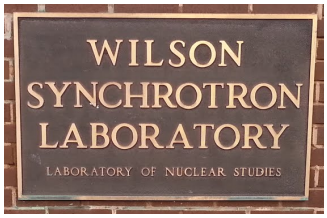
CHESS Run Summer 2014



May 28 - June 3
participants: Alex Barnes, Ken Finkelstein, Liana Hotte, Richard Jones, Brendan Pratt, and Nathan Sparks

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What to do before leaving CHESS

Goals for this run

1. Adjust the focus and zoom of the camera to allow the 7x7mm diamonds to be imaged fully.
2. Take rocking curves of the following diamonds with 4 scans each, one for each position of the (2,2,0) planes. If any do not fit within the view of the camera, take repeated scans to cover the full surface area.
    a. JD70-1 … JD70-10, 10 samples
    b. UC45-6-S200_50 and UC45-7-S200_50
    c. UC30-9-C300_25 and UC30-10-C300_25
    d. UC45-2-C500_300 and UC45-1-C500
    e. UC45-1, UC45-3, UC45-4, UC45-5
    f. UC45-11 … UC45-15
    g. UC30-15 … UC30-25, as many as we get to.
3. Ken suggested we scan a diamond without the beam to get a base for background

Useful information

- Phone numbers
    - C1 hutch: 607-255-0256
    - Ken's office: 607-255-0914
    - See paper under the monitor for Ken's home and cell
- To edit an image in a terminal:
    - display <*filename*>.tiff
    - left click to open menu, click enhance -> normalize
    - click view -> resize -> change to 320x270
    - save -> <*filename*>.png
- To take a screenshot in Linux
    - Whatever window you want a screenshot of, make sure it is visible
    - open a terminal and type xwd > <*filename*>.xwd (xwd file type recommended)
    - The cursor should have changed to a plus sign. Click on the window you want a screenshot of.
- To open the ANDOR GUI
    - In the terminal type the command *andorview*

FourC Commands

Motor names: *tth* - two theta, *th* - theta, *chi* - chi, *phi* - phi, *topsc* - flourescent top screen, *spinz* - high of the hoop along the phi axis

- *ad_lineup_on <time in second>* is the command to turn on the image updater
- *ad_lineup_off* is the command to turn off the image updater
- *tw <motor> <increment>* - tweaks a motor an increment from your current position
- *set <motor> <position>* - declares the current motor position

- *mv <motor> <position>* - moves a motor to a absolute position
- *wh* can be used to check the current values of all settings
- *newfile <filename>* - start a new file for each diamond. The filename should be the diamond name - diamond number - study1 (Ex. JD70-1-study1)
- *ad_on* is the command required before starting a scan
- *ascan <motor> <starting position> <ending position> <number of steps> <exposure time>*

Goals for this run

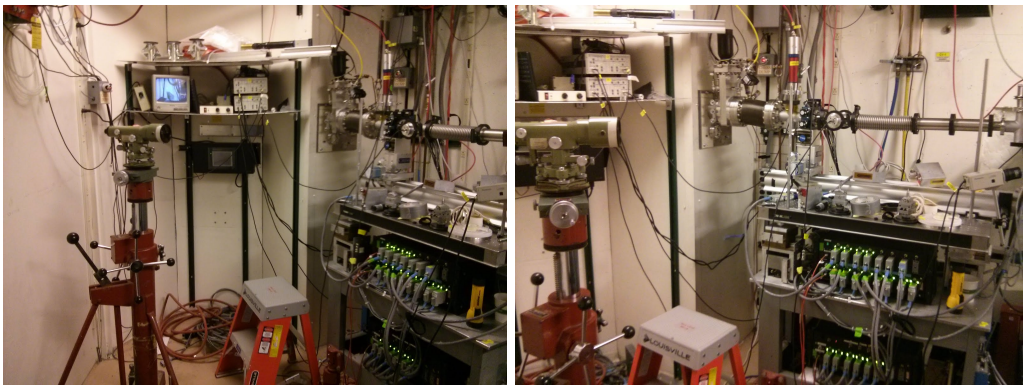
4. Adjust the focus and zoom of the camera to allow the 7x7mm diamonds to be imaged fully.
5. Take rocking curves of the following diamonds with 4 scans each, one for each position of the (2,2,0) planes. If any do not fit within the view of the camera, take repeated scans to cover the full surface area.
    a. JD70-1 … JD70-10, 10 samples
    b. UC45-6-S200_50 and UC45-7-S200_50
    c. UC30-9-C300_25 and UC30-10-C300_25
    d. UC45-2-C500_300
    e. UC45-1...UC45-5 (not including UC45-2 again)
    f. UC30-15 … UC30-25, as many as we get to.
6. Ken suggested we scan a diamond without the beam to get a base for background

Conditions of the experiment

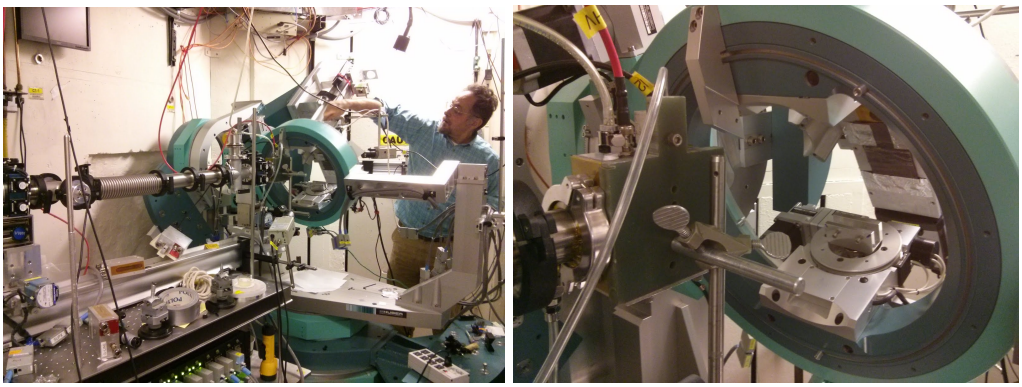
- Beam current:
    - start of fill: 199.35 nA
    - end of fill: 170 nA (electron beam only, positrons are topped up every 3 minutes)
- Beam dimensions:
    - 8mm x 8mm normal to beam
- Camera dimensions:
    - approx 8mm x 7mm
- I0 current:
    - start of a fill: ~55000
    - end of a fill:
- Time between fills: 120 minutes
- Duration of one fill: 5 minutes
- Inside the ANDOR GUI:
    - Image binning: 1x1
    - Image size: 2560x2160
    - PreAmp gain: 16 bit (low noise & high well capacity)
    - Encoding: Mono16
    - Shutter mode: Rolling

- Readout rate: 100MHz
- Overlap: Yes
- Noise filter: Yes

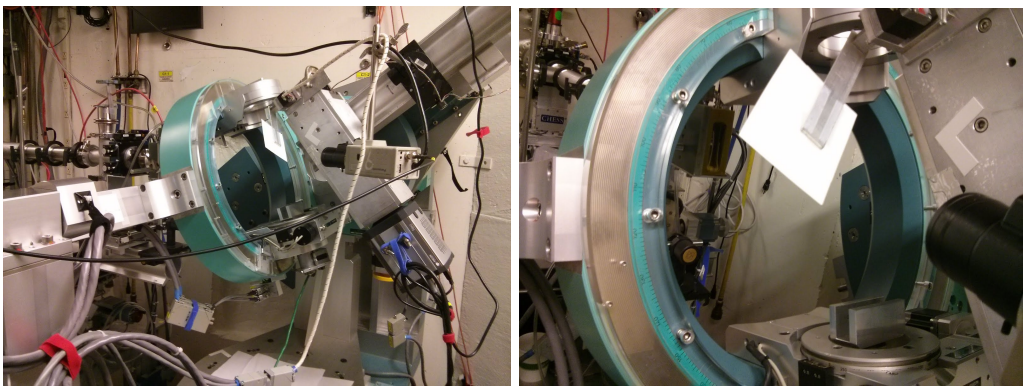
Photos of the C1 hutch and setup



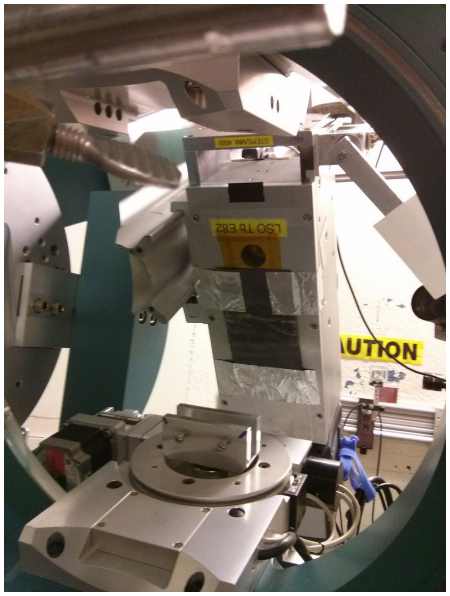
Images showing the scope used to check center of rotation as well as the beamline.



View of the mount from the scope's direction.



View from the opposite side of the mount. The fluorescent paper is connected to a motor to bring it in front of the detector.



Close up of the mount location and detector.

Adjusting the setup

Wednesday, May 28, 14:00

Notes from Ken
Ken will send a report about playing with the Andor camera and adjusting the focus. The previous setting was 3x magnification, for which the nominal field of view was 5.461mm x 4.608mm. Ken measured the vertical dimension of the camera frame by looking at an image and estimated 5.4mm in vertical. Using the nominal scale factor it would be 6.39 in the horizontal, but he's not sure if it's right (for 3x magnification). He looked at the back of the camera and verified it was at the 3x setting. Inside the detector we can see that the horizontal limit is set by the holder at 7mm. The vertical can go further depending on the magnification.

Ken provided this link to a document describing the resolution of the Andor camera.

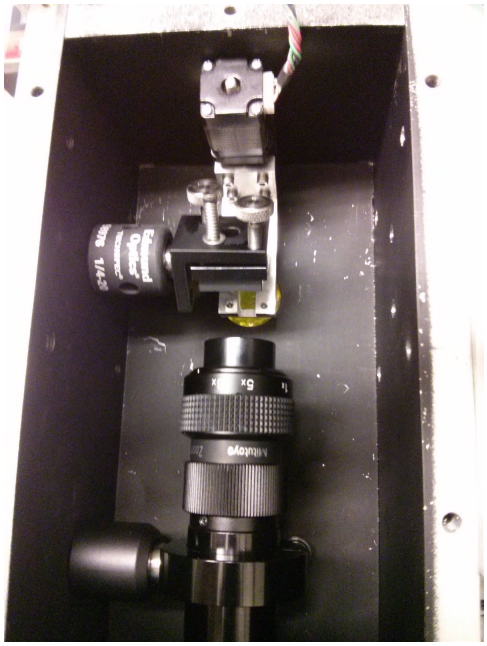


Image of the detector with the back open. The vertical bars near the yellow circle dictate the maximum horizontal size.

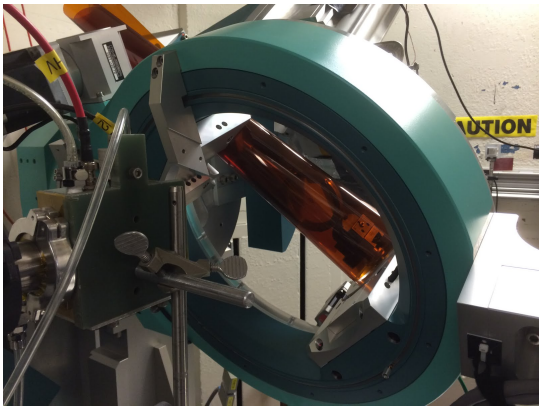
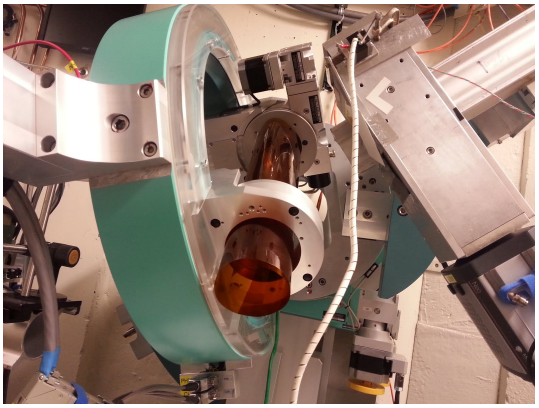
Ken has changed the magnification from 3x to something smaller (he's approximating 2.7 or 2.8 but there's no actual click for that setting). He is looking at the image on the screen to determine how much crystal can be seen. The focus has changed so he is also adjusting *camf* to alter the focus. There is a crystal screen that converts the X-rays to visible light and *camf* is the FOURC control variable used to move the screen.

camf is now at -0.100 and is in focus at this new magnification.

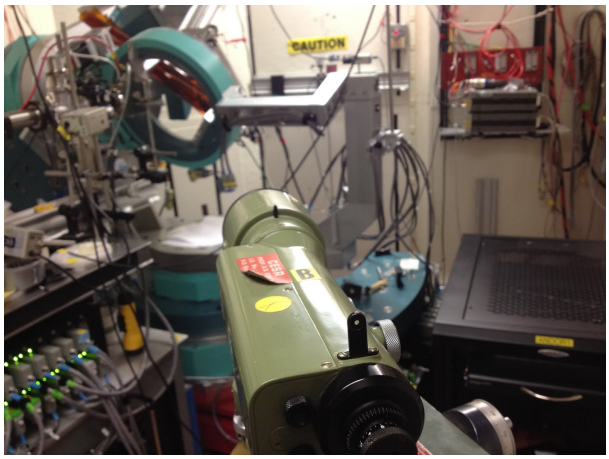
The distance from the center of rotation of the goniometer to the plane of the external camera is now approximately 130 ± 5 mm. We mounted JD70-1 in the small hoop (clear mylar, belongs to Ken) and oriented the goniometer as follows. A 2,2,0 reflection was found at the coordinates,

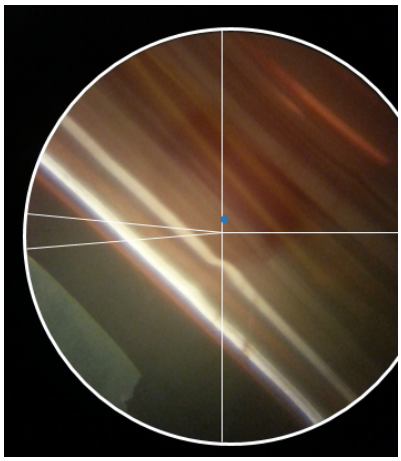
two theta = 38.3, theta = 20.581, chi = 137.25, phi = 180.9

We found that with the current setup and the small clear mylar hoop, there was enough vibration to significantly broaden the rocking curve peak. This was visible in the flutter observed in video mode with the camera, with the diamond oriented on one side of the rocking curve peak to maximize sensitivity to angle fluctuations. We installed a loose cylinder of kapton foil around the target holder, extending over the base of the target mount and up through the ring on the opposite side of the chi circle. A picture of the air current shield is shown below.

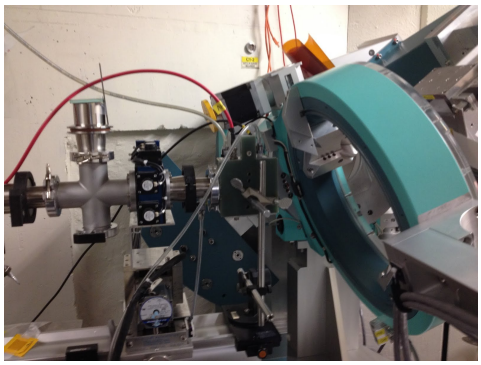
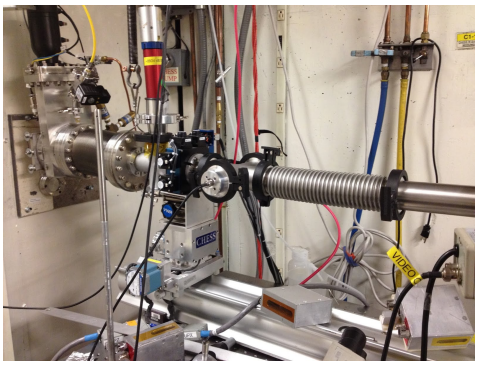



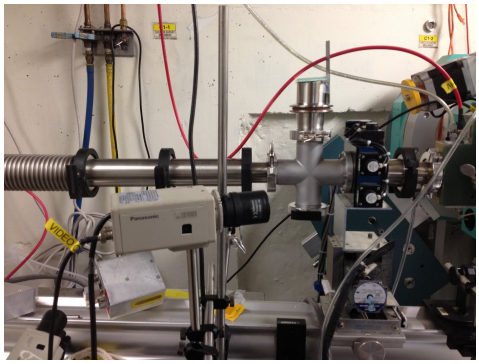
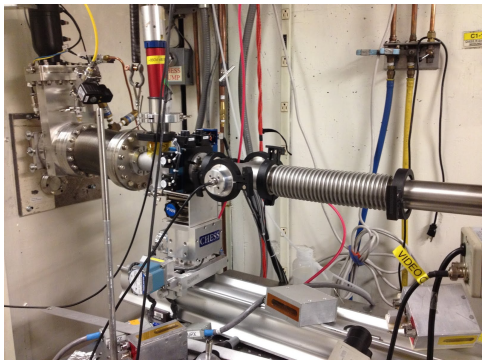
Images of the small clear-mylar hoop target with the cylindrical envelope of 3mil kapton wrapped around it to shield the target holder from air currents, which shake the target and artificially diminish the resolution in theta for the rocking curve scans. Using this kapton shield, the flutter seen when viewing the 2,2,0 reflection at a static angle was diminished to nearly undetectable levels.



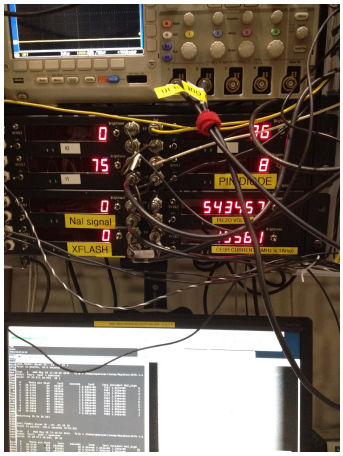


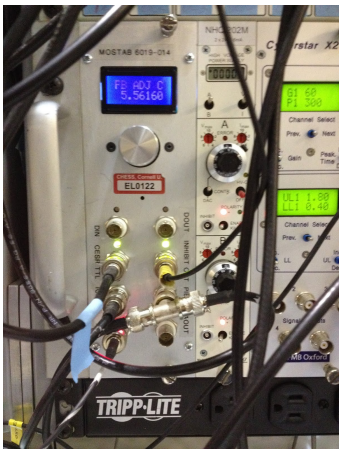
Some views inside the C-line hutch. The telescope shown in the left panel is aligned with the center of rotation of the goniometer. The cross hair image at the right shows what is seen looking through it at a hoop whose center is marked with the blue dot.

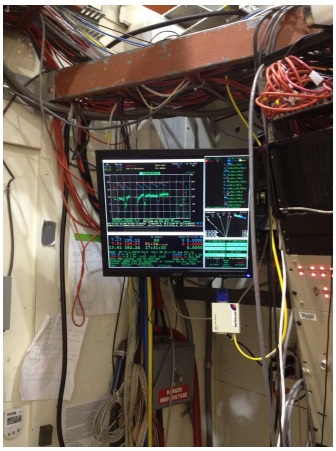



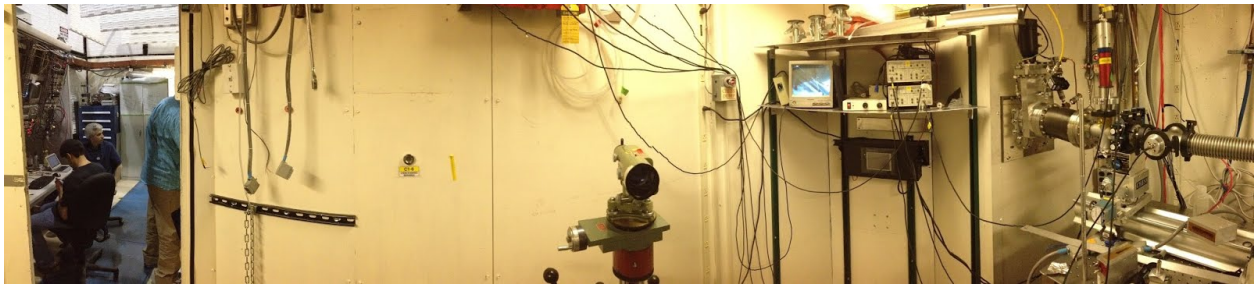
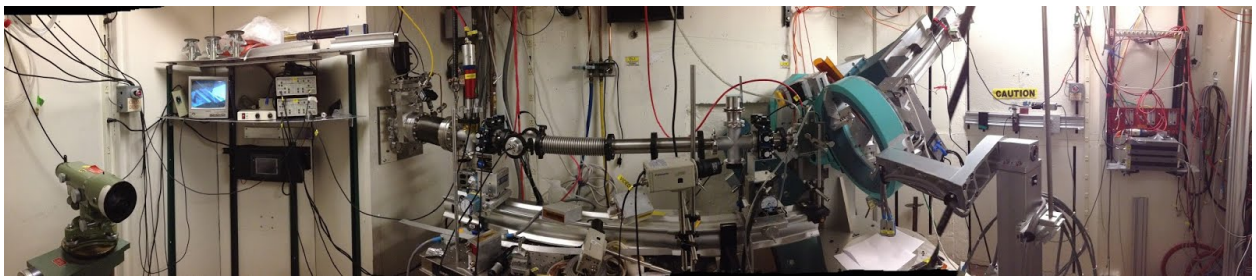
Different views of the beamline inside the C1 hutch. The goniometer is new since the last run, and features 6 circles, including the original 4 (theta, 2theta, chi, phi) plus two more rotations around a vertical axis that allow the target holder and the detector arm to rotate for out-of-plane measurements. We are not interested in those degrees of freedom for our measurements.

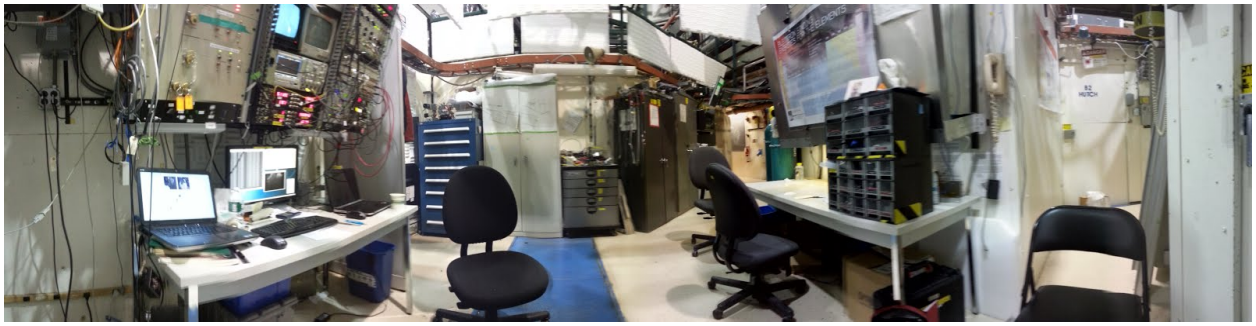




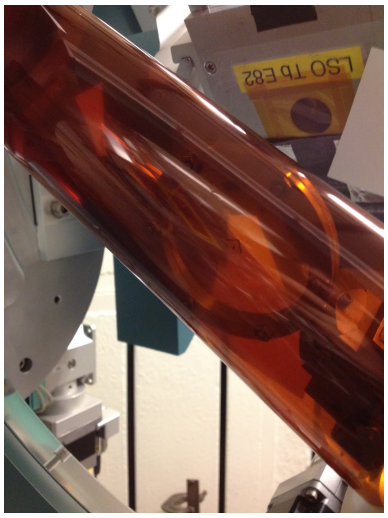


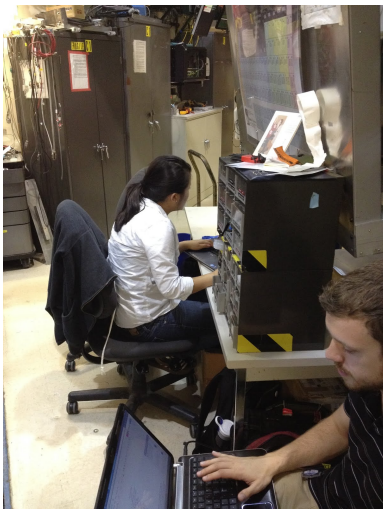
Views of various run conditions displays. The left panel shows the rates in various C-line beam rate monitors. The middle panel shows the front of the module that tweaks the silicon monochomator, to keep the beam spot intensity inform and centered on the aperture. On the right is the accelerator status screen.



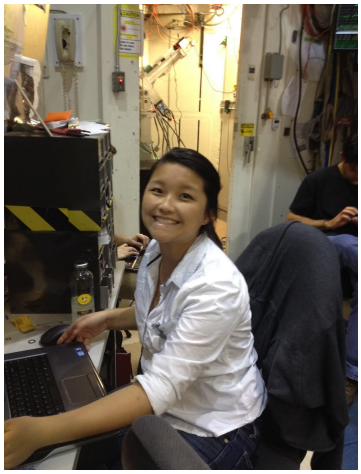
Panoramic views inside the C1 hutch and in the work area outside the hutch.





Close-up of the target hoop inside the kapton air current shield (left panel). In the right panels are shown views of physicists working at the experimental station outside the hutch.










More snapshots of the run crew at work

Scans of JD70-1

We started a scan with tth = 37.900, chi = 137.25, phi = 180.9, th = 20.575..20.590. This provides a step size of 0.0002 degrees with 75 steps. The scan was started at 19:38 using the following command:

ascan th 20.575 20.590 75 10

Between scan003 and scan004 we manually turned phi by 180 degrees. We removed the kapton surrounding the mylar hoop to do this. By rotating by 180 degrees our orientation gives us the negative of our previous measurement. By manually changing phi by 180 we don't have to go to extremes in chi.

After manually changing a motor, the motor must have its position updated! To do this type the following:

set <motor> <new value>

For phi we moved from 180.9 to 0.9 and used the command *set phi 0.9*.

Also between scans we discovered that the air conditioner was turned off so we turned it on with the lowest setting. We will determine the effect of the air currents.

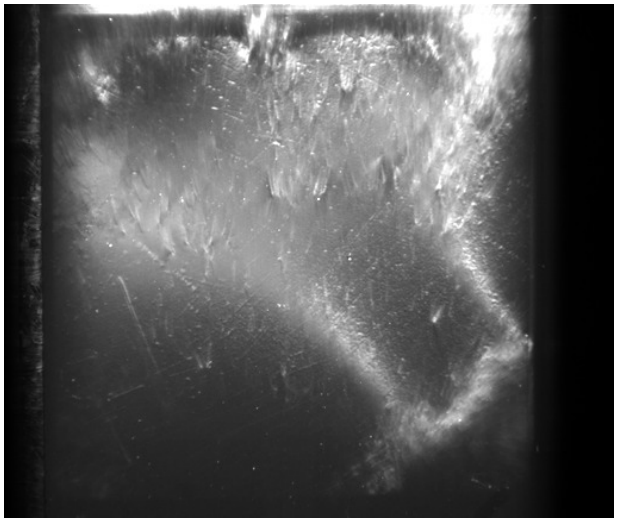
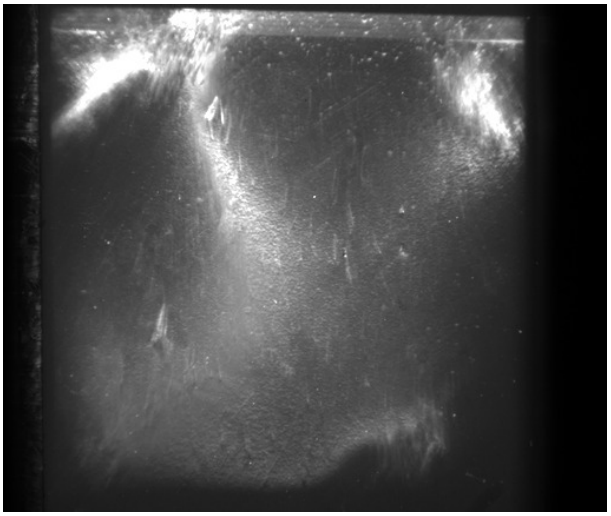
We started scan004 with tth = 37.900, chi = 137.25, phi = 0.9, th = 17.160 - 17.200. The run was started at 20:15. We used 200 steps.

Thursday, May 29, 00:10 [rtj]

Started scan005 with tth = 37.500, chi = -42.75, phi = 0.9, th = 21.02 - 21.06. The run was started at 00:20 with 200 steps. After that scan completed, I repeated it again because it seemed like the mono had drifted out of trim and I0 had slipped down into the mid 40k range. I adjusted the MOSTAB module just one click and the lower half of the diamond image intensified quite a bit in the middle frame of the scan, so I repeated just the middle 100 steps a second time.

Filename	**Orientation**	**Step size (degrees)**	**Exposure (s)**
JD70-1-study1_scan003	(2,2,0)	0.0002	10
JD70-1-study1_scan004	(2,-2,0)	0.0002	10
JD70-1-study1_scan005	(-2,2,0)	0.0002	10
JD70-1-study1_scan006	(-2,2,0)	0.0002	10
JD70-1-study1_scan008	(-2,-2,0)	0.0002	10

Started scan008 with tth = 37.500, chi = -43.05, phi = 180.9, th=19.530 - 19.570. The run was started at 1:50 with 200 steps. I had to abort scan007 because the step size was 0.0020 instead of the intended 0.0002 degrees. Aborting scan007 did not cleanly stop the run, so I had to quit the fourc program and start it up again. After that, scan008 started up fine. Images from the middle of scan006 and scan008 are shown below.

Images taken from the middle of scans 006 (left) and 008 (right). Even though the reflection planes are different, general features are visible which indicate that the two images are related by a rotation by 180 degrees around the 45 degree diagonal axis from lower left to upper right.

Next I unmount JD70-1 and start scans of JD70-2.

Scans of JD70-2

Thursday, May 29, 6:00 - Brendan Pratt and Liana Hotte

We initially had to re-mount the hoop because when it is placed in the wrong orientation, the center of the diamond is not in line with the center of rotation. After re-mounting the hoop, we could see the dots line up in the scope and we could get a reading from the diamond on the detector.

Filename	Orientation	Steps	Step size (degrees)	Exposure (s)
JD70-1-study1_scan009*	(2,2,0)	100	.0002	10
JD70-2-study1_scan001	(2,2,0)	100	.0002	10
JD70-2-study1_scan002**	(2,-2,0)	100	.0002	10
JD70-2-study1_scan003***	(2,-2,0)	100	.0002	10
JD70-2-study1_scan004	(-2,2,0)	100	.0002	10
JD70-2-study1_scan005	(2,-2,0)	100	.0002	10
JD70-2-study1_scan006	(-2,-2,0)	100	.0002	10

*scan009 was saved under the wrong filename so it was repeated

**scan002 was started with the top screen down. We noticed it quickly and restarted.

***scan003 was not in the directory so we re-scanned the plane and saved it under scan005

Started scan009 with tth = 37.500, chi = -43.70, phi = 180.9, th = 19.610-19.630. The run started at 06:40 with 100 steps. We realized that the scan was saved under the wrong filename and so we repeated it with scan001 at 06:58.

We manually rotated phi to 0.9 and afterwards we found that the andor image screen appeared frozen so we closed it. We re-started the andorview window using the following command:

ad_lineup_on 1

After this process, we can now see the full view of the diamond.

We then started scan002 with tth = 37.800, chi= -43.249, phi = 0.9, th = 17.344 - 17.364. The run started at 8:05 with 100 steps. We immediately realized the top screen was down and repeated with the same settings for scan003.

We rotated chi and started scan004 with tth = 37.750 , chi = 137.7, phi = 0.9, th = 20.879-20.899. The run started at 8:46 with 100 steps of 0.0002 degrees.

Scan003 files were not found in the directory so we decided to re-scan the 2,-2,0 orientation. Scan005 was set up with the tth = 37.81 , chi = -42.259, phi = 0.9, th = 17.341 - 17.361. The run started at 9:20 with 100 step of 0.0002 degrees.

After rotating chi to 137.7 and rotating phi to 180.9, we began scan006. Scan006 was set up as follows: tth = 37.81, chi = 136.2 , phi = 180.9, th = 19.119-19.139 with 100 steps and 0.0002 degrees.This run started at 10:15

I summarized these data in the following table.

JD70-2	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.5	-43.70	180.9	19.610 - 19.630	6:58
scan004	(-2,2,0)	37.75	137.7	0.9	20.879 - 20.899	8:46
scan005	(2,-2,0)	37.81	-42.259	0.9	17.341 - 17.361	9:20
scan006	(-2,-2,0)	37.81	136.2	180.9	19.119 - 19.139	10:15

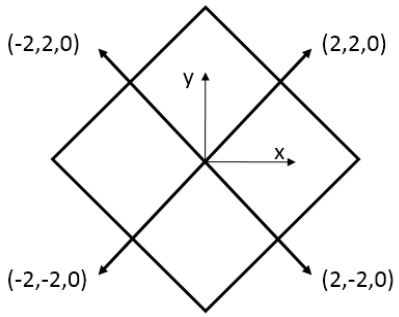
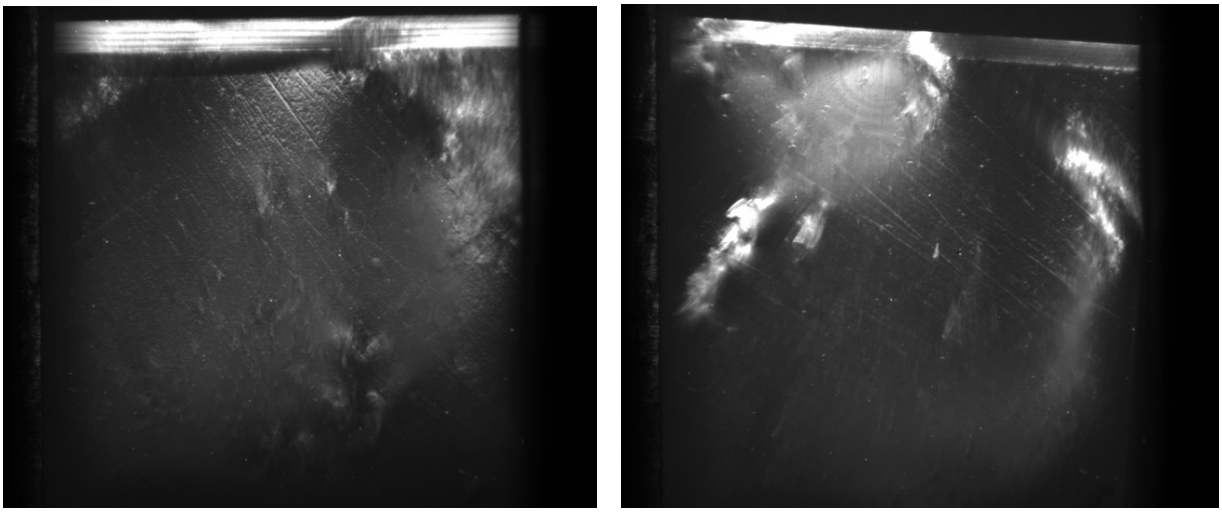


Image used to determine the scanning planes of the diamond.



Rocking curve images taken from JD70-2-study1_scan001 (left) and JD70-2-study1_scan005 (right)

Scans of JD70-3

Thursday, May 29, 10:30 - Brendan Pratt and Liana Hotte

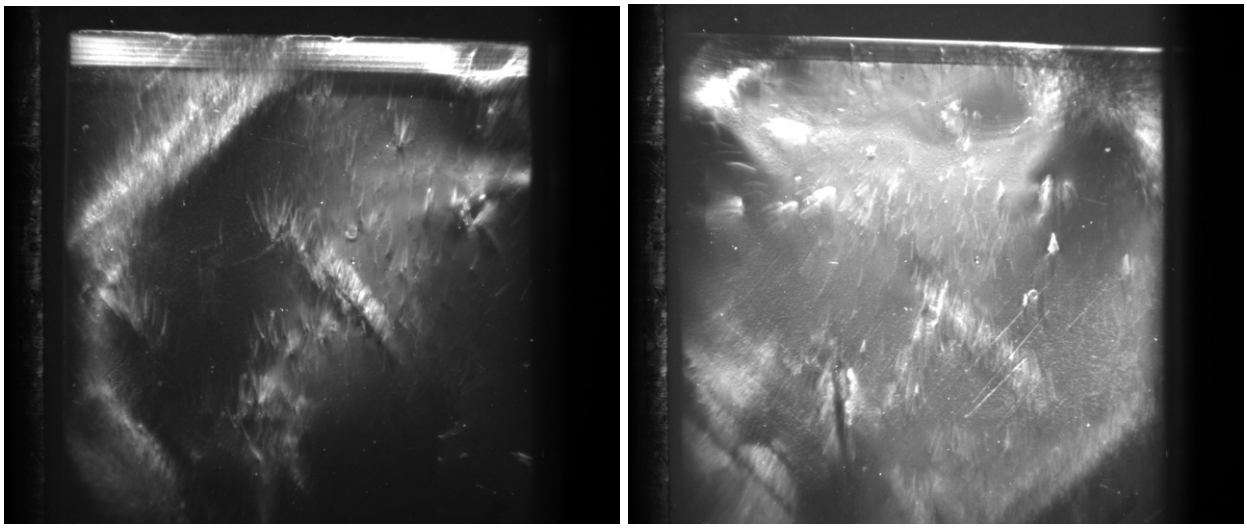
Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-3-study1_scan001	(2,2,0)	100	.0002	10
JD70-3-study1_scan002	(2,-2,0)	100	.0002	10
JD70-3-study1_scan003	(-2,2,0)	100	.0002	10
JD70-3-study1_scan004	(-2,-2,0)	100	.0002	10

As we found earlier today, the orientation of the hoop inside the mount matters. If the hoop mounted in the wrong direction, the center of the diamond will not align with the center of rotation

of the machine. Noting this, we were careful to mount JD70-3 so that the center of the hoop is close to the center of rotation.

Alex and Nathan arrived before scan002 so we used scan002 and scan003 to bring them up to speed and let them get a feel of the process. Then they left for lunch and we finished JD70-3 with scan004.

JD70-3	Orientation	TTh	Chi	Phi	Th	Time
scan001	(2,2,0)	37.81	136.00	180.9	18.678 - 18.698	10:45
scan002	(2,-2,0)	38.04	137.85	0.9	19.156 - 19.176	11:40
scan003	(-2,2,0)	37.54	-42.109	0.9	19.048 - 19.068	12:38
scan004	(-2,-2,0)	37.69	- 43.909	180.9	19.492 - 19.512	13:19



Two images taken from scan001 (left) and scan002 (right) of JD70-3-study1

Scans of JD70-4

Thursday, May 29, 2:00 - Alex Barnes and Nathan Sparks

Brendan and Liana mounted JD70-4 for us while we ate lunch. They adjusted the motors to align the diamond in the center of the screen and then left for lunch. Nathan and I proceeded to find the appropriate theta values for the scan.

While preparing for scan003 we were tweaking chi and the andor camera stopped updating. We canceled our tweaking of chi and restarted the camera with ad_lineup_off followed by ad_lineup_on 1. We then proceeded to tweak chi again and the camera stopped refreshing again. At this point Ken stopped by to see how we were doing and commented that he also has

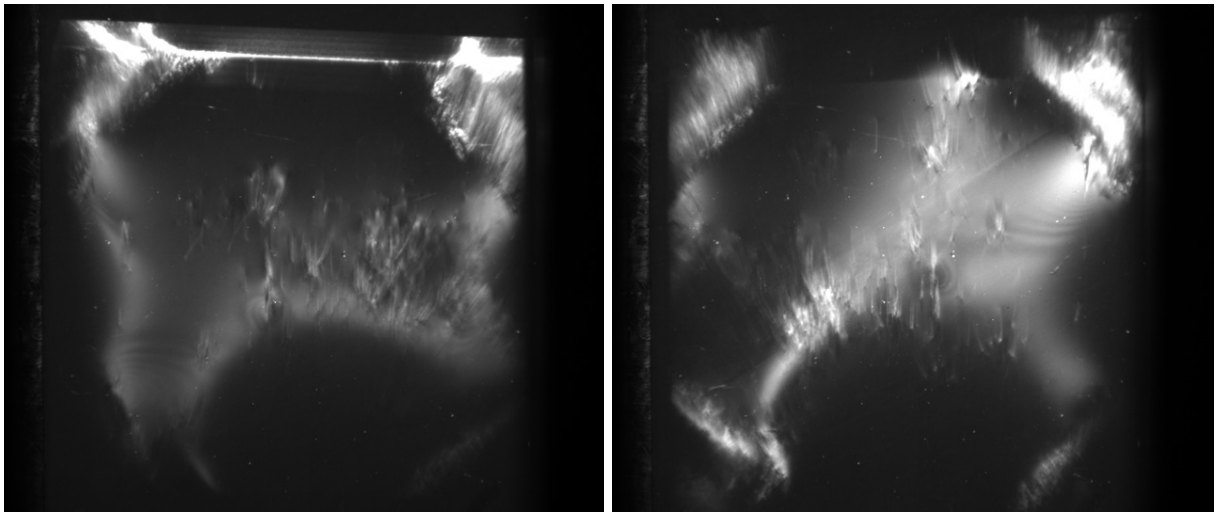
had that problem in the past. He called up Zack and Phil to help diagnose the issue but everything was working for them. They suggested taking a screenshot of the ANDOR GUI the next time the issue occurs and pass it along to them.

Another issue while setting up scan003 was that we couldn't see the diamond in the camera but we could see it on the fluorescent paper. This was after rotating phi by 180 degrees. The solution to this problem is that our chi values were higher for the first two orientations. Now that phi has been rotated we needed to adjust our chi to the other side of 135.

Note: If the image of the diamond while lit up seems to shift a lot, possibly due to air currents, try rotating the kapton so that the tape is on the far side of the hoop, the side opposite of the hutch door.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-4-study1_scan001	(2,2,0)	100	0.0002	10
JD70-4-study1_scan002	(-2,-2,0)	100	0.0002	10
JD70-4-study1_scan003	(2,-2,0)	100	0.0002	10
JD70-4-study1_scan004	(-2,2,0)	100	0.0002	10

JD70-4	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.49	-39.609	180.9	16.803 - 16.823	14:41
scan002	(-2,-2,0)	37.89	140.378	180.9	21.403 - 21.423	15:48
scan003	(2,-2,0)	37.62	133.25	0.9	22.268 - 22.288	18:35
scan004	(-2,2,0)	37.86	-46.45	0.9	15.938 - 15.958	19:18



Images taken from scan001 (left) and scan002 (right) of JD70-4-study1

Scans of JD70-5

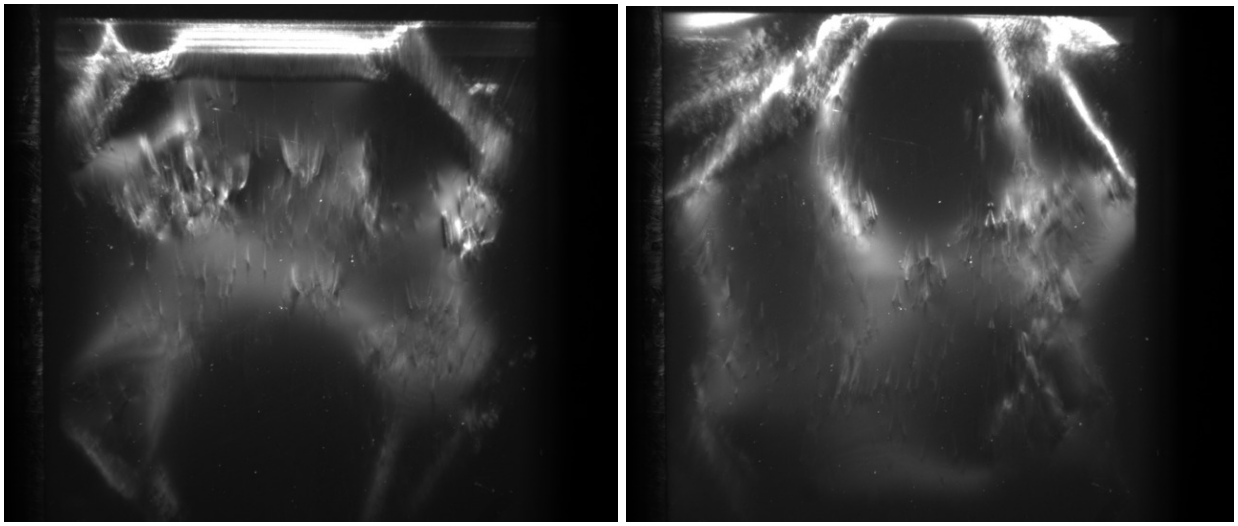
Alex and Nathan have changed the diamond to JD70-5 and checked the alignment in the scope. A newfile JD70-5-study1 has been created.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-5-study1_scan001	(2,2,0)	100	0.0002	10
JD70-5-study1_scan003*	(2,-2,0)	100	0.0002	10
JD70-5-study1_scan004**	(-2,2,0)	100	0.0002	10
JD70-5-study1_scan005	(-2,-2,0)	100	0.0002	10

JD70-5	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.76	-43.05	0.9	19.106 - 19.126	20:22
scan003	(2,-2,0)	37.66	-43.05	180.1	20.681 - 20.701	23:13
scan004	(-2,2,0)	37.71	136.95	180.1	17.518 - 17.538	23:58
scan005	(-2,-2,0)	37.70	135.95	0.9	19.094 - 19.114	00:56

*scan002 was wasted because the upper limit of the scan was mistakenly entered as 21.001 instead of 20.701 in th.

**beam tripped off at scan step 88 out of 100 in scan004, but the rocking curve images were already dark by this point, so I consider this scan intact anyway.



Images taken from scan001 (left) and scan003 (right) of JD70-5-study1.

May 29, 23:00 [rtj]
I came on shift after scan001 and completed the scans of JD70-5. I then dismounted JD70-5 and returned it to its holder in the case.

Scans of JD70-6

I have mounted the JD70-6 diamond in the hoop and checked the alignment in the scope. A newfile JD70-6-study1 has been created, but only after I realized that I had failed to enter the newfile command, after I had already completed 2 of the scans. The first two scans are incorrectly appended to the JD70-5 file, but it is a simple error, so I am leaving it.

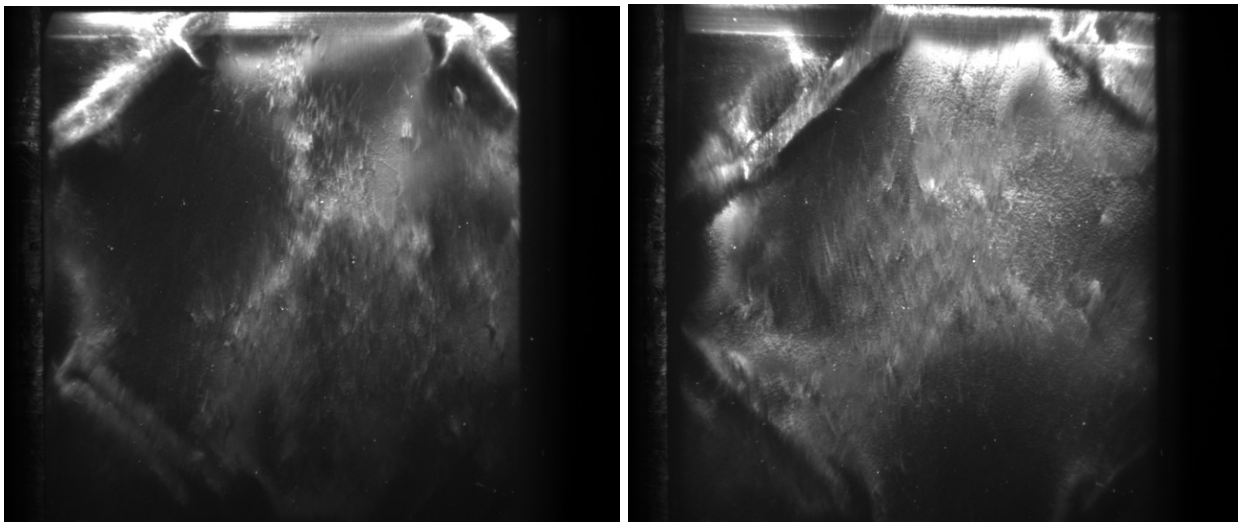
Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-**5**-study1_scan006*	(2,2,0)	100	0.0002	10
JD70-**5**-study1_scan007**	(2,-2,0)	100	0.0002	10
JD70-6-study1_scan001	(-2,2,0)	100	0.0002	10
JD70-6-study1_scan002	(-2,-2,0)	100	0.0002	10
JD70-6-study1_scan004	(-2,-2,0)	50	0.0002	10
JD70-6-study1_scan005	(-2,-2,0)	50	0.0002	10

JD70-6	Orientation	TTh	Chi	Phi	Th	Time
scan006*	(2,2,0)	37.70	135.55	0.9	18.595 - 18.615	1:58
scan007**	(2,-2,0)	37.70	135.55	180.9	17.570 - 17.590	2:30
scan001	(-2,2,0)	37.60	-44.45	180.9	20.604 - 20.624	3:00
scan002	(-2,-2,0)	37.60	-43.90	0.9	21.109 - 21.129	3:30
scan004***	(-2,-2,0)	37.60	-43.90	0.9	21.129 - 21-139	3:50
scan005***	(-2,-2,0)	37.60	-43.90	0.9	21.139 - 21-149	4:00

* incorrectly appended to the JD70-5-study1 dataset, data are ok

** incorrectly appended to the JD70-5-study1 dataset, data are ok

*** continuation of scan002, which failed to contain the rocking curve peak; scan003 was aborted because it was started with the fluorescent screen blocking the view of the camera.



Images taken from scan006 (left) and scan007 (right) of JD70-5-study1. Those scans were incorrectly appended to the JD70-5 run, whereas they actuall belong to JD70-6.

Around step 25 in scan005 above, I obtained some repeated warning messages in the FourC window.

```
Retry 1 on "ANDOR1:cam1:DetectorState_RGB" …
Error on epics_get("ANDOR1:cam1:DetectorState_RGB"): User specified timeout on
IO operation expired (0.5 s).
```

Something in the network or camera i/o system was slowing down and getting stuck. After several steps, the error message stopped being printed. Since the images continue to update in the camera image window, I assume that the camera is still working and move on. The images

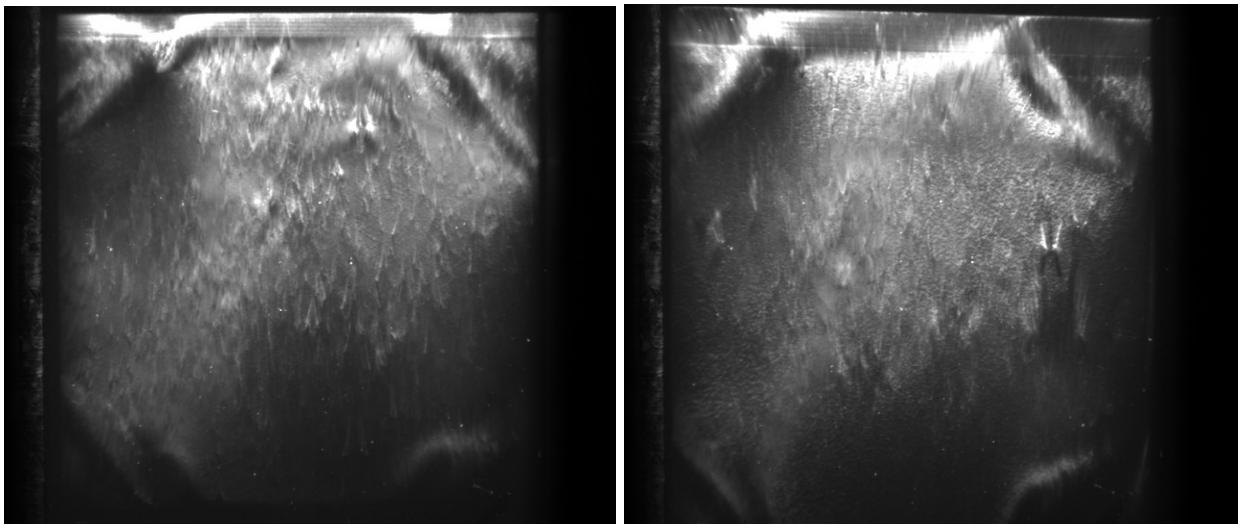
in that section of the scan were all dark anyway because it happened outside the region of the rocking curve peak.

Scans of JD70-7

I have mounted the JD70-7 diamond in the hoop and checked the alignment in the scope. A newfile JD70-7-study1 has been created.

Filename	Orientation	Steps	Step size (degrees)	Exposure (s)
JD70-7-study1_scan001	(2,2,0)	100	0.0002	10
JD70-7-study1_scan002	(2,-2,0)	100	0.0002	10
JD70-7-study1_scan003	(-2,2,0)	100	0.0002	10
JD70-7-study1_scan004	(-2,-2,0)	100	0.0002	10

JD70-7	Orientation	TTh	Chi	Phi	Th	Time
scan001	(2,2,0)	37.50	-46.70	0.9	18.971 - 18.991	4:30
scan002	(2,-2,0)	37.70	-39.25	179.0	19.579 - 19.599	5:08
scan003	(-2,2,0)	37.70	140.75	179.0	18.612 - 18.632	5:42
scan004	(-2,-2,0)	37.80	133.15	0.9	20.578 - 20.598	6:13



Images taken from scan001 (left) and scan002 (right) of JD70-7-study1.

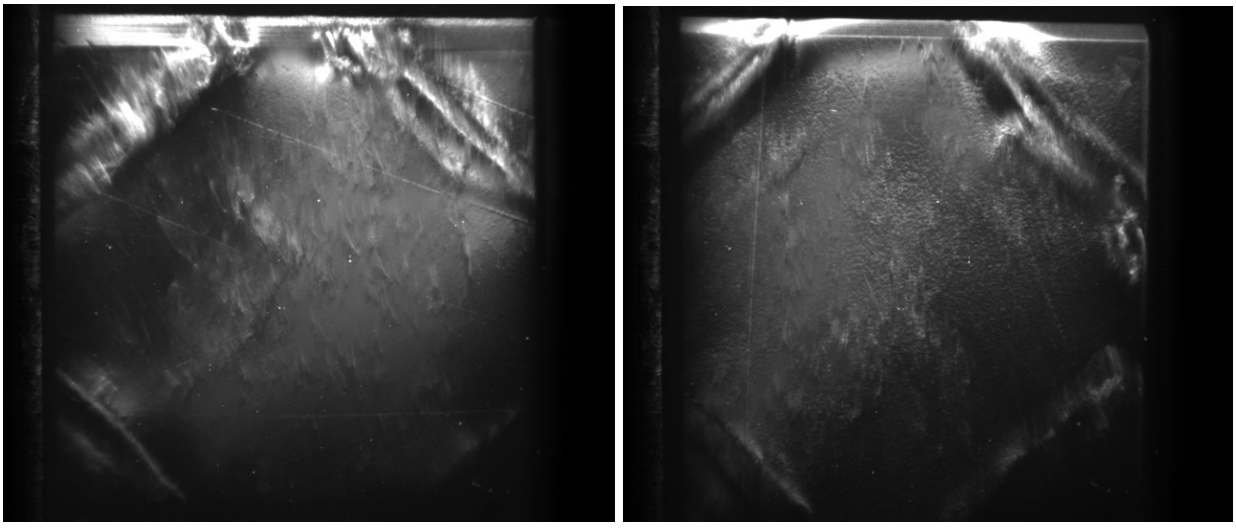
Scans of JD70-8

Friday, May 30, 6:00 - Alex Barnes and Liana Hotte

Filename	Orientation	Steps	Step size (degrees)	Exposure (s)
JD70-8-study1_scan001	(2,2,0)	100	.0002	10
JD70-8-study1_scan002	(2,-2,0)	100	.0002	10
JD70-8-study1_scan003	(-2,2,0)	100	.0002	10
JD70-8-study1_scan004	(-2,-2,0)	100	.0002	10

JD70-8	Orientation	TTh	Chi	Phi	Th	Time
scan001	(2,2,0)	37.498	135.22	0.9	21.439 - 21.459	7:04
scan002	(2,-2,0)	37.698	138.51	180.9	24.195 - 24.215	7:37
scan003	(-2,2,0)	37.698	-41.60	180.9	14.037 - 14.057	8:12
scan004*	(-2,-2,0)	37.898	-44.7	0.9	16.731 - 16.751	9:10

*The beam was filled around the 86th step. The IO level didn't appear to change. We consider this scan to be completed.

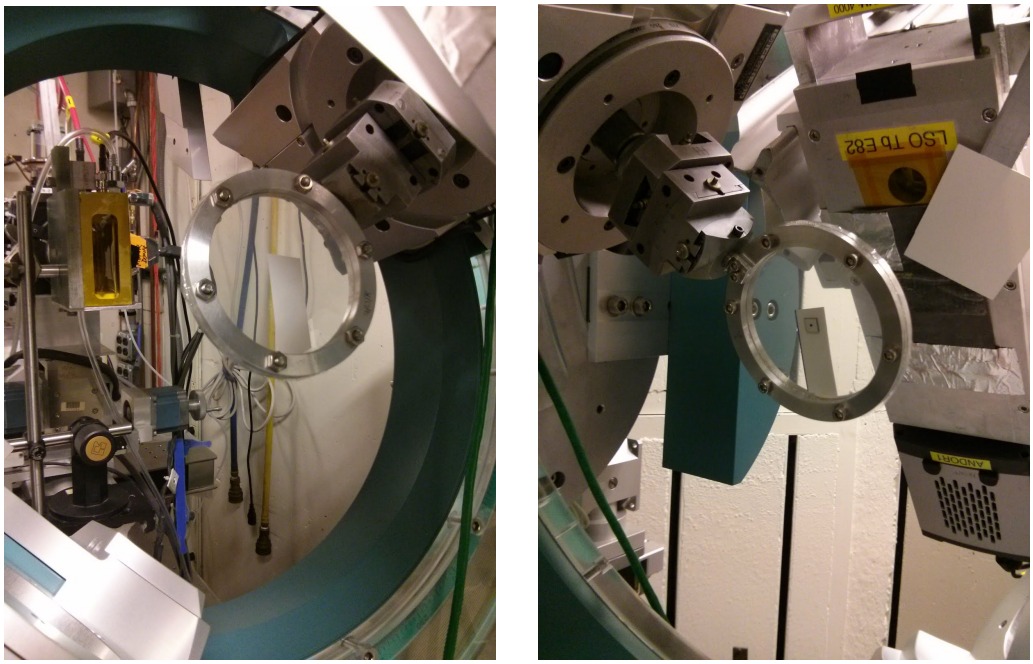


Images from scan001 (left) and scan002 (right) from JD70-8-study1.

Scans of JD70-9

Friday, May 30, 9:30 - Alex Barnes and Liana Hotte

After mounting the diamond in the hoop, we tried to set up the position for a run. We noticed that the size of the diamond in the camera was a rectangular shape that had a narrow width. We asked Ken about this and he did the following to determine the problem. He placed a strip of fluorescent paper behind the diamond on the hoop and shut the hutch. He then left the light on and turned on the beam. The paper will light up and the diamond will still be visible. You can then see how the beam lines up with the diamond. In our case the beam was missing the diamond partially thus giving us a rectangular shape. It seems like the hoop was not pushed in far enough. Once we pushed it in farther the beam lined up with the diamond. We also noticed that the beam does not fully cover the diamond. The beam size is 8mm by 8mm at the normal which means once we tilt our mount we will see less of the beam. However, it appears that it does not miss by any significant amount and would be part of the edge of the frame of the diamond.



Images of the hoop with fluorescent paper placed behind the sample in order to determine where the beam is in relation to the diamond.

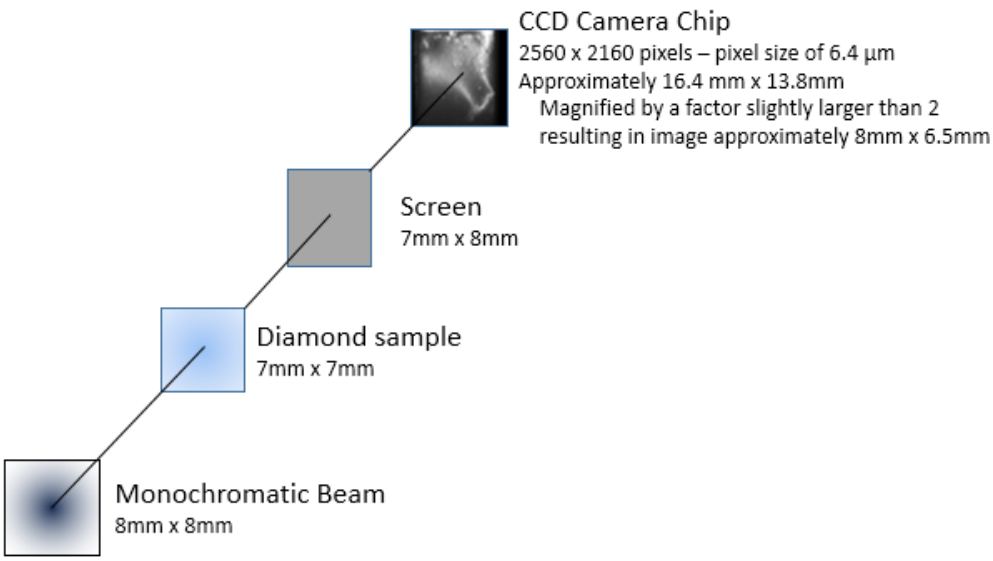


Image of the beam's travel as it passes through to the camera. We don't know the exact magnification, but we know that we can see approximately 8mm in the horizontal direction on any given image.

While running scan002, we found that FourC froze at step 38. We exited FourC, opened a new konsole and executed the command *fourc*. Everything worked again and we re-started the scan.

Before rotating phi to 180.9 for scan004, we realized that the kapton foil was not around the hoop for scan001, scan002, and scan003.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-9-study1_scan001*	(2,2,0)	100	0.0002	10
JD70-9-study1_scan002**	(2,2,0)	100	0.0002	10
JD70-9-study1_scan003***	(2,2,0)	100	0.0002	10
JD70-9-study1_scan004	(2,2,0)	100	0.0002	10
JD70-9-study1_scan005	(2,-2,0)	100	0.0002	10
JD70-9-study1_scan006	(-2,2,0)	100	0.0002	10
JD70-9-study1_scan007	(-2,-2,0)	100	0.0002	10

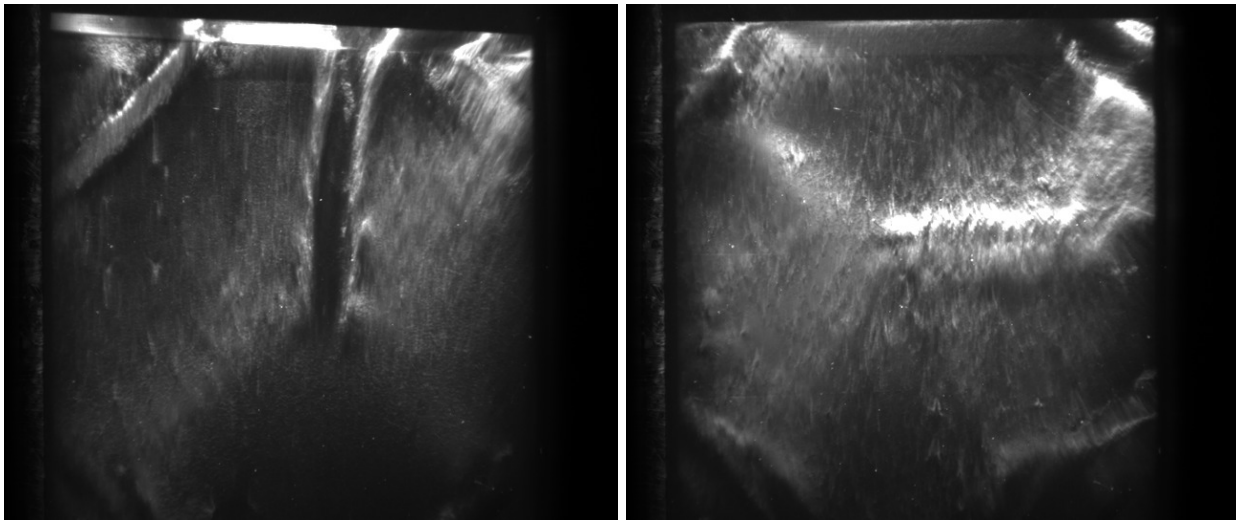
**scan001 was for the wrong range of theta. We used 19.960-19.980 instead of 18.960-18.980.*

***scan002 froze at step 38. The run was restarted.*

****scan003 did not have the kapton cover.*

JD70-9	Orientation	TTh	Chi	Phi	Th	Time
scan004	(2,2,0)	37.788	-41.15	0.9	18.960 - 18.980	11:50
scan005	(2,-2,0)	37.588	-44.85	180.9	19.629 - 19.649	13:26
scan006	(-2,2,0)	37.808	135.1	180.9	18.582-18.602	14:12
scan007	(-2,-2,0)	37.698	138.85	0.9	19.261 - 19.281	14:55

Nathan and Brendan came in for the 2nd shift of the day to relieve Alex and Liana. We started with scan005 of diamond JD70-9 after resetting phi to 180.9. After a good run we are now moving on to the next orientation, changing chi to 138.5 and tweaking it to 135.1. Another rotation in phi brings us to the last orientation for this crystal (-2,-2,0).



Images from scan004 (left) and scan005 (right) for JD70-9-study1.

Just spoke with the beam operator about the error message Alex and Liana received earlier. He didn't know exactly what the cause was, but mentioned that it's common when something is changed during the middle of a scan. We were instructed that it was not cataclysmic and to find him if it happened again. Commencing scan007 at 2:55 pm. This diamond is now completed.

Scans of JD70-10

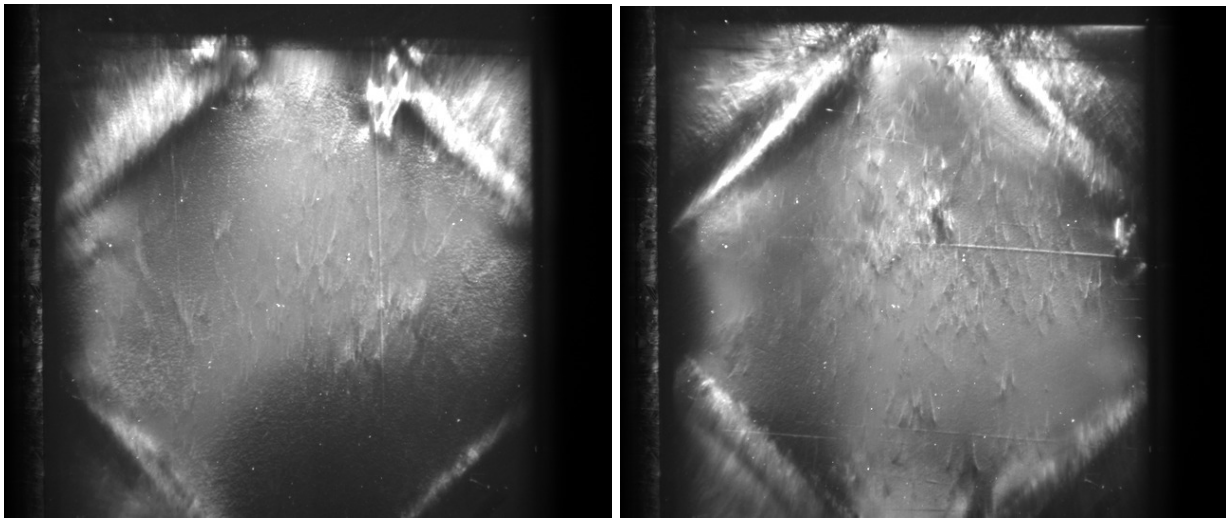
Friday, May 30, 3:15 - Nathan Sparks and Brendan Pratt

Nathan successfully mounted JD70-10 in the mylar hoop which we are currently reinstalling in the goniometer. We will then begin scans starting with the (2,2,0) orientation. Created new file JD70-10-study1. We noticed the same problem Alex and Liana did after remounting a new diamond. In the ANDOR1 screen, the right side of the diamond was cut off. Taking their advice,

we pushed the mount further in, looked at it through the scope (it was much closer to center) and we see the full width of the stone. Rotating phi to 180.9 for the second scan of this diamond (2,-2,0). After rotating in phi we saw the same problem again, so we tried pushing the mount farther down again to better align it with the beam. Because there is aluminum tape wrapped around post it was not seating metal to metal. Brendan used an exacto blade to remove the tape from the upper most portion of the post which was preventing further travel into the mount. The diamond was then sighted again through the scope and it looked to be in much better alignment. We found the peak on the paper and things are looking better again on ANDOR. And just as we attempted to start the scan, FOURC stalled. We hit ctrl c and started over and everything worked. Now that scan002 is finished we are moving chi to -46.1 and setting up for the third scan (-2,2,0).

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
JD70-10-study1_scan001	(2,2,0)	100	0.0002	10
JD70-10-study1_scan002	(2,-2,0)	100	0.0002	10
JD70-10-study1_scan003	(-2,2,0)	100	0.0002	10
JD70-10-study1_scan004	(-2,-2,0)	100	0.0002	10

JD70-10	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.973	140.45	0.9	17.474 - 17.494	15:59
scan002	(2,-2,0)	37.873	133.86	180.9	17.161 - 17.181	16:57
scan003	(-2,2,0)	37.573	-46.09	180.9	21.05 - 21.07	17:35
scan004	(-2,-2,0)	37.6230	-39.910	0.9	19.996 - 20.016	18:27



Images from scan001 (left) and scan002 (right) for JD70-10-study1.

After this scan we will begin looking at the UC45-6-S200_50 and UC45-7-S200_50. Successfully mounted UC45-6-S200_50 and will start a new scan.

Scans of UC45-6-S200_50

Friday, May 30, 19:00 - Nathan Sparks and Brendan Pratt

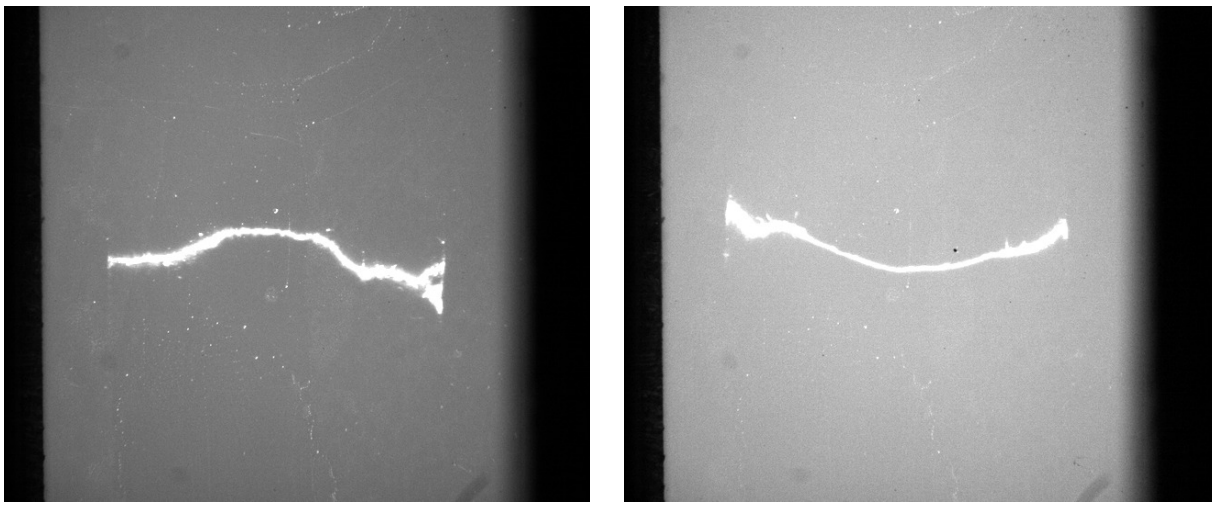
Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC45-6-S200_50-study1_scan001*	(2,2,0)	500	0.0002	10
UC45-6-S200_50-study1_scan003	(2,2,0)	100	0.001	10
UC45-6-S200_50-study1_scan004	(2,-2,0)	550	0.0002	10
UC45-6-S200_50-study1_scan005	(2,-2,0)	110	0.001	10
UC45-6-S200_50-study1_scan006	(-2,2,0)	110	0.001	10
UC45-6-S200_50-study1_scan007	(-2,-2,0)	110	0.001	10

* we lost beam towards the end of the run, so we are starting right at the start of a new injection

UC45-6-S200_50	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001*	(2,2,0)	37.623	-48.11	0.9	16.793 - 16.893	19:15
scan003	(2,2,0)	37.623	-48.11	0.9	16.790 - 16.890	20:52

scan004	(2,-2,0)	37.623	-37.91	180.9	16.440 - 16.550	21:29
scan005	(2,-2,0)	37.623	-37.91	180.9	16.440 - 16.550	23:27
scan006	(-2,2,0)	37.623	142.09	180.9	21.680 - 21.790	0:09
scan007	(-2,-2,0)	37.623	131.09	0.9	21.330 - 21.440	12:40

We mounted UC45-6-S200_50 and found our old friend the worm, in that a bright band of light streaked across this diamond indicating a large rocking curve. The scan interval is a factor of 5 larger than the JD70's and to keep the resolution the same, we increased the step count to 500. Each scan at this interval takes about 87 minutes, so plan accordingly.



One frame from rocking curve scan003 (left panel) and scan005 (right panel) of UC45-6-S200_50

Dr. Jones came in to start the next shift and mentioned that we should keep the number of steps close to 100 and reduce the shutter time from 10 to 5 seconds. This will keep the runs at the same length in time (about 30 minutes).

May 30, 11:30 [rtj]
I take over for the night shift in the middle of the scans through diamond UC45-6-S200_50. From the image below taken from one of the early scans, it is clear that this diamond has a very wide rocking curve. I decide to keep the shutter time fixed at 10 seconds, and make larger steps in theta for these crystals because having high resolution images is more valuable than having more of them if they are of lower resolution.

One difficult thing to understand about these crystals that were etched by Sinmat is how the damage was created in the frame region around the thin window. We discussed as a group how the VPIE process might create etch craters and crevasses in the surface that are deep enough that internal stress might cause the thin diamond to distort, but in the frame regions the

thickness should still be hundreds of microns. We need to check this on the Zygo when we get back to Storrs, but if this is the case, we need to reconsider our plans to have Sinmat thin our thick large-area samples down to a few hundred microns prior to ablating them with a laser. Perhaps the VPIE process has caused some irreversible damage to the crystal that we do not yet understand.

Scans of UC45-7-S200_50

Saturday, May 31, 2:00 - Richard Jones

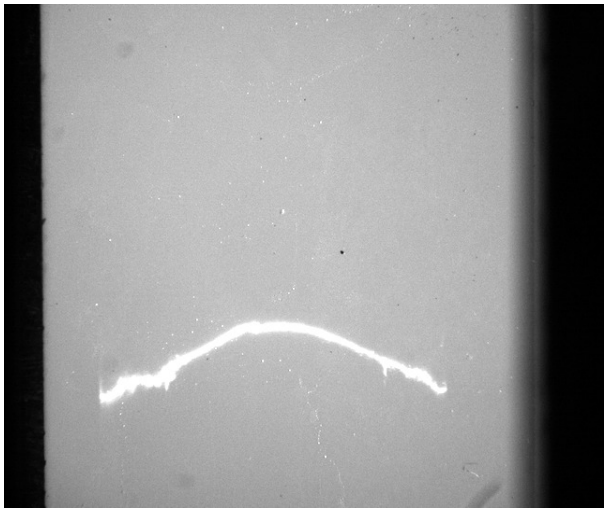
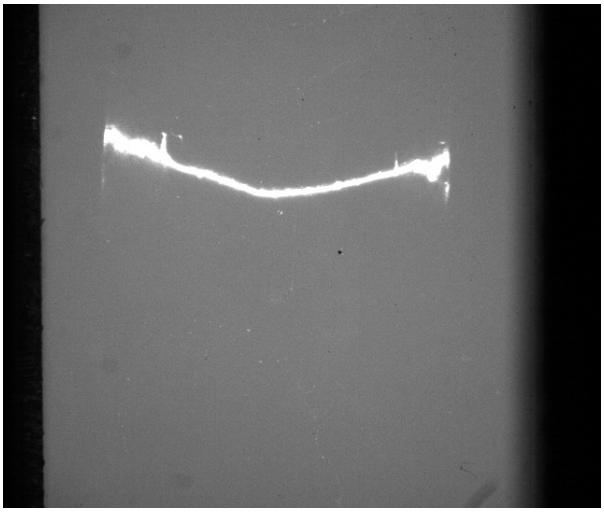
I took out UC45-6-S200_50 from the mylar hoop and returned it to its little clear plastic box with the pink sponge and hinged lid. Then I looked for UC45-7-S200_50 in the case. I found the little hinged box with that name printed on it, but there was nothing inside! However after some searching I found it. It had been put back into the original dimpled plastic carton, together with the others of the set of 15 4.5mm diamonds that UConn obtained from E6. It was different from the others, in that it was a milky white color and translucent, and one could see that a central square region of that diamond was of a different thickness than the outer region. I removed it from the carton, with the intention of returning it to its little clear plastic hinged box after we are done scanning it.

When I mounted it in the holder, I laid it such that the depression was facing up in the mylar hoop, toward the surface marked “upstream” on the hoop and away from the side where the holding post is attached. When I mounted it in the goniometer, it was in the orientation with the hoop “upstream” face looking downstream. That orientation is labeled “2,2,0) in the table below. The opposite orientation is marked with phi = 180.9 deg.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC45-7-S200_50-study1_scan001	(2,2,0)	350	0.0002	10
UC45-7-S200_50-study1_scan002	(2,-2,0)	120	0.0005	10
UC45-7-S200_50-study1_scan003	(-2,2,0)	120	0.0005	10
UC45-7-S200_50-study1_scan004	(-2,-2,0)	120	0.0005	10

UC45-7-S200_50	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.623	135.49	0.9	18.060 - 18.130	2:15
scan002	(2,-2,0)	37.623	138.19	180.9	20.590 - 20.650	3:35
scan003	(-2,2,0)	37.623	-42.21	180.9	17.610 - 17.670	4:13

scan004	(-2,-2,0)	37.623	-44.10	0.9	21.450 - 21.510	4:43

Images from rocking curve scan001 (left) and scan002 (right) of UC45-7-S200_50.

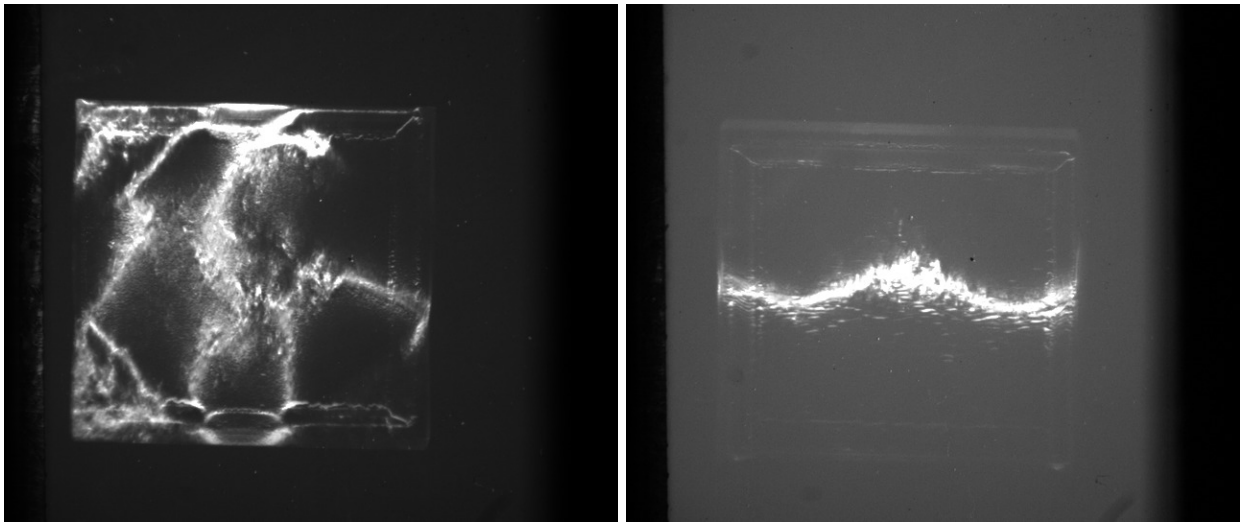
I start off with a high-resolution scan of 350 steps. I do not need all of the scans to have such high resolution, but should have one at this step size to determine if the individual pixel rocking curves have the same width as the original material.

Scans of UC45-2-C500_300

After completing the above scans of UC45-7-S200_50 I returned that diamond to its clear plastic hinged case and put it back in the case. I then got out the UC45-2-C500_300 from the plastic pocket pack holding the UConn 4.5mm diamonds and mounted it in the mylar hoop. The diamond is almost completely black from ablation, so it was difficult to see through it well enough to line it up with the center dot on the mounting mylar, but I did the best I could. The ablated cavity on the diamond is facing the "upstr" side of the hoop.

Filename	Orientation	Steps	Step size (degrees)	Exposure (s)
UC45-2-C500_300-study1_scan001	(2,2,0)	125	0.0002	10
UC45-2-C500_300-study1_scan002	(2,-2,0)	150	0.0002	10
UC45-2-C500_300-study1_scan003	(-2,2,0)	150	0.0002	10
UC45-2-C500_300-study1_scan004	(-2,-2,0)	100	0.0002	10

UC45-2-C500_300	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.623	-42.11	0.9	16.580 - 16.605	5:45
scan002	(2,-2,0)	37.623	-42.21	180.9	16.187 - 16.217	6:18
scan003	(-2,2,0)	37.623	135.79	180.9	22.002 - 22.032	7:00
scan004	(-2,-2,0)	37.623	137.39	0.9	23.055 - 23.075	7:51



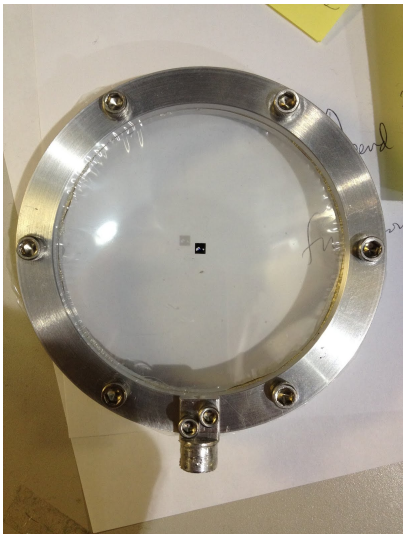
Images from rocking curve scan001 (left) and scan002 (right) for UC45-2-UC500_300.

Scans of UC30-9-C300_25

Saturday, May 31, 8:00 - Brendan Pratt

I removed UC45-2-UC500_300 from the mylar hoop and put it back into its appropriate case. When I mounted it in the holder, I laid it such that the depression was facing up in the mylar hoop, away from the surface marked “upstream” on the hoop and towards the side where the holding post is attached. When I mounted it in the goniometer, it was in the orientation with the hoop “upstream” face looking away from the beam as was its last orientation.

Before leaving, Dr. Jones reminded me that these diamonds have different orientations along the edges, and so they must be mounted “square” in the hoop versus the “diamond-like” orientation the 4.5mm diamonds were given. See the image below.



UC30-9-C300 diamond mounted in mylar hoop

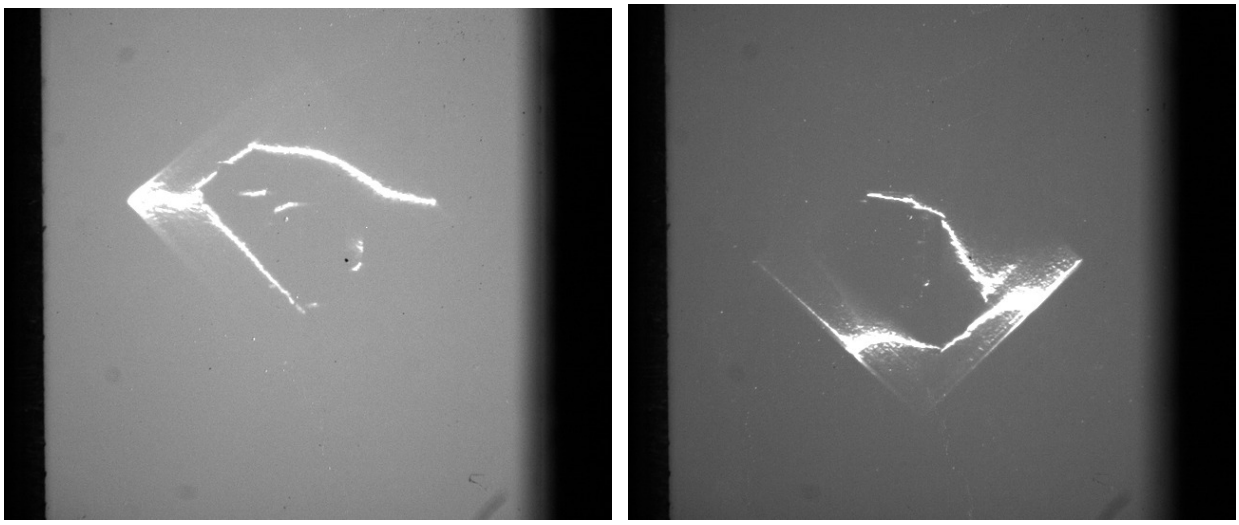
I created newfile UC30-9-C300_25-study001 and will now find the first peak at (2,2,0) orientation. The data from the previous run told us that these 3x3mm diamonds have much broader rocking curves than the previous 3mm samples we obtained from e6. Using the ad_lineup_on with an exposure on 1 second was too short to find the lower limit of the theta spectrum, so I canceled the scan001 and will use an exposure of 3 seconds to find a better range for scan002. Grabbing lunch with Nathan now, be back around 1pm. After reading Dr. Jones' entry I thought it would be a good idea to do one scan with higher resolution (0.0005), we are back from lunch and the scan is nearly done with no apparent loss in beam.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-9-C300_25-study1_scan002	(2,2,0)	170	0.001	10
UC30-9-C300_25-study1_scan003	(2,-2,0)	270	0.001	10
UC30-9-C300_25-study1_scan004	(-2,2,0)	260	0.001	10
UC30-9-C300_25-study1_scan005	(-2,-2,0)	540	0.0005	10

UC30-9-C300_25	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan002	(2,2,0)	37.623	139.19	0.9	14.70 - 14.870	9:11
scan003	(2,-2,0)	37.623	133.9	180.9	15.90 - 16.170	9:54

scan004	(-2,2,0)	37.623	-45.11	180.9	22.10 - 22.360	11:07
scan005	(-2,-2,0)	37.623	-40.51	0.9	23.32 - 23.590	12:10

here is some text



Selected images from scan002 (left) and scan003 (right) of UC30-9-study1.

Scans of UC30-10-C300_25

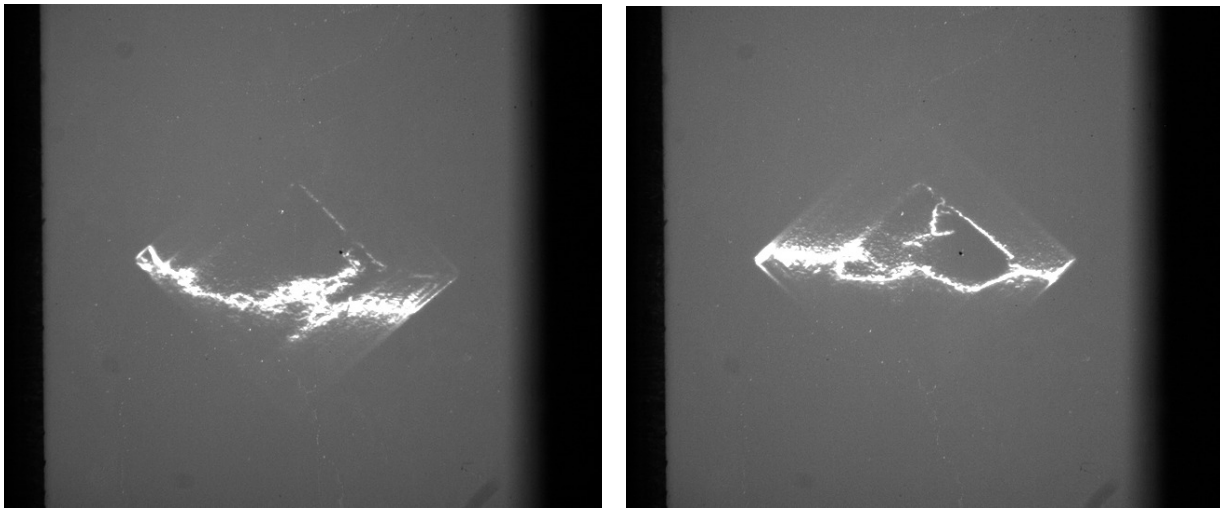
Saturday, May 31, 2:20 - Nathan Sparks

UC30-9-C300_25 was removed from the mount and returned to its case, and was replaced by UC30-10-C300_25, which was mounted in the same orientation as the previous diamond. A new file named UC30-10-C300_25-study1 was then created to store the data. After watching scan 1, it was clear that the chosen theta range was much larger than needed. The theta range for the next scan will be chosen more carefully in order to coincide more closely with the actual edges of the rocking curve, reducing scan time and/or allowing for a finer step size. When searching for the rocking curve edges for scan 2, instead of finding a narrower range, another large range was found. Thus, scan 2 will be executed with this large range. Apparently, small features in the images for the edges of the theta range were not noticed when watching scan 1. After scan 2 was finished, the chi angle was rotated by 180 degrees. There was not enough run time left to complete another scan, so it was decided to wait for the start of a new run to ensure a steady flux of X-rays during scan 3. A typo occurred when entering the command to start scan 3, and this scan was immediately aborted. The command was then entered correctly to start scan 4.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-10-C300_25-study1_scan001	(2,2,0)	260	0.001	10

UC30-10-C300_25-study1_scan002	(2,-2,0)	310	0.001	10
UC30-10-C300_25-study1_scan004	(-2,2,0)	260	0.001	10
UC30-10-C300_25-study1_scan005	(-2,-2,0)	260	0.001	10

UC30-10-C300_25	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.623	-45.21	0.9	17.753 - 18.013	15:18
scan002	(2,-2,0)	37.623	-40.68	180.9	19.070 - 19.380	17:18
scan004	(-2,2,0)	37.623	139.12	180.9	18.900 - 19.160	19:21
scan005	(-2,-2,0)	37.623	134.85	0.9	20.210 - 20.470	20:25



Selected images taken from scan001 (left) and scan002 (right) of UC30-10-study1.

Scans of UC45-1-C500

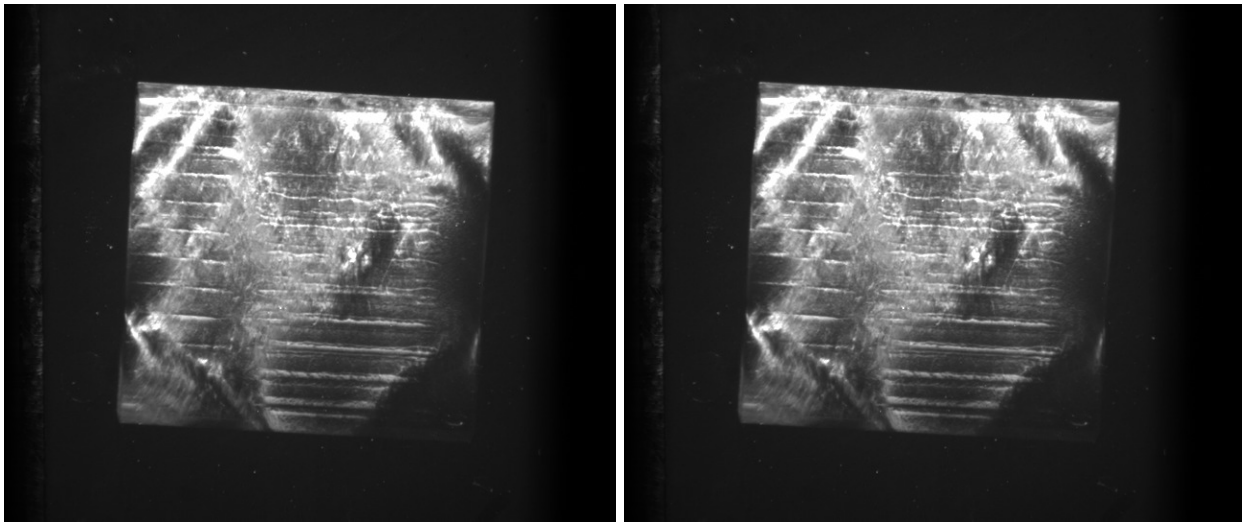
Saturday, May 31, 10:30 - Richard Jones

I put the the UC30-10-C300_25 diamond back into the case, and mounted the UC45-1-C500 diamond into the mylar hoop holder. This diamond was used for laser focal spot studies, so it has one of its two surfaces etched in isolated rows of laser cuts with varied focus settings. We think that the bulk diamond in this thick sample should not be affected by a few passes of the ablating beam over the upper surface. The purpose of this run is to test this assumption, and see if there is any evidence of deep crystal damage caused by the laser. As far as I could tell, the etched surface is facing the side of the hoop that is marked “upstr”. The diamond is so close

to perfect that I only need 50 steps at step size 0.0002 deg to fully span the rc peak for the whole crystal.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC45-1-C500-study1_scan001	(2,2,0)	50	0.0002	10
UC45-1-C500-study1_scan002	(2,-2,0)	50	0.0002	10
UC45-1-C500-study1_scan003	(-2,2,0)	50	0.0002	10
UC45-1-C500-study1_scan004	(-2,-2,0)	50	0.0002	10

UC45-1-C500	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.523	140.0767	0.9	17.624 - 17.634	23:00
scan002	(2,-2,0)	37.623	133.9767	180.9	17.777 - 17.787	23:22
scan003	(-2,2,0)	37.523	-46.3233	180.9	20.474 - 20.484	23:45
scan004	(-2,-2,0)	37.723	-39.6233	0.9	20.602 - 20.612	0:02



Selected images from scan001 (left) and scan002 (right) of UC45-1-C500-study1.

Scans of UC45-3 - UC45-5

Saturday, May 31, 10:30 - Richard Jones

I put the the UC45-1-C500 diamond back into the case, and mounted the UC45-3 diamond into the mylar hoop holder. These diamonds are untouched so far, so these scans should be quick and painless. I find I only need 75 steps at step size 0.0002 deg to fully span the rc peak for the whole crystal. I repeated the identical procedure for the following 3 diamonds.

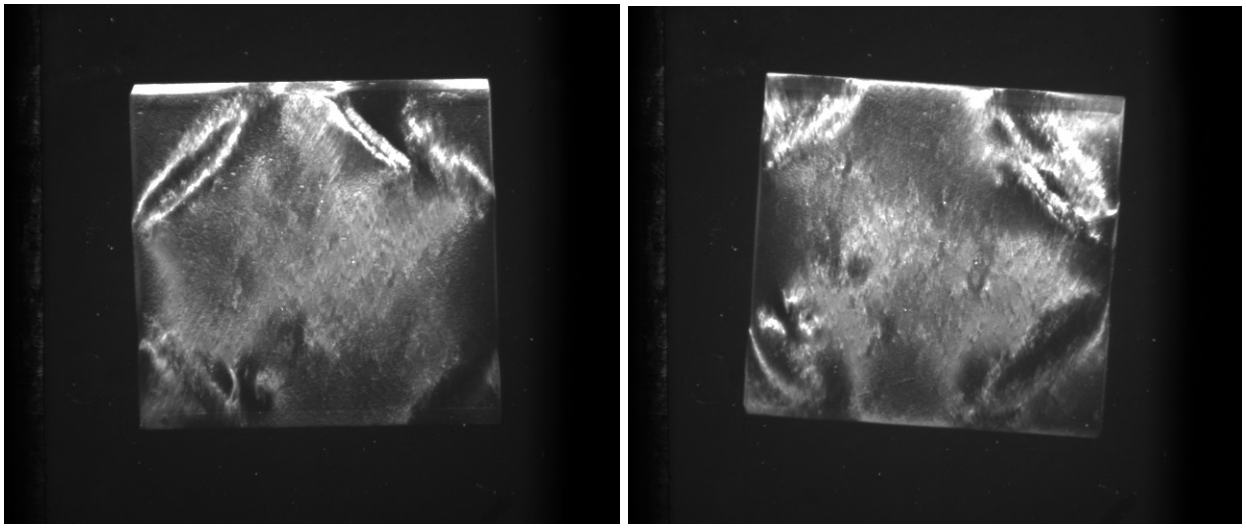
Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC45-3-study1_scan001*	(2,2,0)	75	0.0002	10
UC45-3-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-3-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-3-study1_scan004	(-2,-2,0)	75	0.0002	10
UC45-4-study1_scan001	(2,2,0)	75	0.0002	10
UC45-4-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-4-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-4-study1_scan004	(-2,-2,0)	75	0.0002	10
UC45-5-study1_scan001	(2,2,0)	75	0.0002	10
UC45-5-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-5-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-5-study1_scan004	(-2,-2,0)	75	0.0002	10

* scan001 of UC45-3-study1 was repeated twice because the first time I ran it, I forgot to do newfile and so the first time it was recorded as scan005 of UC45-1-C500-study1.

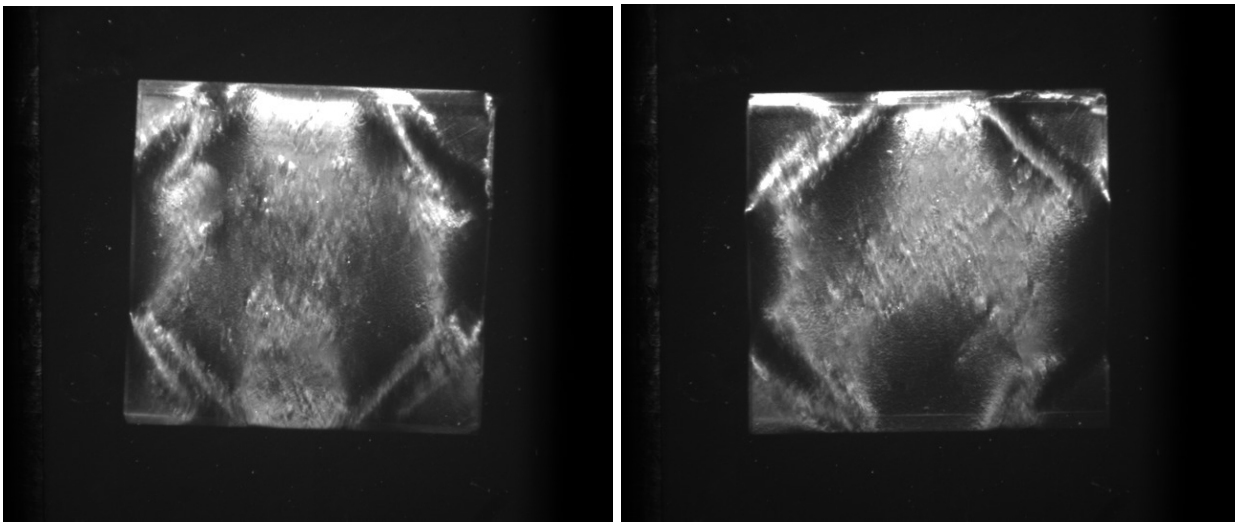
UC45-3	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.573	-46.7733	0.90	17.188 - 17.203	0:34
scan002	(2,-2,0)	37.473	-38.9733	180.9	17.558 - 17.573	1:14
scan003	(-2,2,0)	37.673	141.0267	180.9	20.660 - 20.675	1:42
scan004	(-2,-2,0)	37.573	132.8267	0.90	21.035 - 21.050	2:02

UC45-4	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.523	138.7267	0.9	21.650 - 21.665	2:56
scan002	(2,-2,0)	37.623	135.4267	180.9	18.345 - 18.360	3:20
scan003	(-2,2,0)	37.523	-44.7733	180.9	19.895 - 19.910	3:48
scan004	(-2,-2,0)	37.673	-41.0733	0.9	18.015 - 18.030	4:10

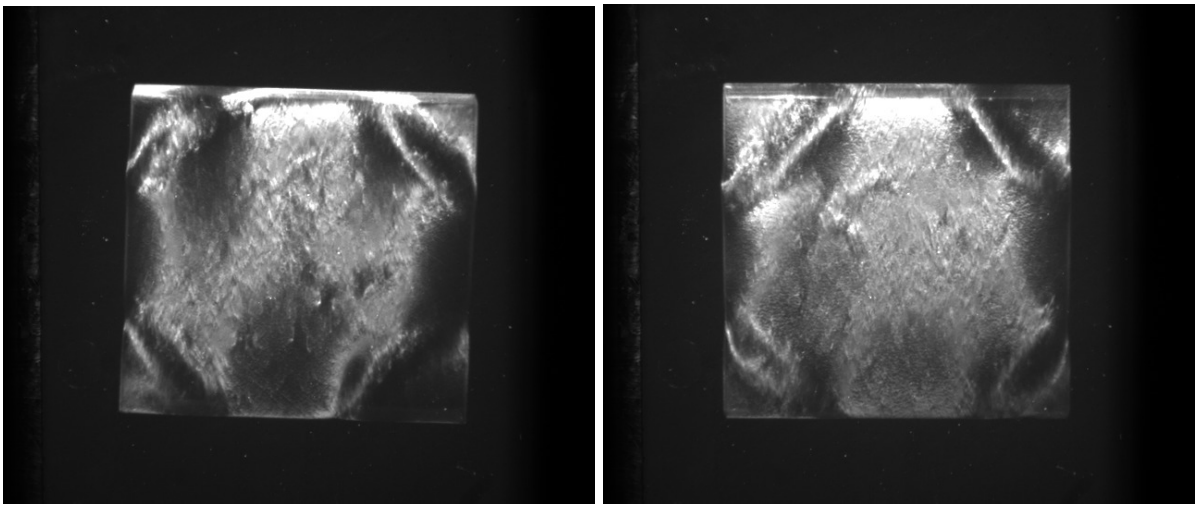
UC45-5	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.623	-42.8733	0.9	19.017 - 19.032	4:55
scan002	(2,-2,0)	37.473	-42.7733	180.9	21.303 - 21.318	5:14
scan003	(-2,2,0)	37.673	137.2267	180.9	16.912 - 16.927	5:40
scan004	(-2,-2,0)	37.573	136.6767	0.9	20.617 - 20.632	6:01



Selected images from scan001 (left) and scan002 (right) of UC45-3.



Selected images from scan001 (left) and scan002 (right) of UC45-4.



Selected images from scan001 (left) and scan002 (right) of UC45-5.

Scans of UC45-11 - UC45-15

Saturday, May 31, 10:30 - Richard Jones

After completing the UC45-3, UC45-4, and UC45-5 diamonds, I continue on here with the 5 at the end of the series. Alex measured UC45-6 … UC45-10 back in May, 2013 so we do not need to repeat those measurements here. In fact, we already looked at UC45-6 and UC45-7 this run, as these have been cut using VPIE by Sinmat (see above). That leaves just the last 5 crystals in the 4.5 mm series to be measured. These have not yet been touched.

Rather than micro-managing the process for each diamond, I stick with 75 steps at step size 0.0002 deg to fully span the rc peak for the whole crystal.

Sunday June 1, 8:00am - Brendan Pratt
I came in and got to work on the remaining 4.5mm diamonds. Not much to report, just getting into a rhythm and trying to get as many scans in as I can. I must be getting tired because I rotated phi 180 degrees instead of rotating in chi.

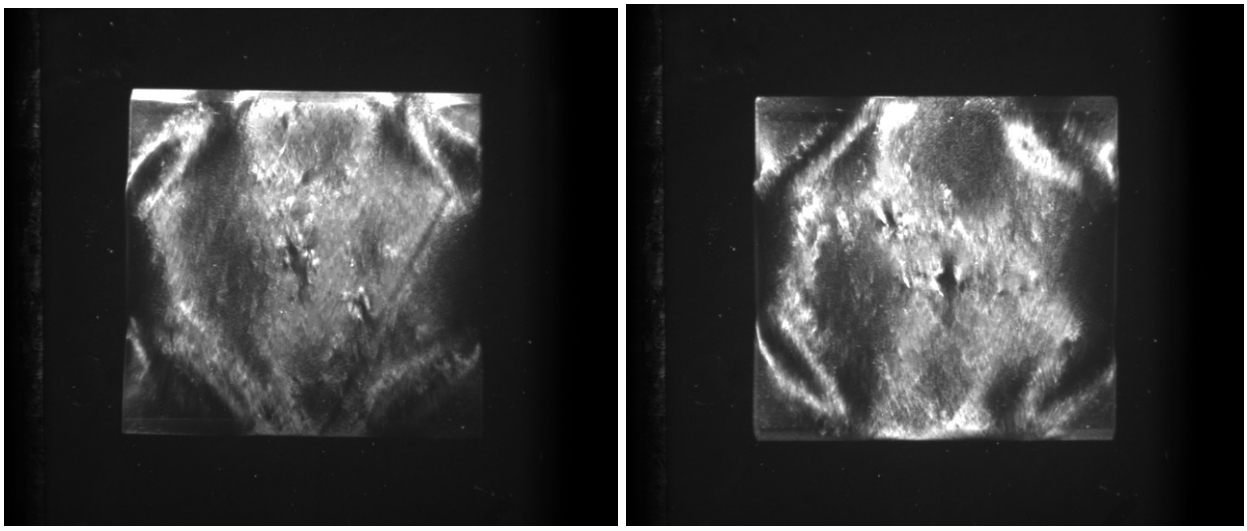
Sunday June 1, 2:00pm - Nathan Sparks
I started with the last scan for UC45-14 and then moved on to the scans of the last 4.5mm diamond, UC45-15. After mounting UC45-15, I forgot to create a new file. I stopped my initial scan about a third of the way through and then waited for about 10 minutes for the beginning of a new run to start a new properly labeled scan. UC45-14-study1_scan007 is the name of the first scan of diamond UC45-15 which was aborted after 30 data points. This aborted scan started at a theta value of 17.704. The other parameters are the same as the first completed scan.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC45-11-study1_scan001	(2,2,0)	75	0.0002	10
UC45-11-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-11-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-11-study1_scan004	(-2,-2,0)	75	0.0002	10
UC45-12-study1_scan001	(2,2,0)	75	0.0002	10
UC45-12-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-12-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-12-study1_scan004	(-2,-2,0)	75	0.0002	10
UC45-13-study1_scan001	(2,2,0)	75	0.0002	10
UC45-13-study1_scan002	(2,-2,0)	75	0.0002	10
UC45-13-study1_scan003	(-2,2,0)	75	0.0002	10
UC45-13-study1_scan004	(-2,-2,0)	75	0.0002	10
UC45-14-study1_scan001	(2,2,0)	75	0.0002	10
UC45-14-study1_scan002*	(2,-2,0)	75	0.0002	10

UC45-14-study1_scan003	(2,-2,0)	75	0.0002	10
UC45-14-study1_scan004	(2,2,0)	75	0.0002	10
UC45-14-study1_scan005	(-2,2,0)	75	0.0002	10
UC45-14-study1_scan006	(-2,-2,0)	75	0.0002	10
UC45-15-study1_scan001	(2,2,0)	100	0.0002	10
UC45-15-study1_scan002	(2,-2,0)	100	0.0002	10
UC45-15-study1_scan003	(-2,2,0)	100	0.0002	10
UC45-15-study1_scan004	(-2,-2,0)	100	0.0002	10

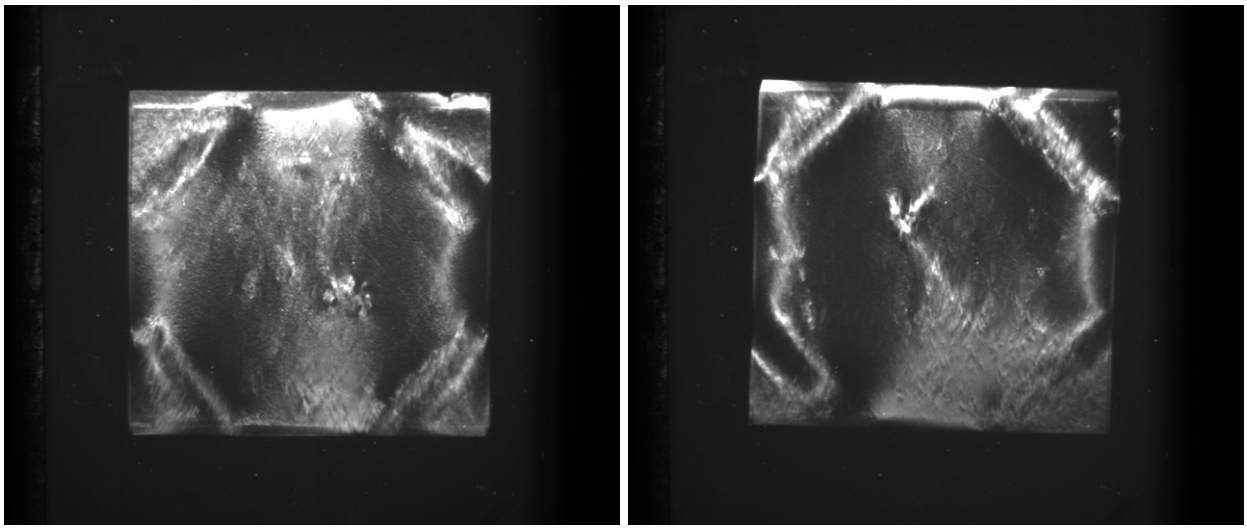
*the range in scan002 was incorrectly set as the peak appears right at the end of the scan, repeated with a better range in scan003.

UC45-11	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.573	136.4767	0.9	20.985 - 21.000	6:41
scan002	(2,-2,0)	37.873	137.1767	180.9	18.922 - 18.937	7:00
scan003	(-2,2,0)	37.323	-42.8233	180.9	19.320 - 19.335	7:32
scan004	(-2,-2,0)	37.623	-42.5733	0.9	17.220 - 17.235	8:00



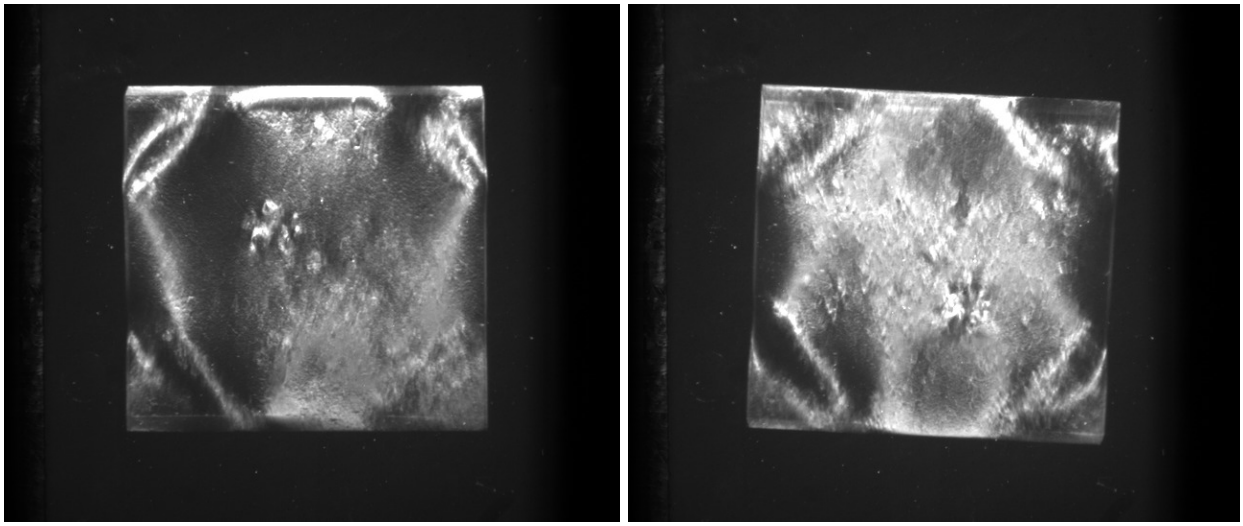
Selected images from scan001 (left) and scan002 (right) from UC45-11.

UC45-12	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.823	-44.3733	0.9	18.189 - 18.204	8:48
scan002	(2,-2,0)	37.423	-41.6233	180.9	18.909- 18.924	9:14
scan003	(-2,2,0)	37.6730	138.9767	180.9	19.308 - 19.323	9:44
scan004	(-2,-2,0)	37.3730	135.2267	0.9	20.019 - 20.034	10:07



Selected images from scan001 (left) and scan002 (right) of UC45-12.

UC45-13	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.523	135.0267	0.9	18.733 - 18.748	10:36
scan002	(2,-2,0)	37.586	139.1267	180.9	17.924 - 17.939	11:04
scan003	(-2,2,0)	37.596	-41.0733	180.9	20.320 - 20.335	11:30
scan004	(-2,-2,0)	37.696	-44.8733	0.9	19.450 - 19.465	11:52

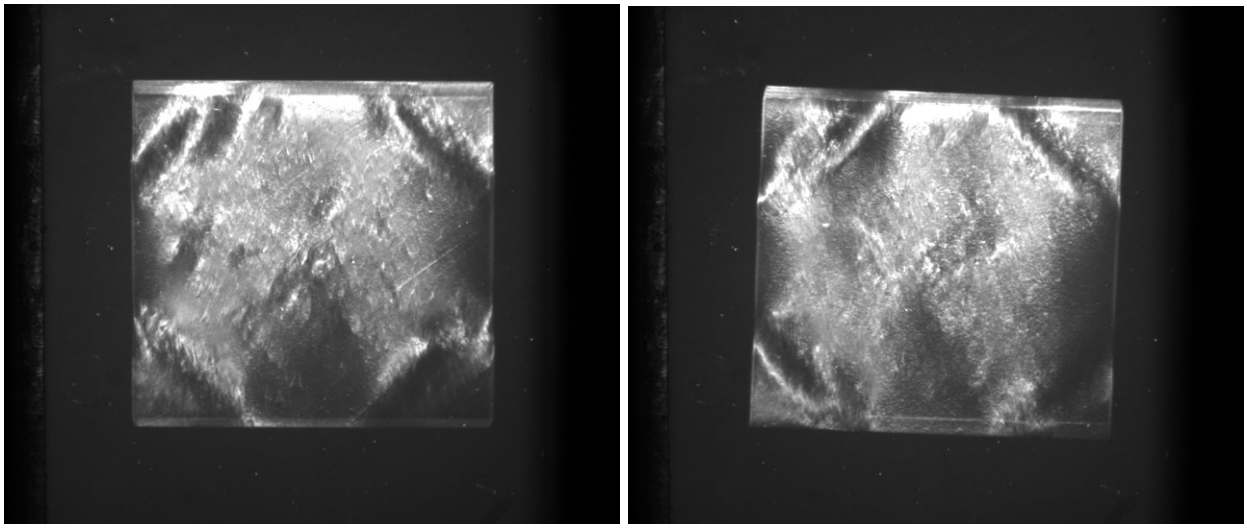


Selected images from scan001 (left) and scan002 (right) of UC45-13.

UC45-14	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.596	-43.0233	0.9	20.224 - 20.239	12:20
scan002*	(2,-2,0)	37.396	-42.3733	180.9	17.408 - 17.423	12:39
scan003	(2,-2,0)	37.396	-42.3733	180.9	17.415 - 17.430	12:58
scan004**	(2,2,0)	37.646	-43.073	0.9	20.186 - 20.201	13:19
scan005	(-2,2,0)	37.746	137.657	180.9	20.808 - 20.823	13:50
scan006	(-2,-2,0)	37.546	136.157	0.9	18.130 - 18.145	14:27

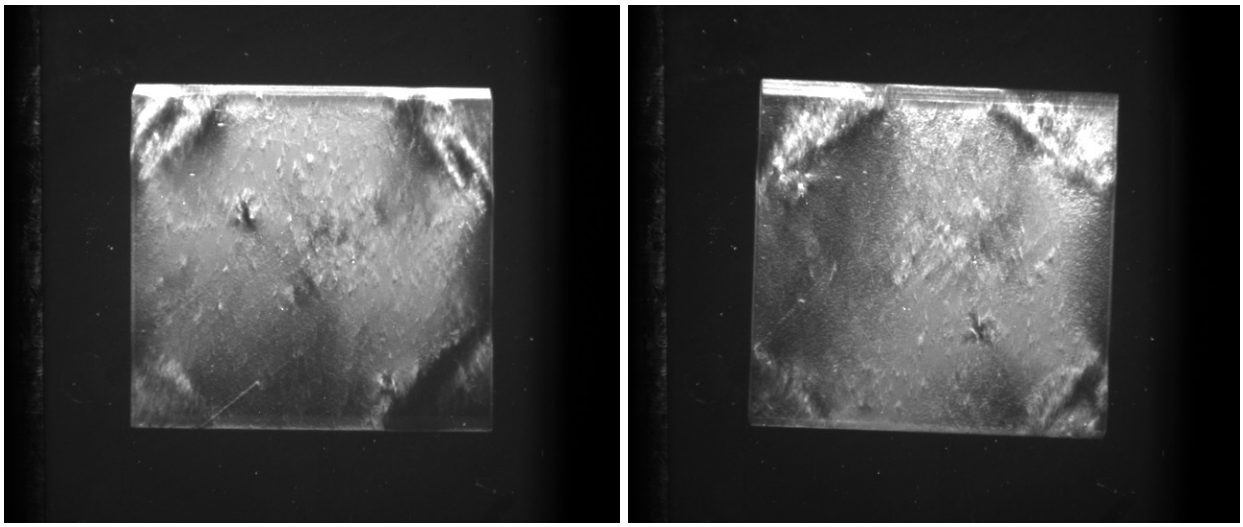
*scan002 was incorrectly set, but the entire scan was completed

**made the mistake of doing the same orientation twice



Selected images from scan001 (left) and scan003 (right) of UC45-14.

UC45-15	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.426	140.907	0.9	17.703 - 17.723	15:52
scan002	(2,-2,0)	37.701	133.137	180.9	19.613 - 19.633	16:32
scan003	(-2,2,0)	37.551	-47.163	180.9	18.614 - 18.634	17:06
scan004	(-2,-2,0)	37.651	-38.698	0.9	20.517 - 20.537	17:54



Selected images from scan001 (left) and scan002 (right) from U45-15.

Scans of UC30-15 - UC30-2

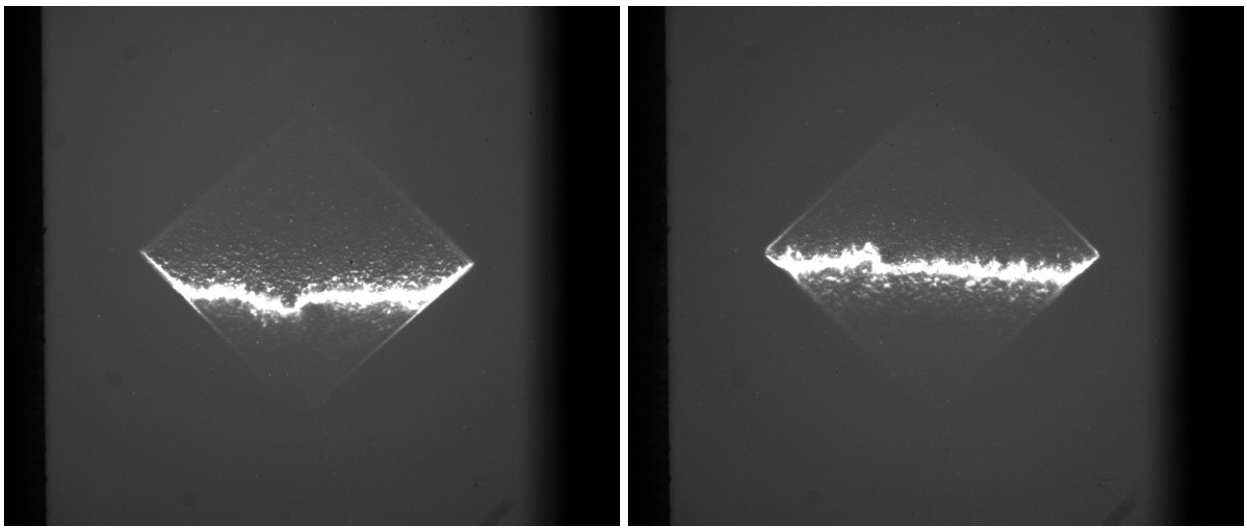
Sunday, June 1, 18:30 - Nathan Sparks

The previous diamond UC45-15 was removed from the mount and put back in its case. I then proceeded to mount UC30-15. I placed this diamond in the “square” orientation, as were the other UC30-diamonds.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-15-study1_scan001	(2,2,0)	200	0.0005	10
UC30-15-study1_scan002	(2,-2,0)	200	0.0005	10
UC30-15-study1_scan003	(-2,2,0)	400	0.00025	10
UC30-15-study1_scan005*	(-2,-2,0)	500	0.0002	10

* scan004 was aborted because the endpoints were misentered at the command prompt

UC30-15	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.651	-43.748	0.9	18.688 - 18.788	19:01
scan002	(2,-2,0)	37.351	-41.948	180.9	19.525 - 19.625	20:10
scan003	(-2,2,0)	37.651	138.152	180.9	18.595 - 18.695	21:00
scan005	(-2,-2,0)	37.551	135.652	0.9	19.470 - 19.570	22:45



Selected images from scan001 (left) and scan002 (right) for UC30-15.

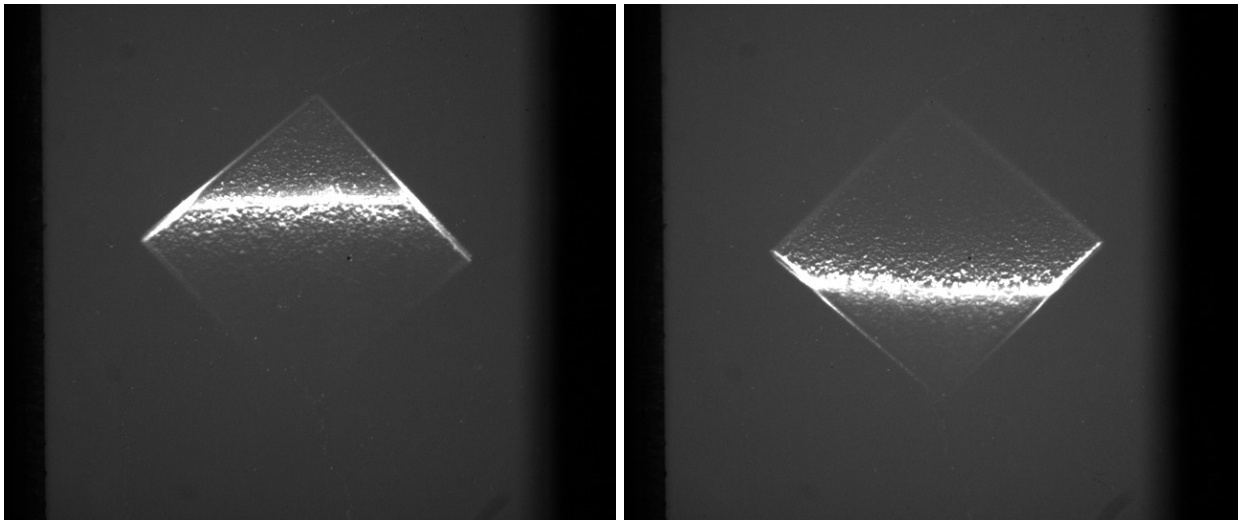
Sunday, June 1, 22:30 - Richard Jones

When I came on shift, scan003 of UC30-15 had just completed. I decided to make the last scan of UC30-15 at full resolution, so I set it up to run 500 steps. After that was complete, I put UC30-15 back in the case and mounted UC30-16. I also decided to continue scanning these samples at full resolution, which gives me enough time to work on analysis of the data we have taken so far, in hopes that if anything surprising shows up we will still have time to go back and measure something again or check vary the conditions.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-15-study1_scan006*	(2,2,0)	350	0.0002	10
UC30-15-study1_scan007*	(2,-2,0)	350	0.0002	10
UC30-15-study1_scan008*	(-2,2,0)	350	0.0002	10
UC30-15-study1_scan009*	(-2,-2,0)	350	0.0002	10

*These scans were actually performed on crystal UC30-16, but I failed to enter the “newfile” command before starting the scans, so they were appended to the runs for UC30-15 instead.

UC30-**16***	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan006	(2,2,0)	37.351	139.152	0.9	17.780 - 17.850	12:58
scan007	(2,-2,0)	37.651	134.852	180.9	16.670 - 16.740	2:10
scan008	(-2,2,0)	37.451	-45.748	180.9	21.520 - 21.590	3:28
scan009	(-2,-2,0)	37.751	-40.348	0.9	21.810 - 21.880	4:53

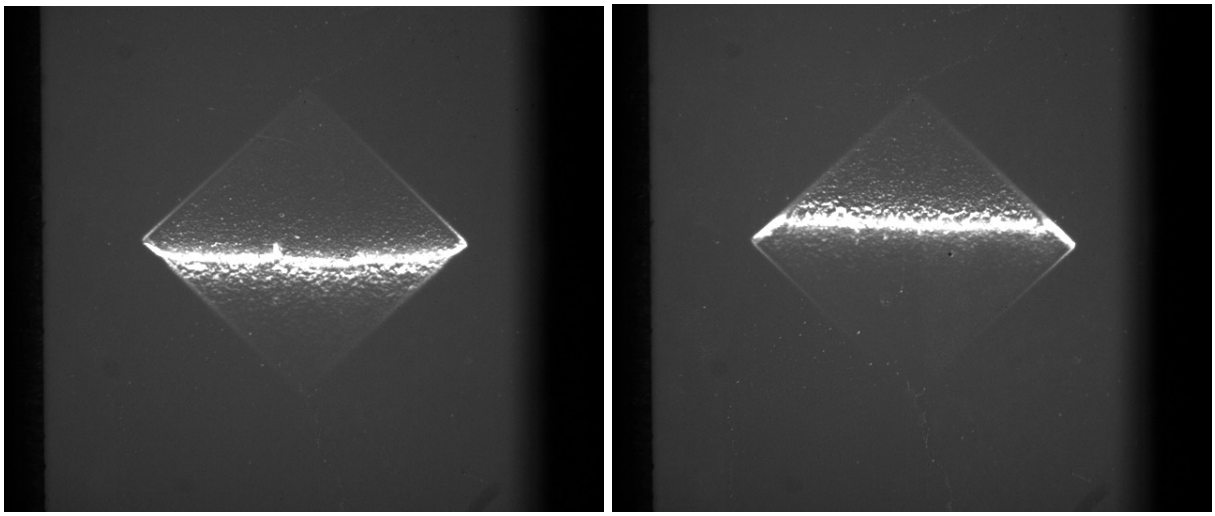


Selected images from scan005 (left) and scan006 (right) of sample UC30-16. These data were from a dataset named UC30-15 because I failed to remember to set the “newfile” to UC30-16 before scanning.

I took crystal UC30-16 out of the mount and returned it to the case, replacing it in the mount with UC30-17. The following scans were then performed on this crystal.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-17-study1_scan001	(2,2,0)	350	0.0002	10
UC30-17-study1_scan002	(2,-2,0)	350	0.0002	10
UC30-17-study1_scan003	(-2,2,0)	350	0.0002	10
UC30-17-study1_scan004	(-2,-2,0)	350	0.0002	10

UC30-17	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.551	-41.548	0.9	21.150 - 21.220	6:30
scan002	(2,-2,0)	37.843	-44.048	180.9	20.010 - 20.080	7:44
scan003	(-2,2,0)	37.6510	135.952	180.9	18.160 - 18.230	9:14
scan004	(-2,-2,0)	37.551	137.852	0.9	18.450 - 18.520	10:25



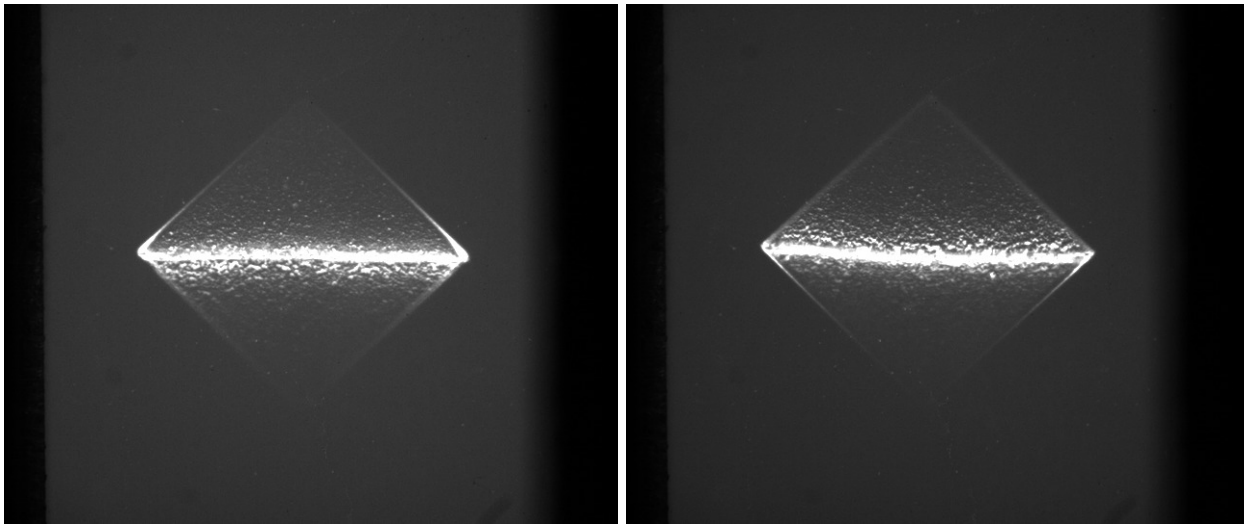
Selected images from scan001 (left) and scan002 (right) of UC30-17.

Monday, June 2, 14:45 - Nathan Sparks

My first scan of the day was scan 3 of UC30-18. I am sticking with the same scan parameters which were used by Richard and Brendan on the most recently scanned UC30-diamonds. I am no longer timing my scans to avoid electron beam fill periods, since for most of the fills the I0 current level stays relatively constant (around the expected value, ~50,000).

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-18-study1_scan001	(2,2,0)	350	0.0002	10
UC30-18-study1_scan002	(2,-2,0)	350	0.0002	10
UC30-18-study1_scan003	(-2,2,0)	350	0.0002	10
UC30-18-study1_scan004	(-2,-2,0)	350	0.0002	10

UC30-18	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.5510	137.652	0.9	19.07 - 19.140	11:41
scan002	(2,-2,0)	37.651	136.152	180.9	20.350 - 20.420	13:06
scan003	(-2,2,0)	37.451	-43.848	180.9	17.841 - 17.911	14:58
scan004	(-2,-2,0)	37.601	-41.848	0.9	19.075 - 19.145	16:17

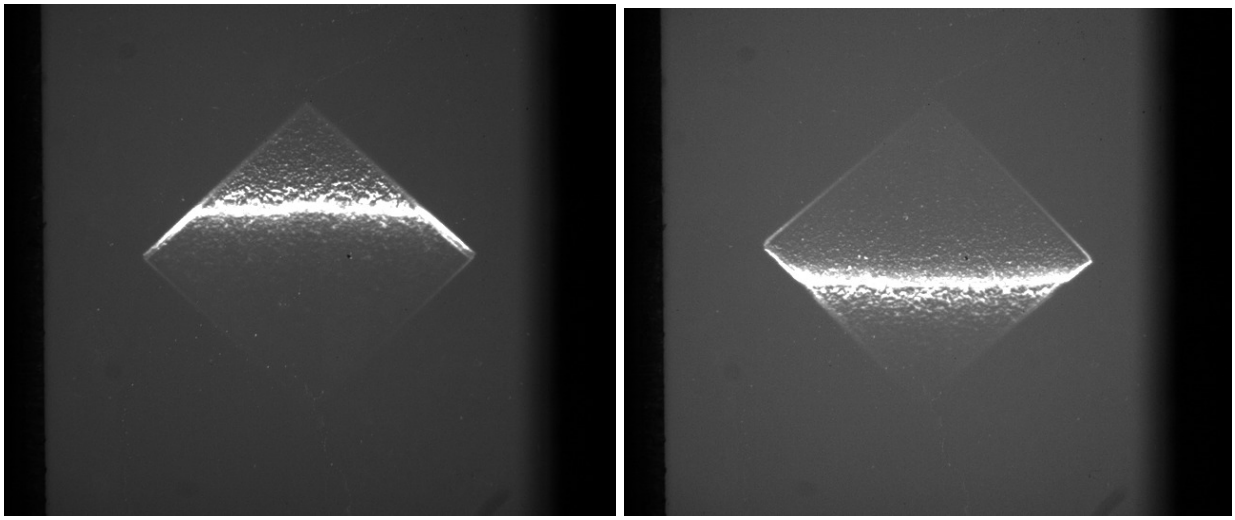


Selected images from scan001 (left panel) an scan002 (right) for UC30-18.

The first scan of UC30-19 paused after 17 points so I aborted it. I tried another scan and it also froze. Then I restarted “fourc” and tried another scan which proceeded to immediately hang as well. The ANDOR camera appeared to be unresponsive to all commands, so I asked the beam operator for help. The beam operator found that the camera was in an error state and restarted the detector. After the detector was restarted, the camera became responsive again, and I started a new scan. However, I forgot to turn-off the hutch light, so I had to abort scan 4.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-19-study1_scan005	(2,2,0)	350	0.0002	10
UC30-19-study1_scan006	(2,-2,0)	350	0.0002	10
UC30-19-study1_scan007	(-2,2,0)	350	0.0002	10
UC30-19-study1_scan008	(-2,-2,0)	350	0.0002	10

UC30-19	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan005	(2,2,0)	37.701	-45.148	0.9	19.765 - 19.835	19:33
scan006	(2,-2,0)	37.371	-40.548	180.9	17.338 - 17.408	20:57
scan007	(-2,2,0)	37.646	139.582	180.9	20.823 - 20.893	22:19
scan008	(-2,-2,0)	37.546	134.382	0.9	18.410 - 18.480	23:34

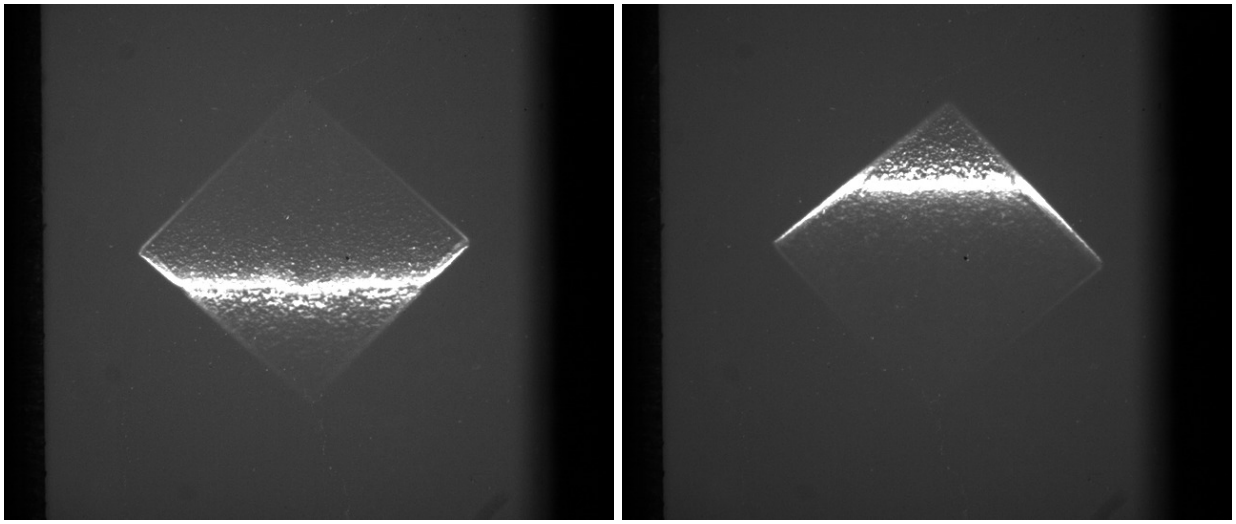


Selected frames from scan005 (left) and scan006 (right) from UC30-19.

June 2, 2014, Richard Jones
I came on shift at 11:00, right around the start of the last scan of UC30-19. I continued with the previous run, then changed over to diamond UC30-20 and continued scanning as usual.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-20-study1_scan001	(2,2,0)	350	0.0002	10
UC30-20-study1_scan002	(2,-2,0)	350	0.0002	10
UC30-20-study1_scan003	(-2,2,0)	350	0.0002	10
UC30-20-study1_scan004	(-2,-2,0)	350	0.0002	10

UC30-20	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan001	(2,2,0)	37.546	134.282	0.9	19.650 - 19.720	0:53
scan002	(2,-2,0)	37.646	139.682	180.9	19.580 - 19.650	2:07
scan003	(-2,2,0)	37.446	-40.418	180.9	18.610 - 18.680	3:22
scan004	(-2,-2,0)	37.646	-45.318	0.9	18.490 - 18.560	4:42



Selected frames from scan001 (left) and scan002 (right) of UC30-20.

Filename	**Orientation**	**Steps**	**Step size (degrees)**	**Exposure (s)**
UC30-20-study1_scan005*	(2,2,0)	175	0.0002	10

* This should have been UC30-21-study1_scan001, but I forgot to enter the newfile command after I changed to the new diamond.

UC30-21	**Orientation**	**TTh**	**Chi**	**Phi**	**Th**	**Time**
scan005	(2,2,0)	37.646	-43.518	0.9	14.440 - 14.510	6:15

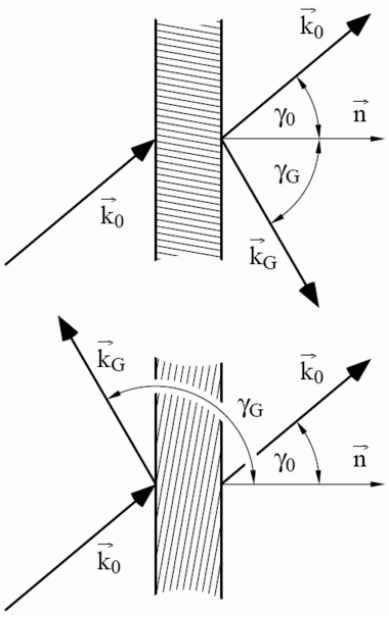
The run ended at 7:00am on June 3. We had time to complete the first scan of UC30-21 but only at half-resolution, and without any complementary scans. We probably need to come back and rescan this one next run, if we decide we need it.

Understanding the grainy appearance of some topographs

Looking at these rocking curve images for the 3mm diamonds, they look grainy as if they were composed of small grains of size on the order of 10-25 microns. This gives the image a texture like pebbles on a beach, or gravel on a driveway. The images of the 4.5mm and 7mm diamonds also have a roughness to them, but the grainy texture is unique to the 3mm diamonds in this set. I wonder if there is something fundamentally different about this material, that sets it apart from the material used to make the 4.5mm and 7mm stones, apart from their size and thickness.

One obvious possibility is that these 3mm diamonds are not polished on both sides, whereas the 4.5mm and 7mm diamonds are polished on both sides. This difference is apparent just looking at the 3mm stones: one side looks glossy when reflecting light, whereas the other looks matte. I wonder if this texture could be the result of the “pendelloesung” effect in transmission-mode diffraction where the intensity of the flux in bulk of the crystal oscillates between the two modes of X-rays propagating through the diamond, one with direction Kinc and the other with direction Kref, like a pair of coupled oscillators. Without a detailed calculation, I would guess that the pendellosung wavelength is on the same order as the extinction length. I found an online calculator of X-ray diffraction properties of materials here. This tool gives two answers for the extinction length of 15 keV X-rays from 2,2,0 planes in diamond: one for Bragg geometry and one for Laue geometry. How these are related is not explained. There are also slightly different answers depending on the polarization of the X-rays, but these differences are not important in the particular case of interest here. Here are the results.

geometry \ polarization	sigma	pi
Bragg	4.17 um	5.31 um
Laue	37.6 um	48.0 um



A comparison is shown at the left of Laue (top) and Bragg geometries (bottom) for diffraction. In the Laue case the transmitted and reflected waves both emerge from the opposite side of the crystal from where the incident beam enters, whereas in Bragg geometry the reflected wave emerges from the same surface where the incident beam enters.

From these pictures, I was able to figure out how to relate the two extinction lengths. For the symmetric Bragg case, the extinction length is the depth below the surface where the incident beam intensity drops to 1/e of its value on at the surface. If L is the distance along the original k vector inside the crystal where the incident wave intensity drops by a factor 1/e, then $X_{Bragg} = L \sin\theta_B$ By contrast, in the Laue case the same factor L appears in an oscillation phase factor, so extinction occurs when the wave advances by a phase of π, leading to $X_{Laue} = \pi L \cos\theta_B$ where the trig factors appear because the extinction length is measured in terms of sample thickness instead of distance along a kinematic vector in the sample. These formulas predict that $X_{Bragg} / X_{Laue} = (\tan\theta_B)/\pi$ which actually holds for the above values, and the given Bragg angle $\theta_B = 19.174$ degrees.

For the symmetric Laue case, which corresponds to our own geometry actually, the extinction length is the depth below the surface where the intensity of the incident beam crosses through zero because of the Pendellosung effect. The full Pendellosung period is thus twice the Laue extinction length quoted above, which is around 100 microns. This is too large to explain the texture observed in the 3mm diamond topographs. To rule out any effects from absorption affecting this comparison, I checked the absorption length in diamond at this wavelength and it is 5mm or so, too large to have a significant effect on scattering at or near the 2,2,0 diffraction conditions. So the question remains unanswered what is causing the strong contrast on the two sides of the rocking curve peaks in the scans of the 3mm samples.

At optical wavelengths this effect might be due to destructive/constructive interference between waves that pass through different lengths of material to arrive at the same position on the viewing screen. How much thickness variation would be required to use this explanation? The real part of the refractive index at this wavelength is 1-3.3e-6 according to the above-mentioned online calculator, so to get 100% contrast would require a full 2π phase shift would require a path length difference $\Delta k_R L = 2\pi$, or L = 25 microns. This seems like too large a thickness variation. Given that the transverse feature size of these grains is around 25 microns, this would imply that the surface features are as deep as they are wide, which would be surprising if true.

The wikipedia page on Diffraction Topography lists a number of possible causes for contrast that appears in a topograph.

1. voids and inclusions in the crystal
2. phase boundaries (regions of different crystallographic phase, polytype, ...)
3. defective areas, non-crystalline (amorphous) areas / inclusions
4. cracks, surface scratches
5. stacking faults
6. dislocations, dislocation bundles
7. grain boundaries, domain walls
8. growth striations
9. point defects or defect clusters
10. crystal deformation
11. strain fields

I think I can rule out 1, 2, 3 because the dark regions in the topographs occupy a significant fraction of the entire crystal volume, and the excellent global crystal quality would seem to exclude that. Possibility 4 is what I have been dealing with above, and think I have ruled out. Features related to 5-8 would not be uniformly distributed, and these are clearly seen in the 4.5mm and larger samples forming a characteristic hexagonal pattern of striations with high defect density in certain regions. Number 10 is what causes the global curvature seen in the rocking curve peak displacement across the crystal, which is the principal feature that these rocking curves are collected to measure.

Number 9 is a possibility. If that is the case, the dark regions in one part of the rocking curve might show up as light regions in another part, as their peak has simply shifted elsewhere in angle, indicating a d-spacing variation that might be the result of inclusions or other types of point defects. Another possibility is that the contrast is due to rocking curve width variations, rather than peak shifts, with the light spots corresponding to regions with larger rc widths that tend to scatter a larger fraction of the beam than regions with a very narrow rc width. We know that our beam, even though it is monochromated, still has a considerable energy width, only a fraction of which may be within the scattering peak of perfect diamond at any given scattering angle. In addition, even if the beam were perfectly monochromated, there are still vibrations in the target angle within its mount that can penalize the integrated light intensity for pixels that view highly perfect crystals in contrast to those that view crystals that diffract within a broader angular band. I don't see how numbers 10 and 11 are really distinct, from an operational point of view. To make any progress, we need to make fits to the individual pixel rocking curves and do detailed comparisons between them to see what the data are telling us.

What to do before leaving CHESS

- Make sure all the diamonds and diamond accessories (tweezers, cleaners, etc) are put back in the safe box
- Place the hoop and mount in their storage box
- If Dr. Jones isn't there at check out be sure to call him before turning in badges.
- Make sure all image files and log files have been pushed to our servers.
- Leave the scan files on the computer so that in the future we can remember the naming convention.
- Remove the image files from the Andor1 computer (computer in the hutch)