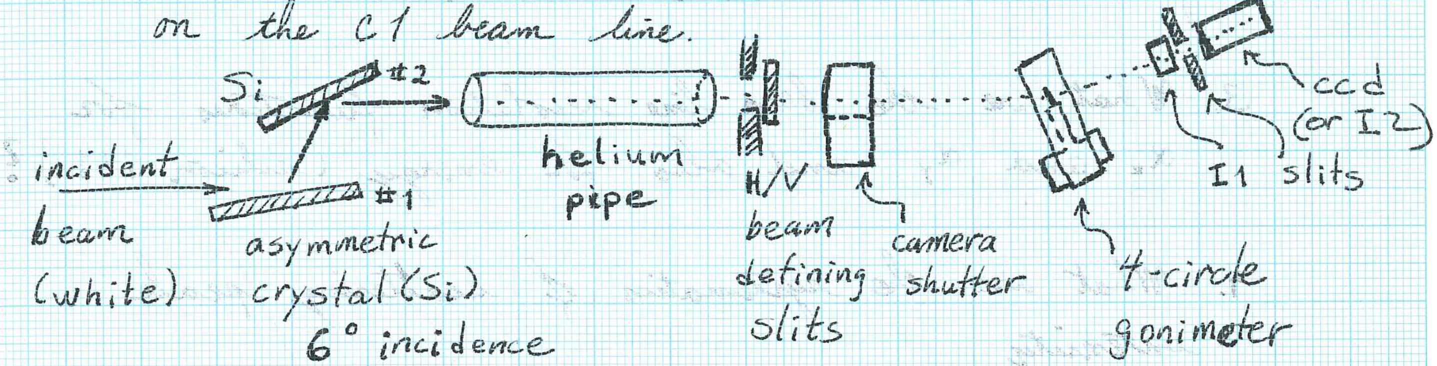
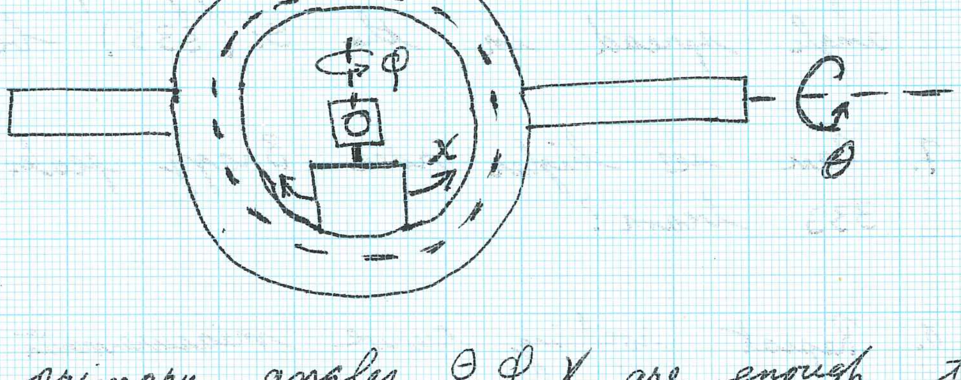
Feasibility Measurements on Diamonds at CHESS

Nov. 1 2006 We arrived at CHESS today around 1:30 pm and were given a safety walk-through of the facility. We did our online quiz and each received a TLD badge.

Ken Finkelstien then gave us a tour of the set-up on the C1 beam line.



Blow-up of goniometer



The three primary angles θ, φ, χ are enough to orient the diamond at a random orientation. A telescope pointing at the crossing of the three goniometer axes allowed us to keep the sample fixed while turning it.

Nov 2
2006

Questions to be addressed during this run.

1. What are the goniometer offsets of the #3 crystals in the target ladder?

2. What are the rocking curves for these 3 crystals for (220), (2-20), (400), (040)

3. What are the beam line resolution functions for α_x and α_y and also for energy (silicon 111)?

4. What are the systematics of scattering peak intensity

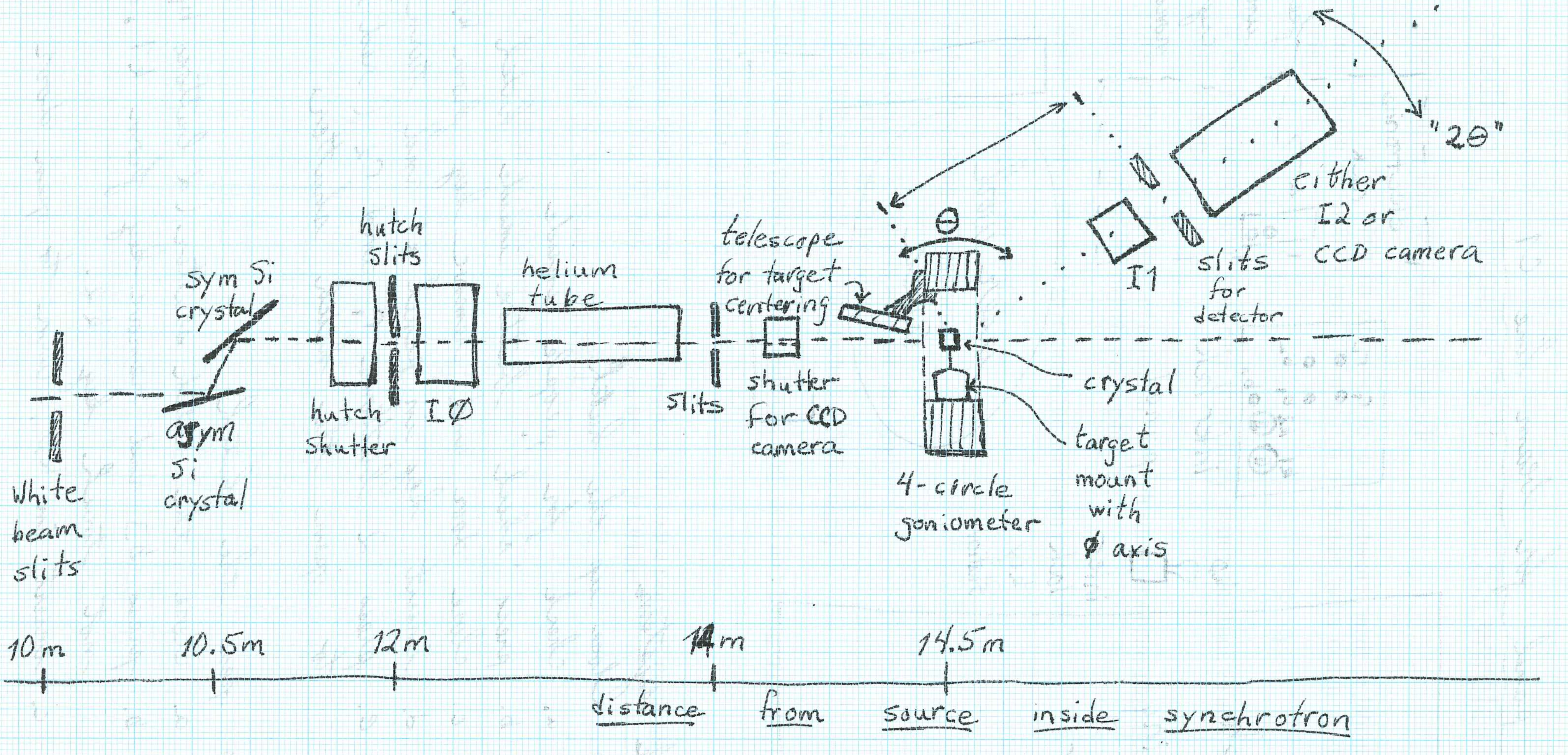
5. Can we see a Bragg peak from the (333) planes of silicon?

6. If so, what is the energy spread and the angle spread in the Si (333) beam.

7. Can see higher order Bragg peaks in the 333 beam?

8. Repeat rocking curve measurements in the Si 333 beam.

CHESS Beam Line C1 (vertical plane)



from CHESS web site (C1 beam line page)

source size: 1.486 mm horizontal (FWHM)
1.39 mm vertical (FWHM)

Detectors:

- ion chambers I0, I1, I2
- CCD camera (in place of I2)
- I1 removed during run 10/06

Control panel for beam line C1

Interlock system

1. To close up the hutch
   a. check for people inside
   b. press "check" button inside hutch
   c. close door and remove key from k0
   d. insert key in panel k1 and turn
   e. press 2 buttons r_1 and r_2 (red) to open the hutch shutter.

2. To open the hutch
   a. close the hutch shutter (2 buttons b_1, b_2)
   b. press b3 and remove key from k1, do not touch key k2.
   c. insert in k0, turn to open door

Adding a computer to the CHESS wireless network

Accessing the wireless network

All users who wish to connect to our network with a Windows computer must have an active real time virus scanner with current virus definitions installed.

If they cannot show you one then they must contact Frank Labonte before connecting.

Due to the large number of wireless network cards that are available for computers we cannot support the hardware in general.

Settings users will need to access the wireless network.

1.Set connection for DHCP

2.Network name - chess wap

3.The connection key is a 64 bit hex key
  a.) ab03e4f842 for windows
  b.) $ab03e4f842 for Mac (note the dollar sign)

The Wireless network is a "B" network and runs at 11meg maximum.

Connecting with cable.

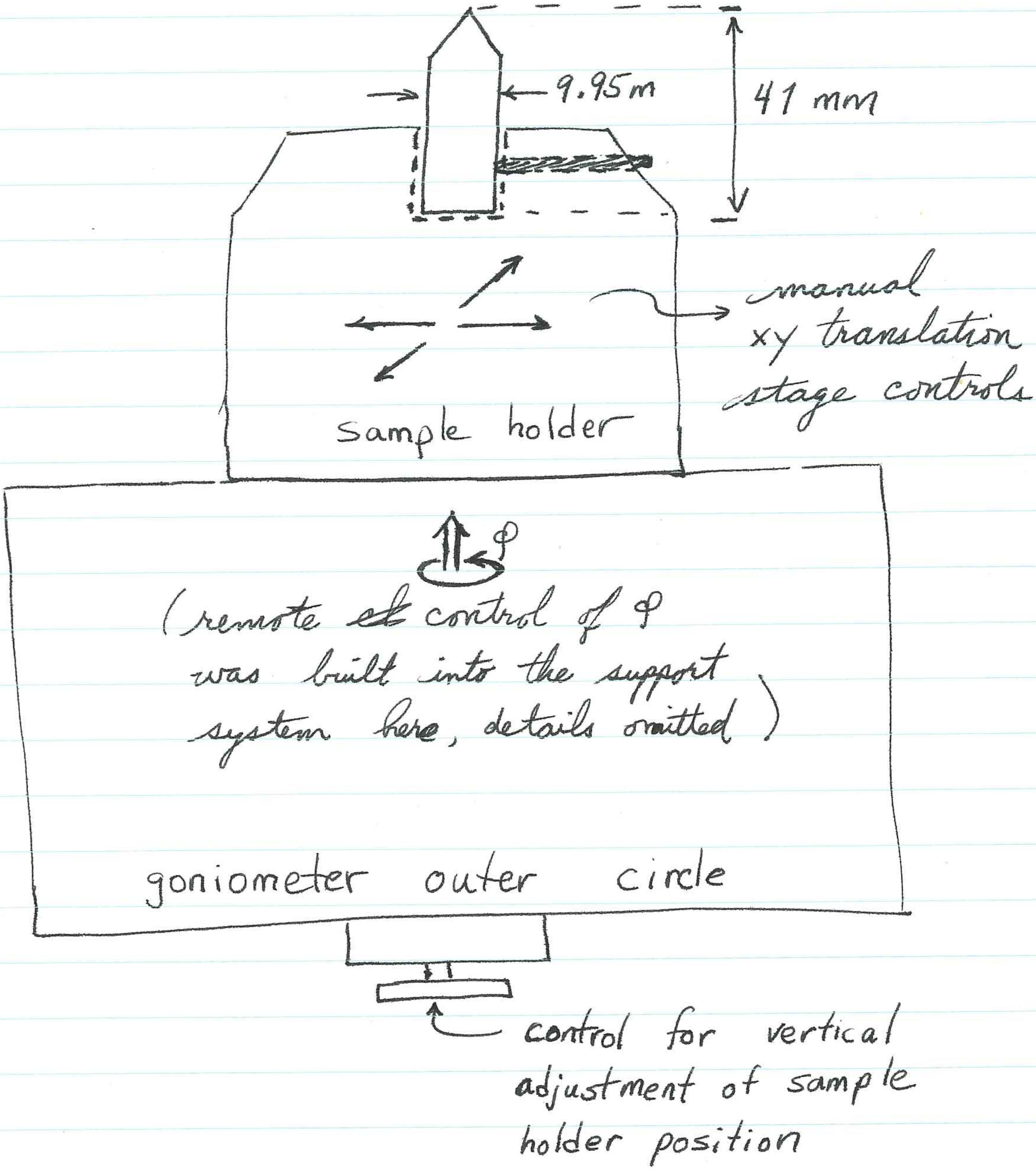
If users need a faster connection there are extra ports at each station that a user can plug into. All they have to do is set up for DHCP.

Created By: Lee Shelp 2-21-2004

Last Updated 07/14/2005

Sample mount and goniometer

First, the sample must be placed inside the mount and located so that it stays at the sample position as the goniometer rotates.



(remote ~~et~~ control of φ was built into the support system here, details omitted)

The above picture shows the goniometer target mount with the positioning pin installed in the place of an actual target. We used an optical sample mount and fabricated a post of appropriate length to hold it in the sample fixture hole. There was a set screw to secure it.

Using the manual translation controls on the target holder we were able to locate several crystals on the goniometer center point without removing them from the target ladder (see below). The Φ control for the goniometer is a part of the target support not shown in the figure on p. 103.

Beam Line Setup

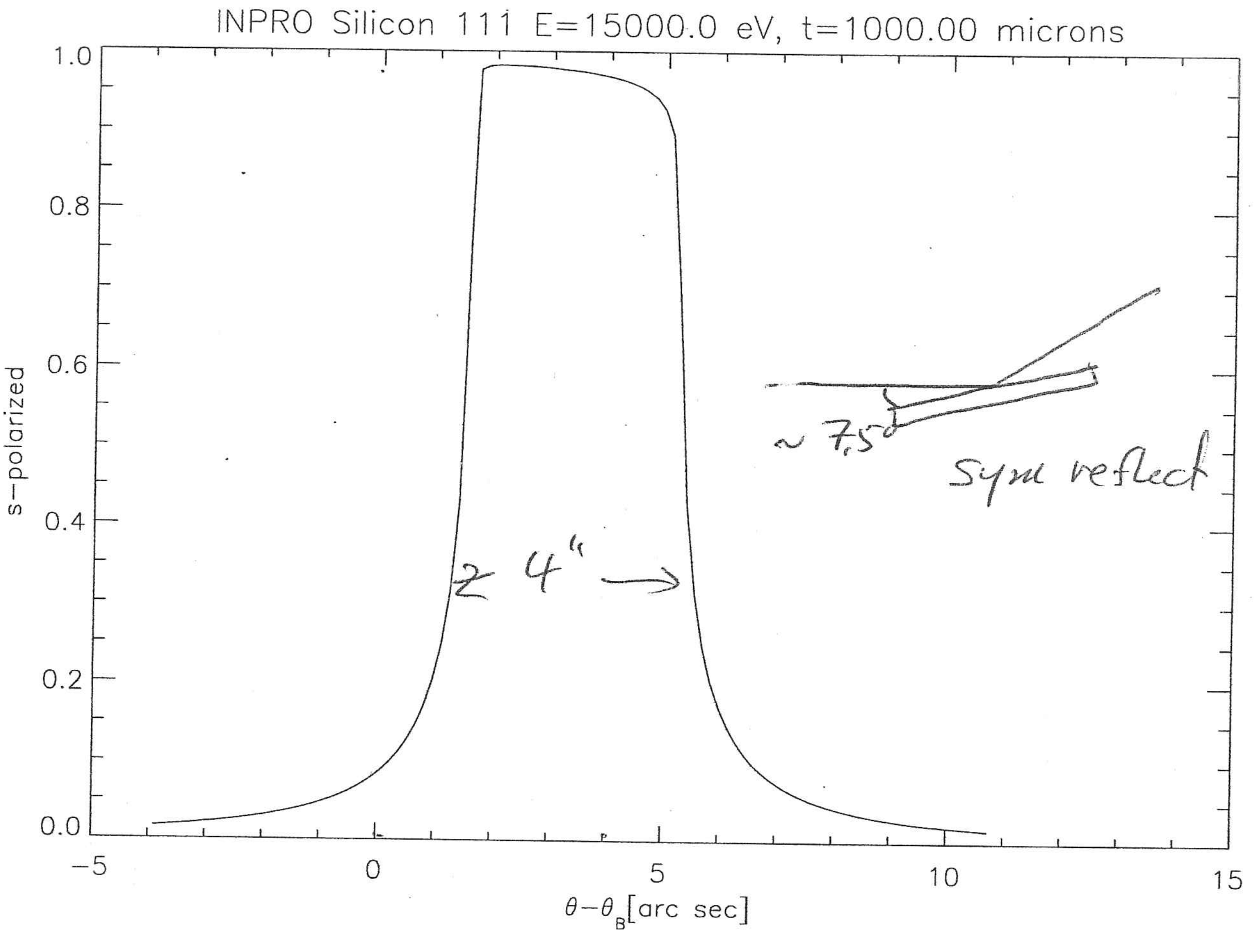
The definition of the beam is determined by

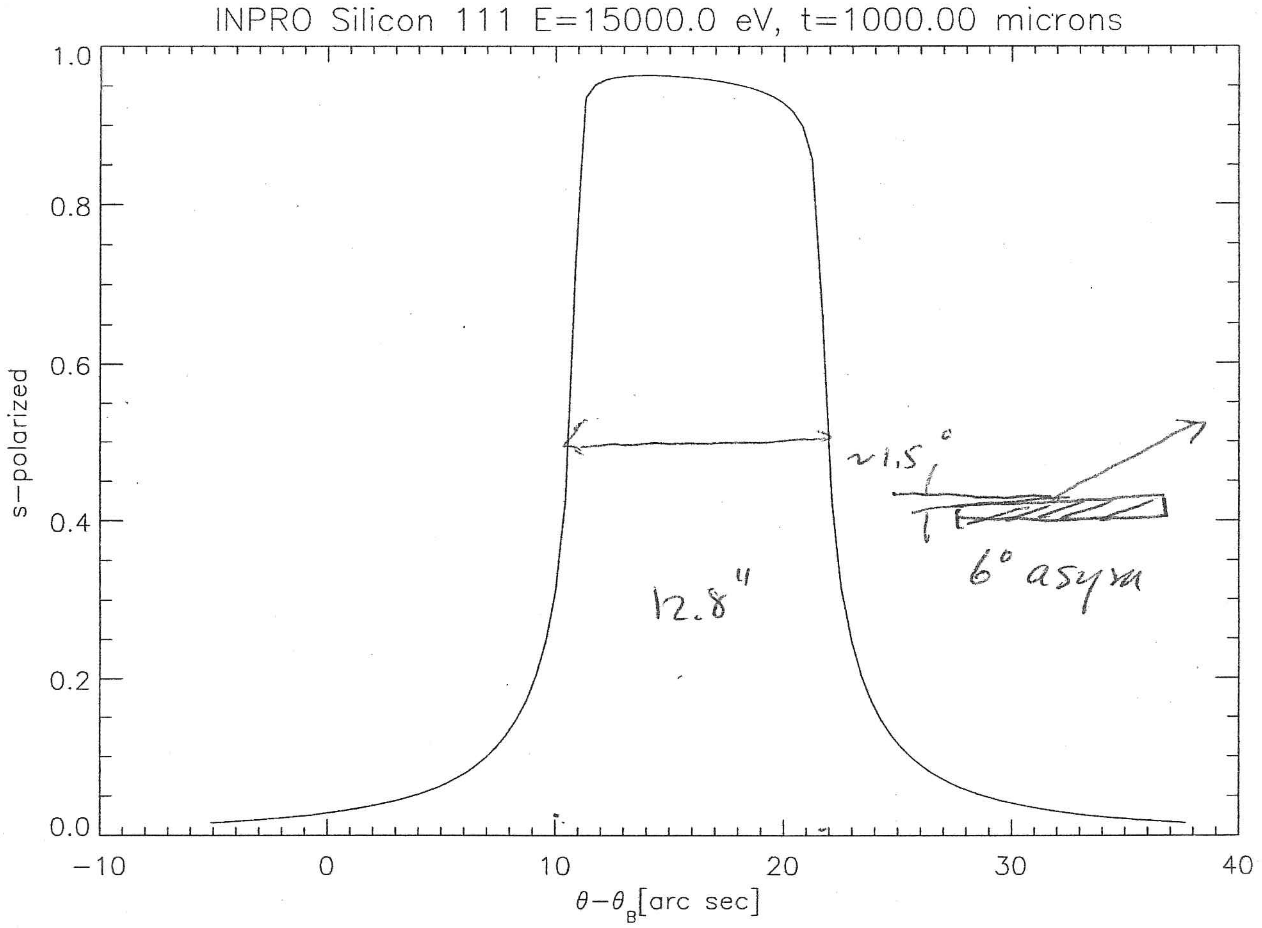
1) The synchrotron source size
2) The slits just before the target (at 14 m, p. 100)
3) The kinematics of scattering from the 111 planes of silicon in the asymmetric crystal. Or, one may look at the 333 planes

The intrinsic divergence of the beam (without the asymmetric crystal) at the target position is given by the source size.

horizontal : $1.03 \cdot 10^{-4}$ r FWHM
vertical : $9.59 \cdot 10^{-5}$ r FWHM

The actual vertical emittance is about a factor 3 better than this because of the asymmetric crystal monochromator described in the following plots by K. Finkelstein.

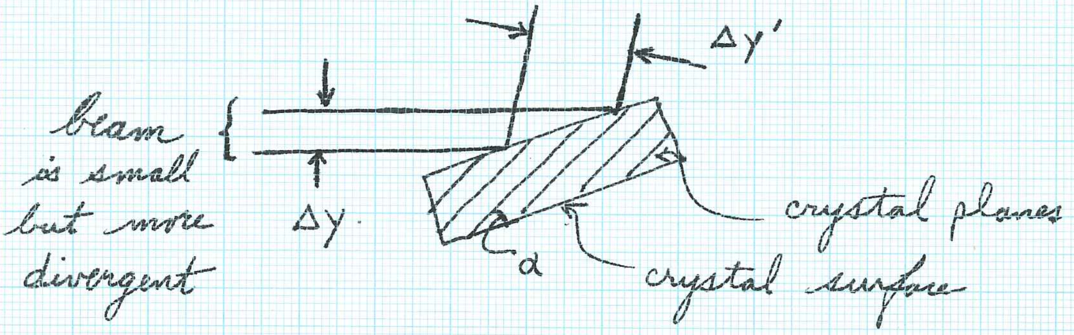

INPRO Silicon 111 E=15000.0 eV, t=1000.00 microns
s-polarized
1.0
0.8
0.6
0.4
0.2
0.0
−5
0
5
10
15
θ−θ_B[arc sec]
≈ 4"
~7.5°
sym reflect


INPRO Silicon 111 E=15000.0 eV, t=1000.00 microns
s-polarized
1.0
0.8
0.6
0.4
0.2
0.0
−10
0
10
20
30
40
θ−θ_B[arc sec]
~1.5°
6° asym
12.8"

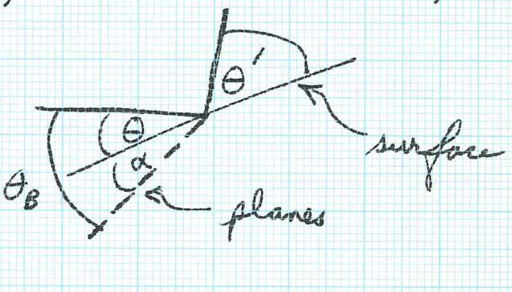
The curves on p. 105 are theoretical rocking curves for Silicon 111 planes in a 15.0 keV x-ray beam. The thickness (1 mm) sets the scale for the tails for thin crystals (thin compared to isotropic absorption depth).

1. The rocking curve width for the symetric crystal (top) is about 20 μr.

2. For the asymmetric crystal the rocking curve width depends on which direction is chosen.



From geometry, we see that Δy' is related to Δy



$$\frac{\Delta y}{\sin\theta} = \frac{\Delta y'}{\sin\theta'}$$

$$\Delta y' = \frac{\sin\theta'}{\sin\theta}\,\Delta y$$

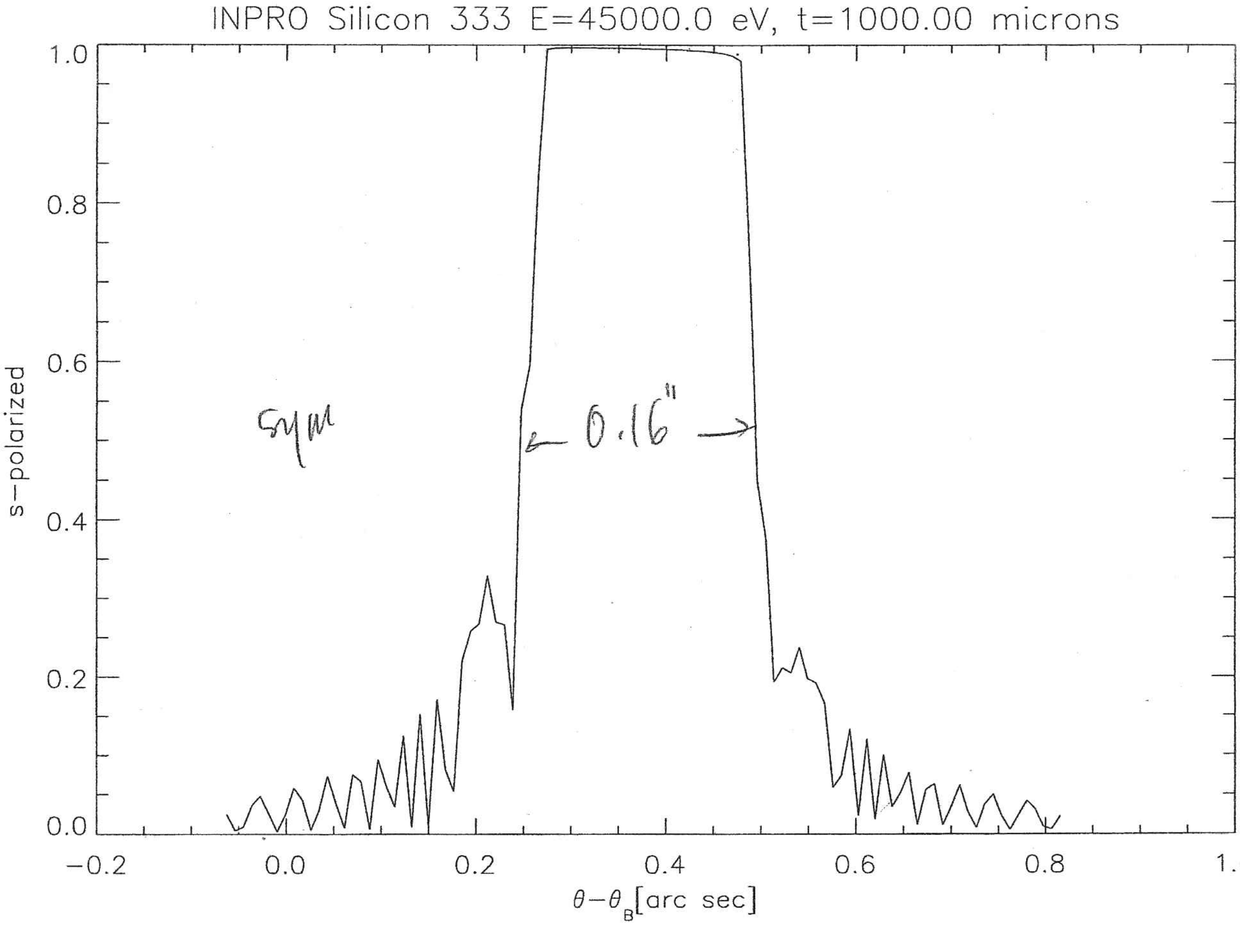
$$\Delta y' = \underbrace{\left(\frac{\sin(\theta_B+\alpha)}{\sin(\theta_B-\alpha)}\right)}\Delta y$$

θ_B is "Bragg angle"

<u>asymmetry parameter</u> "b" of monochromator

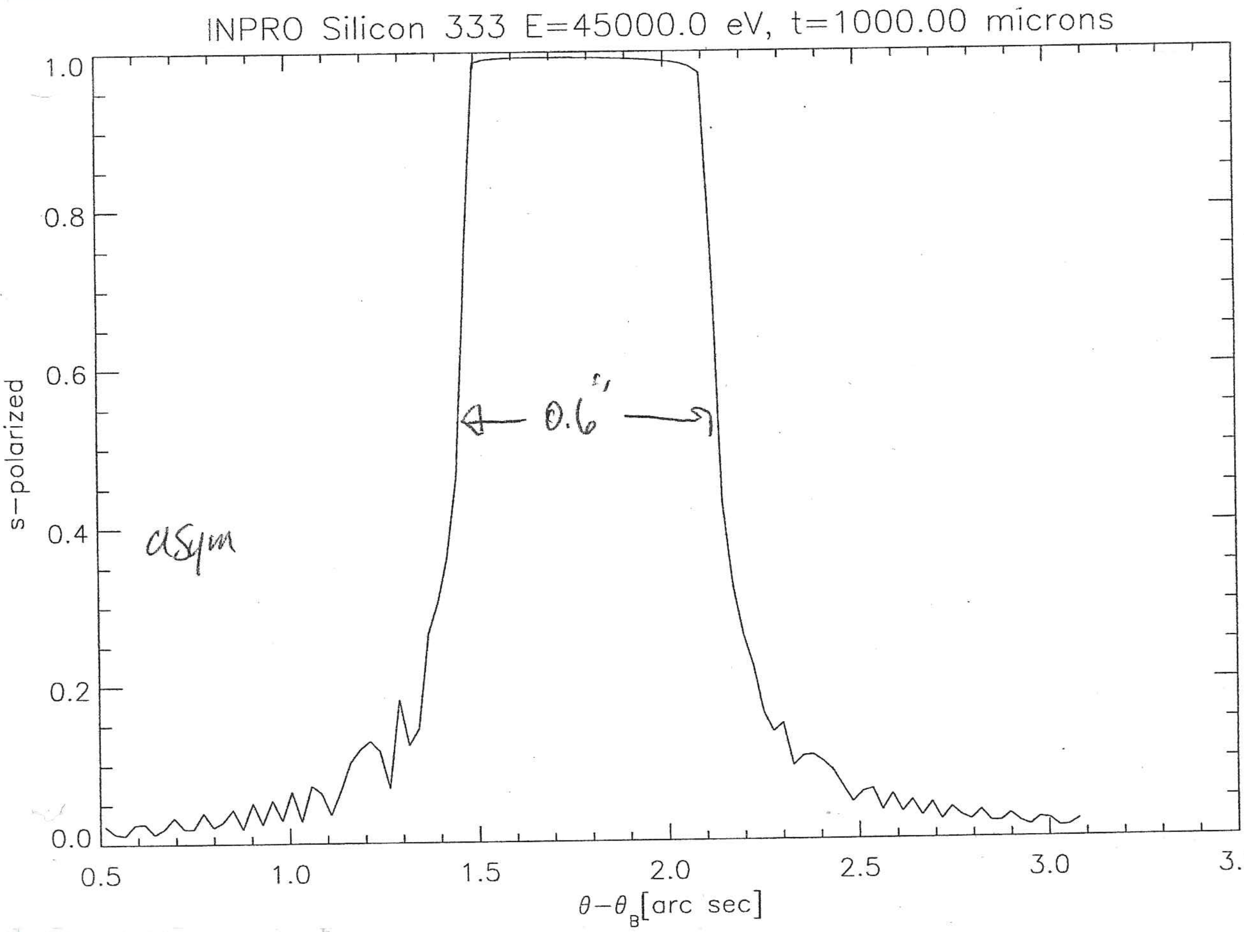
So to maximize the effect, one wants ~~θ_B small and~~ α close to θ_B : here it is <u>$\alpha = 6°$</u>; <u>$\theta_B = 7.5°$</u>

★ Liouville's theorem: $\boxed{\Delta\theta'_y = b^{-1}\,\Delta\theta_y}$ ← <u>reversibility</u>



sym

← 0.16" →



asym

← 0.6" →

The effect on the rocking curve is split between the two sides.

1) large-angle incidence side $\Delta\theta_{rc} = \frac{1}{\sqrt{b}}\,\Delta\theta_0$

2) small-angle incidence side $\Delta\theta_{rc} = \sqrt{b}\,\Delta\theta_0$

From the figures we see the effect for $\underline{\underline{b = 8.7}}$ where the beam enters from the small-angle side (case 2)

$$\text{Apparent source distance} = \underbrace{10.1\,m}_{\text{source distance at monochromator}} \times b = \underline{\underline{89m}}$$

Adding in the 4.4 m to the target station gives $\underline{\underline{93.4m}}$ which is comparable to what we had at Daresbury.

$$\left.\begin{array}{ll} \star\ \text{Vertical divergence at target:} & 1.51\times10^{-5}\,r \\ \star\ \text{Horizontal divergence at target:} & 1.03\times10^{-4}\,r \end{array}\right\}\text{FWHM}$$

To see how beam divergence translates into rocking curve width, we compare the q vector magnitudes in the monochromator and the diamond.

$$\delta\theta_{rc} = \left\{ 1 - \frac{\sin\theta_m}{\sqrt{\left(\frac{q_m}{q_d}\right)^2 - \cos^2\theta_m}} \right\} \delta\theta_\gamma$$

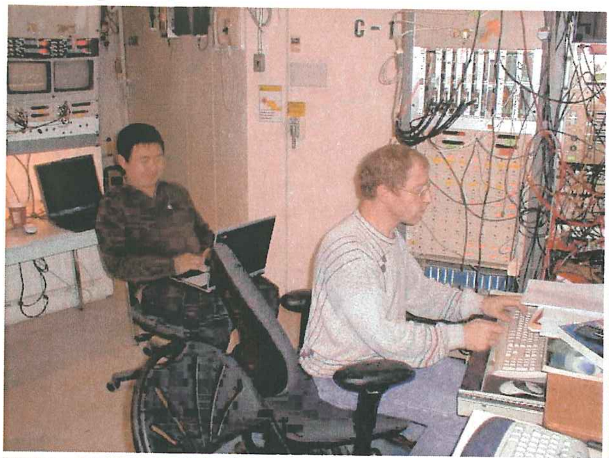
← divergence of beam at second monochromator crystal

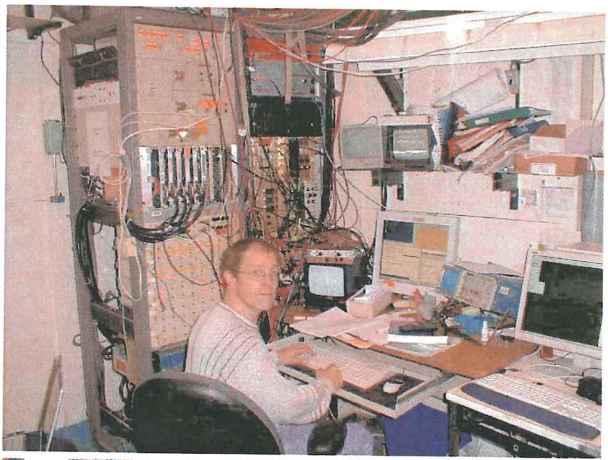
q_m = monochromator q value, eg. for Si (111)

q_d = diamond q value, eg. for diamond (220)

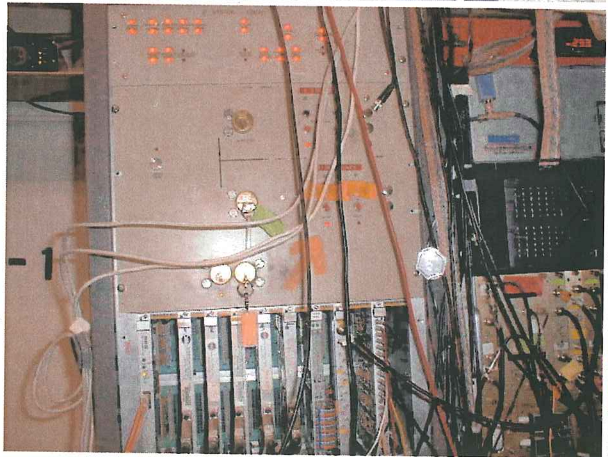
θ_m = Bragg angle setting of the monochromator.

This δ_{rc} must be combined with other (i.e. natural) contributions.

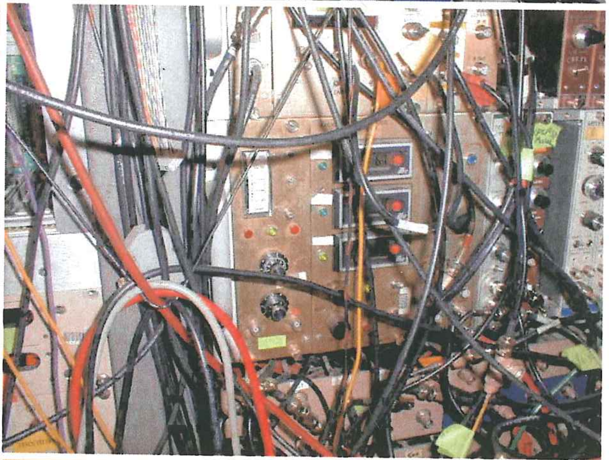




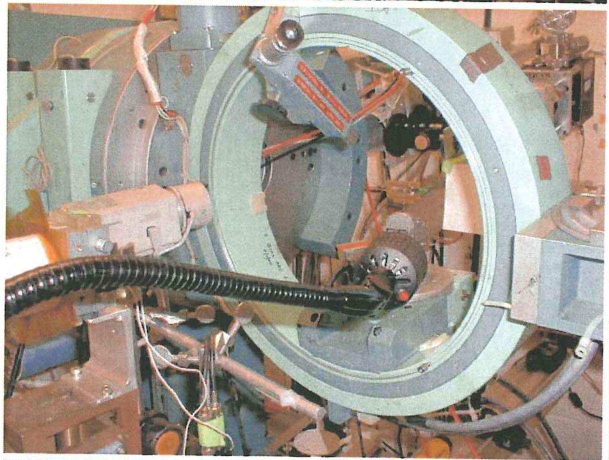
beam King at the controls



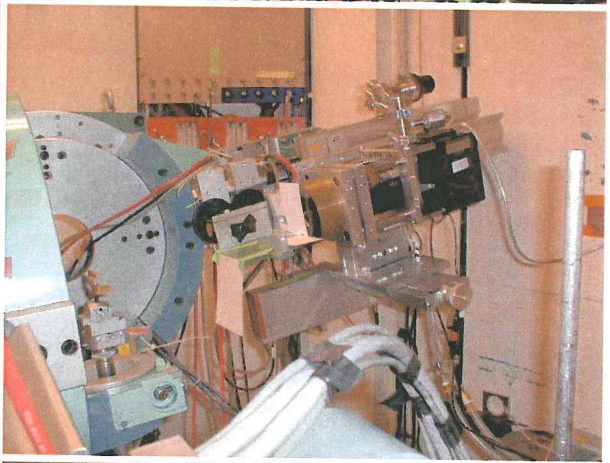
interlock system
left



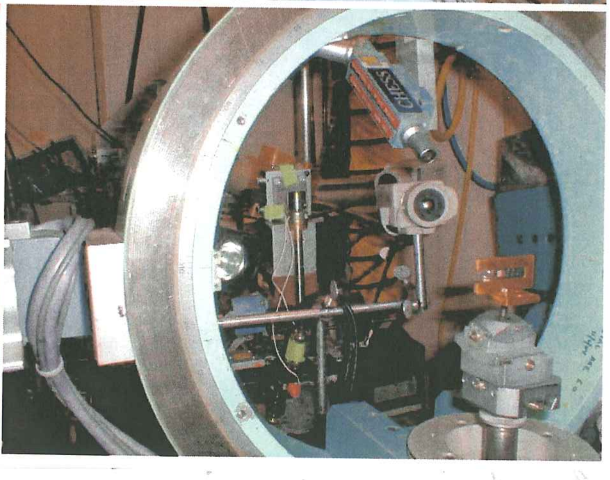
feed back monochromator control
right



goniometer looking downstream
left



CCD camera
right



target mounted inside goniometer
(2 views)

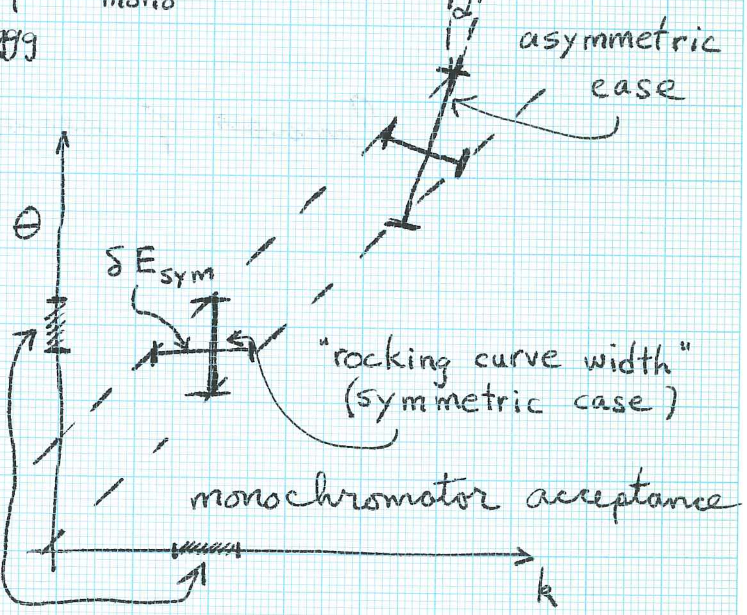
It turns out to be small (10μr) for Si(111) and diamond (220) and even smaller (3μr) if we switch over to Si (333). This is <u>not</u> the #1 reason for using 333, however. As seen on p. 107, the natural width of 333 for silicon is a factor 20 smaller than 111 ⟹ much better energy definition in the monochromator

Si (111) : δE = ~~51μr~~ 2.2eV

$\delta\theta_{rc}$ from this source = 51μr !

To find this, consider the rocking curve on p. 107.

$$\delta E_{mono} = \left|\left(\frac{dk}{d\theta}\right)_{Bragg}\right| \delta\theta_{mono}$$



As shown in the figure, the asymmetric crystal has a wider energy bite for a given source size. Therefore I use the second monochromator crystal (symmetric case) to define the energy resolution

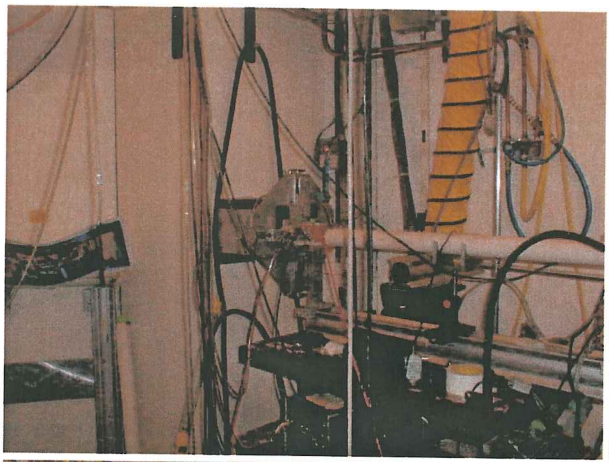
$$\frac{q}{2} = k \sin\theta_B$$

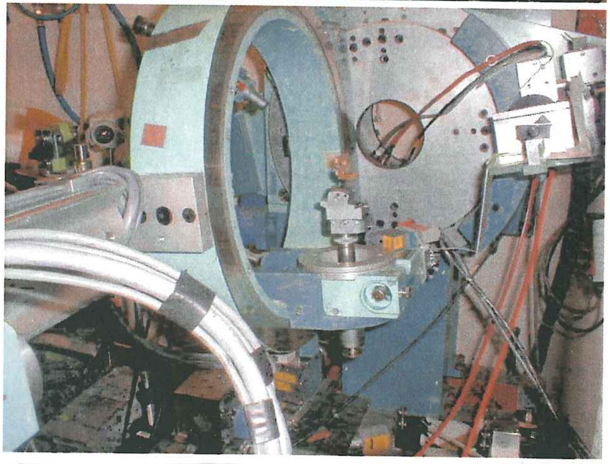
$$\sin\theta_B \, dk + \cos\theta_B \, k \, d\theta = 0$$

$$\frac{dk}{d\theta} = -k \cot\theta_B$$

The improvement at 333 is δE = 0.8 μr !

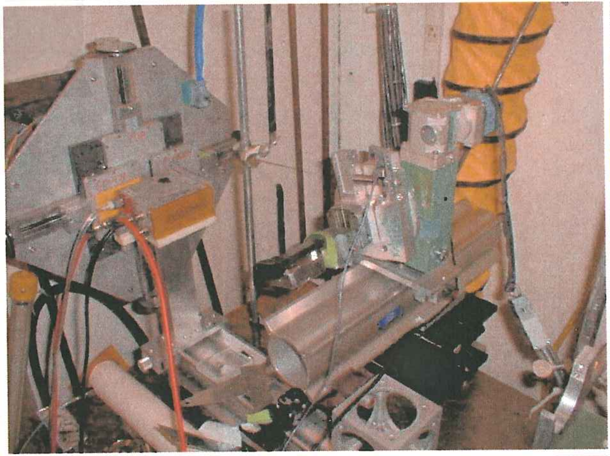
Part of the improvement comes from moving to smaller angles as a part of using 45 keV x-rays at 333.

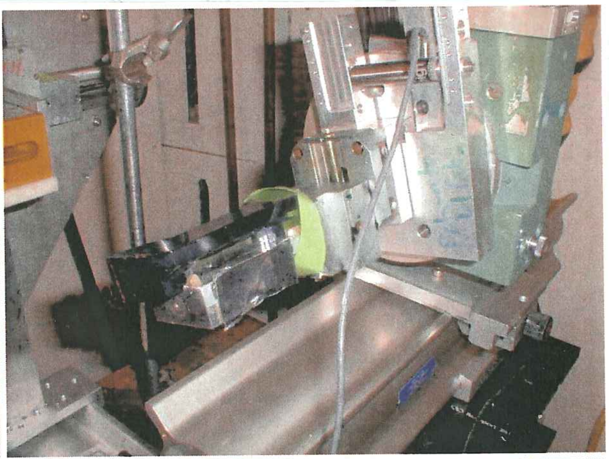


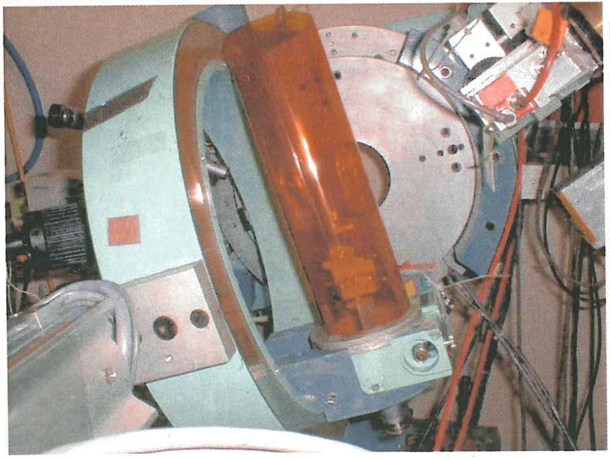


more views
from inside the
hutch

Images of second monochromator ↓





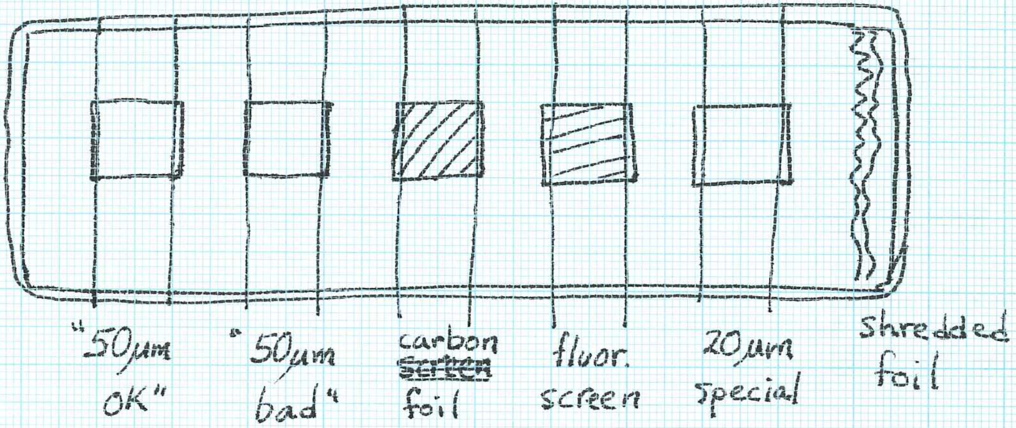


vibration-reducing "tent"

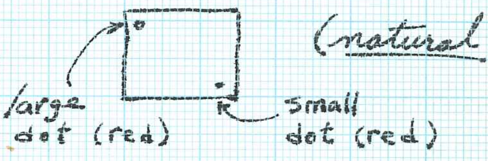
Crystals

Nov 5, 2006

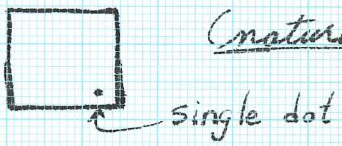
The diamonds for study are mounted in the g8 target ladder.



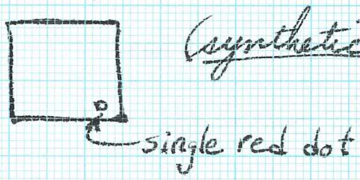
50µm OK : type IA diamond, 53 µm (natural)
ZA1440812 ref. 9823853



50µm bad : type IIA diamond, 40 µm (natural)
belongs to Glasgow, now Jlab

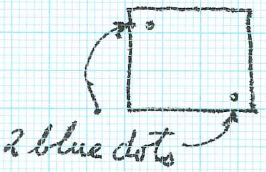


20µm special : type IB diamond, 18 µm (synthetic)
A001505 Glasgow, now Jlab

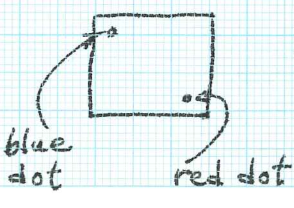


We also have 2 stand-alone diamonds in single holders that we have brought along for study.

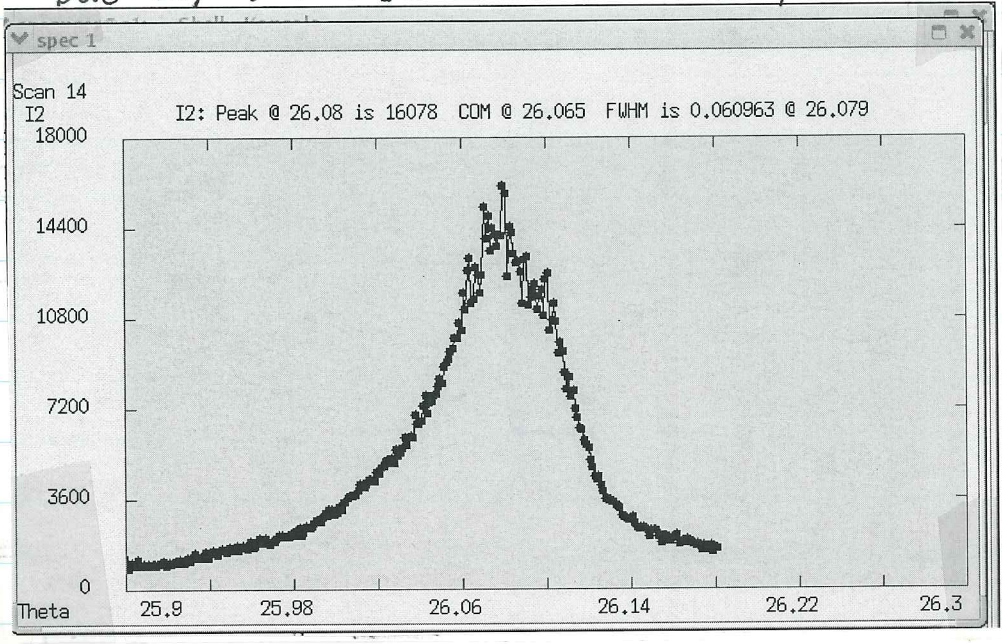
~~bad boy 50?~~ twin 50 : type IA diamond, 50µm (natural)
ZA1440812 ref. 9823853



~~split 50?~~ ~~("twin")~~ bad boy : type II A diamond, 50 µm (natural)
ZA2440810 ref. 9823853



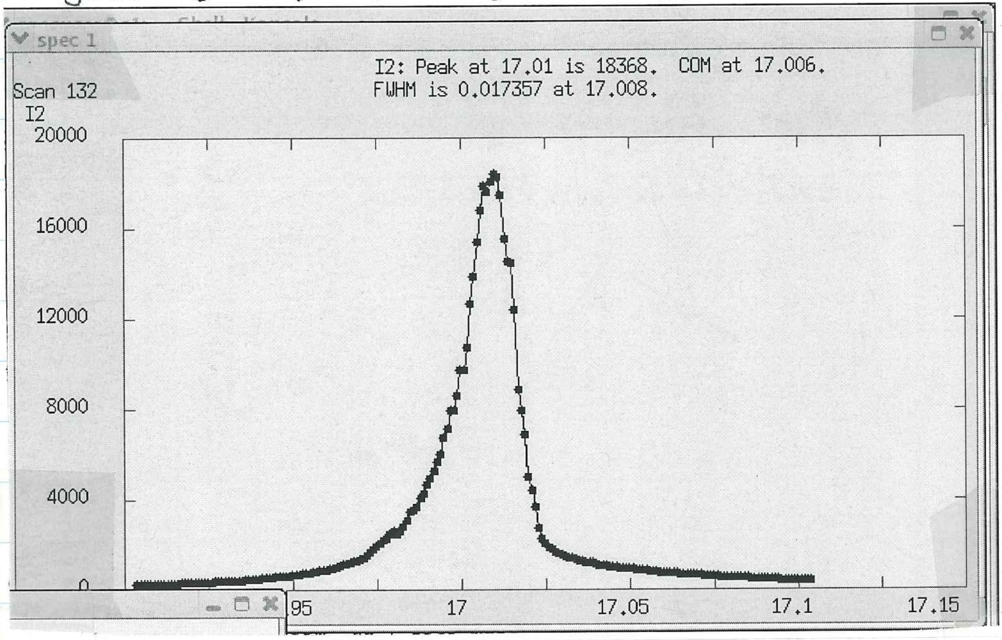
"bad boy" . [040] @ 15KeV   first peak found


spec 1

Scan 14

I2: Peak @ 26.08 is 16078 COM @ 26.065 FWHM is 0.060963 @ 26.079

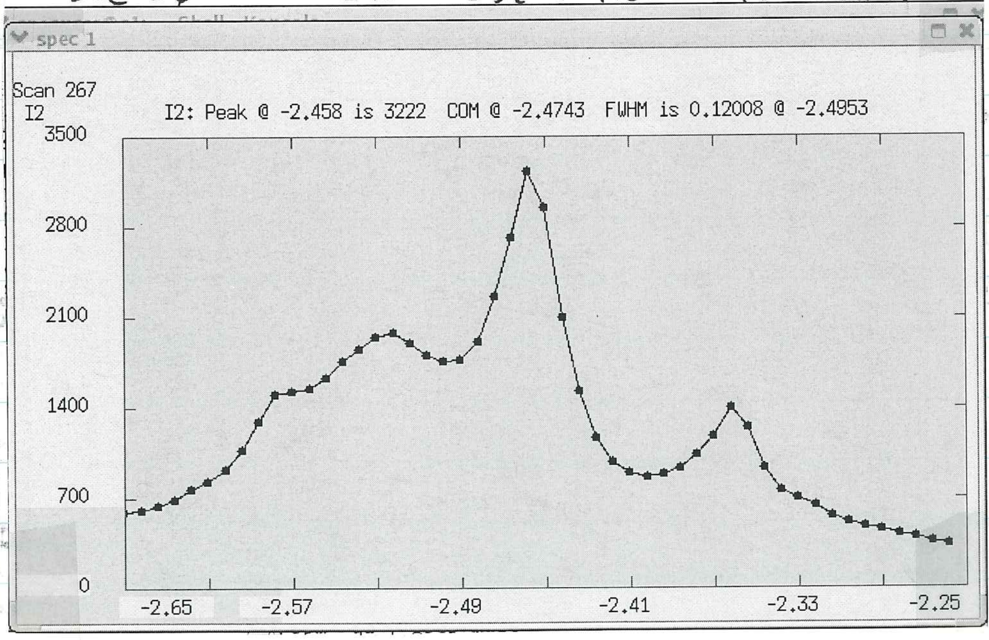
"good 50" , [2 2 0] @ 15 KeV


spec 1

Scan 132

I2: Peak at 17.01 is 18368. COM at 17.006.
FWHM is 0.017357 at 17.008.

"bad 50"   [$\bar{2}$ 2 0]@15KeV   (φ-scan)


spec 1

Scan 267

I2: Peak @ -2.458 is 3222 COM @ -2.4743 FWHM is 0.12008 @ -2.4953

<u>Goniometer</u> : Made by Huber (Germany)

4-circle : $\theta, \chi, \varphi, 2\theta$.

Most precise axis is $\theta/2\theta$: $\Delta\theta = 0.8\,\mu r$! using what they call "micropulse mode" of the stepping motor. The rocking angle for Bragg curves is θ.

<u>CCD camera</u> : Stella CAM (home-built) CCD with a $CdNO_3$ phosphor, I guess, optimized for ~12 keV. 1024 × 1024 pixels, 16 bits, variable shutter time.

<u>Instructions</u> : Program that controls data acquisition - all motors, camera, I0, 1, 2 ionization chambers, camera shutter, etc - is called SPEC. Motor control commands include

`> mv <motor name> <angle setting in degrees>` - move to pos.

`> mvr " "` - move relative to cur. pos.

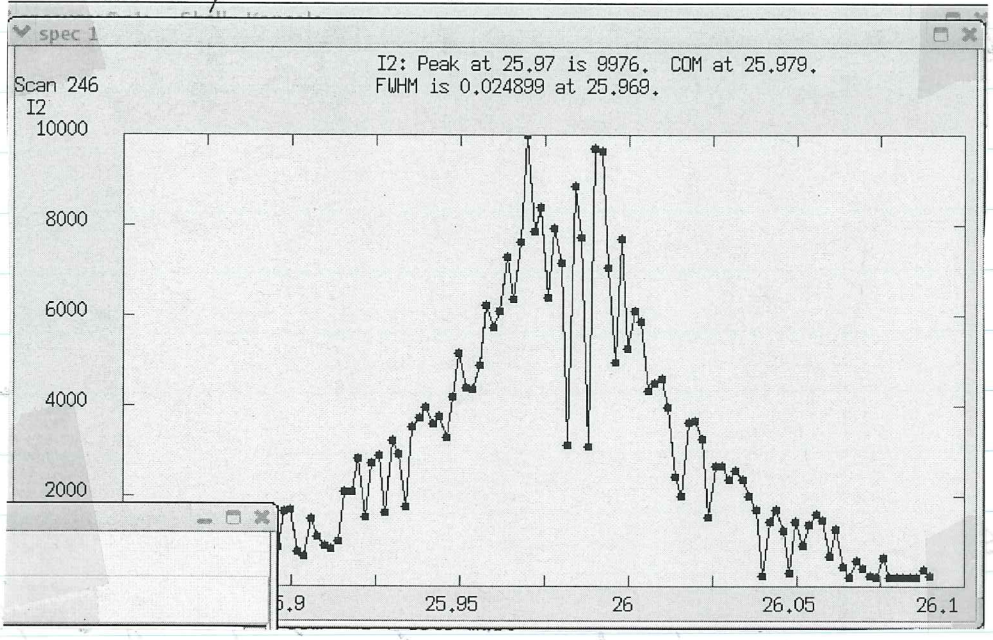
`> tw <motor name> <delta in degrees>` - interactive motion by steps of size delta, with user prompts on each step. One can also reverse direction during a "tweak" sequence, useful for peak finding.

To print out the current position of all motors, use

`> we`

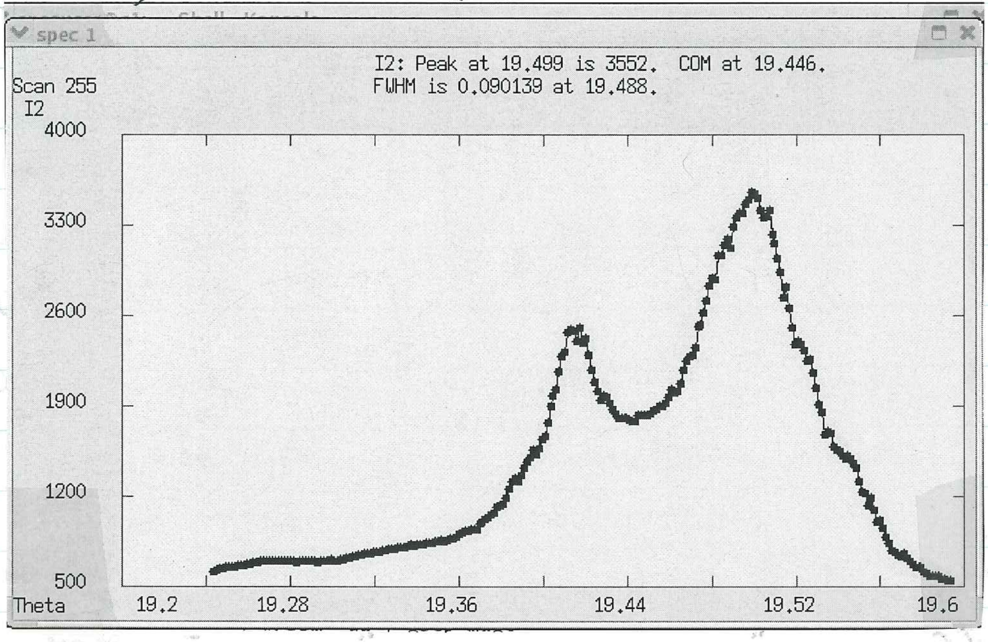
Motor names include th (θ), tth (2θ), chi (χ), phi (φ) and many many more that we were told to leave alone.

"bad bay"   [040] @ 15 keV



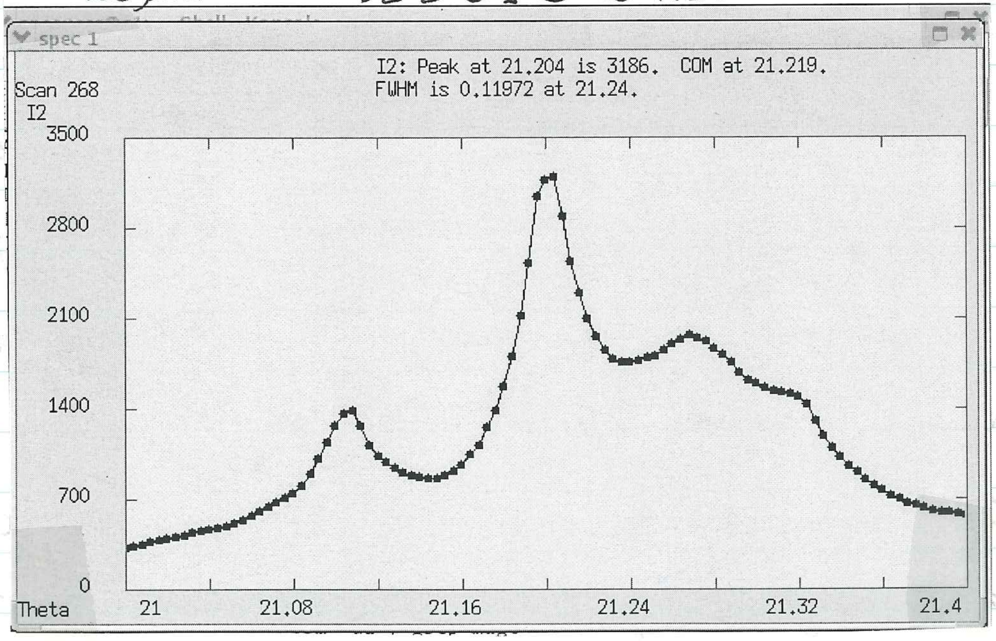
I2: Peak at 25.97 is 9976. COM at 25.979.
FWHM is 0.024899 at 25.969.

"20 µm"   [2 2 0] @ 15 KeV



I2: Peak at 19.499 is 3552. COM at 19.446.
FWHM is 0.090139 at 19.488.

"20 µm"   [$\bar{2}$ 2 0] @ 15 keV



I2: Peak at 21.204 is 3186. COM at 21.219.
FWHM is 0.11972 at 21.24.

For doing scans, the following commands are used.

>ccd_on - arms the camera for action

>ccd_off - do this before moving the CCD or changing beam conditions that might expose it.

> ~~starts~~ opens - force the shutter open (camera shutter) used during setup and alignment scans.

> ~~stops~~ closes - let the shutter close, get ready to take pictures

> dscan <motor name> <start angle> <end angle> <n points> <exposure> - do a scan, see below.

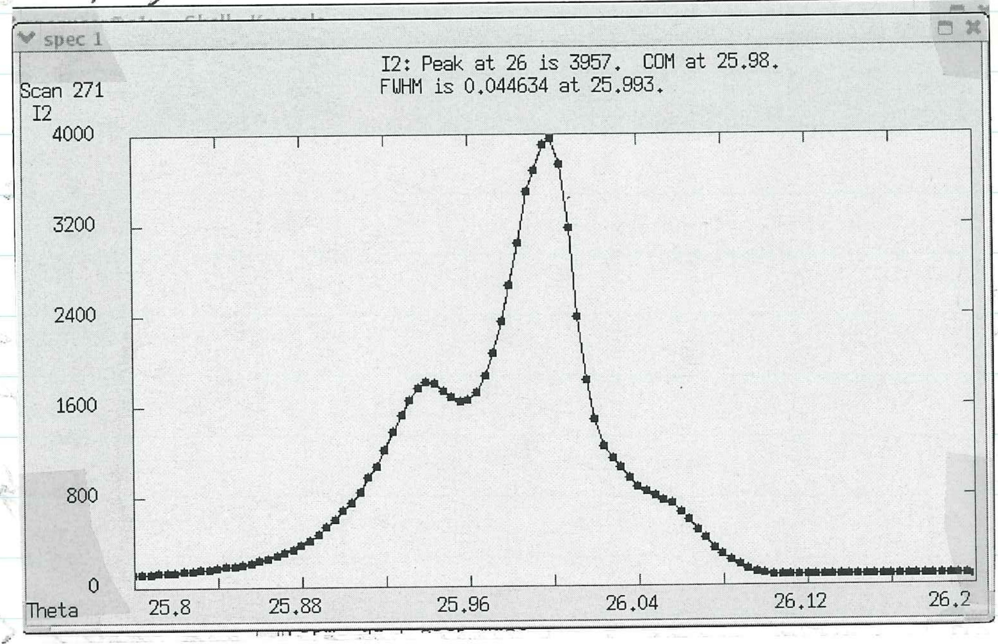
> ascan " " " "

> tseries <# snapshots> <exposure> - take CCD images, no motion

> ct <#> - chose counter channels for scan plot and exposure normalization ("monitor") If # < 0, chose monitor, otherwise chose detector. or leave blank for interactive choser.

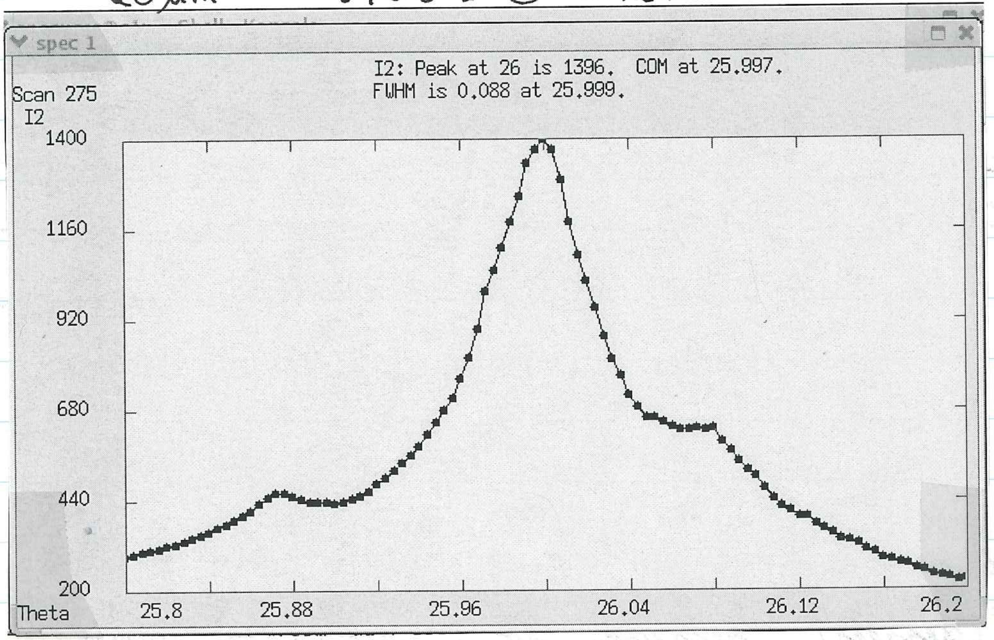
The scans are done in equal steps between the specified limits (degrees) relative to (dscan) or absolute (ascan) the current position. The exposure is either a duration in seconds (> 0) or a number of counts seen in the monitor detector (< 0). The detectors are always read out and the camera is also triggered on each step (slower) if ccd_on has been issued. Running with the camera requires the second computer be running the camserver program under the root account. Output files are stored on the dedicated ccd readout computer.

"20 μm" [0 4 0] @ 15 keV



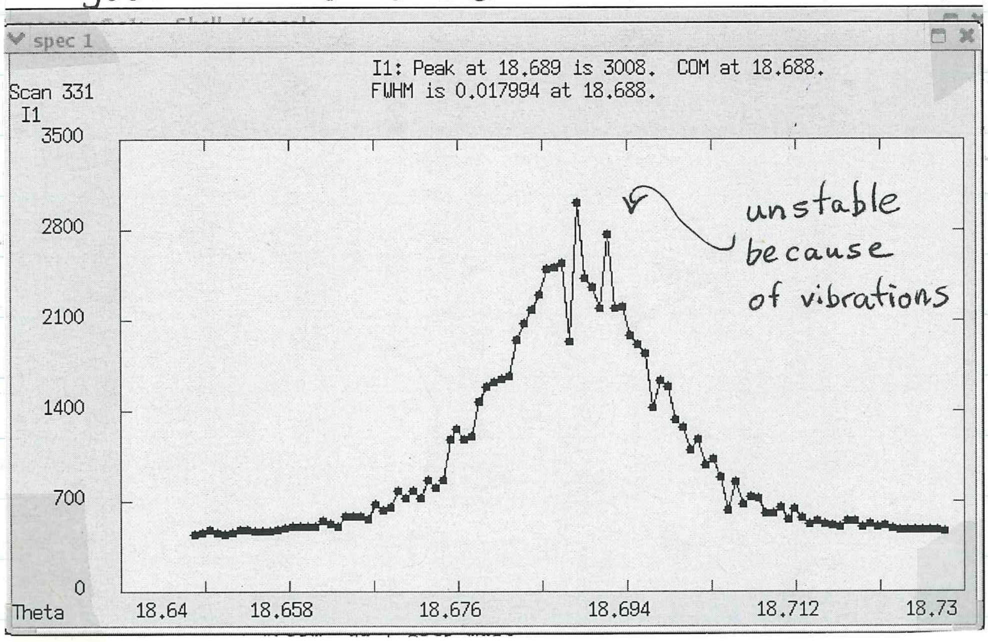
I2: Peak at 26 is 3957. COM at 25.98.
FWHM is 0.044634 at 25.993.

"20 μm" [4 0 0] @ 15 keV



I2: Peak at 26 is 1396. COM at 25.997.
FWHM is 0.088 at 25.999.

"good 50" [2 2 0] @ 15 keV, double mono, no "tent"



I1: Peak at 18.689 is 3008. COM at 18.688.
FWHM is 0.017994 at 18.688.

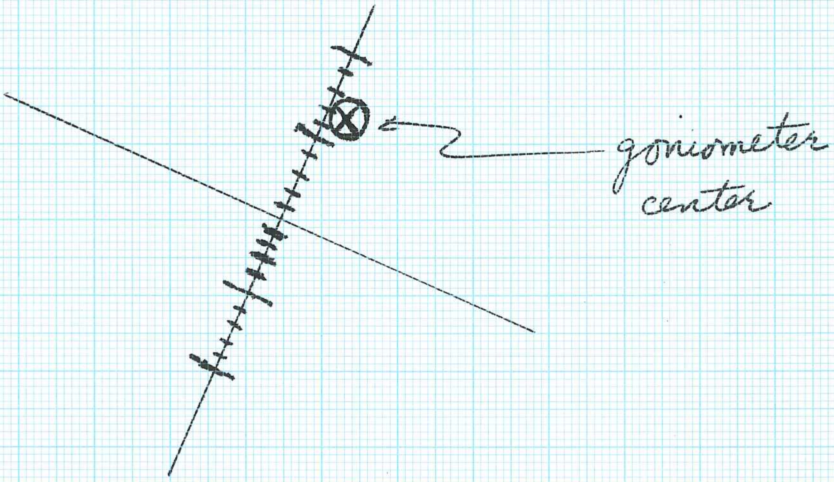
unstable because of vibrations

Crystal sample alignment

One aligns the sample at the goniometer center by the following procedure.

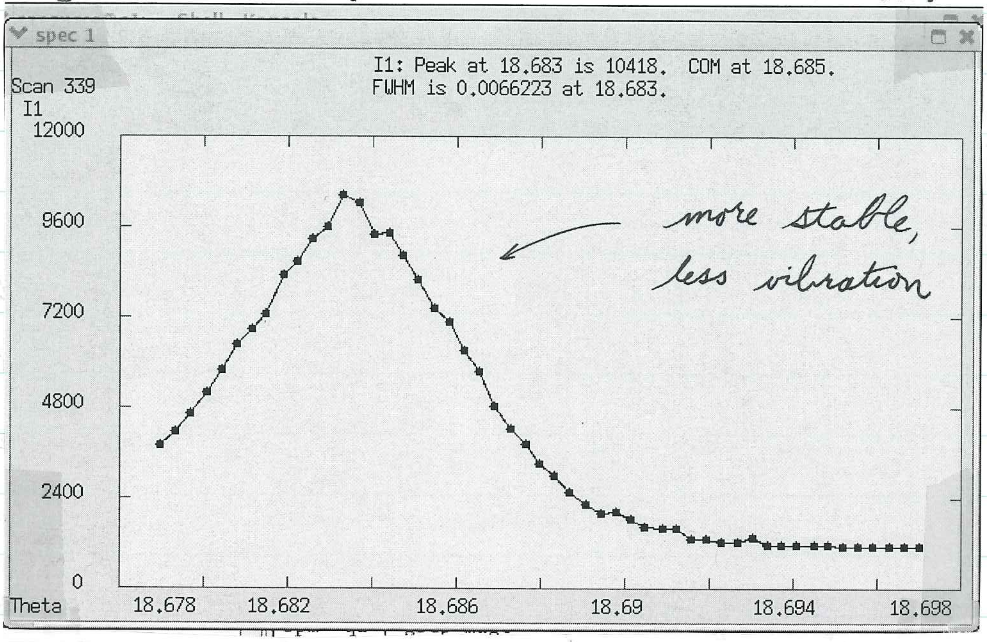
1) check that the φ axis post center axis passed through the center of the target. A translation stage on the sample holder gives manual controls to make it possible to do this without physically touching the sample in its holder. This part is performed visually; an accuracy of ± 1mm is generally sufficient

2) Look through the alignment telescope and see that the center of the target is at the point shown below on the telescope cross hairs.



3) Now rotate the sample in χ and adjust Z until the center of the target remains on the cross hair centering point indicated above. The Z control is below the χ carriage on the θ circle and is a knob that turns about the φ axis.

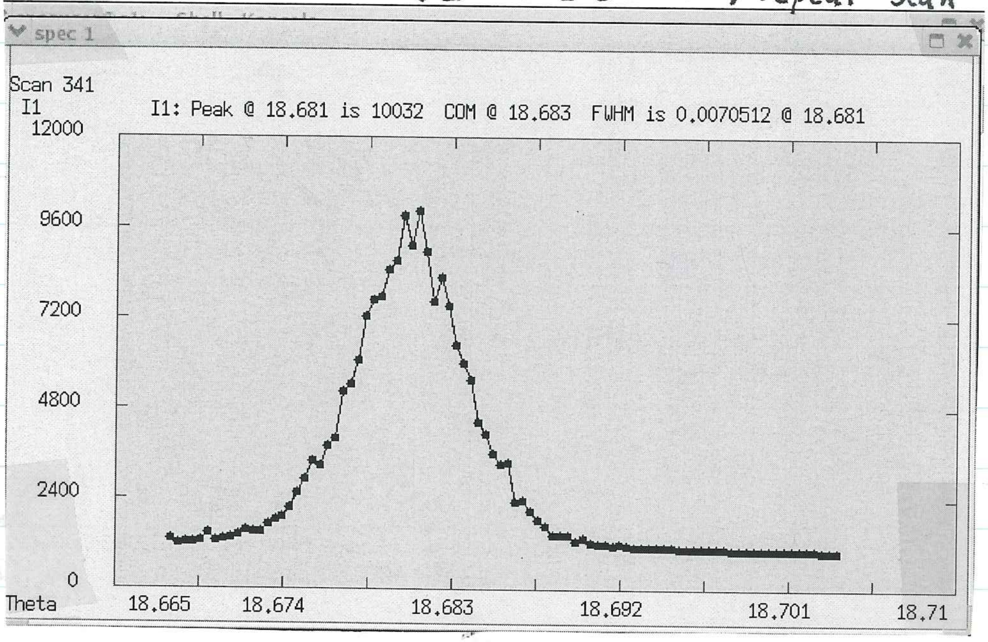
"good 50" [2 20] @ 50 keV with "tent"



```
I1: Peak at 18.683 is 10418.  COM at 18.685.
FWHM is 0.0066223 at 18.683.
```

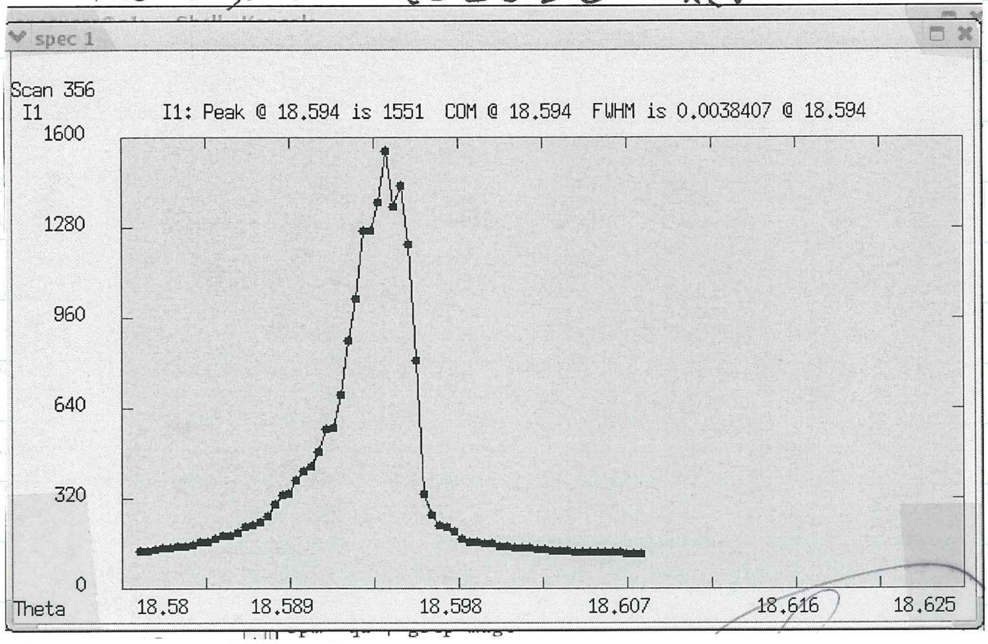
more stable, less vibration

"good 50" [$\bar{2}$ 2 0] @15 keV, repeat scan



```
I1: Peak @ 18.681 is 10032  COM @ 18.683  FWHM is 0.0070512 @ 18.681
```

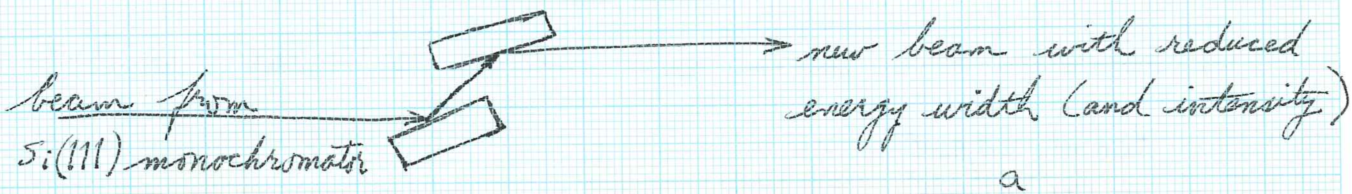
"bad 50 μm" [2 2 0] @ 15 keV



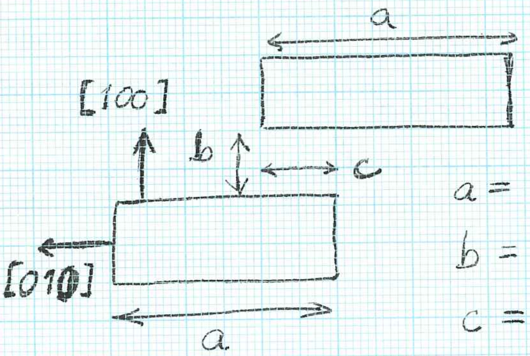
```
I1: Peak @ 18.594 is 1551  COM @ 18.594  FWHM is 0.0038407 @ 18.594
```

Second monochromator

With the first monochromator we have good angular divergence properties (10μr FWHW vertical) but not very good energy resolution (2.2eV FWHM @ 15 KeV). This results in a broadening of the rocking curve width for diamond [220] of about 50μr. We can reduce this by using a second double-crystal monochromator, as shown below.



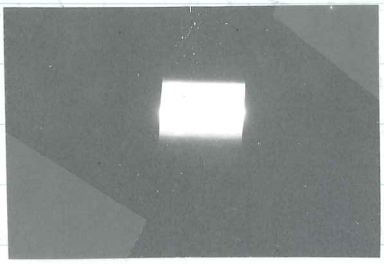
This is a so-called [400] channel-cut (all one Si crystal)



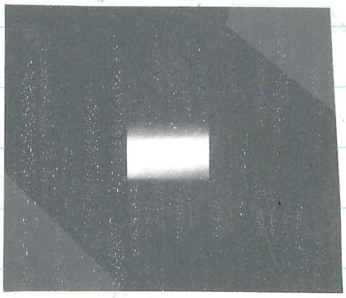
a = 17 mm
b = 5mm
c = 5 mm

At nominal alignment and $\theta_B = 17.722°$ we found 3 q-values contributing at once: [400], ~~[illegible]~~, ~~[illegible]~~ [202], [20-2]. By rotating the crystal about [100] by a few degrees we were able to isolate a clean beam of only [202] reflections. Scans after #331 (marked "double mono") use this improved beam. The energy is 15 keV and energy width <u>0.8eV</u>, about a factor 3 improvement over previous running with only a single monochromator. The way this results in extra diamond rocking curve width is angle-dependent and happens to cancel completely in the limit where the two crystal $\vec{q}$ vectors are parallel. For Si 220 and diamond 220 this dispersive contribution to $\underline{\Delta\theta_c = 6\mu r}$!

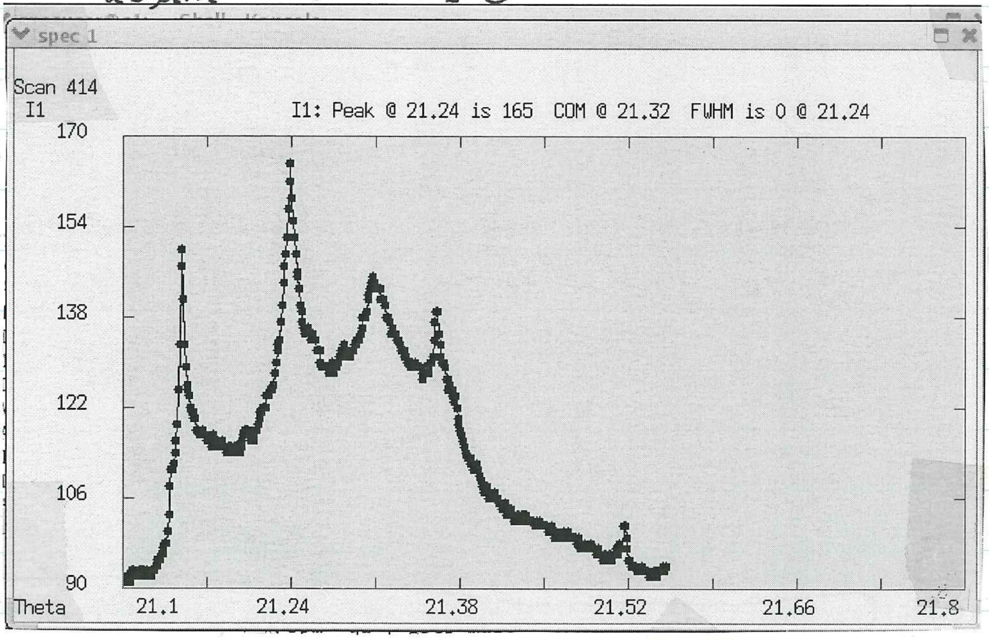
Beam spot
without
second
monochromator →



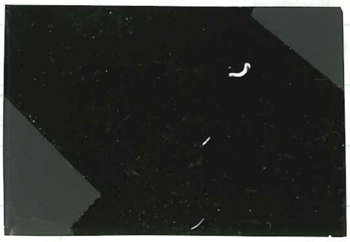
Beam spot
with
second
monochromator →



"20μm" [-2 2 0] @ 15KeV



```
Scan 414
I1: Peak @ 21.24 is 165   COM @ 21.32   FWHM is 0 @ 21.24
```



← from scan 414
@ θ = 21.16°

For [400] diamond this is somewhat worse at 9 μr but still it is an improvement of a factor 6 over what we had with only the Si (111) monochromator.

We also tried the option of using Si (333) radiation from the first (asymmetric) monochromator at 45 keV but the beam was very low-flux and the camera was not very sensitive - very noisy. We decided to stay at 15 keV and use the 2-monochromator scheme described above. One scan was recorded at 45 keV however.

username: specuser
Password: CThrooMe

CHESS Access & Phone Numbers

After hour building access: From the "W" parking lot, access is located at the bottom of the loading ramp the left side door. **Keypad Code: 34565**

CHESS Main Phone: **(607) 255-7163**
CHESS Fax #: (607) 255-9001

CHESS: www.chess.cornell.edu
MacCHESS: www.macchess.cornell.edu

Station Phone Numbers & Staff Scientists (All phones in 607 area code):

A1	**255-0149**	See MacCHESS Listing
A2	**255-0197**	Alex Kazimirov, 255-2538
B1	**255-0243**	Zhongwu Wang, 255-3551
B2	**255-0247**	Zhongwu Wang, 255-3551
C	**255-0256**	Ken Finkelstein, 255-0914
D	**255-0281**	Detlef Smilgies, 255-0917
F1	**255-0584**	See MacCHESS Listing
F2	**255-0614**	See MacCHESS Listing
F3	**255-0631**	Darren Dale, 255-3819
G	**254-5499**	Arthur Woll, 255-3617