Characterization of Diamond Samples -- CHESS Run Summer 2012

6/6-6/12/2012

Goals for this run

1. Center the target holder in the goniometer and align a sample crystal for diffraction from 2,2,0 planes to prove that everything is working. Make sure rocking curve widths are as expected, proving that the monochromator is configured for dispersionless diffraction from diamond 2,2,0.
2. Follow the standard procedure to optimize the focus of the ccd camera, and measure its edge resolution using a knife edge.
3. Measure the rocking curves of the following samples in both x and y orientations.
   - dave-30
   - casey-300
   - sinmat-100
   - sinmat-50
   - sinmat-25
   - e6-10
4. Do systematic studies of the shape of dave-30 and e6-10 by shifting it around in the mount and looking for mount-induced changes
5. Take any measurements that might be useful to show the quality of the xray data in our upcoming paper.

1. First rocking curve to check setup

aligning the goniometer and mounting the sample holder

June 6, 2012, RTJ, BJP, AEB

We arrived at CHESS and checked in around 11:00am. Ken Finklestein showed us around end station C. He had already set up the Si331 mono at 15KeV and aligned the beam in the hutch. We noticed the following rates were being recorded in the ion chambers with the beam on.

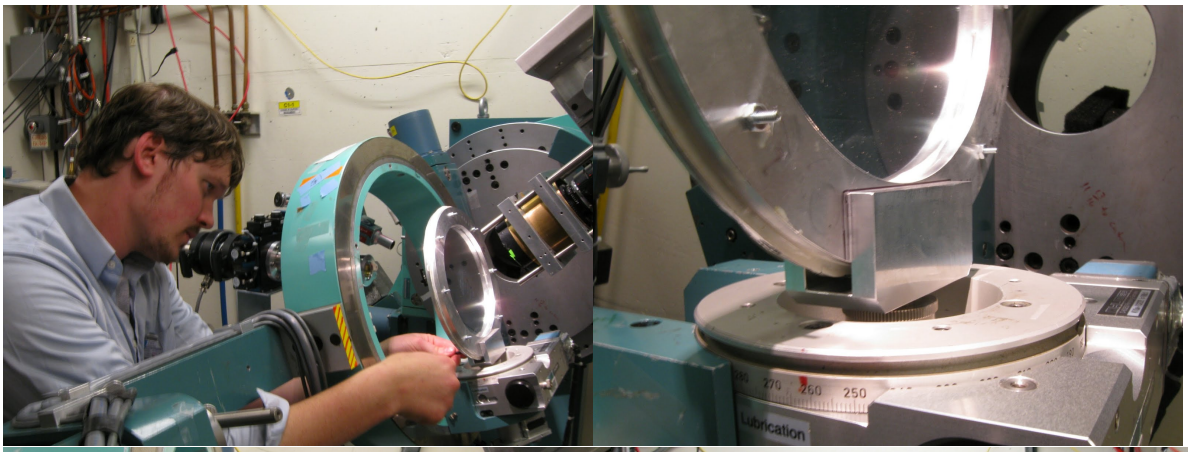
Ion0: 258 kHz Ion1: 326 kHz

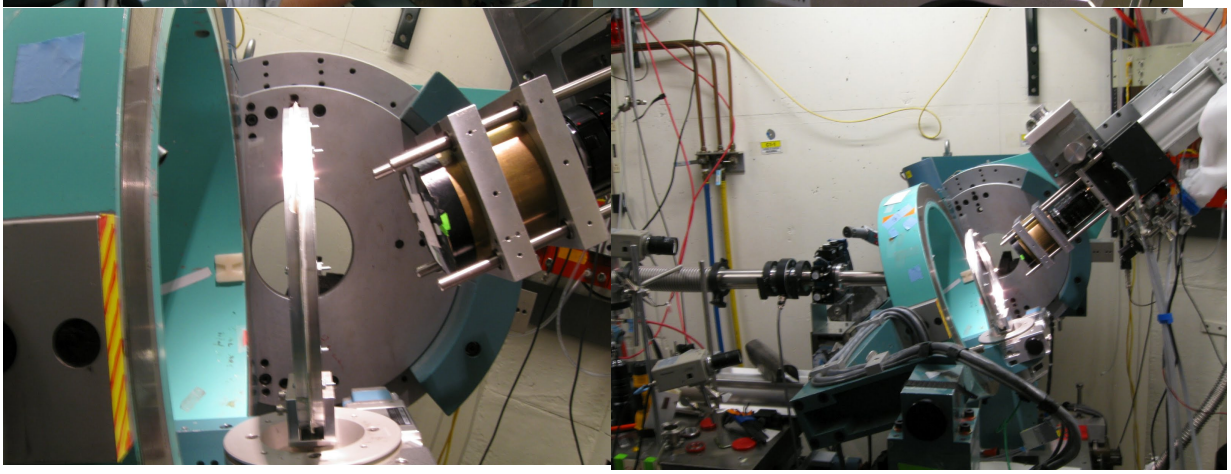
The target telescope had been removed from the 4-circle goniometer, but it was found lying nearby in the hutch. Ken put a manual stage on the goniometer sample post and locked the alignment pin (pointed post) in its chuck. He then used the following procedure to mount the telescope and find the center in the viewfinder.

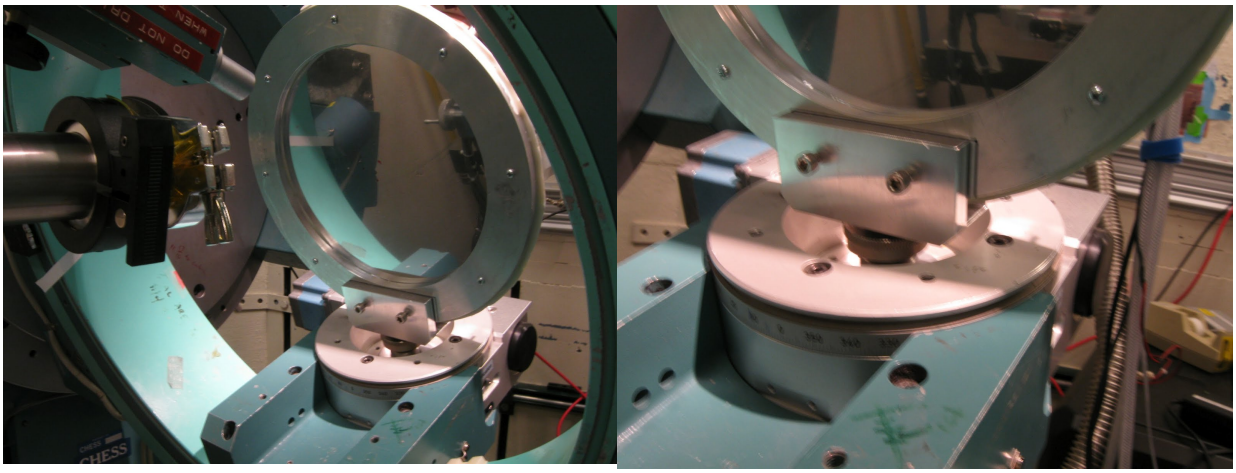
1. Find the screws that hold the telescope on the upstream face of the chi circle upper arc and loosely tighten the telescope in place, so that it can still be moved within the slack allowed by the screw clearance holes.
2. View the tip of the alignment pin through the telescope. There is a lamp on a flexible tube mounted to the ceiling that can be pointed at the tip to make it more visible. Placing a hand behind the tip helps improve the contrast.
3. Loosen the knurled nut that engages the gear on the phi motor, so that the phi pivot can be rotated freely by hand. Rotate the phi pivot while watching the pin tip through the telescope. If the tip moves as you rotate phi, the pin is off axis. Manually adjust the x and y stages that hold the pin until the pin tip remains fixed in the telescope viewfinder as you rotate phi.
4. To re-engage the phi motor, place a hand on the motor itself and wiggle it while rotating the phi axis a small amount, until you feel the gear engage. Tighten the knurled nut to fully engage the gear to the drive motor. Check that the phi axis is no longer free to rotate.
5. Loosen the knurled nut that engages the gear on the chi motor, so that the sample holder rotates freely on the chi ring. The word “freely” is used in a relative sense here, as the chi motion tends to be stiff. Rotate the chi axis through large angles (more than 180 degrees) while watching the pin tip in the telescope. The position of the tip should not move. If it does, turn the large nut that lies on the bottom of the phi axis to move the pin radially in and out until the tip remains fixed through a full range of chi motion.
6. Re-engage the chi motor, using a similar procedure as described in step 4 above. For chi, there is an orange indicator that points to an engraved angle scale on the chi ring. Ken says that the convention is to always make that orange indicator point “just past 270 degrees” when re-engaging the chi driving gear. Adhering to this rule prevents us from accumulating a new chi offset for the motor control software every time we perform another alignment.
7. Tighten down the screws holding the telescope, and record where the tip of the alignment pin appears in the telescope viewfinder.

Adopted alignment position for goniometer center in telescope viewfinder

Next we needed to adapt our mylar hoop target holder to fix it to the 4-circle target post. We found the clamp that we used last run (April, 2011) but the plates that squeezed the two sides of the hoop were missing, and the design was defective because the plane of the mylar sheets did not coincide with the phi axis. Ken had a CHESS machinist create a new clamp for us out of a block of aluminum. He cut a channel through the block that was just the right size to insert the plate sandwich into, and had two threaded holes in one side through which we placed bolts tightened just finger-tight to clamp the hoop in place. When we need to change out the samples, we just loosen those two bolts and remove the hoop, leaving the clamp on the 4-circle post. This works much better than the fixture we had last year, and it places the sample on the phi axis for more flexibility in adjusting phi. Here are some pictures of the hoop mounted on the 4-circle.







first rocking curve of the run

We took the mylar hoop out of the goniometer mount and brought it to the chem prep room. There we separated the two mylar halves and cleaned the middle region of the interior mylar surfaces using a few drops of alcohol and a lens tissue. We then took sample **dave-30** and placed it on the center of the hoop. The center was located by holding a ruler above the hoop and aligning one edge with the centers of opposite holes in the outer ring. The orientation of the crystal was chosen so that one of the diagonals, which corresponds to one of the 2,2,0 directions in the crystal, is along the phi axis of the mount. We then tightened 7 of the 8 screws holding the two hoops together (the remaining screw is omitted so that it does not interfere with the clamp that holds it on the 4-circle target post, as shown above.

To help in the initial search for a 2,2,0 reflection, we taped a phosphorescent screen to the front of the ccd camera and set up a video camera to view it. The best view was obtained by having the camera view a reflection of the screen in the aluminized mylar of the sample holder. We then closed up the hutch and began the search. Here are some commands issued to the Spec program that were useful to help us get started.

- *ccd_off* -- turns off the ccd camera, not needed during visual search
- *opens* -- opens the beamline shutter, otherwise x-rays do not reach the diamond
- *mv th/chi/phi/tth angle* -- moves one of the 4-circle motors to the absolute position given by argument *angle* in degrees.
- *tw th/chi/phi/tth dangle* -- moves one of the 4-circle motors in increments of *dangle* in degrees, stopping after each step to allow the operator to continue in the same direction or reverse directions.

Watching the video camera carefully as the motor swept through the theta angular region 18° - 22°, we saw nothing during the first pass. We then advanced chi from 90° to 95° and repeated the same mv command. A faint blip was observed in the expected region of the video frame as theta passed between 18° and 19°. The calculated value for theta at 15KeV for the 2,2,0 planes in diamond is 19.2° so this is not far off. Such offsets are due to a combination of imperfections in the mount and miscut of the diamond away from perfect alignment of the normal to the crystal face with the 0,0,1 direction.

We then turned on the ccd with *ccd_on* and closed the shutter with *closes*, and took a ccd image with *tseries 1 30*. The image was so bright that it saturated many pixels in the image, so we reduced the shutter time to 10s. Adjusting chi allowed us to center the rocking curve image in the frame of the camera. We then took two rocking curves, one each along the two in-plane 2,2,0 directions of the crystal. We created a new folder under the specuser home directory called uconn-6-2012 and started a new scan sequence with the *newfile setup1* command.

- **setup1_022** : chi=94.75°, tth=38.338°, th=18.574° .. 18.597° in 115 steps
- **setup1_033** : chi=2.75°, tth=38.338°, th=18.715° .. 18.745° in 150 steps

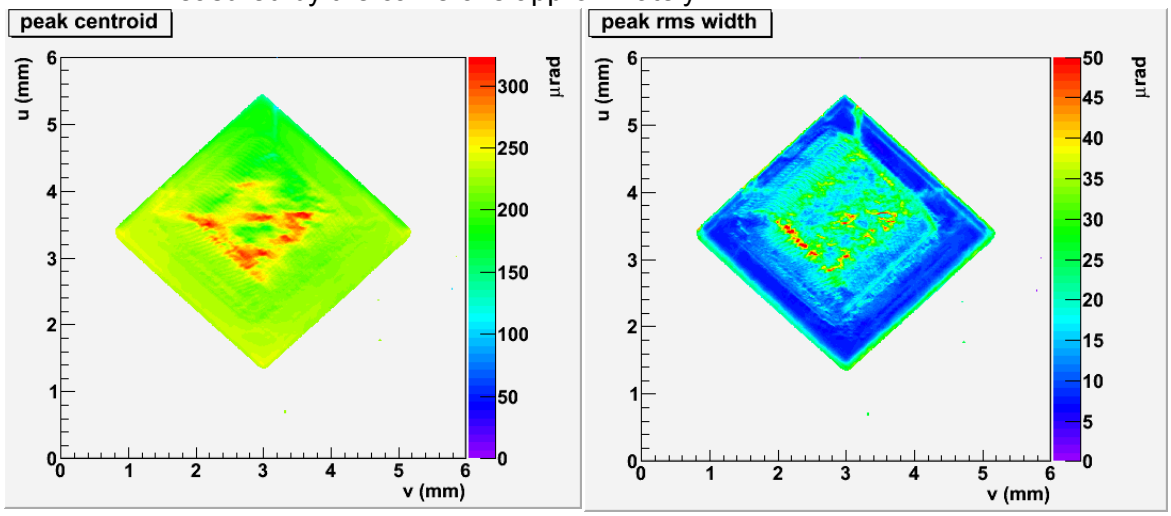
June 7, 2012, RTJ (night shift)

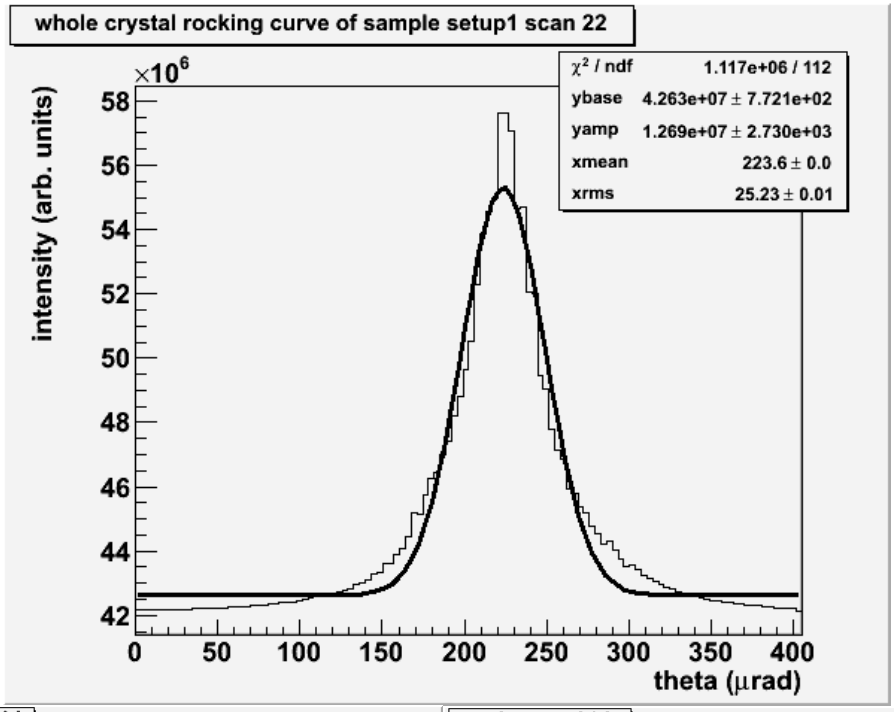
The following plots show the results of these two initial rocking curve scans of this sample. There are two pairs of plots for each rocking scan, laid out in the following manner.

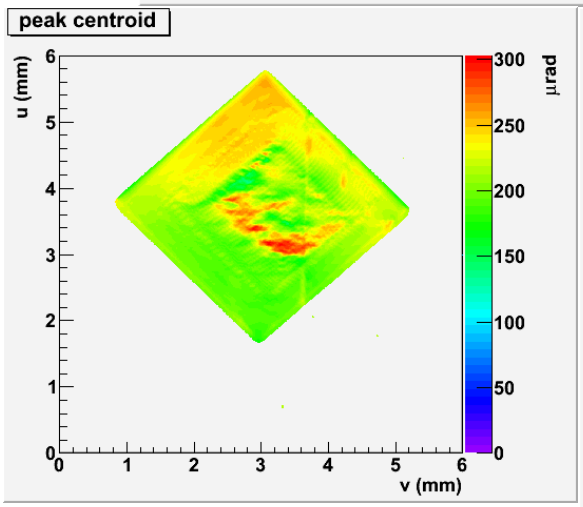
2D plot of rocking curve peak centroid, from Gaussian fit

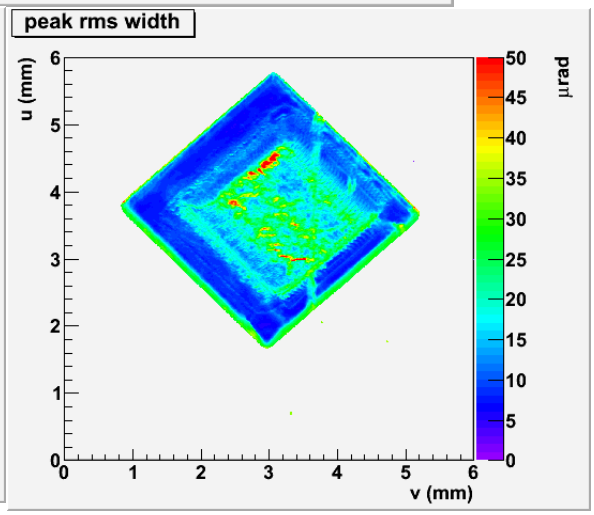
2D plot of rocking curve peak rms, from Gaussian fit

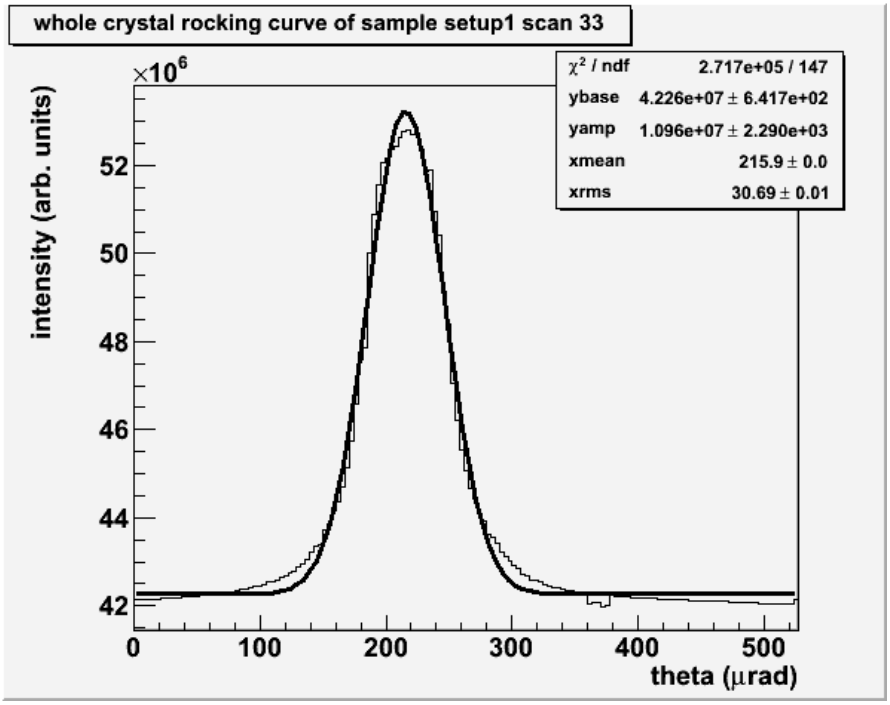
1D plot of whole-crystal rocking curve weighted by intensity without zero subtraction. The zero of the intensity distribution measured by the camera is approximately 42.



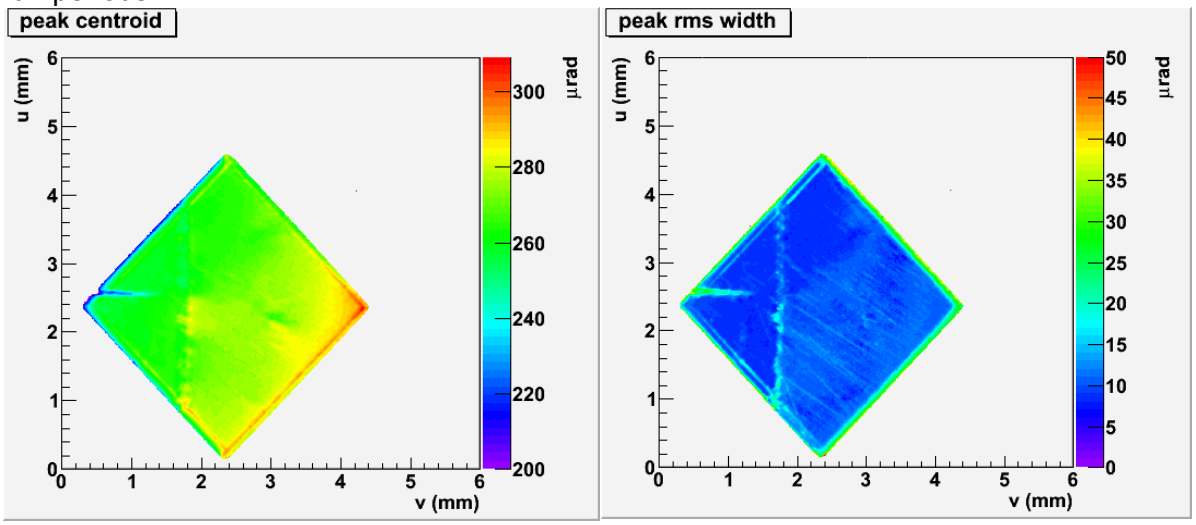

whole crystal rocking curve of sample setup1 scan 22
χ^2 / ndf 1.117e+06 / 112
ybase 4.263e+07 ± 7.721e+02
yamp 1.269e+07 ± 2.730e+03
xmean 223.6 ± 0.0
xrms 25.23 ± 0.01
×10^6
intensity (arb. units)
theta (μrad)

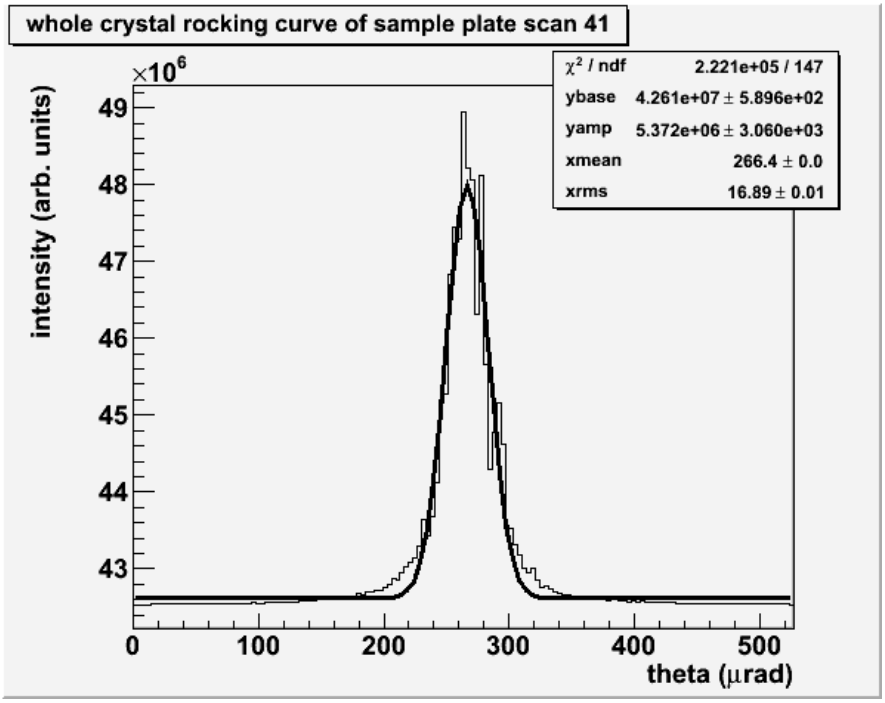

peak centroid
u (mm)
v (mm)
μrad


peak rms width
u (mm)
v (mm)
μrad

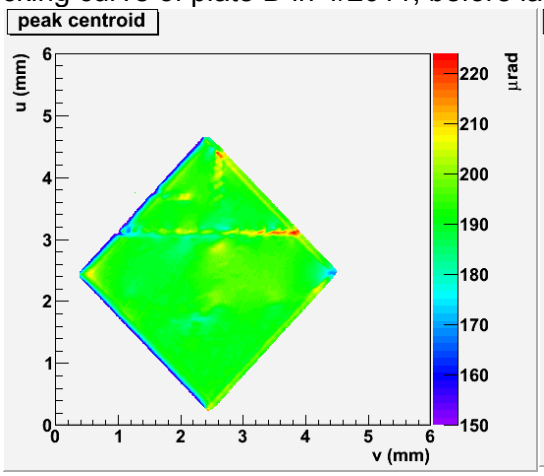


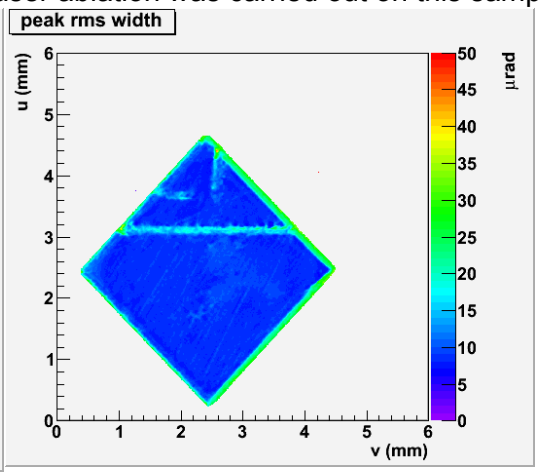
For comparison, I show the scans of this same diamond that were taken in 4/2011 before laser ablation was started on this sample. This comparison makes clear which aspects of the sample are original artifacts and which are the result of damage from the laser. It is clear that in the region where significant amounts of material have been removed, the rocking curve widths have substantially increased. Perhaps this is a form of radiation damage. The original markings are conveniently located in the corners so they can be used to orient the sample between different run periods.

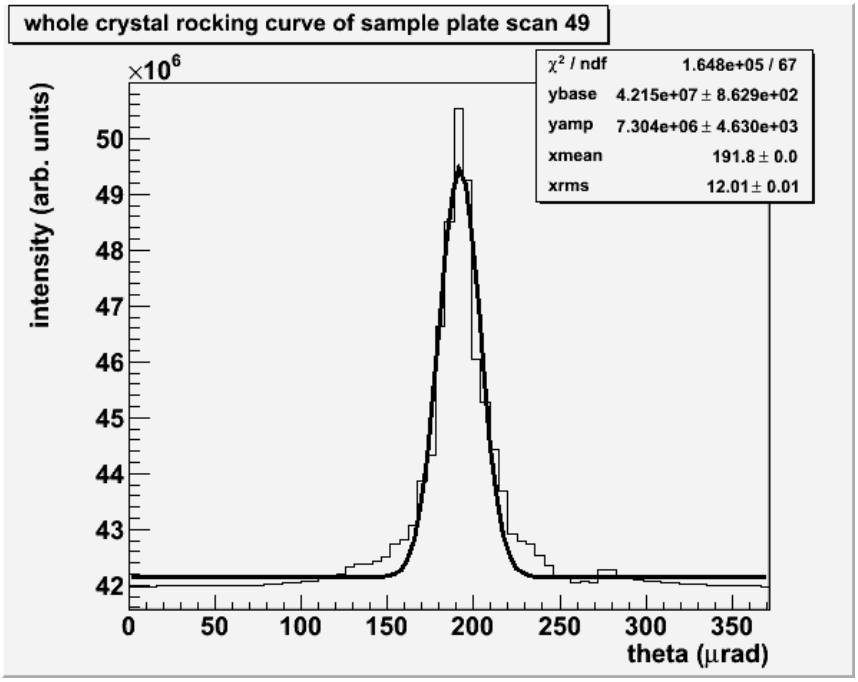




Rocking curve of plate D in 4/2011, before laser ablation was carried out on this sample.







Rocking curve of plate D in 4/2011 along the orthogonal axis, before laser ablation was carried out on this sample.

For the first time, I now have the tools in place to take rocking curve topographs from two orthogonal directions and combine them to form a single strain image of the crystal, under the interpretation that large-scale strain explains all of the structure seen in the pixel-to-pixel variation in the rocking curves. These are big maps, 1000x1000 pixels so I must be patient to wait for the PoissonSolve method to solve for the strain map whose gradient components are the two rocking curve topographs. I launched PoissonSolve 25 minutes ago and it is still running. I project that it should complete within an hour or so.

2. Focusing of ccd camera using knife edge

June 7, BJP, AEB, 9:00am

The first task on this shift is to optimize the focus of the camera and measure the response to a knife-edge boundary. After that, proceed down the list of samples and run rocking curves over x and y directions for all of the remaining samples.

Since this is both Alex and Brendan's first time performing this procedure we will document in extensive detail. The first step is to locate the diamond imaged on the scintillator so we know where to place the knife edge. The diamond was in screen, but the intensity was low and so we are altering theta. We were unsure on how to create a new directory and didn't want to mess anything up. We will continue to save the images to setup1 starting with the following file, setup1_034_000.tif

We then realized it would be much quicker to simply replace the fluorescent paper and watch

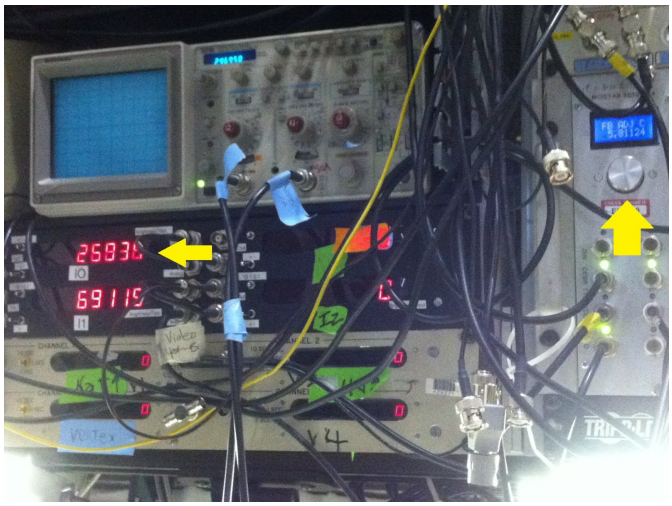
the screen for the bright flash (the diamond). Trying to get the entire diamond to image has been very difficult. Using RTJ's past runs as indicators, we were only able to image half of the diamond. We will try increasing the exposure time to 20 seconds in hopes that we will see more of the diamond. Changing the exposure time did not help. We're not sure if the images produced are good enough to focus the ccd with. We are going to run a rocking curve scan through the present theta (18.7270) with the following features:

ascan th 18.7250 18.7300 50 +10

Watching the scans as they came through it was clear that increasing theta was producing lower intensity results. We aborted the scan (ctrl c) and decided to start at 18.7250 and work our way down with increments of 0.0002. Compared to yesterday's images we are getting poor results. Our plan is to take a larger rocking curve while we grab a quick bite to eat. The scan is as follows:

ascan th 18.715 18.745 120

Coming back we learned our mistake. Ken showed us a dial which maximizes the I0 gauge below the oscilloscope. Apparently, the setup is aligned with this value at max and for some reason this changed overnight. Adjusting the knob (shown below) brought this value back up to >25K which is where we were yesterday. Great, we deleted the above run from setup1 since it was useless. Now we will attach the knife edge and proceed with focusing the camera.



We now attached the knife edge to the scintillator using scotch tape and took our first image at the following micrometer reading 0.102" (this is the distance at which all previous measurements were taken)

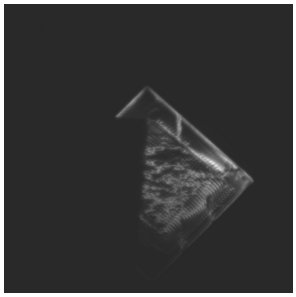


Image taken at 0.102"

coarse adjustment of the camera focus

We then advanced the micrometer 0.050” in the forward direction so that we can perform the focussing at 0.01” increments as suggested in the focusing instructions. Below is the image of the knife edge clearly out of focus.

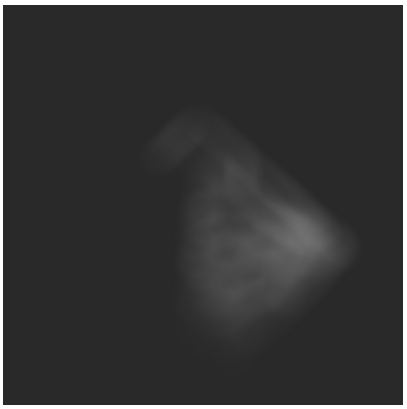
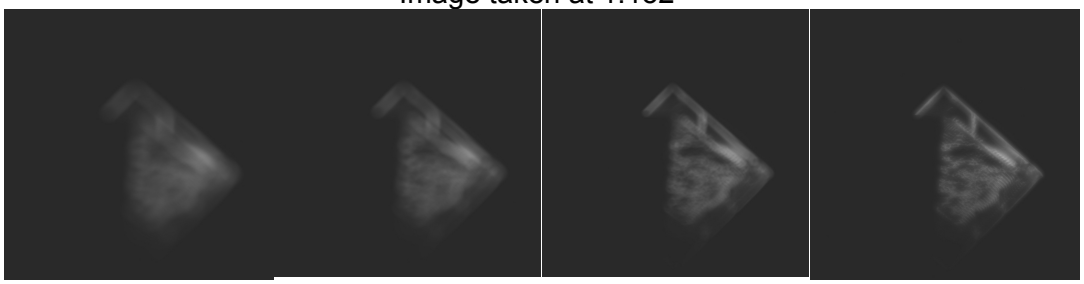


Image taken at 1.152”



The following images of the diamond at 1.142”, 1.132”, 1.122”, and 1.112” respectively

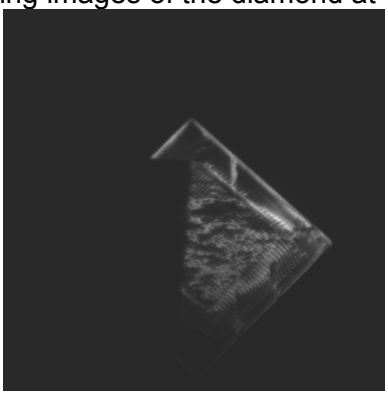


Image taken at 1.102”

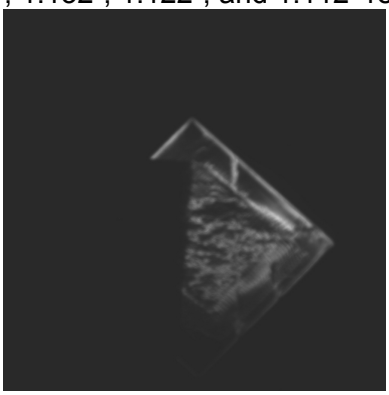


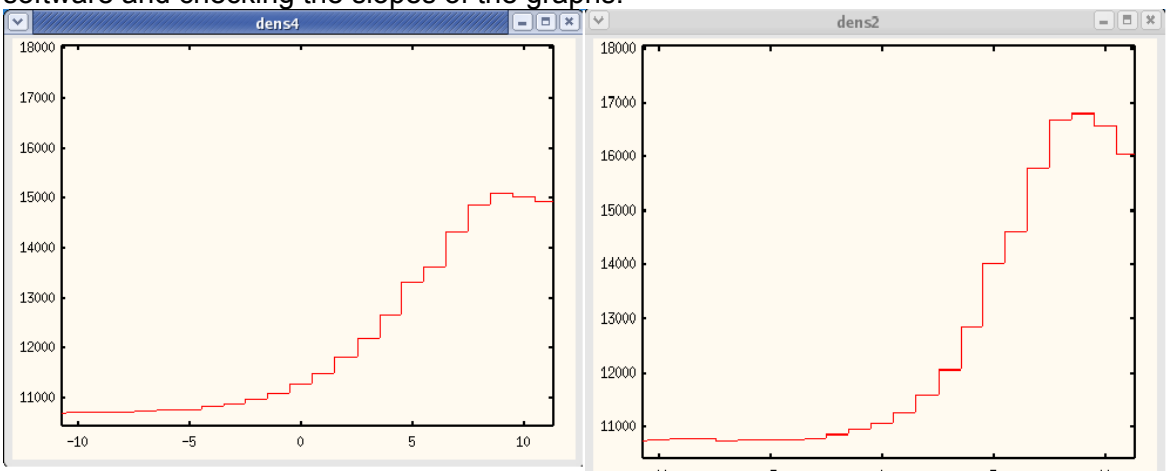
Image taken at 1.092”

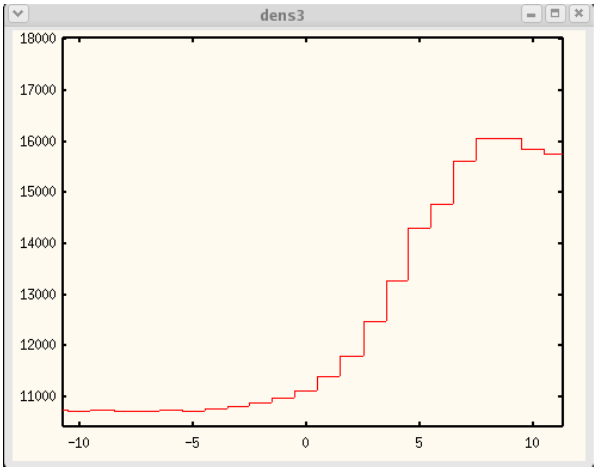
The coarse adjustment is completed since we are now again out of focus at 1.092”. The focus is somewhere between 1.102” and 1.092” and we will use these as the starting points for the fine adjustment. To account for the backlash of the micrometer, we will advance past 1.102”, reimage and continue in 0.002” increments towards 1.092”.
Edit: After running the scans again it was found that the focus was infact between 1.108” and 1.102”.

fine adjustment of the camera focus

We are now attempting to use the TVX program listed in the camera focusing manual. Using the camera's laptop we cd into tvx followed by the command ./tvx . We ran into an error when trying to run the command:
disp /home/specuser/uconn-6-2012/setup1_078_000.tif 10400 65535 5
As both Alex and I are newbie Linux users, it is taking us a little longer to figure this out. We moved the image file to the the home directory of spec user (/home/specuser) and then cd into tvx followed by ./tvx . Next we type the command
imagepath /home/specuser <return>
disp /home/specuser/setup1_078_000.tif 10400 65535 5 <return>
and then a display window appears. Great, now we can properly compare scans.

In the middle drop down menu below the sliders above the image, select "butterfly". You will now see a yellow circle with lines intersecting it, click on the lines and make them parallel so that the window on the bottom labeled "splay" equals 0.0 . There should be now two parallel lines on one side of the circle and three on the other (the extra line in the middle of the two is your directional ray). Click on the knife edge and the center of the circle moves to that location. Now click and drag the ray so it is perpendicular to the vertical knife edge. A trick is to make the ray parallel (easier to see) and then subtract 90 from it. Mouse over the lines within the circle until you get the "double arrow" and bring the lines in close to zero (if not zero exactly). Type integrate into the tvx terminal and you'll see a graph pop up. To keep measurements consistent, put the center of the circle in the same x/y location and angle for each image.
Also, when zooming into the graphs make sure the zoom box areas are the same. We found the focus to be slightly offset at a micrometer reading of 1.104". This was done using the TVX software and checking the slopes of the graphs.





The images above show the plots made using the TVX software for micrometer readings of 1.103”, 1.104”, 1.105” respectively. The steepest slope appears in the middle picture (1.104”) and so this is where we have set the focus.

Below is a table indicating the image number with its corresponding micrometer reading.

Image #	Micrometer Reading		Image #	Micrometer Reading	
070	1.102”		083	1.104”	✳
071	1.152”		084	1.112”	
072	1.142”		085	1.111”	
073	1.132”		086	1.110”	
074	1.122”		087	1.109”	
075	1.112”		088	1.108”	
076	1.102”		089	1.107”	
077	1.092”		090	1.106”	
078	1.102”		091	1.105”	
080	1.100”		093	1.104”	
081	1.098”		094	1.103”	

3. Rocking Curve Measurements

rocking curves of dave-30 in a pitchfork mount

Before moving on to take rocking curves of our remaining samples, we decided to take one more pair of scans on dave-30 with the improved camera focus. At the same time, we also take this opportunity to rule out a possible flaw in our technique. After noticing the prominent bulge in the mylar caused by the edges of the dave-30 diamond, I started to worry that the mylar might actually be putting pressure on the diamond surfaces. In particular, the back wall of the thin section of dave-30 might be flexible enough to be strained by the pressure of the mylar. What if small flecks of dust or irregularities in the surface protrude above the height of the edges and so bear a load of pressure from the mylar? Also, the mylar surface itself might be rough enough to have ridges that exert force on the back of the thin window. To rule this out, a CHESS machinist has fabricated a special aluminum post that has a slot in it just large enough to hold the diamond. There are ridges on either wall of the slot that prevent the diamond from falling out.

We had to file down the aluminum walls of the slot a wee bit to allow the diamond to sit freely inside without pinching. There was also the problem that the 2,2,0 direction is along the diagonal of the diamond rectangle, so the walls of the holder are potentially in the way of the x-ray diffraction path. Below is a picture illustrating the mount.

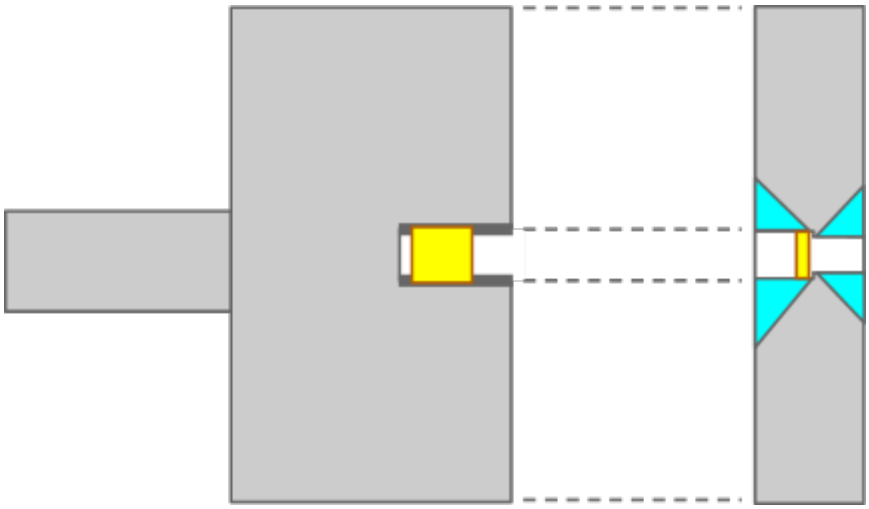
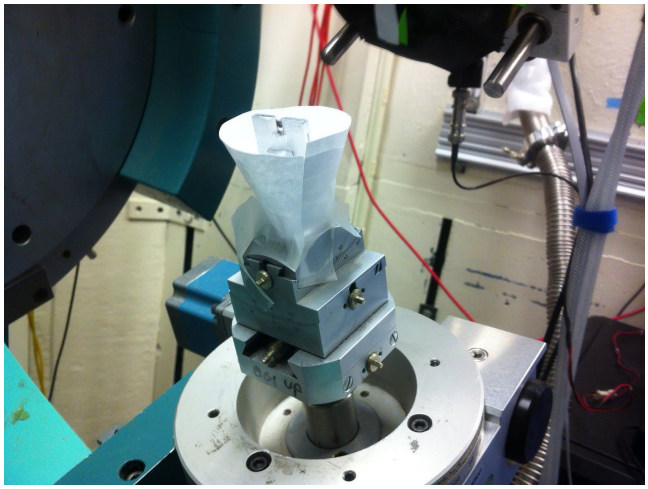


Illustration of the pitchfork mount made for us by the CHESS machinist. The yellow rectangle is the dave-30 diamond sample being held in place in its slot only by gravity and van der Waals forces. The blue trianges are the corners of the original piece that we filed off so that the diamond is not shadowed in its transmission diffraction geometry by the mount material.

To keep the path of the diffracted x-rays clear, I need to cut away the sections of the mount indicated in blue. The angle of the wedges should be close to 45 degrees to be sure to be out of the way.



Dave placed in new mount is shown above. the paper “dog cone” is around the mount’s base to catch the diamond in case it falls out. A second camera was also positioned in view of the diamond so we could check if it has fallen out.

June 7, 2012, RTJ (night shift)

After aligning the new slotted mount for sample dave-30 in the goniometer, I was unable to find any diffraction peaks, searching high and low. Finally, I decided to pull back the target and mount a phosphorescent screen to view the beam spot. There was no beam! The ion counters were counting fine, however, so I suspected the shutter. Sure enough, the control box for the shutter has a bnc cable running to it from a camac control board outside the hutch. That cable had somehow come loose and gotten disconnected. We found another loose cable on the floor of the hutch that contained a barrel, and determined that the two were supposed to be connected. After that, the *opens* and *closes* commands in **fourc** were again able to turn on and off the lights on the shutter control box. Now we are seeing a bright beam spot in the forward direction. This is progress!

Once the shutter issue was resolved, it was trivial to find the 2,2,0 diffraction peak. I started at 18° and scanned downward. There was no trouble seeing the peak, even with the motors advancing at full speed. I adjusted chi to center the image in the center of the ccd camera viewfinder, and started a new scan. Ion chamber rates are now I0=256kHz, I1=210kHz.

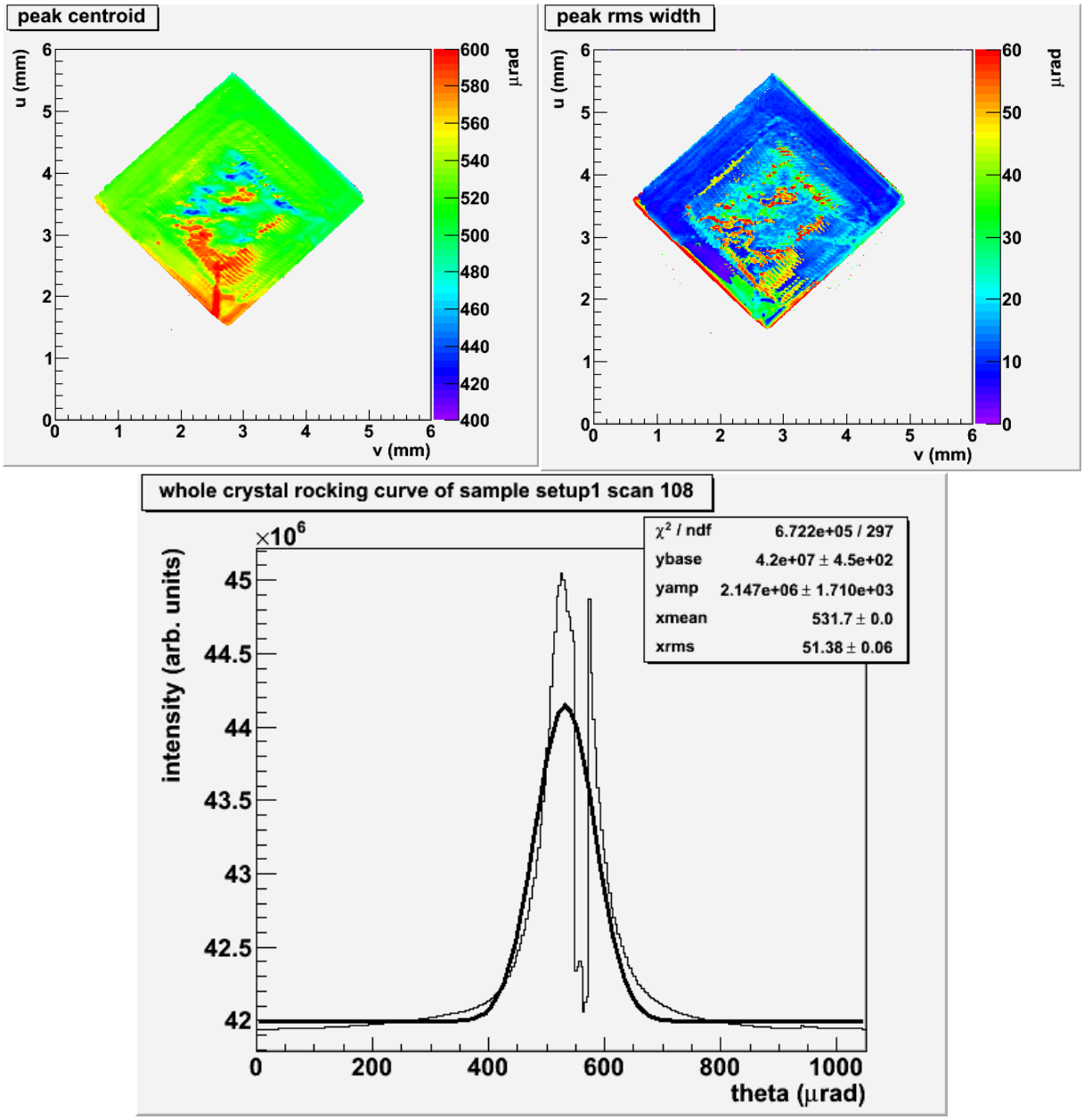
- **setup1_108** : chi=51.0°, tth=38.338°, th=15.9580° .. 16.0180° in 300 steps

I left this scan running at about frame 10, and everything looked stable. I checked conditions again around frame 265 and found that ion chamber rates had dropped to I0=170kHz. I tweaked the mono, and the ion chamber rates immediately returned to I0=258kHz, I1=225kHz. When I first found the peak, it was down near 14° which indicated that I had a large phi offset in the mount. I moved phi by 5 degrees in the direction that shifts the peak theta back closer to its ideal value of 19.2°, then scanned th to find the peak again. Then I realigned the image in the center of the ccd imaging area by moving chi a few degrees.

- **setup1_116** : chi=143.0°, tth=38.338°, th=17.738° .. 17.778° in 200 steps

I chose a shorter range in th for the second scan because there is nothing out beyond 50 steps either side of the peak in scan 108. Rocking curve topographs for these two scans are

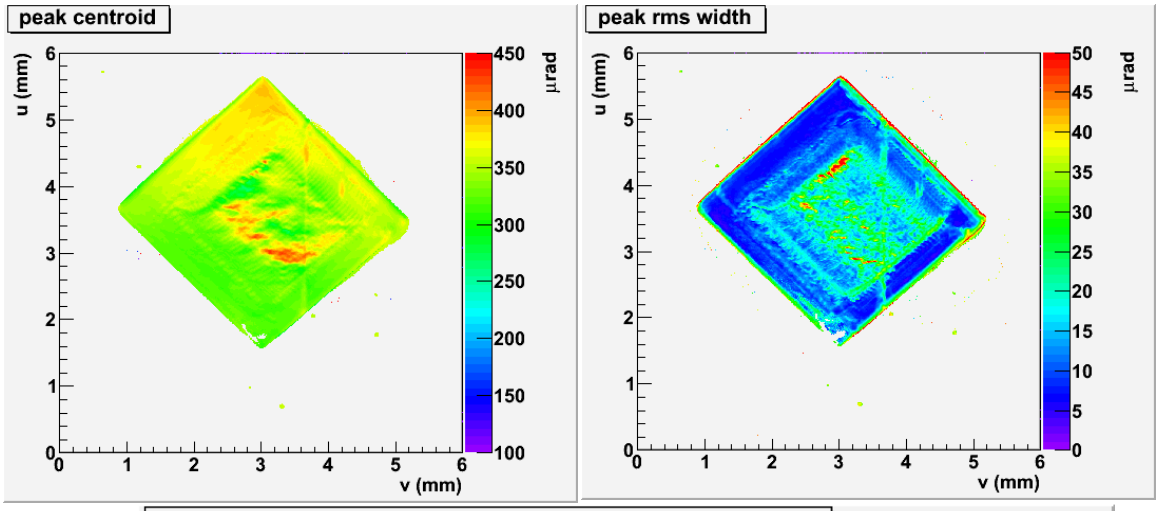
shown below. During the middle of scan 116 I had to tweak the mono to get the rates in the ion chambers back to nominal. I see in the intensity pattern in the ccd image that this created a shift in the pattern seen in the topograph, suggesting that the energy accepted by the mono have shifted when I adjusted it. We may need to repeat this scan later, depending on what is seen in the peak position and rms plots. I took a look at scan 116 up to frame 150, and it looks in the rocking curve peak mean and rms plots. There is no need to redo scan 116.

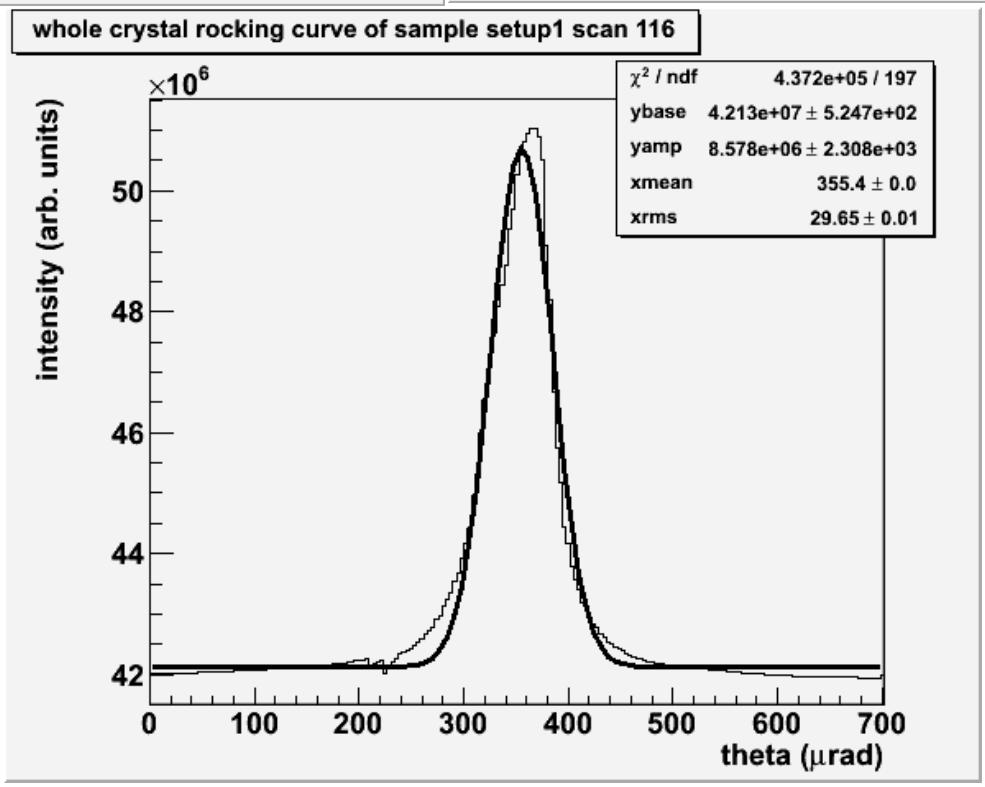


Bad luck with scan 108, it looks like there was a loss of beam (or a refill of the machine) right in the middle of the maximum. This is artificially broadening the measured widths.

There seems to be some accumulated strain along the lower edge toward the corner that is at the bottom in these pictures, suggesting that this mount is putting force on the corners of the sample. If this is the physical bottom of the crystal, this may be due to that corner bearing the

weight of the crystal in the sample holder.

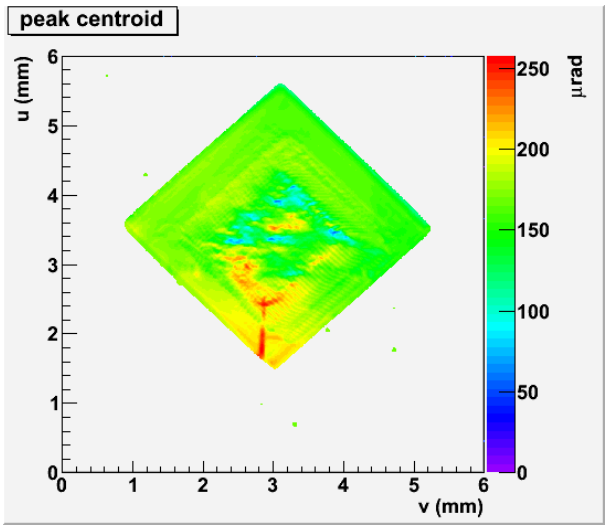


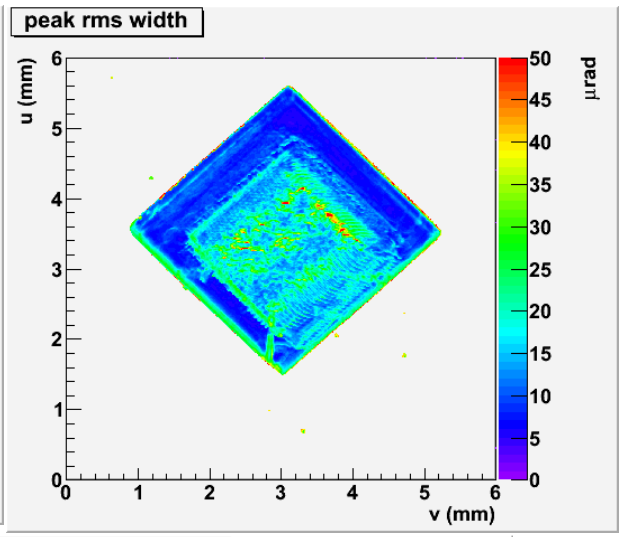


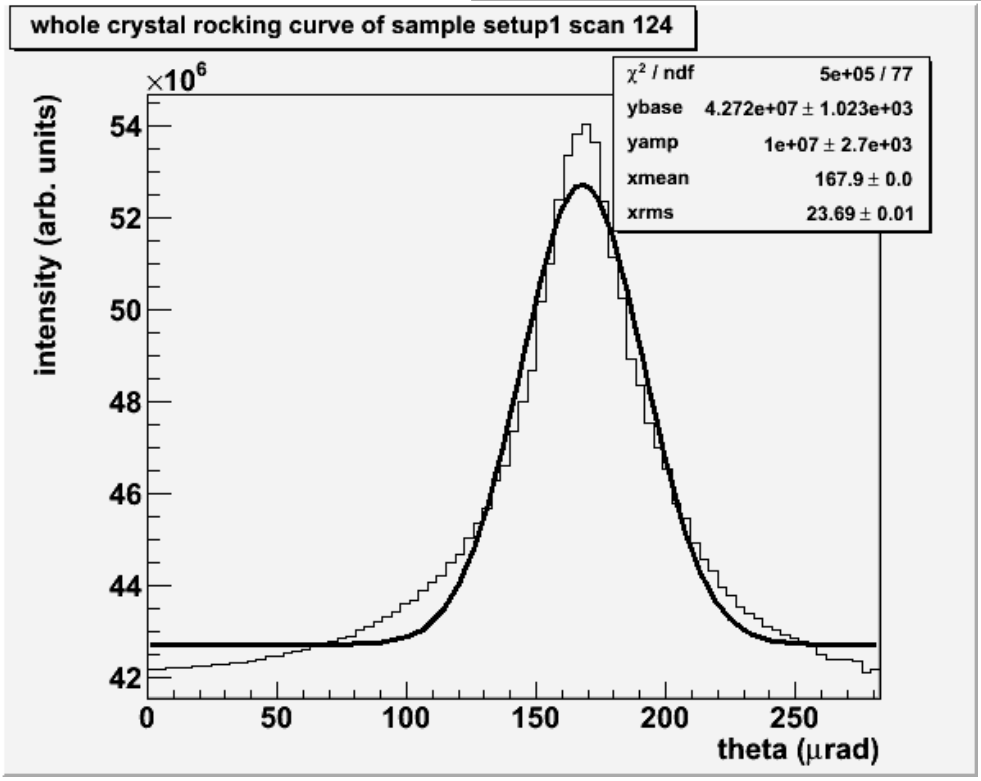
There is a striking resemblance between these images and the ones shown above for scan 33. Apparently the mount (or mylar hoop) has very little effect.

- **setup1_124** : chi=51.5°, tth=38.338°, th=12.5240° .. 12.5400° in 80 steps

Once again, we ran into the end of a fill before reaching the end of the scan. We will take what we get for this one, and then start a new scan at the beginning of the new fill.







These images from scan 124 match very nicely with scan 22 above, except that the sample is rotated by 180 degrees. This provides a confirmation of our interpretation of the rocking curve peak positions as vector components of the unit normal vector to the crystal planes.

- **setup1_125** : chi=51.5°, tth=38.338°, th=12.5120° .. 12.5520° in 200 steps, 10s shutter exposure time. *Note added later by RTJ -- the data from this run were deleted accidentally before they could be analyzed. Will have to come back and repeat later.*

June 8 2012, BJP, AEB 10:00am

measurements of the Sinmat samples

While scan 125 was running with **dave-30** in the pitchfork mount, we mounted **sinmat-25** in the mylar hoop. We created a new file directory using the newfile command called **sinmat25**. After scan 125 for setup1 using dave-30 finished we mounted **sinmat-25** and adjusted the setup so that we can take images.

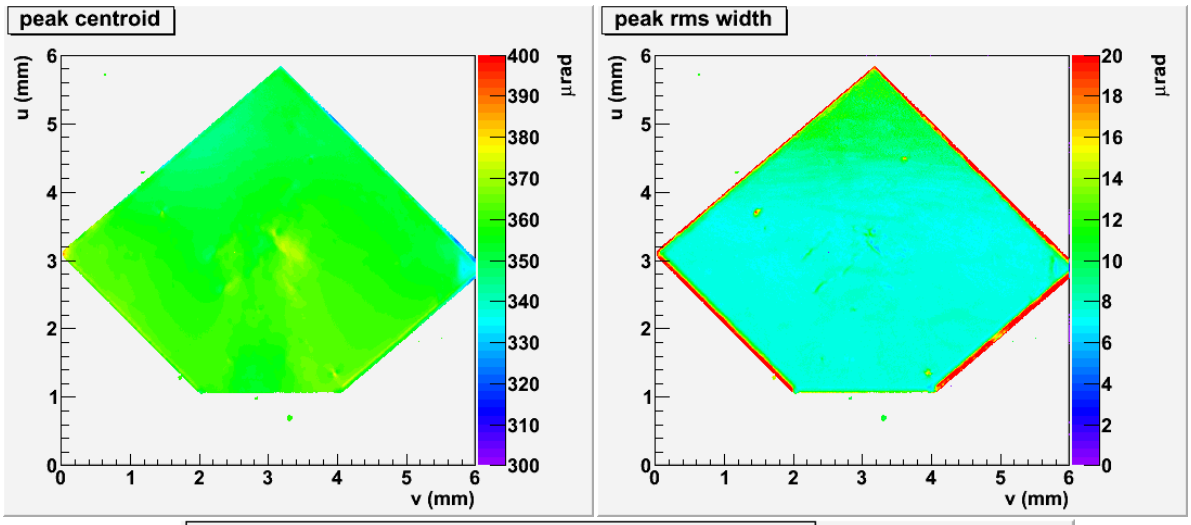
Our first attempt to take images failed after sweeping through theta and chi. We checked the diamond in the mount and noticed that it had slipped down about a cm. We then unmounted the ring and repositioned the diamond. However the diamond was sliding through the mount regardless of how tight it was clamped. This mylar hoop was used for 10 micron diamonds, so it must work. I realized the diamonds were previously moved onto the hoop using di water and carbon fibers and so I used the water to help the diamond cling to the mylar surface...it's still in place. Ken came by to listen to our troubles and tried to help with the sweep. We first put fluorescent tape over the backside of the mylar and we clearly saw the outline of the diamond surrounded by beam. After about an hour of sweeps we still had no luck. It may have something to do with the sample (dubious?) and so in light of getting data, we are moving on to the thicker sinmat samples starting with **sinmat100**.

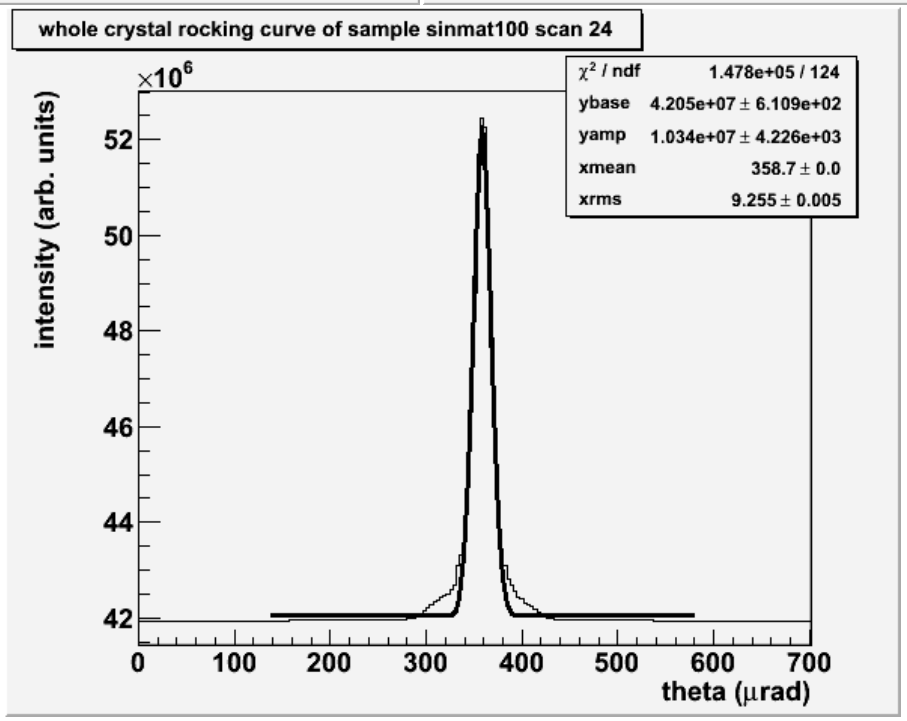
After mounting **sinmat100** it took only a minute to find the peak. This suggests something funny is going on with **sinmat25**. Perhaps this particular diamond was cut in a different orientation and Ken optioned for use to use a Laue camera to find the proper orientation of the planes. We are currently bringing the diamond into the camera's view and will begin a rocking curve measurement shortly. These diamonds are much larger and are clipped in the view of the camera. To bring the diamond into the camera's field of view we need to adjust twotheta. We will have to take two rocking curve measurements and stitch them together. We are starting our first run...(finally!)

- **sinmat100_024** chi=87.2730°, tth=39.0880°, phi -1.7500, th=19.2280° .. 19.2680° in 200 steps, 10s shutter exposure time.

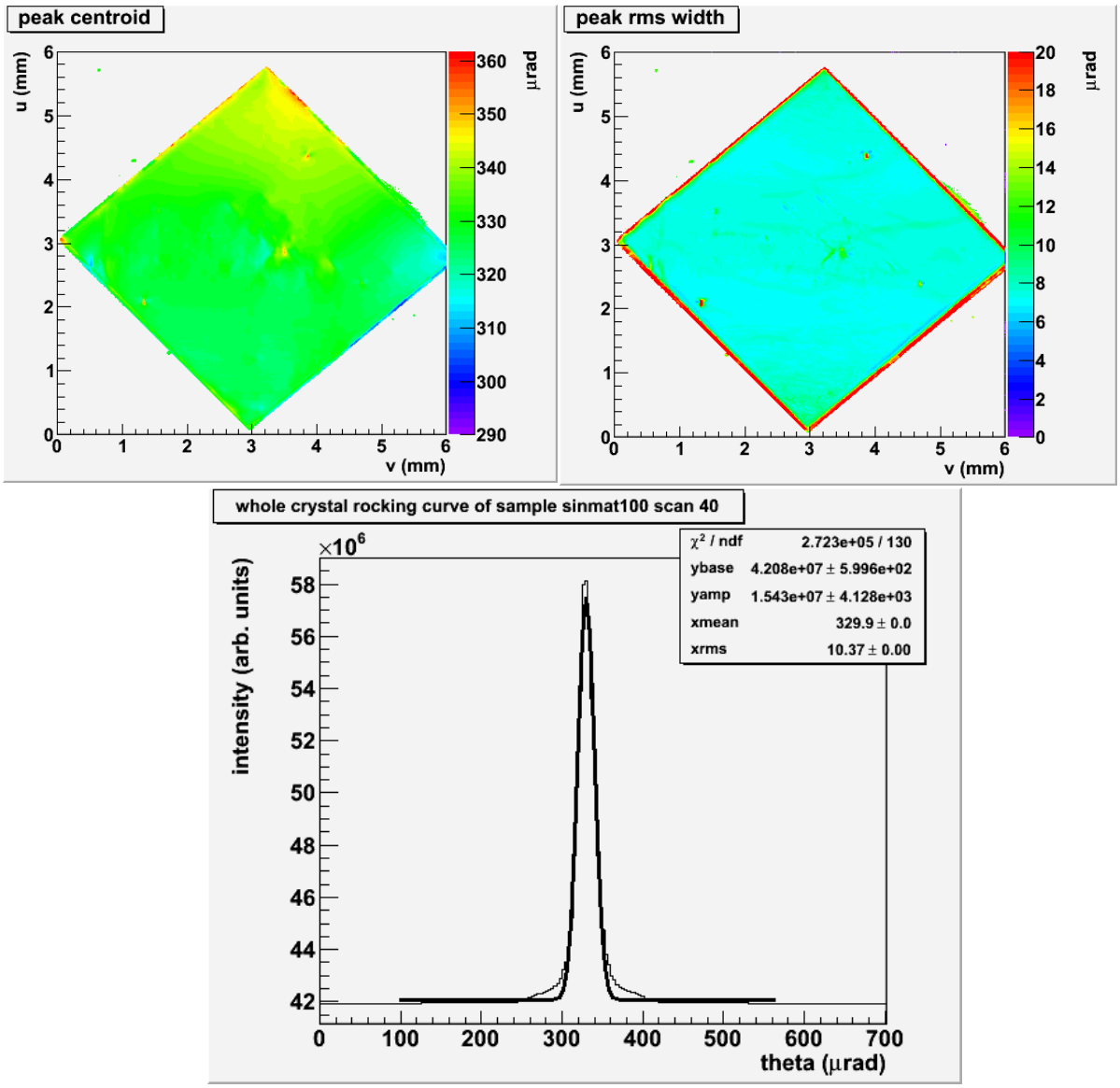
We started the run and left to get coffee and see a little bit of Cornell but when we came back Ion0 had dropped to 173kHz. The image of the diamond is also cutting off at the top. We originally thought the clipping was due to theta and that by rocking through theta we would image the whole diamond but it appears that the diamond is at the edge of the ccd. Throughout the run Ion0 has slowly been decreasing. By scan number 165 out of 200 it has dropped to 140kHz. Even with low IO we are still able to get a usable peak. Next up is rotating around chi to -2.7270° and taking the complementary rocking curve while two theta is still 39.0880°.

- **sinmat100_040** chi=-3.0270°, tth=38.2880°, phi -1.7500, th=19.0600° .. 19.1000° in 200 steps, 10s shutter exposure time. started at 172 minutes left to beam time at 7:42





This is probably the cleanest crystal we have ever assessed. The entire crystal has a rocking curve width that is close to the ideal for single-crystal diamond for the 2,2,0 planes at 15 keV and room temperature. The lower corner was clipped off in this scan by placement of the sample too close to the edge of the beam where it is cut off by the slits at the entry to C1.



This scan is perpendicular to the previous one and shows the same features (of lack thereof!). This time the sample was better centered in the beam so it does not have any corners cut off in the topographs.

It is interesting to consider what reasons might lie behind sample sinmat100 being so perfect. This is the first sample we have studied that has been polished on both sides with the RCMP process. All of the others are thinned using an ion mill / vapor etching process after the surface has been cleaned up using RCMP. One explanation might be that one needs to have the RCMP process applied to both surfaces of a thin diamond sheet, as the last step in the thinning process. On the other hand, this diamond is very thick at 100 microns, so it may have more to do with the bulk stiffness and uniform crystal quality of this particular sample than anything that is done to the surface. Hints on this question should be forthcoming for the thinner samples sinmat50 and sinmat25 that we study next.

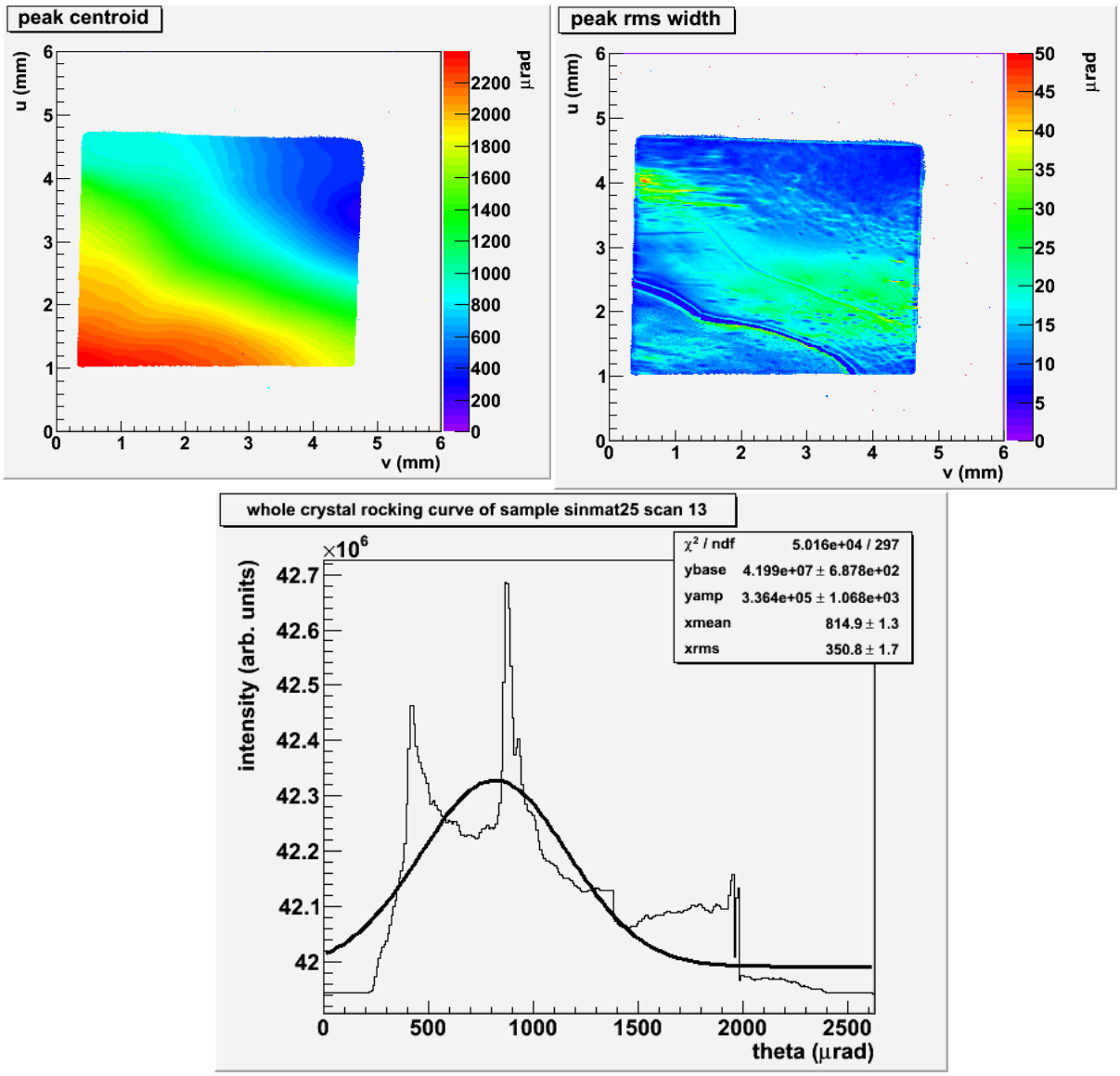
June 9 2012, RTJ 12:00am

measurements on the Sinmat-25 sample

The first challenge was to orient the sample. As Brendan and Alex reported earlier, initial attempts to find the 2,2,0 diffraction peaks for this sample were not successful. Brendan mounted the **sinmat25** sample in the mylar hoop, using de-ionized water as an adhesive to keep the diamond from slipping down inside the mount. This sample was then mounted on the 4-circle and centered. Centering was accomplished by viewing the mylar hoop through the telescope and shining a flashlight through the mylar from the back side. Even though it is aluminized, enough light gets through to illuminate the outline of the diamond sandwiched between the two mylar sheets. After making sure that the diamond was on axis, we mounted a phosphorescent screen on the front of the ccd camera and set up a mirror mounted under the camera to project up toward the ceiling an image of the screen. A video camera mounted on a rod suspended from the ceiling allowed us to view the phosphorescent screen continuously in real time, as the goniometer rolls through a range of angles. This is the most efficient way to orient the diamond in a monochromatic x-ray beam. Even for very perfect diamonds, the width of the rocking curve peak is sufficient to show a clear flash on the video screen whenever one of the diffraction peaks crosses through its 15 keV scattering condition.

We started out with a the diamond oriented with a diagonal pointing up, as was observed to be the 2,2,0 direction in our CVD plates (eg. dave-30). The first peak we saw as around 11° in theta, and turned out to be a 1,1,1 reflection. It was within 10° of the vertical in chi, which rules out an orientation similar to the CVD plates. A second scan was taken at 90° away in chi angle from the first one, and a second 1,1,1 diffraction peak was seen around 12.5° in theta. So we moved to the chi angle located midway between those two scans, and found our first 2,2,0 reflection at around 20° in theta. This turned out to be within a degree of parallel to one of the sides of the sample. Perhaps the edges of the sample are aligned with the (2,2,0) directions. We took a couple of coarse scans around this peak position, and then started a fine scan. Along with these scans we started running a script called **pusher.sh** which uses rsync to make copies of the .tif files on the CHESS computer and send them to gluey.phys.uconn.edu. Rsync looks at various properties of the files and determines if the files have been updated compared to the files in the destination folder. By doing so we are able to backup our data and can analyze the rocking curves through VNC on our own laptops rather than the experiment's computer.

- **sinmat25-013** : chi=46.3°, tth=39.0°, th=19.7450° … 19.8950° in 300 steps, exposure time 10s for the ccd camera.



Scan 13 shows the sinmat25 diamond oriented so that the vertical edges coincide with one of the 220 directions of the crystal lattice. The horizontal edges in this picture correspond to a 0,0,1 direction.

It was very difficult to find the second 2,2,0 direction in this diamond. The clue to this was that we found a 1,1,1 reflection close to the in-plane diagonal direction. This means that the direction along the edge perpendicular to the 2,2,0 edge must be the 0,0,1 direction, as shown below.

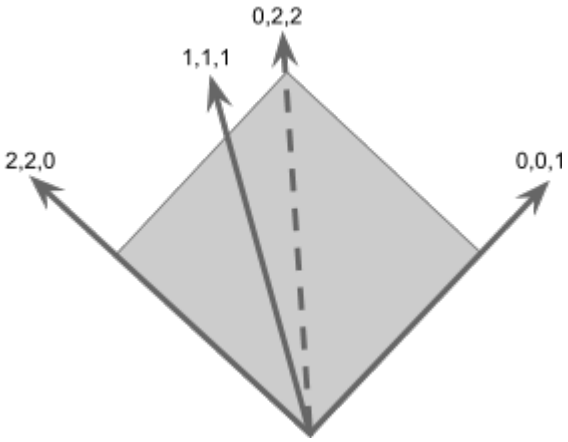
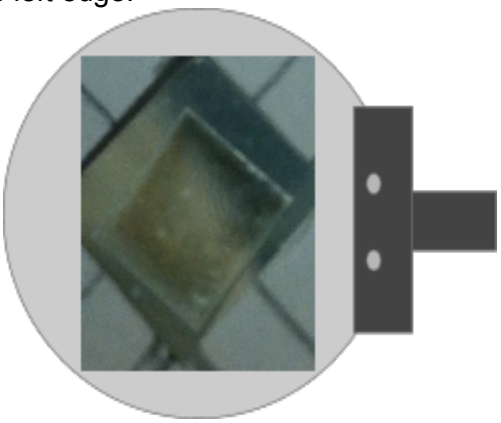


Diagram of crystal orientation of sinmat25. The solid arrows represent vectors that are in the plane of the surface, whereas the dashed vector is 30° above (or below, because of symmetry) the surface plane.

To get access to this out-of-plane 0,2,2 lattice vector, we had to remove the hoop from the four-circle and move the missing screw position from the six o'clock to the nine o'clock position. The chi angle was then rotated to near 0° and the hoop reattached with the mount gripping the hoop at the nine o'clock position. Now we could use the phi axis to rotate the hoop past the present limit of theta=23 that is imposed by the geometry of the beam line. I advanced phi to -31° with theta fixed at 20° and then started a search for the out-of-plane 0,2,2 reflection indicated by the dashed arrow in the above figure. It was there, within a couple degrees of where I expected it. We ran a scan of the rocking curve at the 0,2,2 position.

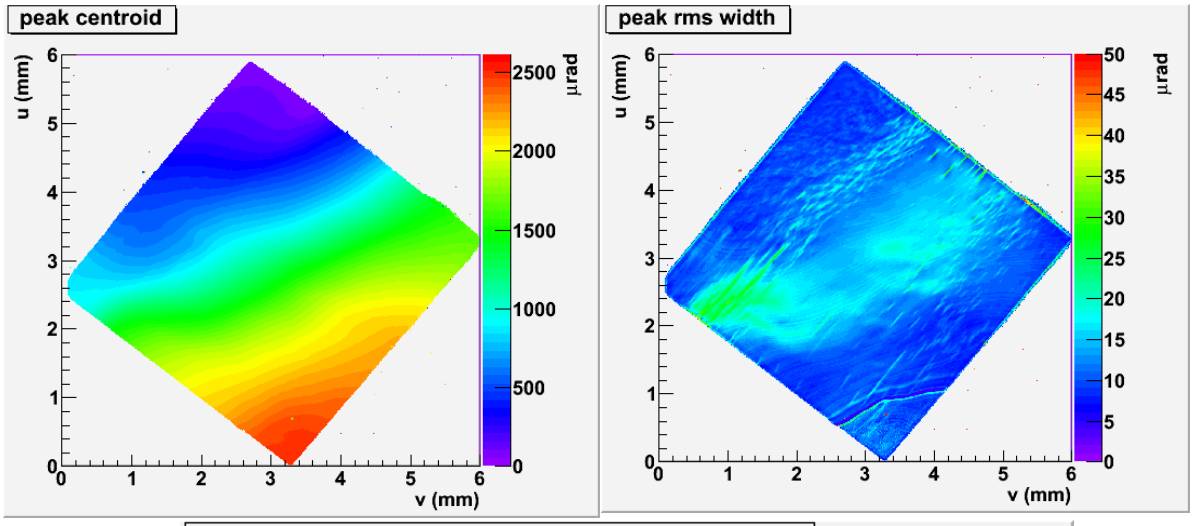
Brendan noticed for this particular diamond, a set of features that help finding the correct orientation of the diamond in the mylar hoop for 0,2,2 lattice vector. First, you need to find the side of sinmat25 that has the tinted markings on it. This will be easy to do since one side is free from any residuals the vapor ion etching left. Now, look at the diamond on the mylar hoop and you should see one half of the diamond that has a window free from tint (see picture below). Put this half in the upper half of the hoop, in the normal "square" oreientation. Now rotate the diamond clockwise 45° so it's in the "diamond" orientation. When mounted as depicted above by RTJ (see picture below) this will give the orientation shown above with the 0,2,2 along the diagonal and the 2,2,0 on the left edge.

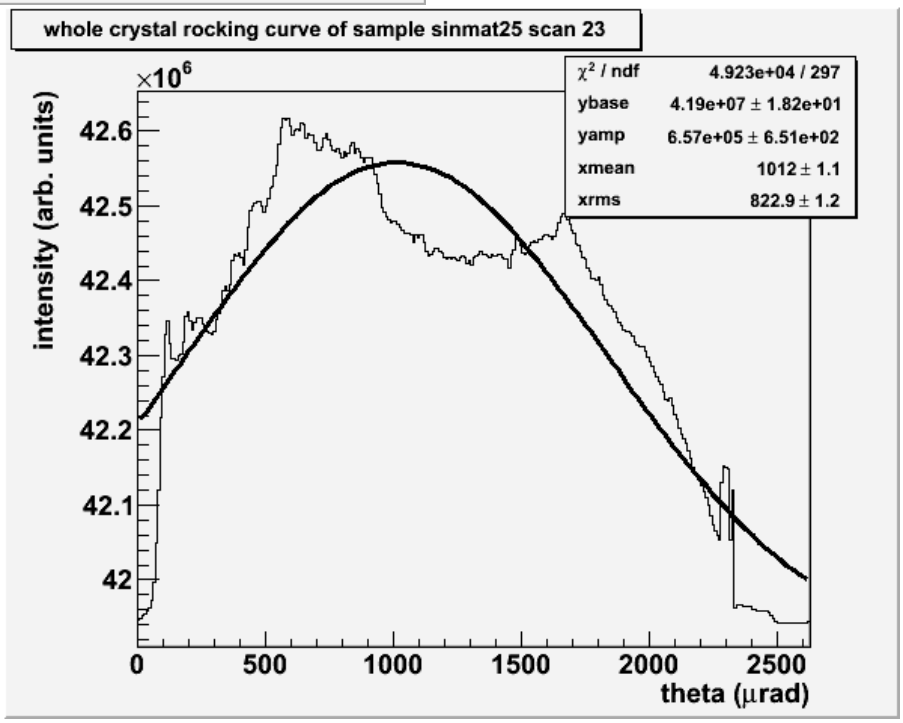


Orientation of diamond (not to scale) in the mylar hoop. In this view, the X-ray beam is coming into the paper from the above outer plane. Remember the hoop is being mounted from the side (as shown) instead of the bottom in this orientation so that the out of plane 0,2,2 can be found

through the adjustment of phi. Note the clear region inside the tinted window of the diamond.

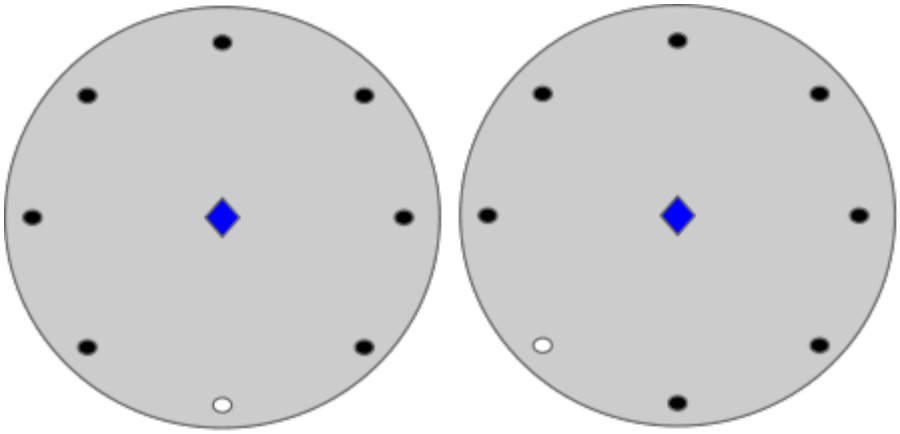
- **sinmat25-023** : chi=1.6°, tth=38.338°, phi=-31.074°, th=21.000° … 21.015° in 300 steps, exposure time 10s for the ccd camera.





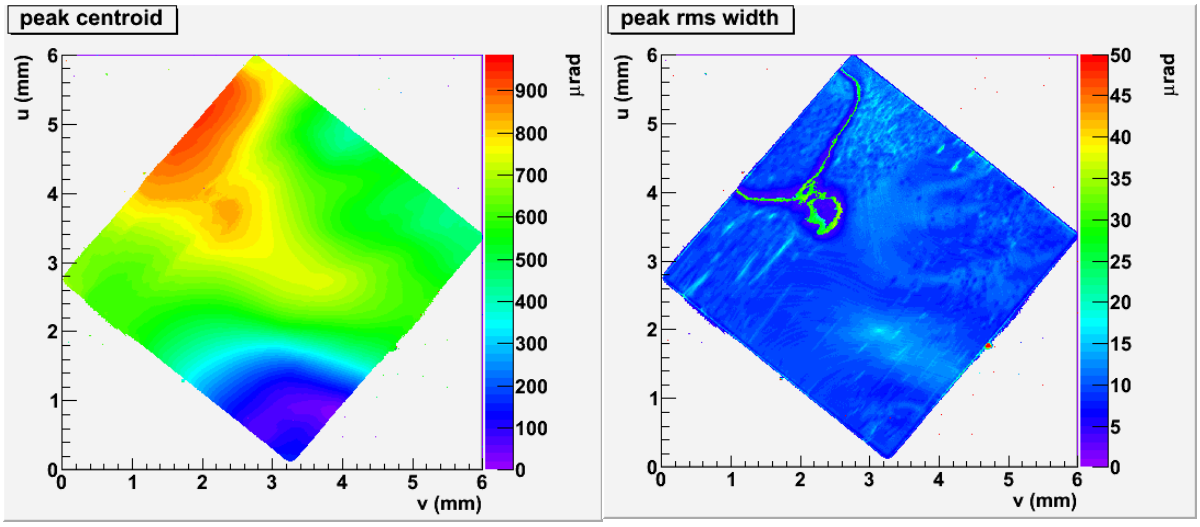
In scan 23, the sinmat25 diamond is being viewed against an out-of-plane 0,2,2 crystal axis which projects onto the plane of the crystal surface along one of the diagonals. Because the angle to the camera is theta=19.2° plus the out-of-plane 30° angle offset, the image looks almost square in the ccd.

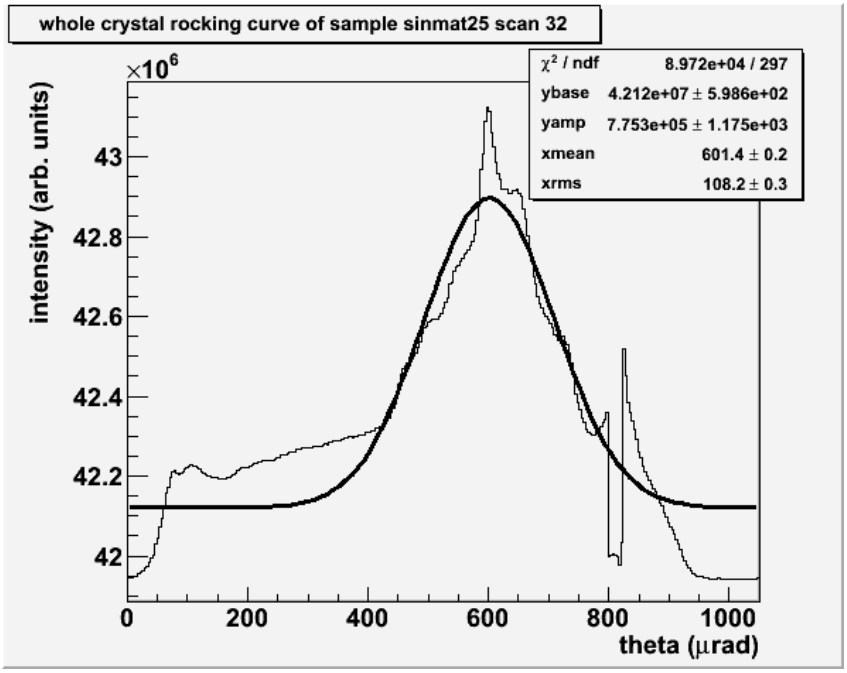
To test if the mylar hoop was creating strain and warping sinmat25 we rotated the hoop while keeping the orientation of the diamond the same. The diamond was remounted and a few coarse scans were taken before beginning the fine scan.



Rotation of mylar hoops keeping diamond orientation fixed. Of course the the thru hole without a bolt after rotation was filled so the hoop could be mounted again.

- **sinmat25_032** chi=9.6°, tth=38.738°, phi -31.074, th=20.990° .. 21.050° in 300 steps, 10s shutter exposure time.





If the mylar surface were not having any distorting effect on the sinmat25 diamond, these rocking curve topographs should be identical to what is seen in scan 023. There are similarities, but there are differences that are clearly seen, indicating that the mylar hoop mount is inducing a non-neglible strain.

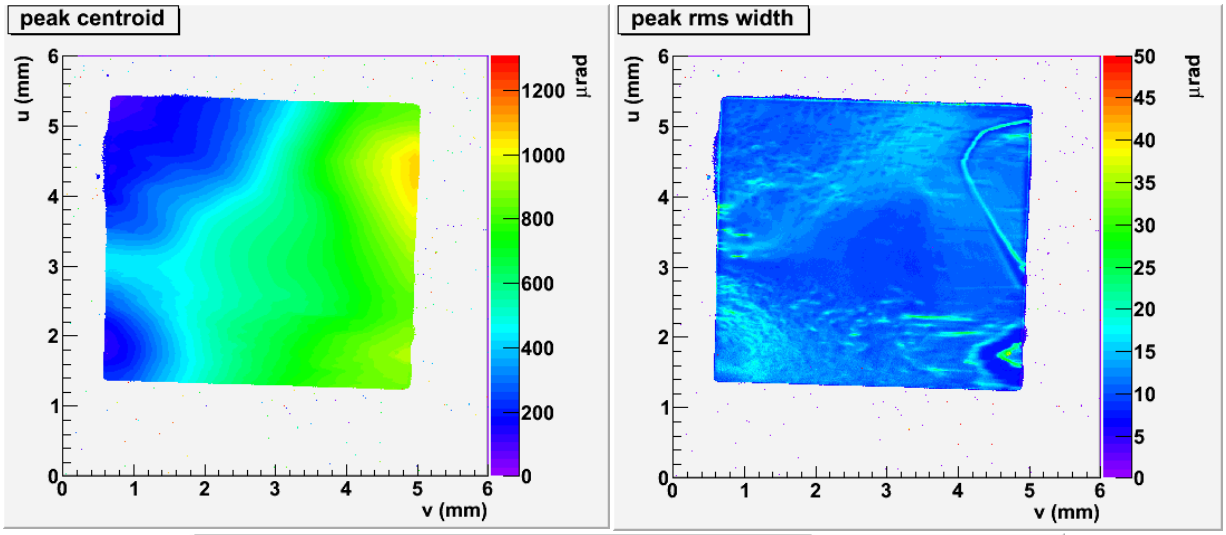
June 9, 2012, BJP, AEB (night shift)

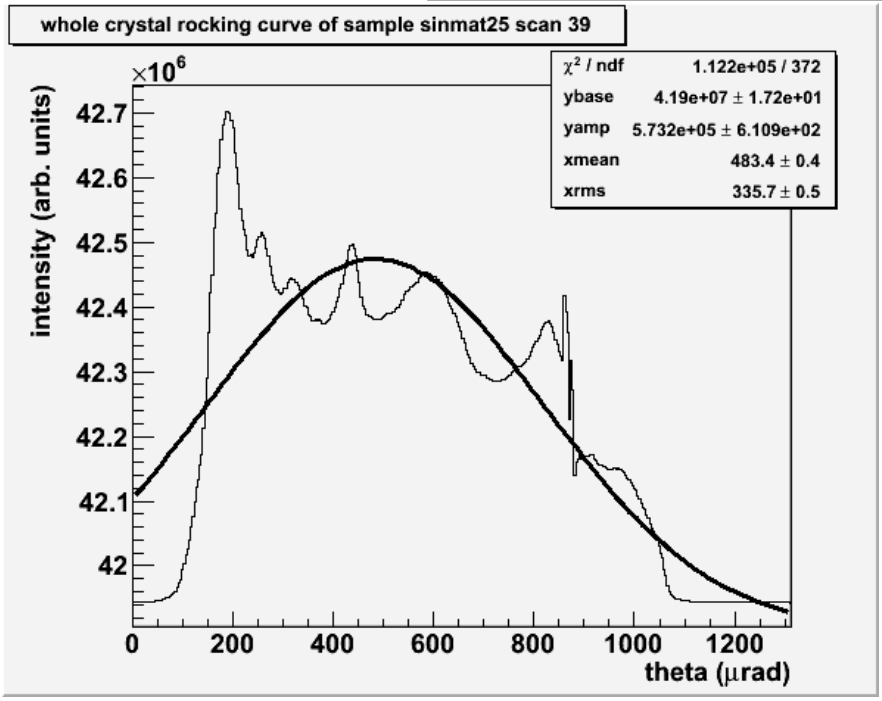
Now we want to look back at the (2,2,0) plane so we can rotate the hoop about the diamond like before. We will rotate chi to about 136°, run a coarse scan followed by a fine scan. Then we will remove the hoop, rotate the empty hole again, scan and go back to the 0,2,2 plane. We moved chi to 136° and phi back to 0° then swept through theta and found the peak.

- **sinmat25_035** : chi=136.00°, tth=38.738°, phi 0.0°, th=17.395° .. 17.455° in 300 steps, 10s shutter exposure time.

We looked at the first image of the scan above to make sure we were starting outside a peak. We were just hitting it, so we broadened the range, keeping the resolution the same. Disregard run 035.

- **sinmat25_039** : chi=136.00°, tth=38.738°, phi 0.0°, th=17.385° .. 17.460° in 375 steps, 10s shutter exposure time.

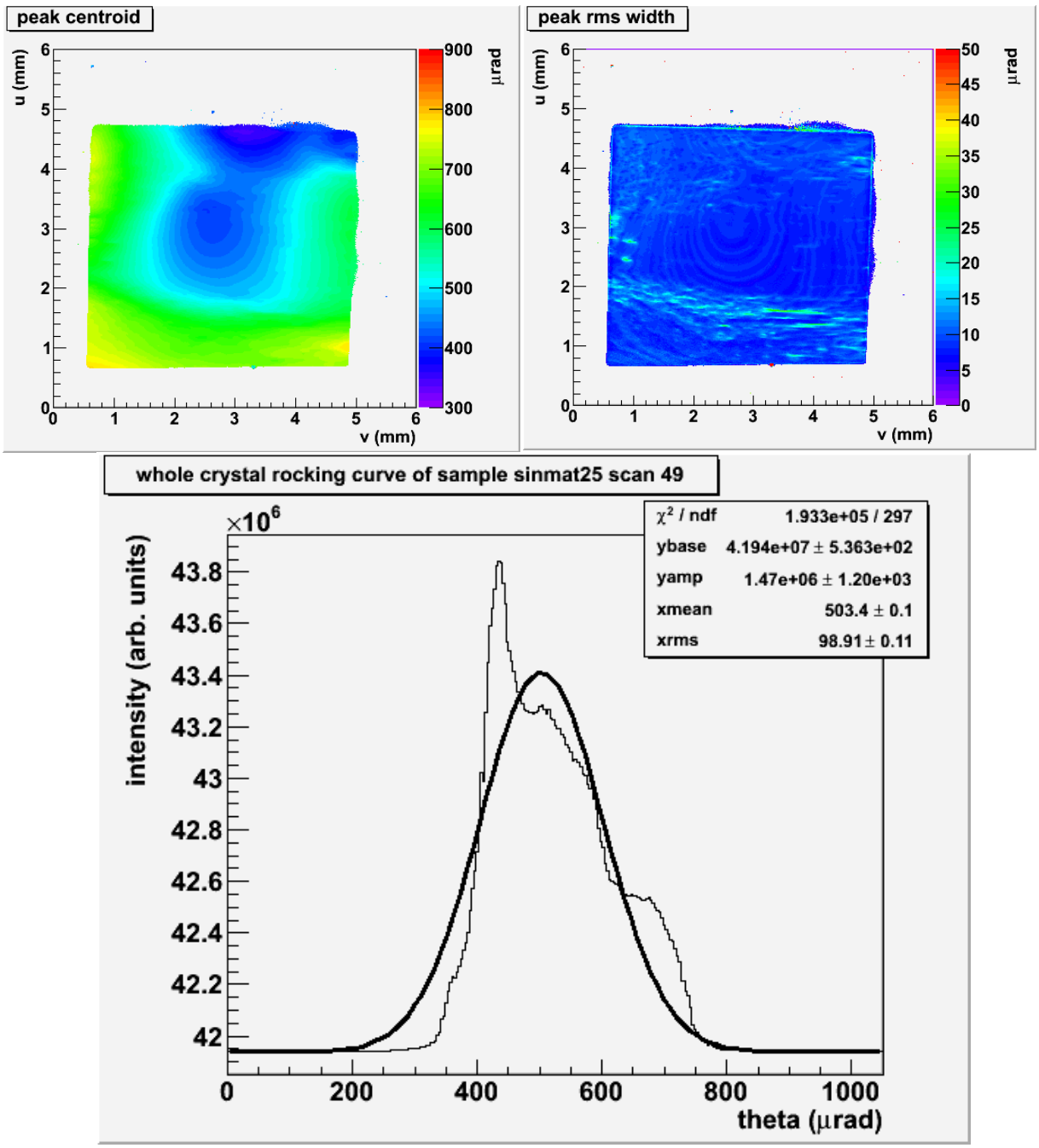




Scan 39 shows sample sinmat25 slid around by about 45° on the mylar hoop, relative to scan 13. The 2,2,0 axis is the one of the orthogonal edges of the crystal.

The mylar hoop has been rotated counterclockwise (same direction as previous rotations) to the next bolt hole. We will scan the 0,2,2 plane again.

- **sinmat25_049** chi=129.50°, tth=38.738°, phi 0.0°, th=17.8420° .. 17.9020° in 300 steps, 10s shutter exposure time.

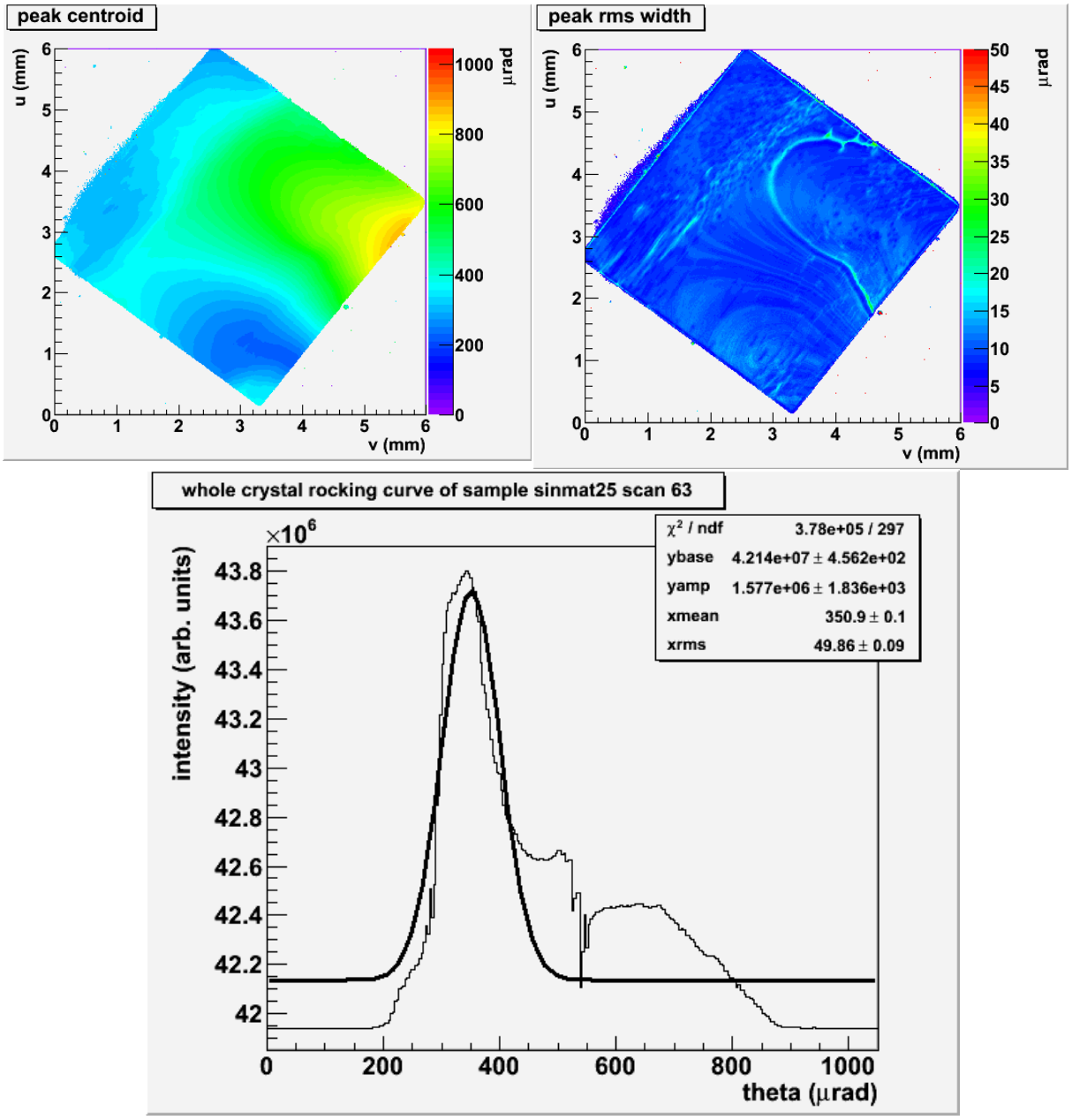


Scan 39 shows sample sinmat25 slid around by a second 45° on the mylar hoop, relative to scan 13. The 2,2,0 axis is the one of the orthogonal edges of the crystal.

Now we go back to the 0,2,2 orientation to take a third scan of sinmat25. Once remounted, we scanned through theta and chi without finding a peak, not good. Then I remembered the 0,2,2 plane is on a particular edge of the diamond and I probably rotated in the wrong direction. Sure enough, after moving the diamond 90° in the opposite direction and remounting the peak was instantly found. We are learning! Note: See above for finding correct orientation

- **sinmat25_063** chi=4.7250°, tth=38.4880°, phi -31.0740°, th=21.020° .. 21.080° in 300

steps, 10s shutter exposure time.



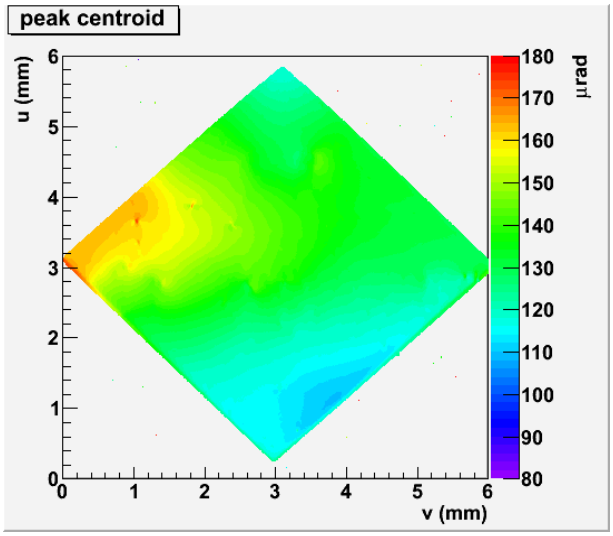
Scan 63 was taken along one of the out-of-plane 2,2,0 axes of sinmat25.
It is the second of a pair with scan 49, in that only the goniometer changed.

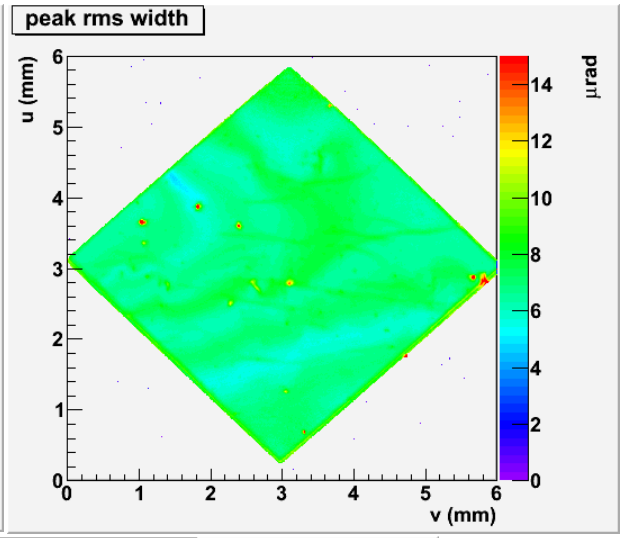
That's it for scanning the sinmat25 diamond and its various repositionings in the mylar hoop. The final diamond to be characterized for its first time is sinmat50. It will be interesting to see if this diamond is also warped. The sinmat100 was excellent...the best we've seen, and the sinmat25 is obviously potato chip-esque in the mylar hoop, I'm guessing the 50 will show signs of warpage too (I was wrong:)). But, that's the end of our shift and we pass the torch to RTJ.

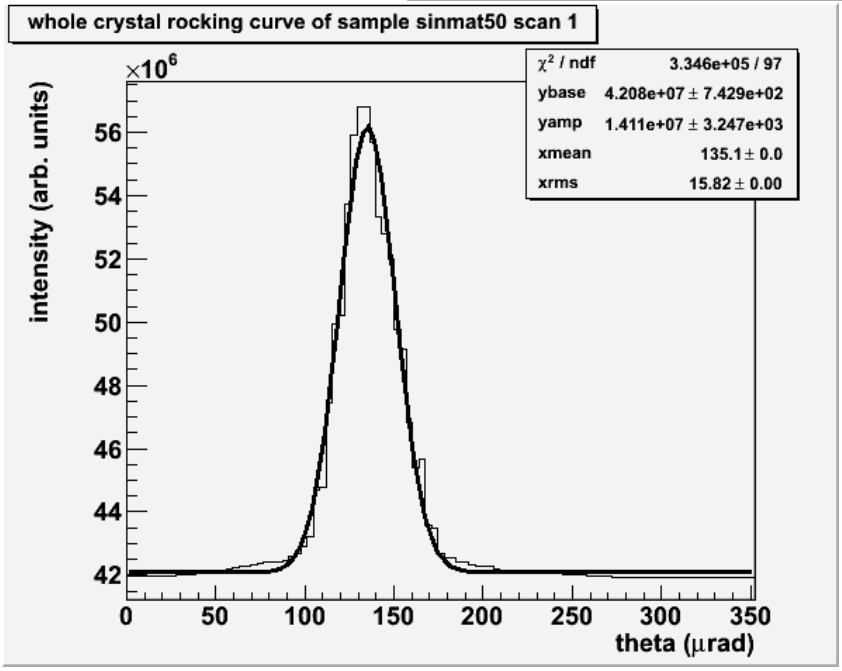
June 10, 2012, RTJ (afternoon shift)

Brendan and Alex mounted the sinmat50 diamond in the mylar hoop in the orientation such that the diagonal of the diamond points up when set at chi=90, theta=0. This was the orientation in which we found the 220 directions in sinmat100 (but not sinmat25). The 2,2,0 reflection was quickly found in this orientation. I had to adjust z (height of the post holding the mylar hoop) to center the image in the ccd frame vertically. I adjusted chi to center it horizontally in the ccd frame. Now I do the first scan of sinmat50. In an initial coarse scan, it looks very narrow. The new series was started, called sinmat50.

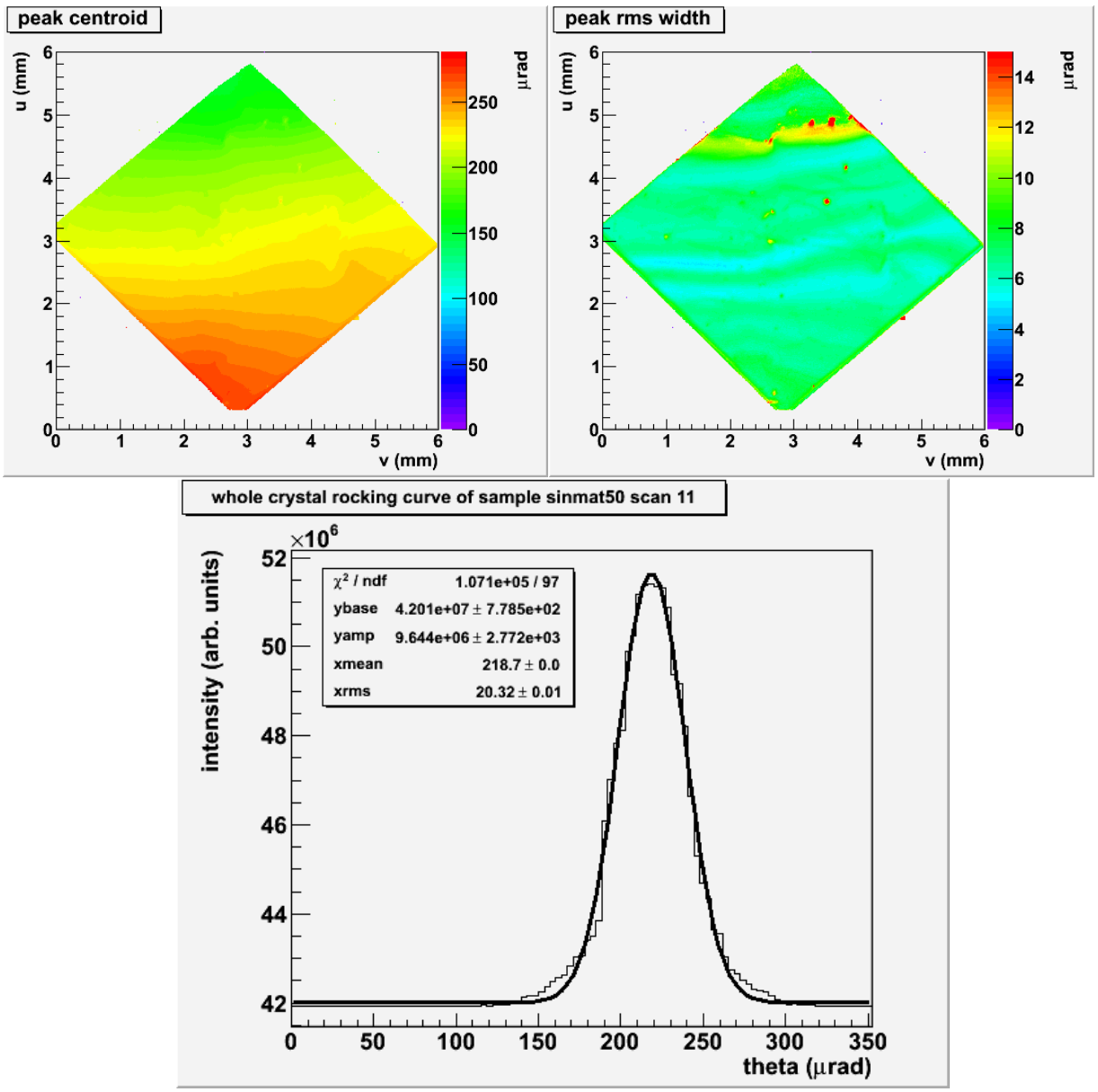
- **sinmat50-001** : chi=91.50°, phi=0°, tth=38.338°, th=18.3900° .. 18.4100° in 100 steps, 10s camera exposure time.







- **sinmat50-011** : chi=0.70°, phi=0°, tth=38.538°, th=20.7500° .. 20.7700° in 100 steps, 10s camera exposure time.

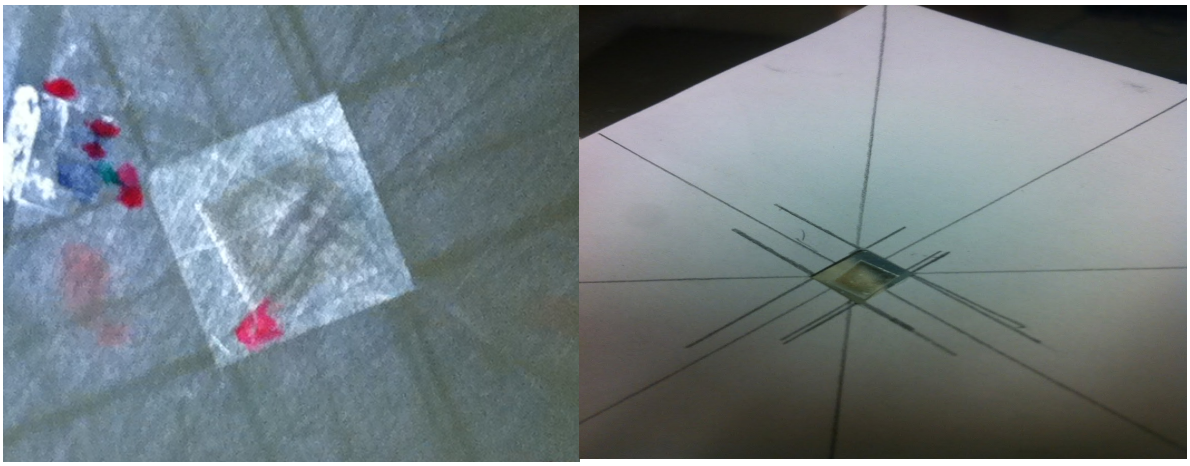


The sinmat50 diamond is an outstanding sample in terms of intrinsic rocking curve and extrinsic curvature. It would meet the GlueX requirements if it could be mounted in a strain-free fashion. Certainly the mylar hoop mount meets this criterion, but unfortunately it cannot be used in an electron beam. Important note: water was not used in mounting the sinmat50 diamond inside the mylar hoop to make it adhere to the mylar. Apparently 50 microns is enough thickness to allow the mylar to grip the crystal without additional adhesives such as a layer of water between the mylar and the diamond. It would be helpful in understanding the shape of the sinmat25 sample if we could mount such that it does not slip, without having to use water to force it to adhere to the mylar.

4. Systematics studies with sinmat25

June 10, 2012, RTJ, BJP (night shift)

During dinner we were spitballing ideas for the remainder of our beam time. We came up with a plan to remove the water component of mounting the sinmat25 diamond in the mylar hoop. There were suspicions that the water was causing some (if not most) of the strain that we see in the diamond. Previously, water was used because the thin diamond would slip between the mylar sheets and the extra surface tension from the water glued the diamond in place. A 9x9cm frame was constructed out of regular printer paper with a 4.5mmx4.5mm diamond shaped hole cut through the middle. The diamond was mounted for the 0,2,2 orientation and the paper was placed on top of it making sure it was not making contact with any of the diamond's edges. Then the top hoop was positioned and fixed. Below you can see pictures taken of the setup including an image showing the shadow of sinmat25 inside our paper frame.



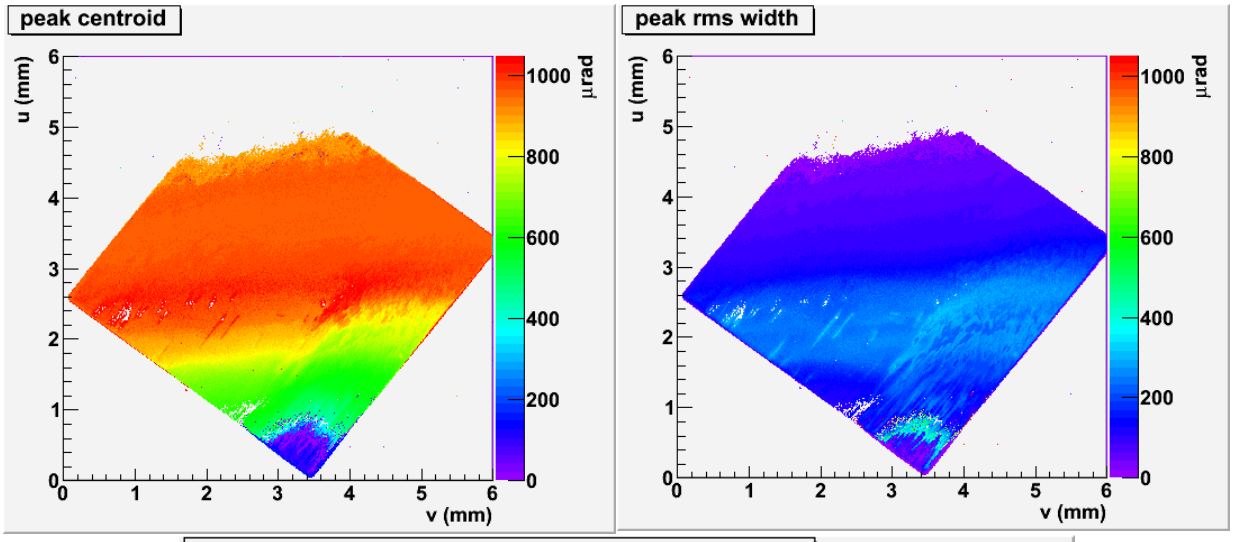
The image on the left shows the diamond in the mylar hoop (using a flashlight to backlight the mylar) and the surrounding paper frame. Once placed on the goniometer the diamond settles into one of the corners. The image on the right shows the basic design for our paper frame, notice the diamond has plenty of space so that it is not overlapping with the paper.

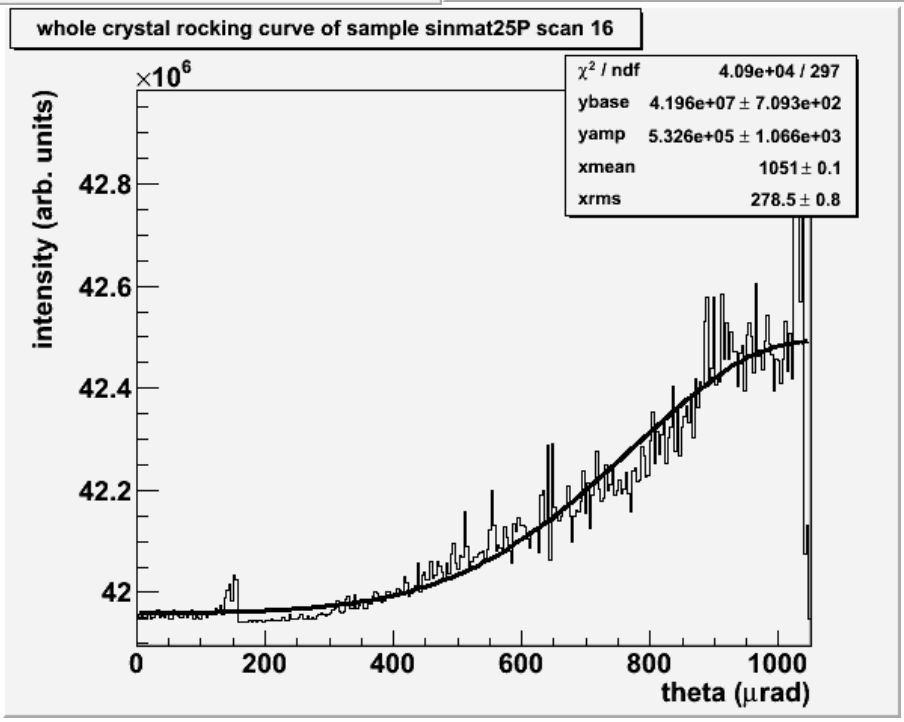
sinmat25 with the paper insert

Sinmat25 was remounted in the mylar hoop, this time using our paper method. A new series was started called sinmat25P (P for paper) and a coarse scan was completed which appeared to showed a much flatter diamond. Next, we proceeded with a fine scan.

- **sinmat25P-016** : chi=1.75°, phi=-31.0740°, tth=38.8380°, th=18.9080° .. 18.9680° in 300 steps, 10s camera exposure time.

The sinmat25P scans are done with sample sinmat25 mounted inside the mylar hoop with another material filling the gap around the diamond to keep it from slipping between the mylar sheets when in the vertical orientation.





Scan 16 was done with the out-of-plane 0,2,2 reflection and office bond paper (nominal 100 microns thickness) in the gap. The huge widths of the local rocking curves shows that this diamond is loose in the mount and moving in the mount in an irregular fashion. This scan is useless.

Unfortunately, what we thought looked like a flatter diamond turned out to be a diamond that was doing a random walk in angle during the exposure time, so that most of the crystal was visible in a single image at fixed theta. However, the image was pretty much invariant with theta, so it was an illusion. Apparently there is too much space being created by the paper in the mylar gap, so the diamond is bouncing around in the box created for it by the square hole in the paper.

sinmat25 with the kapton insert

Maybe the paper is too thick. If we could find a material that is thinner than the diamond, we might be able to box it in with a sheet of this thin material, but still have the diamond in contact with both mylar sheets. To test this idea, we found a sheet of 25 micron kapton and Brendan cut a square hole out of the middle of the sheet, in a similar fashion as he did for the case of the paper insert studied in scan sinmat25P_016 described above. He then installed sinmat25 in the square cutout and put the two mylar halves back together in the same geometry as scan 16. The results are recorded in scan 29. The broadening of the rocking curve from random motion in the mount is reduced relative to scan 16, but it is still present and dominating over the intrinsic rocking curve width measured earlier in the sinmat25 scans.

ALERT -- scan 29 shows no diffraction. There is the faint silhouette of the diamond in the noise of the ccd, but no peaks are visible in the rocking curve. We had seen clear bands of diffracting beam in the coarse scan 27 that covered some of the same range as scan 29 covered. So the crystal moved between the time the peaks were observed in scan 27 and the time that scan 29 reached the same theta. My guess is that sinmat25 was able to wedge a corner between the kaptan insert and the mylar walls, so that it got squeezed into a slightly different orientation than it had when we aligned it. The lesson from this is that trying to stabilize the 25 micron diamond inside the mount by setting it into a square cutout cavity inserted between the two mylar planes is not successful. Either the cutout spacer is too thick, and the diamond is loose inside the cavity and smears out the rocking curve beyond recognition as it jumps around, or the spacer is too thin and the diamond somehow slips into the gap between the spacer and the walls.

June 11, RTJ, BJP (day shift)

For an ideal elastic sheet, the compression force on the diamond is proportional to the thickness (linear restoring force). The force of static friction should be proportional to the compression force, so linear in the thickness. The force of gravity or instantaneous acceleration must be less than the static friction limit if the diamond is to remain fixed in the mount. That force should also be linear in the thickness. If the static friction between the mylar and the diamond is sufficient to keep the first two in position, it should also keep sinmat25 from slipping. Moreover, the areas of sinmat100, sinmat50, and sinmat25 are all the same, so even corrections to the simple model of static friction being independent of the contact area for constant normal force should not contribute. This argument indicates that if sinmat100 and sinmat50 have enough static friction from the compression force produced by the tension on the mylar sheet to keep them from slipping, then the same should hold for sinmat25. However, it doesn't.

Therefore there must be something else at work with sinmat25 that makes it slip when sinmat50 and sinmat100 do not. Here are some possibilities to consider, for why sinmat25 is slipping inside the mount when sinmat50 and sinmat100 do not.

1. Samples sinmat50 and sinmat100 are planar, whereas sinmat25 is curled and so has smaller surface area in contact with the mylar than the former two.
2. Imperfections in the surfaces between the two mylar hoops keep the two sheets from having a truly zero gap when there is no sample inside. Thus there is positive x-intercept in the linear relation of compression force vs sample thickness. Thus the compression force drops faster than linearly at small sample thicknesses, and sinmat25 has too small a compression force to sustain sufficient static friction to withstand gravity.

Hypothesis 1 is plausible, but unlikely for the following reasons. Curling of the sample would actually make its effective thickness greater than its weight would imply, giving the advantage

to static friction over gravity, relative to the case of a planar sample. Simple models of static friction assume that the static limit is independent of area and depends only on the normal force. This is over-simplified, but probably a good approximation for small normal forces and smooth hard surfaces, which is very much the case here.

Hypothesis 2 is clearly correct at some level. The equilibrium separation between the two mylar sheets at the center of the hoop is set by their average separation at the rim. However their separation at the rim is set by their maximum height difference, which is greater than zero and has a scale set by the precision of the machining and gluing process. It is hard to believe that this would be smaller than 0.001" (25 microns) which is the thickness of the diamond. I conclude that hypothesis 2 is most likely the correct explanation.

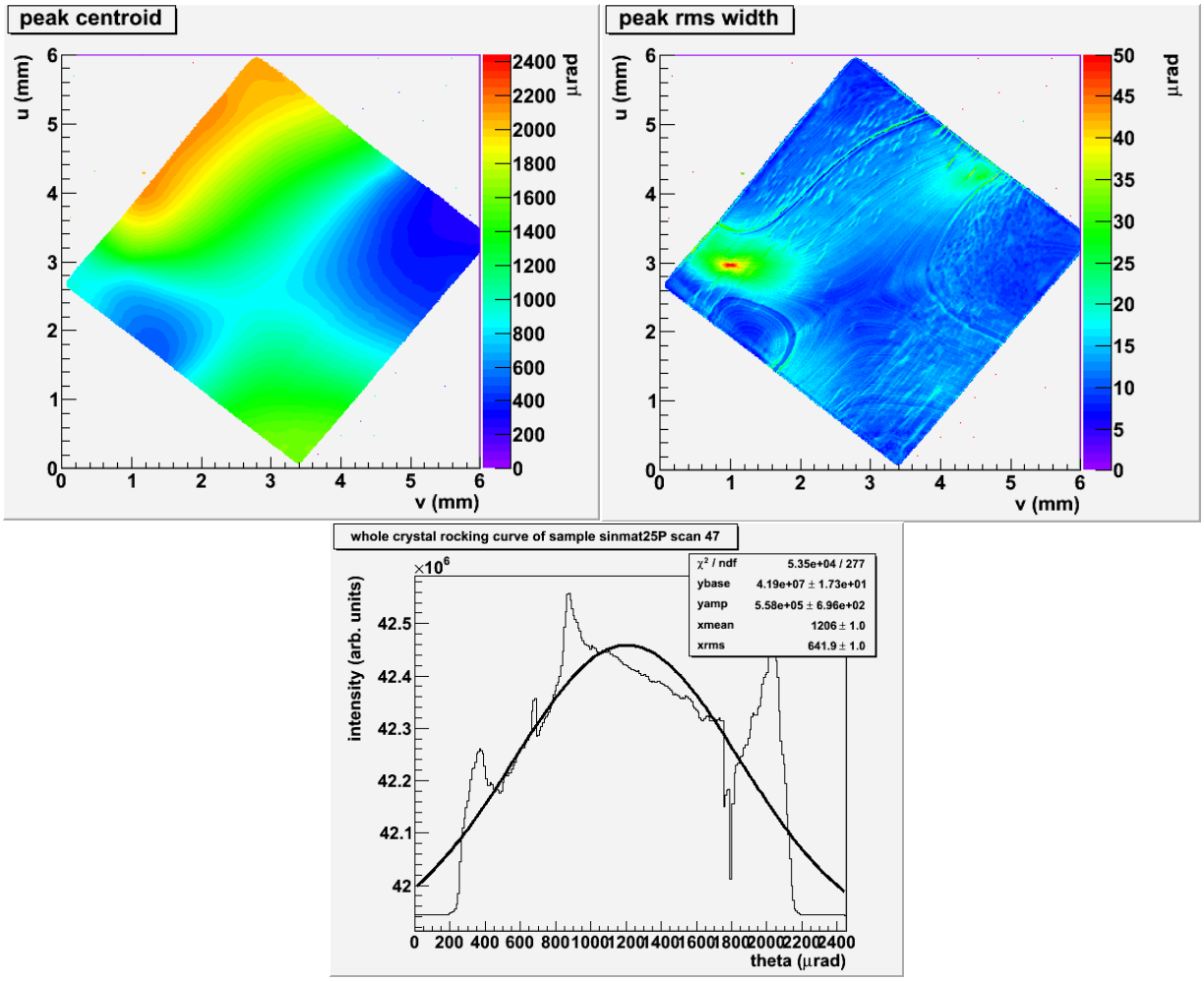
To test hypothesis 2, I need to shim the sinmat25 sample so that it feels the compression force necessary to hold it in place. One way to do this would be to place it on top of a flat sheet of similar thickness. Here I run the risk that the flat sheet will not be flat enough, and will deform the diamond as the two are pressed together. To minimize this effect, I want to have as thin and flat a "shim" as possible. Why not use one of the other diamonds? For example, I can stack the sinmat50 and sinmat25 on top of one another in the mount and scan both of them in place. The compression force should be large enough because the combined thickness of sinmat50 and sinmat25 is 75 microns. This is intermediate between sinmat50 and sinmat100, neither of which slipped in their respective scans.

sinmat25 shimmed with sinmat50

In the stacked configuration, we should sequentially scan both sinmat50 and sinmat25 samples. The scan of sinmat50 should show that it is still flat in spite of being mounted next to sinmat25 (should be the case) and the scan of sinmat25 will be the first measurement we have been able to make of its shape in a stable mount configuration without involving water as an adhesive. The nice thing about this configuration is that the cuts of sinmat50 and sinmat25 are so different that there is no chance of overlap between their diffraction topographs. By aligning the two diamonds exactly on top of each other, their 2,2,0 directions will be at least 30° apart.

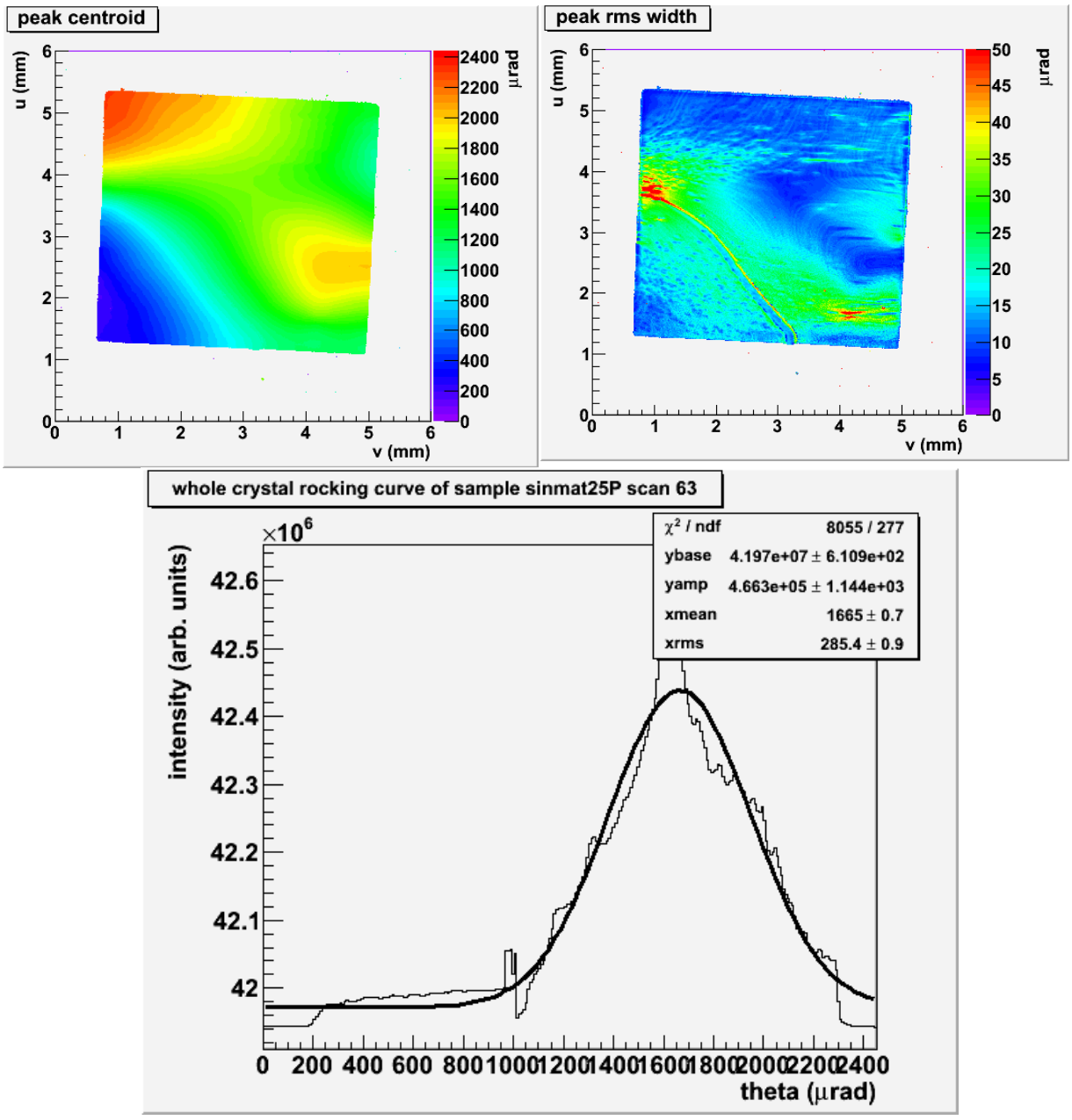
- **sinmat25P_047** : chi=7.30°, phi=-31.074°, tth=38.838°, th=20.480° .. 20.620° in 280 steps, 10s shutter interval. This run stopped after 80 frames because the camera had to be rebooted. The same scan continues from frame 80 in scan number 55.

- **sinmat25P_055** : chi=7.30°, phi=-31.074°, tth=38.838°, th=20.520° .. 20.620° in 200 steps, 10s shutter interval.



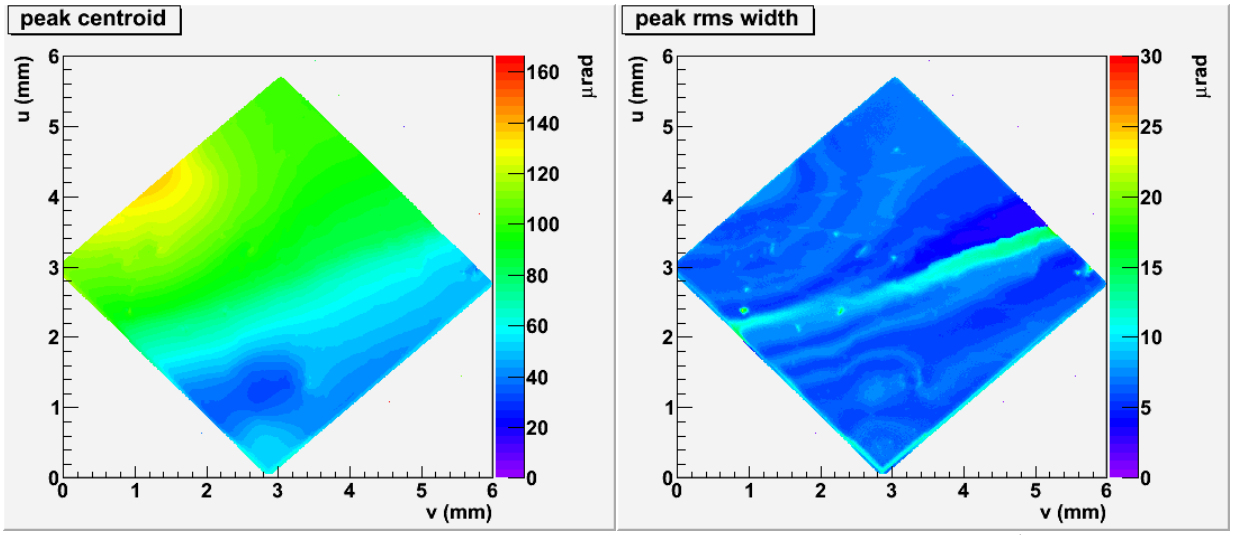
After scan 47/55 finished, I rotated the goniometer back to the chi=135° position and found the other 2,2,0 reflection corresponding to sinmat25. I did this without moving the mount. I now take a second scan of this configuration around a different 2,2,0 reflection

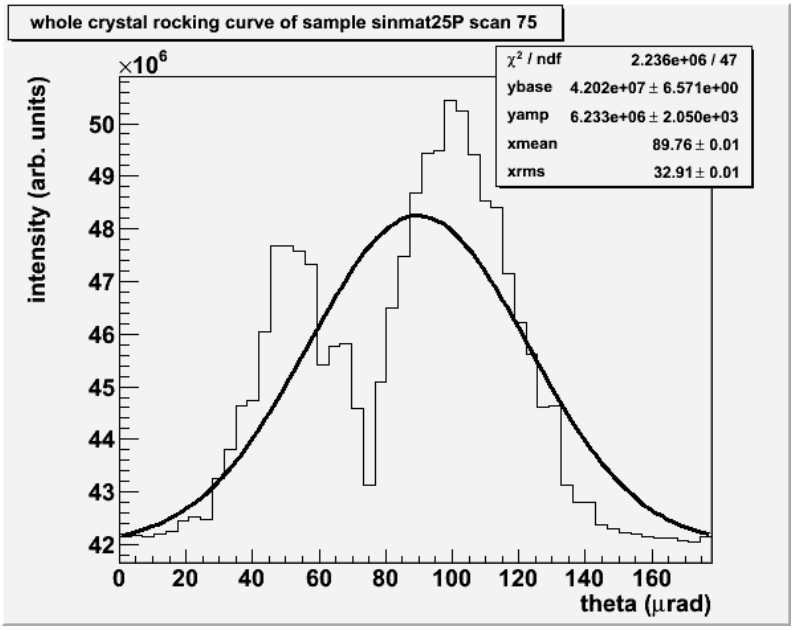
- **sinmat25P_063** : chi=132.50°, phi=-2°, tth=38.838°, th=18.850° .. 18.990° in 280 steps, 10s camera exposure time.



Now we switch over and do a couple of quick scans of sinmat50 in its current position under sinmat25. This is to show that the forces from the mount are not sufficient to appreciably distort the 50 micron diamond.

- **sinmat25P_075** : chi=88.70°, phi=-2°, tth=38.338°, th=18.3660° .. 18.3760° in 50 steps, 10s camera exposure time.



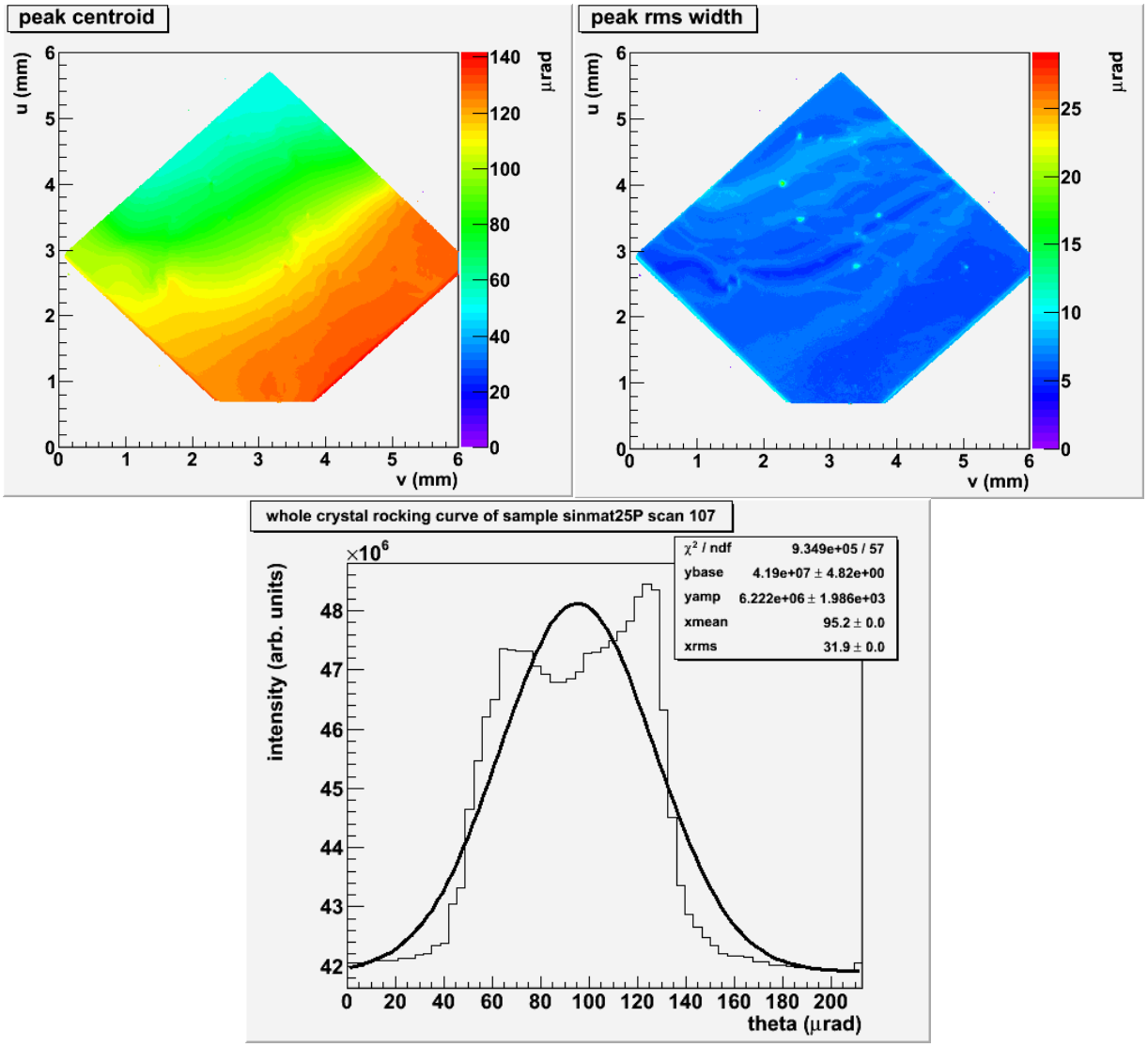


Here we see that sinmat50 is strained, relative to what he looked like when he was mounted alone in the mylar hoop. The shape is close to a single fold along a line through the center at 20° from horizontal.

June 11, BJP (night shift)

I'll begin with picking up where RTJ and AEB left off, scanning the 2,2,0 plane of the sinmat50 sandwiched between sinmat25 and mylar. Using a coarse scan I will find the scan length (which is rather short due to the low r.c. of this particular diamond) and begin the fine scan.

- **sinmat25P_107** : chi=-0.3°, phi=-2°, tth=38.738°, th=19.318° .. 19.330° in 60 steps, 10s camera exposure time.



sinmat25 with kapton shim

Okay, this scan is finished and I will now try a new technique suggested by Ken which involves replacing sinmat50 with a sheet of kapton which has a thickness of about 25 microns. I will first image in the 0,2,2 plane and then take the complementary rocking curve at 2,2,0.

Taking the mount apart in order to remove sinmat50 from the hoop I noticed that the diamonds are not clinging to each other (which is what I thought previously). Sinmat50 was freely sliding across the surface of sinmat25.

Once sinmat50 was removed, I cut a large piece of the thin Kapton film and cleaned it on the thick, lint free cloth making sure it was free from wrinkle. I then reoriented the diamond and placed his Kapton blanket over him. Using a flashlight, I inspected the diamond transition from horizontal to vertical, wanting to make sure he didn't slide...he didn't. I then remounted him in the goniometer, centered him in the scope and found a quick peak. I'm currently running the coarse scan which will of course (no pun intended) be followed by the fine.

Just looking at the initial images, the worms are fat again. Presumably, the diamond is still vibrating inside the mount. I don't think it's worth running another scan of a vibrating diamond, so I will remove the hoop and place a second sheet of kapton underneath the diamond, creating a kapton, diamond, kapton sandwich, mmmm. Looking at the kapton Ken gave us, it is thinner than the mylar sheet RTJ and I spec-ed out last night which may explain why we were still seeing vibrations. I will try to take a reading of it during the scan, I also put a sample away for use to take home.

After remounting the diamond with the two layers of kapton, I was able to find the peaks again. Taking only one image already shows a large decrease in the worm's width. I was correct in assuming their broadening was due to vibrations, and not intrinsic shape. Now I am realigning the diamond in the ccd and will begin a new fine scan.

- **sinmat25P_133** : chi=4.45°, phi=-31.074°, tth=38.638°, th=20.010° .. 19.060° in 250 steps, 10s camera exposure time.

More frustration, after 133 points, there is no data. Theta values that had clear peaks are now showing nothing; meaning the diamond changed position during the run. I will add another two sheets of kapton to be sure it won't budge. Increased the number of sheets to four, remounted, realigned and found peaks. Brought into ccd camera and began coarse scan which looked fine. Then, started the fine scan, got to thetas that showed images before and again found nothing. The gremlins are starting to bug me. Going to add two more sheets of kapton and try this all over again.

Mounted and scoped I found the peak after applying a total of 6 Kapton layers. For some reason the image was so far over on the detector I hit the limit of its motion. I believe this material is my kaptonite. I decided to switch over to using one thin layer and one layer of the thicker material we used previously to make the Kapton insert in the interest of time.

Time is running out and I need to start a scan now if I'm going to start one at all.

- **sinmat25P_161** : chi=3.7°, phi=-31.074°, tth=39.5130°, th=20.450° .. 20.50° in 160 steps, 10s camera exposure time.

Not exactly how I imagined ending our time here at CHESS, but at least we learned that it takes more than 6 layers to dampen the vibration of the diamond, if not more.

85 images in and at least we have some data finally. Just got word that it's quarter past 7am and they will be shutting the beam down shortly, where did the time go???? Thumbing through the images it looks like we made it into the black which is a relief. But, somehow I missed that the diamond was clipped again at the top corner, I can't seem to win today. Note, when they shut the beam down, they really mean business..alarms and sirens that certainly wake you up.

Possible things to try next time

- The main question this visit has been how to hold and analyze a thin diamond without altering its intrinsic shape. We've tried multiple versions of trapping the diamond between sheets of highly tensioned mylar. One thought (which may be ridiculous) I had was to suspend the diamond in a mount that was filled with a viscous fluid like oil. This mount would mimic the pitch fork idea used for dave, allowing the diamond to retain its

original form, but the fluid would help dampen the vibrations. Although, the more rest I get the crazier this sounds.
- Definitely revisit the use of kapton as a “shim” within the mylar hoop. Given more time, I think we could have seen some interesting results.
- If we get more samples from e6, it would be nice to first analyze the pristine versions and then ablate them to varying depths. This would allow us to not only study how ablation damages diamonds as a function of depth, but we can also look at how impurities and strains are propagated throughout the crystal.
- It appears that etching is in our group’s future, and I would like to etch Dave and reimage him to see if we can eliminate the strain shown in the rocking curve measurements.
- In terms of equipment. We should seriously consider investing in the new camera. Currently we can process about one image a minute. As we all know, this takes forever and greatly reduces the amount of data we can take throughout our beam time.

Lessons learned after first visit to CHESS - BJP

- First and foremost, it is really important to have a knowledge of crystal orientations and to use this picture in your mind to troubleshoot when you’re not seeing a peak (or other issues).
- When tightening the two halves of the mylar hoop, make sure you don’t over do it. Finger tightening with just a turn or two with a wrench afterwards is plenty otherwise you begin to ruin the threads, or worse, crack the adhesive. This is also true when mounting the hoop to the goniometer, less is more.
- To get nice, quick peaks, make sure the diamond is in line with the telescope. A helpful tool is using a flashlight to illuminate the diamond (which is otherwise not visible) in the mylar hoop and putting it in the telescope’s sight.
- When adjusting chi and theta to get the diamond peaks into view, be mindful of the typical scale of adjustment for each angle. Chi adjustments (which moves the image either left or right in the camera ccd depending on sign) can be on the order of a degree, while theta (adjusts intensity) and twotheta (adjusts vertical displacement on ccd) adjustments are 0.1° for coarse and 0.01° for fine.
- When everything seems to be set up correctly and you still can’t find a peak, you’ve most likely done something wrong. Starting from the beginning and working your way through instead of just wildly scanning chi and theta will end up saving you time. First check to see if your target is in the telescope. Then see if you are getting beam by using fluorescent paper against your target. When looking for the peaks in the monitor, make sure you have typed **ccd_off** and **opens** commands into your fourc terminal. If you still can’t find a peak, reconsider the orientation of the diamond. You should roughly know what theta and ttheta values are required for each orientation. It’s always possible (as we have seen) that the diamonds are not as simple as you’d like.
- Be careful when using the mv command to adjust theta, chi, or phi. It’s easy to confuse it with the tweak command (tw), but could have huge consequences. For example, if you intend to tweak theta by 1°, but use the mv command, you could send the goniometer into the detector (which didn’t happen, just to be clear :)).
- Placing the diamonds on the mylar hoop can be stressful, especially with the 10-20 micron pieces. What I like to do first is clean the central region of the hoop using a few drops of ethyl alcohol and a lens tissue. Once this is dried, lightly wet the corner of a Kim wipe and use it to pick up the diamond and place it on the hoop. To detach the diamond from the moist paper, I use one of the thin plastic sheets that cover the diamonds in their box and hold the diamond down while I pull up on the tissue. Removal is the same but

reversed and sometimes a little water is necessary to get the diamond to detach from the mylar.

- Finally, get some sleep if you can during long runs. That way when they're finished you'll be a bit more fresh and can get more data through the day.