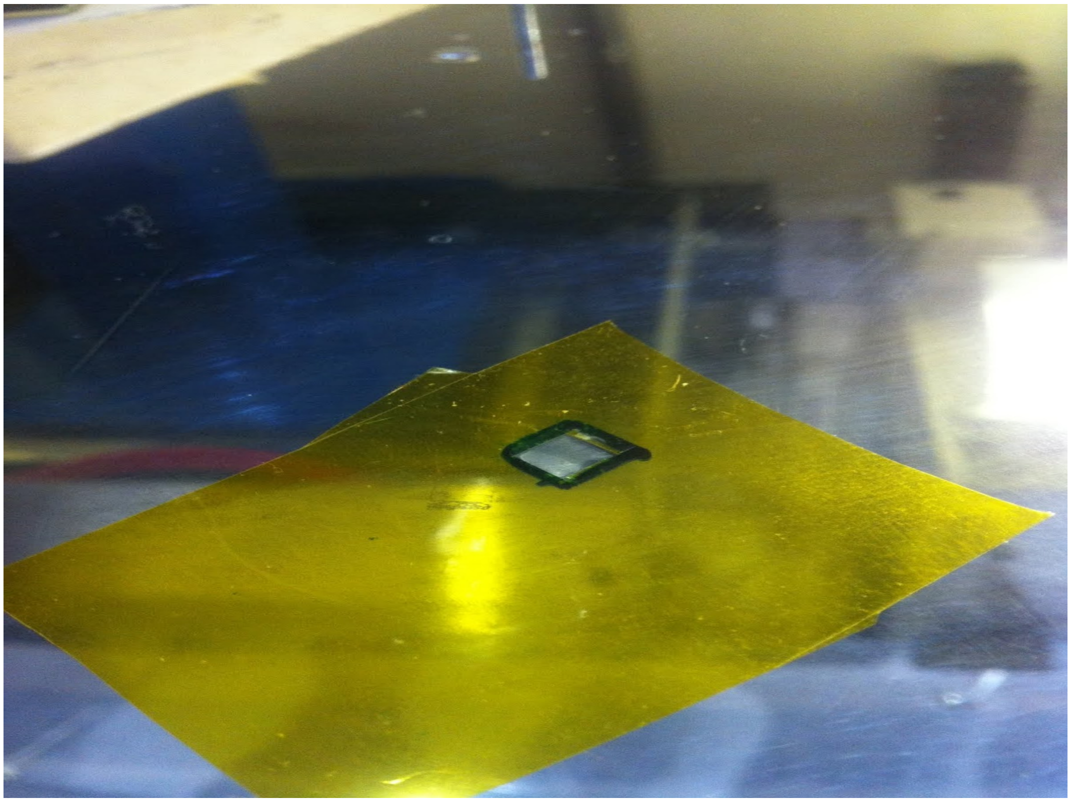
Rotated phi. New values: tth = 40.131, th = 19.999, chi = 182.96, phi = 90

Nov. 10, 2012, BJP, AEB
Hello world. I'm continuing Alex's runs of J2a100study with a rotation in chi as previously stated.
Past scan finished and I'm back in the saddle finding the next peak at chi=93.2, starting a scan.
Rotated chi. New values: tth = 40.481, th = 17.6556, chi = 93.28, phi = 90

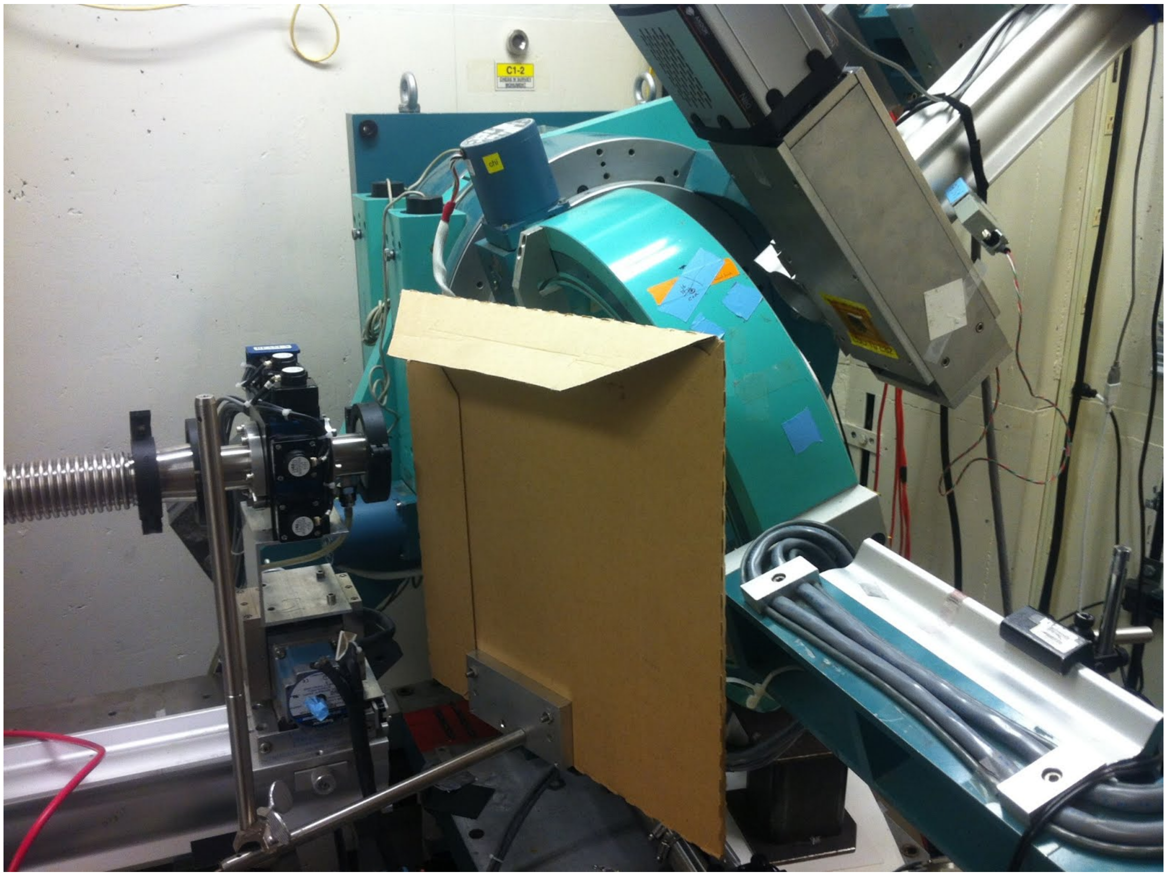
Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
J2a100study02_scan001	2,2,0	front	0.0002
J2a100study02_scan002	2,2,0	front	0.0002
~~J2a100study02_scan003~~			
J2a100study02_scan004	2,-2,0	front	0.0002
J2a100study02_scan005	2,-2,0	back	0.0002
J2a100study02_scan006	2,2,0	back	0.0002

Scan completed I will now mount J1a50 and follow Alex's lead with front and back scans of the 2,2,0 and 2,-2,0 orientations.

Ran into a familiar problem when trying to mount J1a50 (assumingly 50microns). The diamond slips between the mylar sheets and I was lucky enough to find it sitting on the edge of the hoop. So, I created a frame for it to rest on out of thin kapton sheets (like before) and it holds the diamond in place (for now).



I positioned the diamond with its corners pointing to the vertical and horizontal axis and had to rotate chi by 45 to find the 2,2,0 peak. Also, once the peak was established, I watched as it’s intensity decreased (on the fluorescent paper), Ken came over (perfect timing) and suggested vibrations from air flow. We turned off the air conditioner and the image maintained its intensity over a period of a minute, so we think that solved it. But, the air conditioner needs to be on, so we are devising an air dam to stop the vibration.



Everything looks good, ready to scan...

J1a50Study01_001 values: tth = 40.311, th = 19.271, chi =138.08, phi = 90

Rotated chi by 90 and found the peak, running the following scan.

J1a50Study01_002 values: tth = 40.111, th = 18.233, chi =48.6, phi = 90

Since scans are so quick I decided to do a total of 3 per orientation. I will go back and do 2 more scans for the 2,2,0, sorry for the confusion.

J1a50Study01_05...07 values tth = 39.9610 th = 19.604 chi = 135.86 phi = 90

Rotated in phi
J1a50Study01_07...10 values tth = 40.361 th = 18.9402 chi = 142.5 phi = 270.0

Tweaked Chi by 90 and will take 3 scans of the 2,-2,0 orientation for the back
J1a50Study01_011..013 values tth = 40.161 th = 17.598 chi = 52.26 phi = 270.0

File Name	Crystal Orientation	Face Orientation	Step Size (mm)
J1a50study01_scan001	2,2,0	front	0.0002
J1a50study01_scan002	2,-2,0	front	0.0002
J1a50study01_scan003	2,-2,0	front	0.0002
J1a50study01_scan004	2,-2,0	front	0.0002
J1a50study01_scan005	2,2,0	front	0.0002
~~J1a50study01_scan006~~	2,2,0	front	0.0002
J1a50study01_scan007	2,2,0	front	0.0002
J1a50study01_scan008	2,2,0	back	0.0002
J1a50study01_scan009	2,2,0	back	0.0002
J1a50study01_scan010	2,2,0	back	0.0002
J1a50study01_scan0011	2,-2,0	back	0.0002
J1a50study01_scan012	2,-2,0	back	0.0002
J1a50study01_scan013	2,-2,0	back	0.0002

Brendan exchanged J1a50 for J2a50. The diagonal of the diamond is pointed up. New values: tth = 40.521, th = 20.573, chi = 133, phi = 270. The theta range is large, 20.423 - 20.743 (0.32). With a step size of 0.0002 it will take 1600 steps. This is a very long scan (2:40:06) so we will not scan 3 times per orientation.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
J2a50study01_scan001	2,2,0	front	0.0002

The 2,-2,0 orientation for J2a50 would take far too long (theta spread of 0.55 degrees, 2700 steps) so we decided to switch diamonds to S150.

9. Scans of S150 (central region thinned with laser)

New values: tth 40.5210, th = 18.5970, chi = 99.8, phi = 270. Rotated chi by 90. New values: tth = 40.021, th = 20.127, chi = 189, phi = 270. There was a beam dump during scan006. Rotated phi 180. New values: tth = 40.421, th = 20.1791, chi = 178.2, phi = 90. Rotated chi by 90. New values: tth = 40.421, th = 18.7863, chi = 88.832, phi = 90. Scan012 was aborted because the beam intensity decreased below 10,000.

Filename	Crystal Orientation	Face Orientation	Step Size (degrees)
S150study01_scan001	2,2,0	front	0.0002
S150study01_scan002	2,2,0	front	0.0002
S150study01_scan003	2,2,0	front	0.0002
S150study01_scan004	2,-2,0	front	0.0002
S150study01_scan005	2,-2,0	front	0.0002
~~S150study01_scan006~~	~~2,-2,0~~	~~front~~	~~0.0002~~
S150study01_scan007	2,-2,0	front	0.0002
S150study01_scan008	2,-2,0	back	0.0002
S150study01_scan009	2,-2,0	back	0.0002
S150study01_scan010	2,-2,0	back	0.0002
S150study01_scan011	2,2,0	back	0.0002
~~S150study01_scan012~~	~~2,2,0~~	~~back~~	~~0.0002~~
S150study01_scan013	2,2,0	back	0.0002
S150study01_scan014	2,2,0	back	0.0002

Offline Analysis

Resolution of Camera Images Reconsidered

4/19/2013 - *RTJ, BJP*
Above under the caption for the image of the alignment pin, we wrote that the pixel size in the images is 6.5x6.5microns. This appears to be the camera resolution, but between the detector and the camera is a lens that varies in magnification from x1 to x5. To find the true pixel size we compared the Zygo image of sample S90 with the X-ray images from S90study01 scan 7. The measured pixel width of the S90 was 2132 ± 7 pixels. Physically this corresponds to 4.55 ± 0.05 mm. Therefore the resolution turns out to be 2.13 ± 0.02 microns/pixel. The file rcpicker.C was edited to reflect the corrected image resolution. The lens magnification factor was 3.05 ± 0.03 during the November, 2013 run. Next run we should request that the lens factor be reduced to the range 1.5 - 2.0 so that the sample images are not clipped at the corners.

Extracting a 2d shape from orthogonal topographs

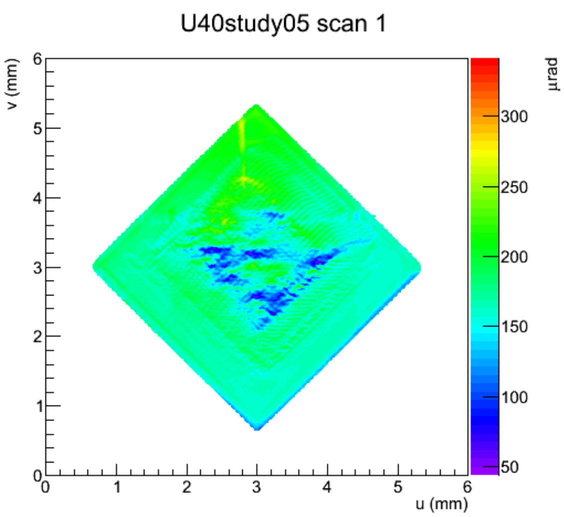
4/22/2013 - *RTJ*
The steps to prepare a pair of orthogonal topographs for shape analysis are as follows. I chose the orthogonal pair U40study04 and U40study05 to use while prototyping the analysis procedure.

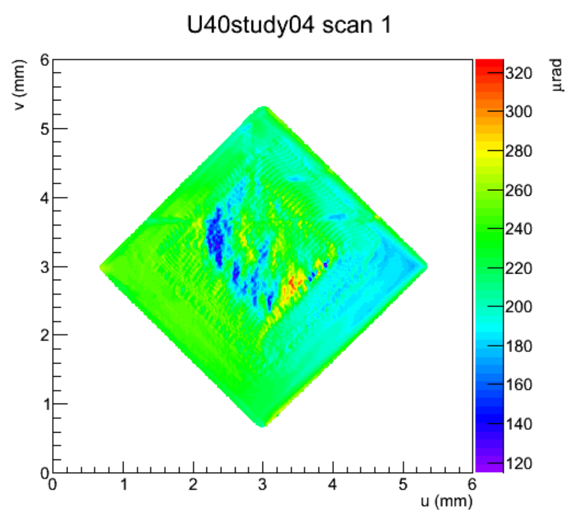
1. Start with the rocking curve mean (peak angle) 2d histogram images for the (2,2,0) and (2,-2,0) scans. Make sure the entire crystal is visible in both images.
2. Rescale both images in the vertical (v) direction by 1/cos(theta) to correct for the projection onto the detector plane. Nominally theta should be the Bragg angle (19.2° for diamond 2,2,0). I found that with 21.5° I was able to obtain right angles in the corners of the images of square diamonds. This might be explained by a slight aspect ratio asymmetry in the optics of the detector. There is also a small offset of a couple degrees between the 0,0,1 direction and the normal to the crystal surface, but that offset would be different for the 2,2,0 and 2,-2,0 scan directions. Within error, I found that the angle 21.5° is the correct value for all of the images in the U40study scanset.
3. Rotate one of the pair by 90 degrees so as to make the two coincide in sample orientation. Now the scan direction is horizontal for the rotated map. If “up” in the original unrotated image is now pointing to the left in the rotated image, the rocking curve data need to be inverted to make the “up” direction point to the right along +x.
4. Overlay the two images to make sure that they are optimally aligned with one another. Make any small adjustments necessary to bring the edges into alignment.

Here is the log of a root session that carries out the above steps for U40study04 and U40study05.

```
[jonesrt@gluey Analysis]$ root -l
root [0] .L Map2D.cc+O
root [1] .L rcpicker.C+O
root [2] .L rockmean.C
root [3] .L overlay.C
root [4] Map2D *u4=rockmean("U40study04",1)
root [5] Map2D *u5=rockmean("U40study05",1)
root [6] c1->Print("scan1_5.png");
root [7] u4->Rescale(1,1,-1)
root [8] u4->Shift(0,0,400)
root [9] u4->Normalize()
root [10] u4->SetContour(100)
root [11] u4->Draw("colz");
root [12] c1->Print("scan1_4rot.png");
```

The results of the above root session are shown in the following graphs.

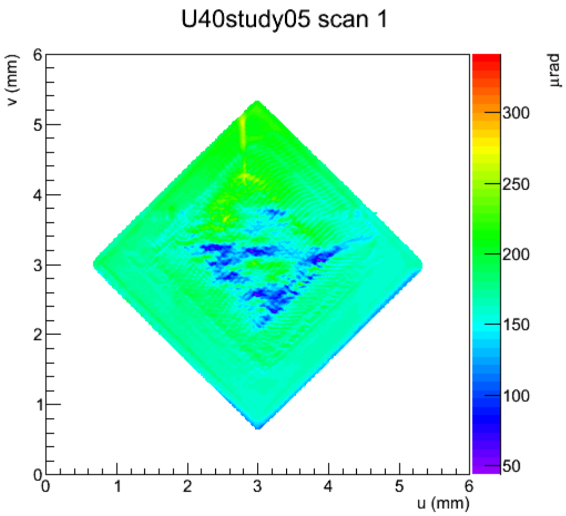


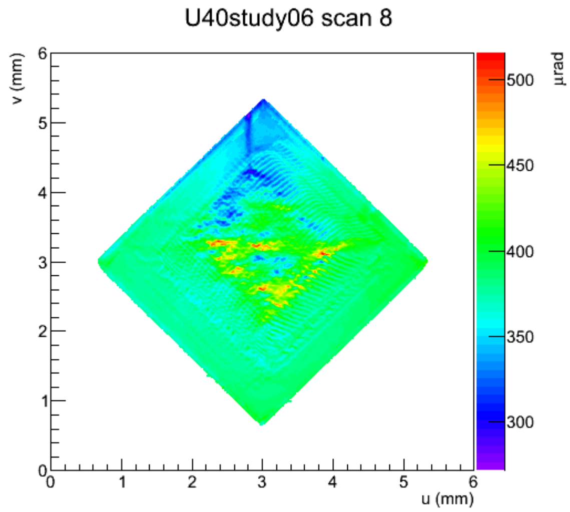


The scan direction in the left image is up, and in the right image it is to the right. One can see from a visual comparison of the shape features in the two images above that they are the same crystal in the same (post-rotation) orientation, even though the color features of the two scans are quite different. Nevertheless there are faint traces of certain crystal defects and artifacts of the laser cuts that show the two images are in the same orientation.

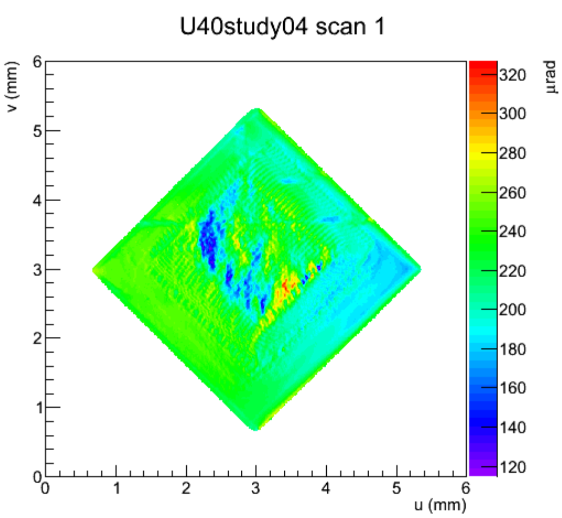
In addition to the finer features, both images show a gradient in the mean peak angle along the scan direction. However in the left image the gradient is positive and in the right image it is negative. I take this to mean that the curvature is not a feature of the beam, but it must belong to the sample. If this is real, is a saddle-shaped distortion of the outer frame of the diamond. It is not of concern because the magnitude is less than that of the local variations in the central region of the crystal, but it is worthy of note.

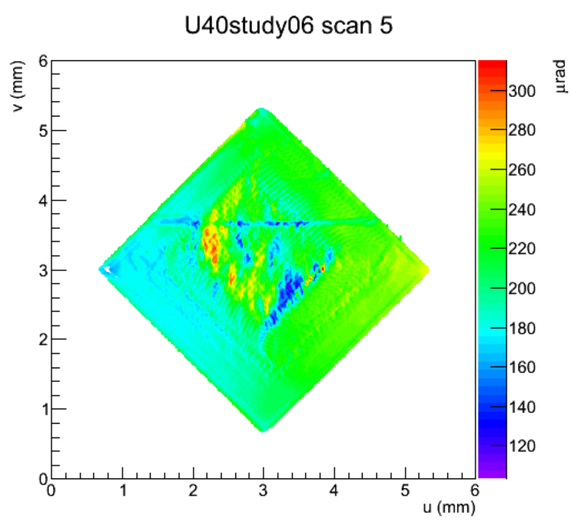
Now to see how much of the observed variation in the peak angle is due to residual warpage and how much due to other defects such as d-spacing variation, I compare the “front” and “back” images of the same scans. Again I look at the U40study scans and always transform the images to present them in the same orientation as shown above.





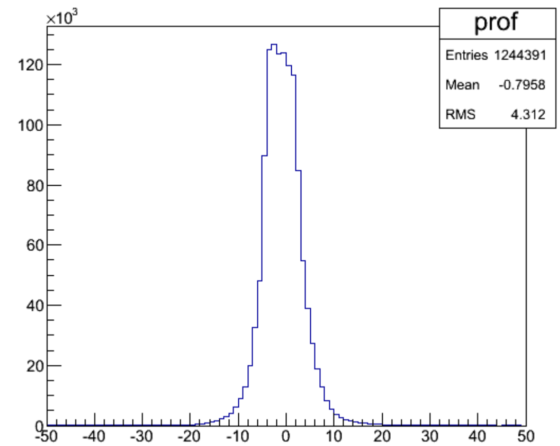
Notice that in the study6/scan8 image the background gradient has reversed direction, as expected if it is due to an external bowing of the sample in the vertical direction.





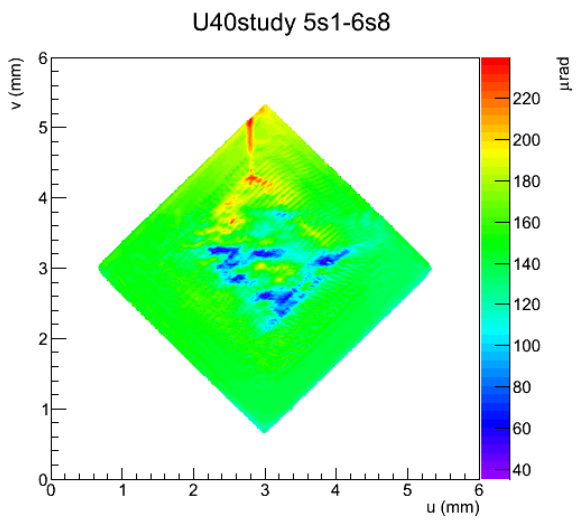
The correlation factors between the pairs are -0.77 (top pair) and -0.74 (bottom pair). This is not a strong correlation, but it confirms the qualitative visual impression that the two pairs of images are complementary to one another. This complementarity is what one would expect if the shifts were due to warpage of the crystal planes (parity-odd) rather than d-spacing changes (parity-even).

By contrast, the correlation between two images from repeated scans of the same crystal orientation are on the order of 98%. The single-pixel error estimated by the difference between the peak angles extracted from two repeated scans is shown in the following histogram. All pixels within the diffracted image are included in the histogram.

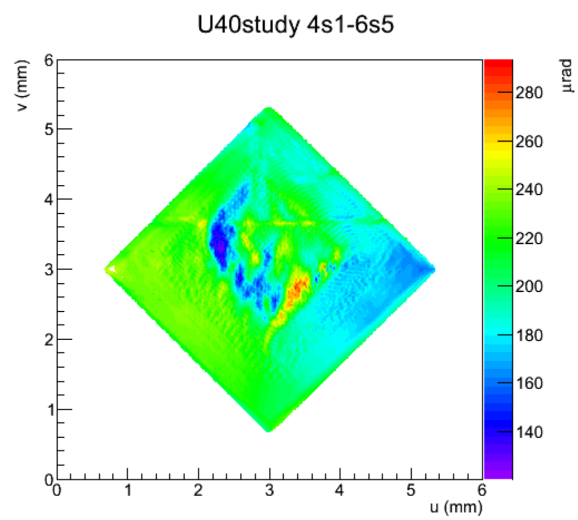


Single-pixel rocking curve peak centroid error distribution extracted from U40study06 scans 6 and 7 by taking the difference between all illuminated pixels in the two images.

To interpret the peak shifts as tilt of the planes, I take the difference between the front and back images in each scan direction shown above and divide the result by two. The new pair is plotted below, with their central values shifted away from zero so they display on a white background.

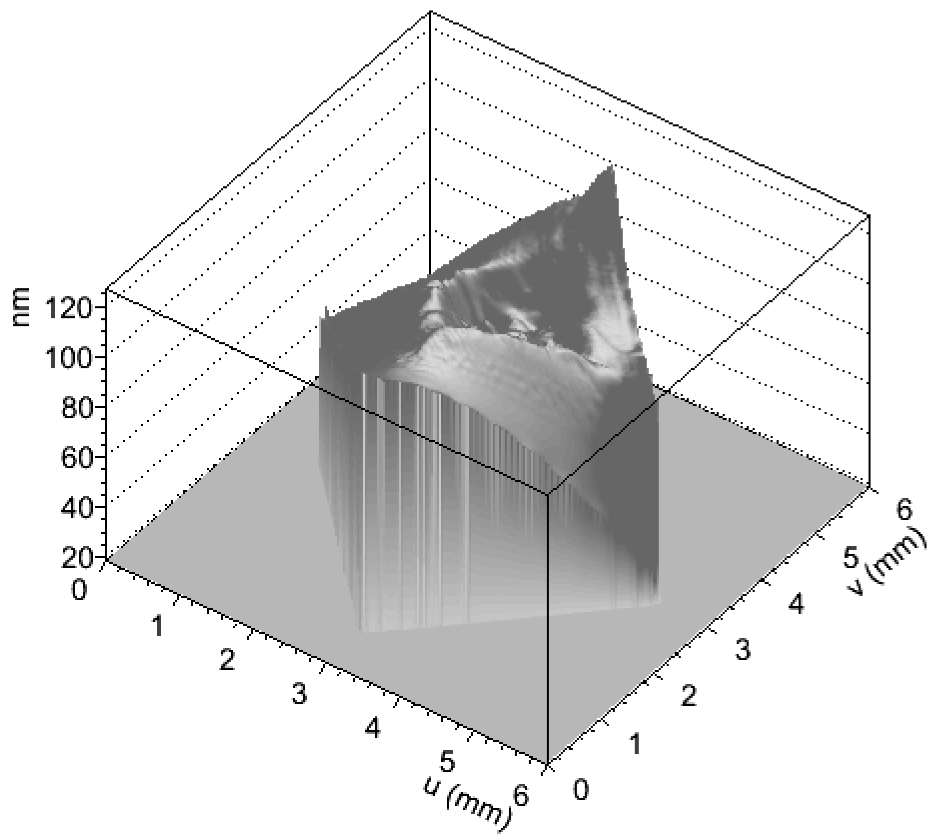


tilt angle along vertical direction



tilt angle along horizontal direction

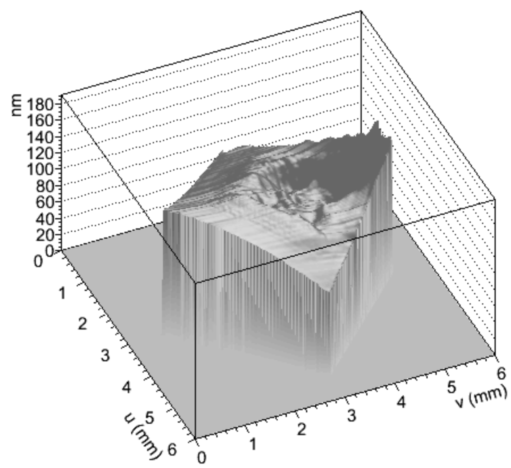
The two images are very nearly aligned, but there are small differences in the boundaries that are eliminated by masking the images with each other's silhouette. Once this is done, a path integral is performed around the outer perimeter of the vector field formed by the two images. The Map2D class has a method TotalCurl(gx,gy) to find this path and compute the path integral. The result for the above images is 52nm. To see the error on this value, I mask off the cells in the gx,gy maps that formed the outer path and created a new one just inside those. Now the path integral is -53nm. Repeating gives a consistent answer with -53nm, which shows that the result has converged once the pixels along the ragged edge have been trimmed away. With a zero curl, the maps are now ready to be integrated to find the height profile of the planes.

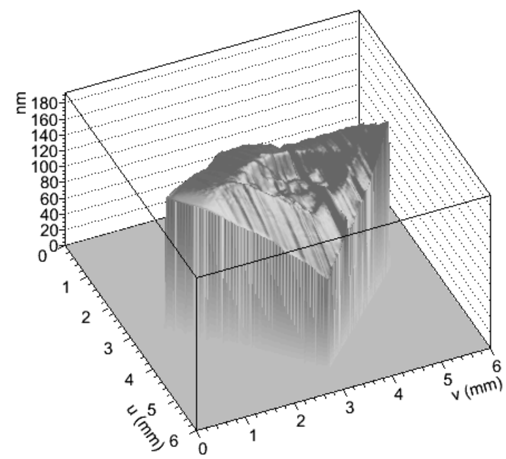


The general saddle-point profile of the diamond frame is apparent in this integrated height profile image of U40. The strains within the central cut-out region are also visible. This image is 250x250 pixels, reduced from the original image dimensions of 2000 to reduce the overall integration time needed to solve Poisson's equation.

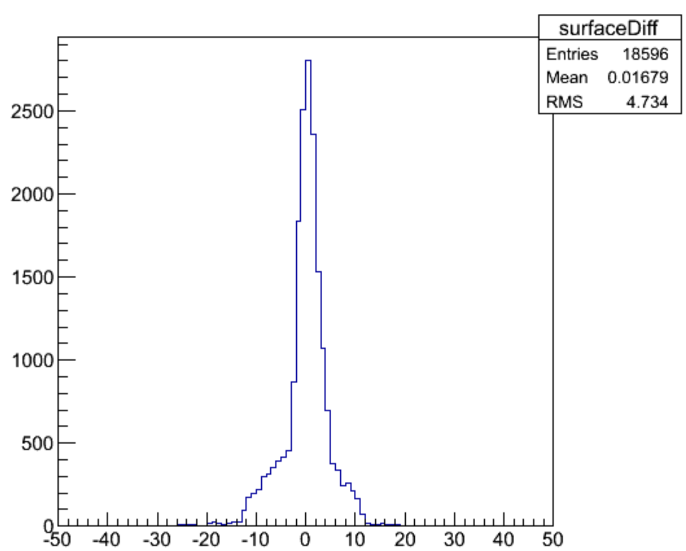
This result is not fully satisfactory because the total curl of 53nm is a substantial fraction of the total height profile, which makes the problem ill-posed. Right now the non-zero curl shows up as a discontinuity at the edge of the image near the right-most corner of the sample.

To resolve the inconsistency in a more regular fashion, I propose the following solution. In 2D, the zero-curl constraint effectively removes one of the two degrees of freedom. Either one can rely on the x-scan and compute the y-tilt from the zero-curl condition, or vice versa. Some information from the complementary scan is needed to connect the stripes, but only a single row of pixels can be used. From this one can solve for the shape independently, using the two scan images, and then use the difference between the two solutions as an estimate of the error in the approximation that the only contribution to rocking curve spread is external curvature. I wrote a new method of the Map2D class called Uncurl(Vx,Vy,comp) which computes one of the two components of the field (Vx,Vy), selected by the comp argument, from the other component and the zero-curl condition. To connect the stripes, I require that the mean value in a given stripe equal the mean value in the corresponding measured scan image. The two solutions for the two scan differences for U40 are shown below. One can see that they are largely consistent. The striations visible in the surface show the direction of the measured scan that was analyzed to form the image.





The difference profile histogram of the two above shape solutions is shown below.



Difference between the surface height computed using the x-scan and the y-scan for sample U40. The resolution is comparable in nm to what is obtained in microradians for the measured peak position, which is what one would expect for a sample size on the order of 1mm.