Optimizing Monocapillary Optics for Synchrotron X-ray Diffraction, Fluorescence Imaging, and Spectroscopy

CHESS

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1. **Introduction to monocapillary optics**
2. **Capillary design principles**
3. **State of the art of capillary drawing**
4. **CHESS applications experience**
   - **a) high pressure diffraction and EXAFS**
   - **b) microbeam protein crystallography**
   - **c) fluorescent imaging of fish ear stones**
   - **d) confocal x-ray microscopy of historic paintings**
   - **e) Micro/Optoelectronics applications**
5. **Hasylab applications experience on Beamline L**
6. **Simulation of gain and beamsize**
7. **Future capillary developments & conclusions**
8. **Brief overview of ERL and prospects for making 1 nm diameter x-ray beam at 10 keV**

Introduction to Monocapillary Optics

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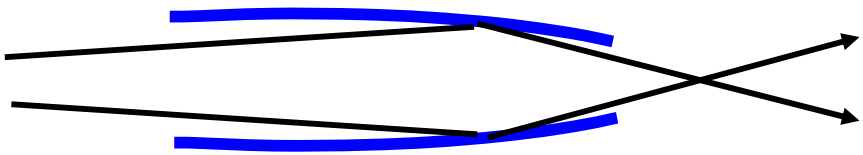
Why do we need a capillary ?

To make small beams and to increase the beam intensity (flux/μm^2)

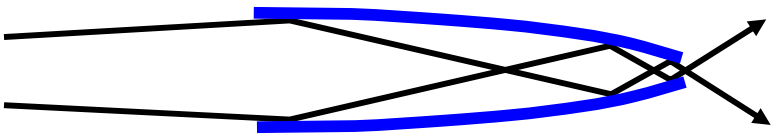
What kind of capillaries we are talking about ?



One-Bounce Glass Capillary

Large working distance (cm scale); Good
•Near 100% transmission; Good
•Divergence controlled by making ideal mathematical shape; Good
•More difficult to make small focal spot, (such as 1 μm); Challenge!
•Short and compact - may fit in spot that is too short for KB mirror assembly! Good

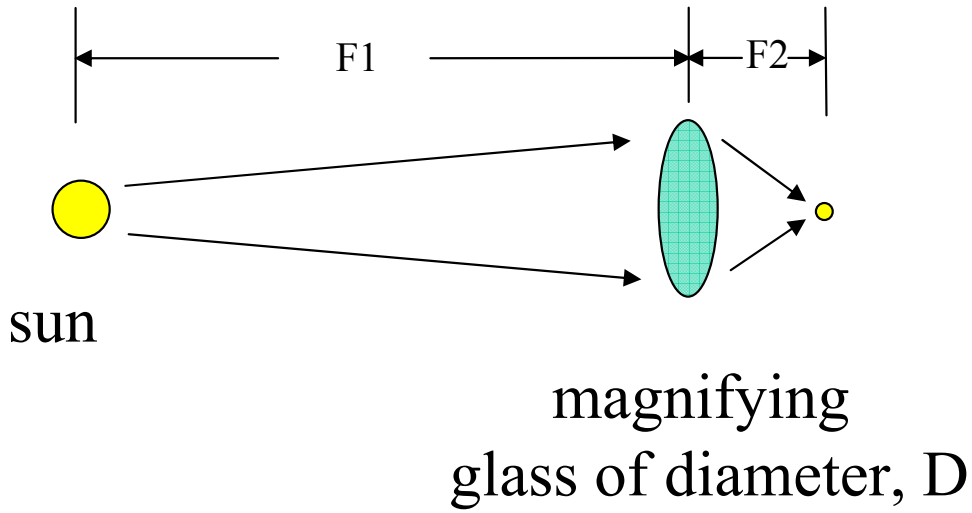
•First really useful elliptical x-ray optic - Balaic D., Nugent, Barnea, Garrett, Wilkins, J. Synch. Rad. 1995; 2: 296.



Multi-Bounce Condensing Capillary

•Easier to make with small openings of < 1 μm; Good
•Short working length of 100 μm; Not enough for many applications

What can optics do?



optics exchange divergence for size

lens transmission is around 100%

Magnification=F2/F1; Divergence ~ D/F2 ~ 0.5° (9 mr)

Elliptical Focusing One-bounce Geometry

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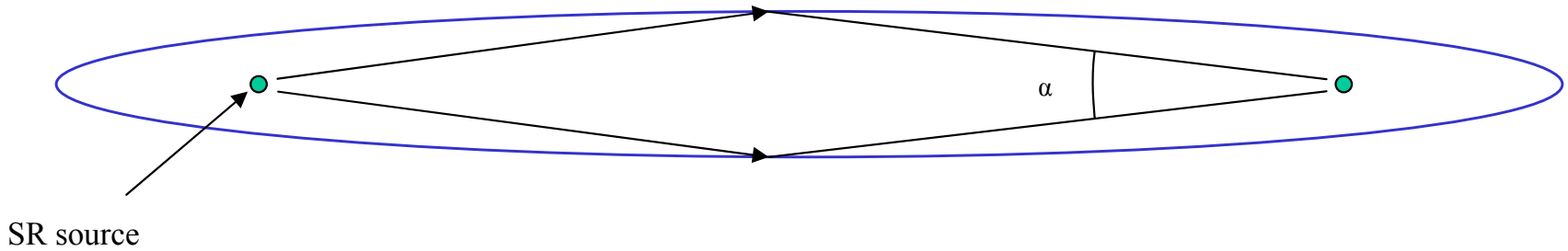
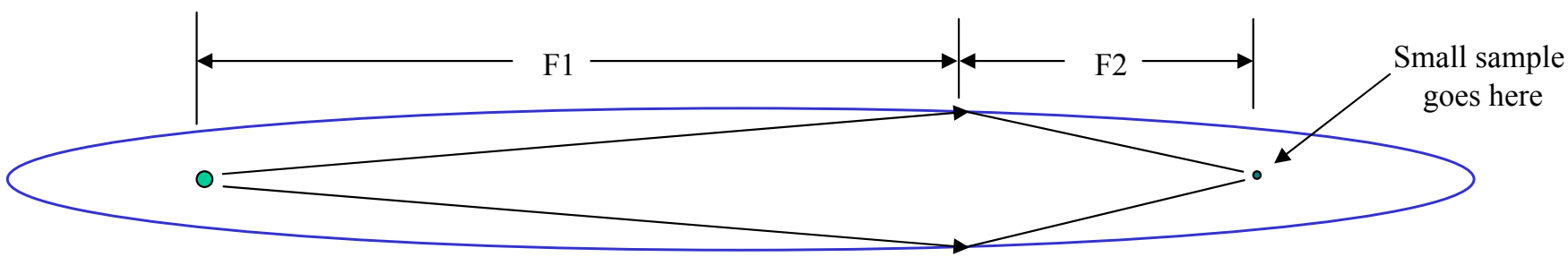
Principle: Demagnify source to create microbeam



1:1 focusing example, source size & divergence unchanged

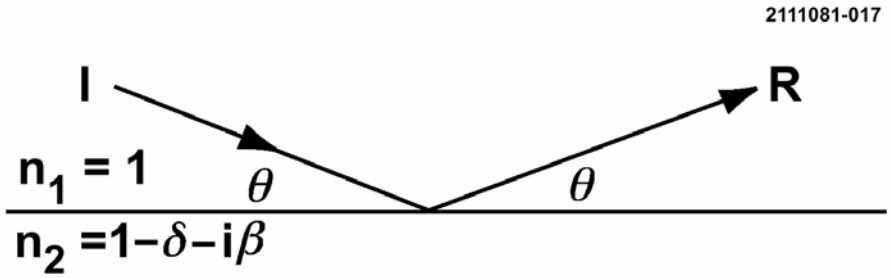


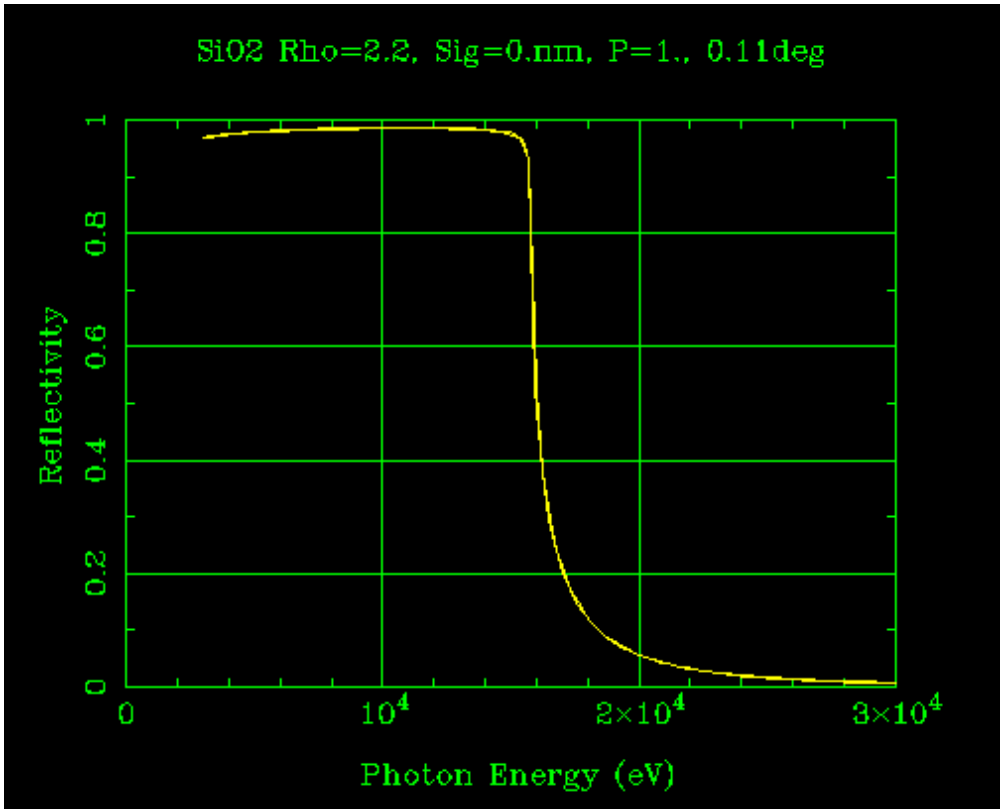
M=F2/F1

1:2 focusing example, 1/2 source size & 2x divergence

Disclaimers: concept only presented. Real demagnification factors can be 1/100 to 1/1000 in practice, other optics maybe employed to make the beam monochromatic, etc. A 'pancake' shaped source maps into a round image point.

Principle of total reflection of x-rays





For Glass:

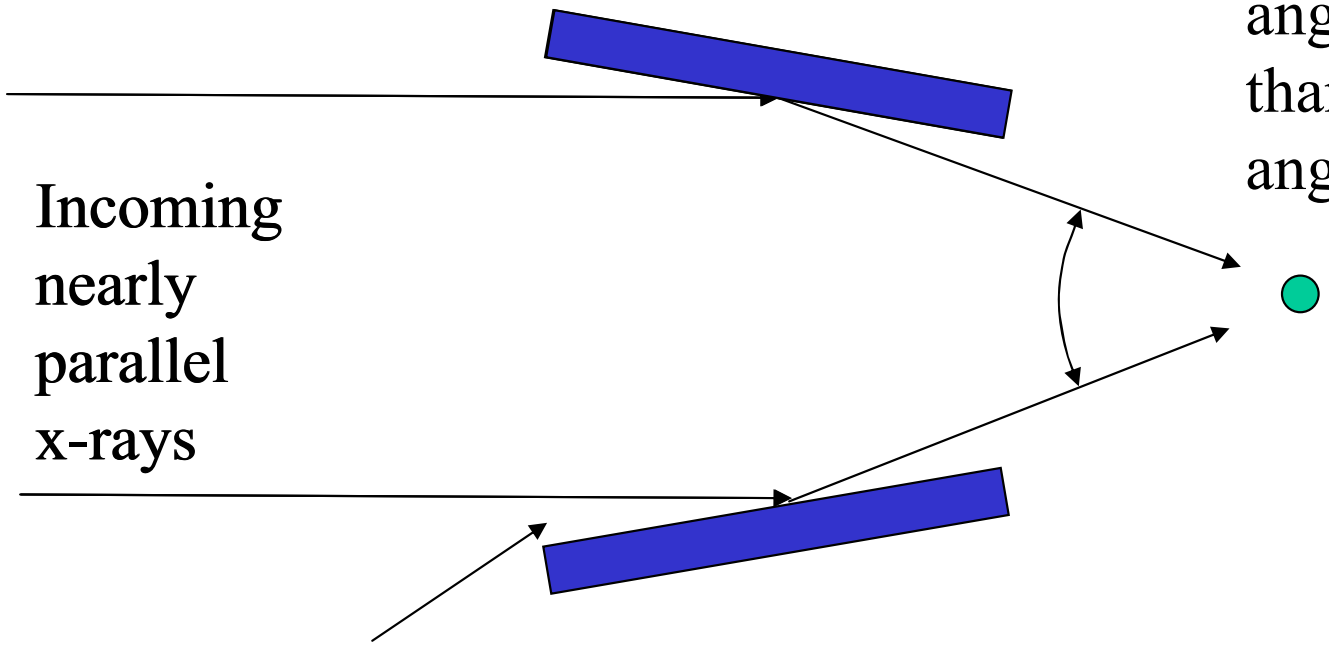
θ_c=32 keV * milliradians

i.e. 2 mr @ 16 keV,
1 mr @ 32 keV
0.5 mr @ 64 keV

Reflectivity as calculated for theta=.11 degrees (2 mr) at http://www-cxro.lbl.gov/optical_constants/mirror2.html

Divergence & Critical Angle

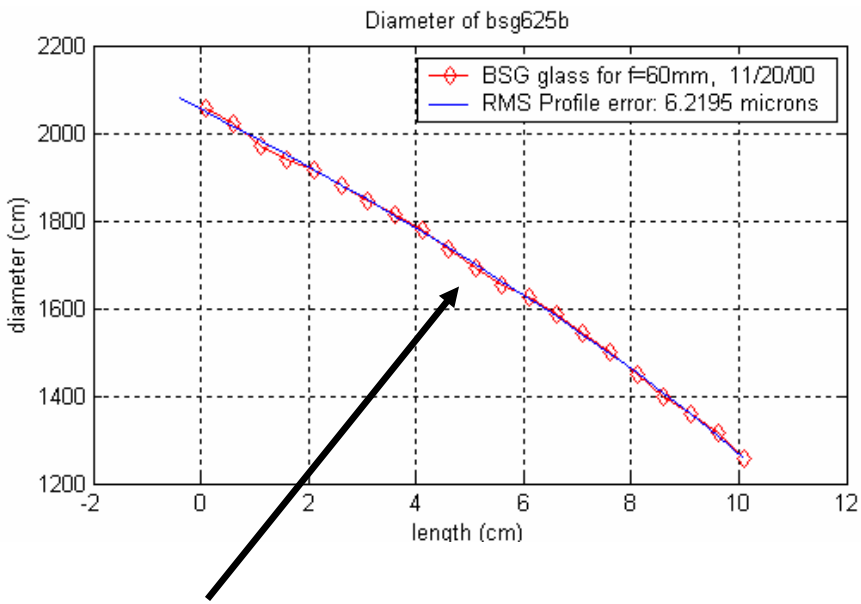
Thus the convergence angle should be less than 4 times the critical angle!

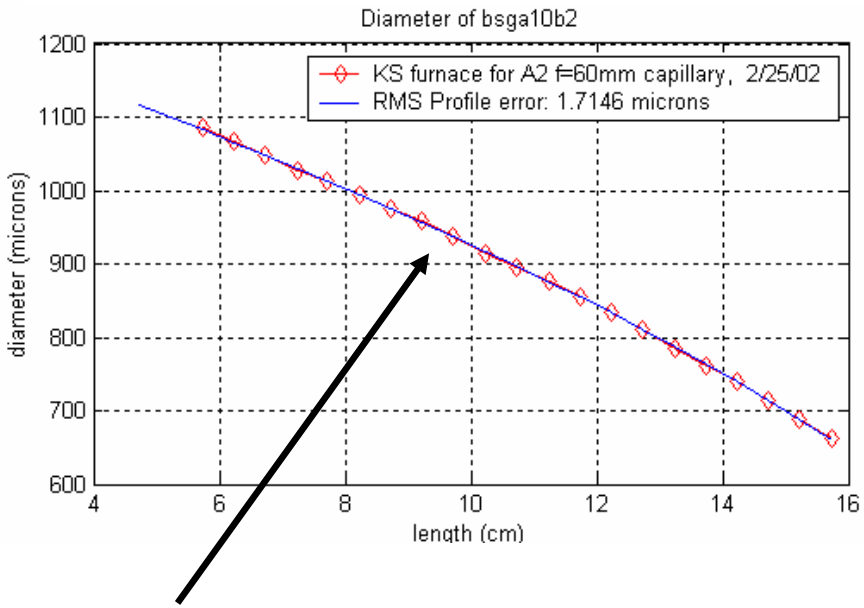


Stay below critical angle on local capillary surface

High-pressure example:
2 millirad A10 capillary will have a 0.5 milliradian critical angle and work to 64 keV!

How good are the capillaries now?





Several years ago:
figure errors of ~ 6.2 microns
slope errors of ~140 microradians

Currently:
figure errors of ~1.7 microns
slope errors of ~ 70 microradians

Present Capillary Drawing Technology

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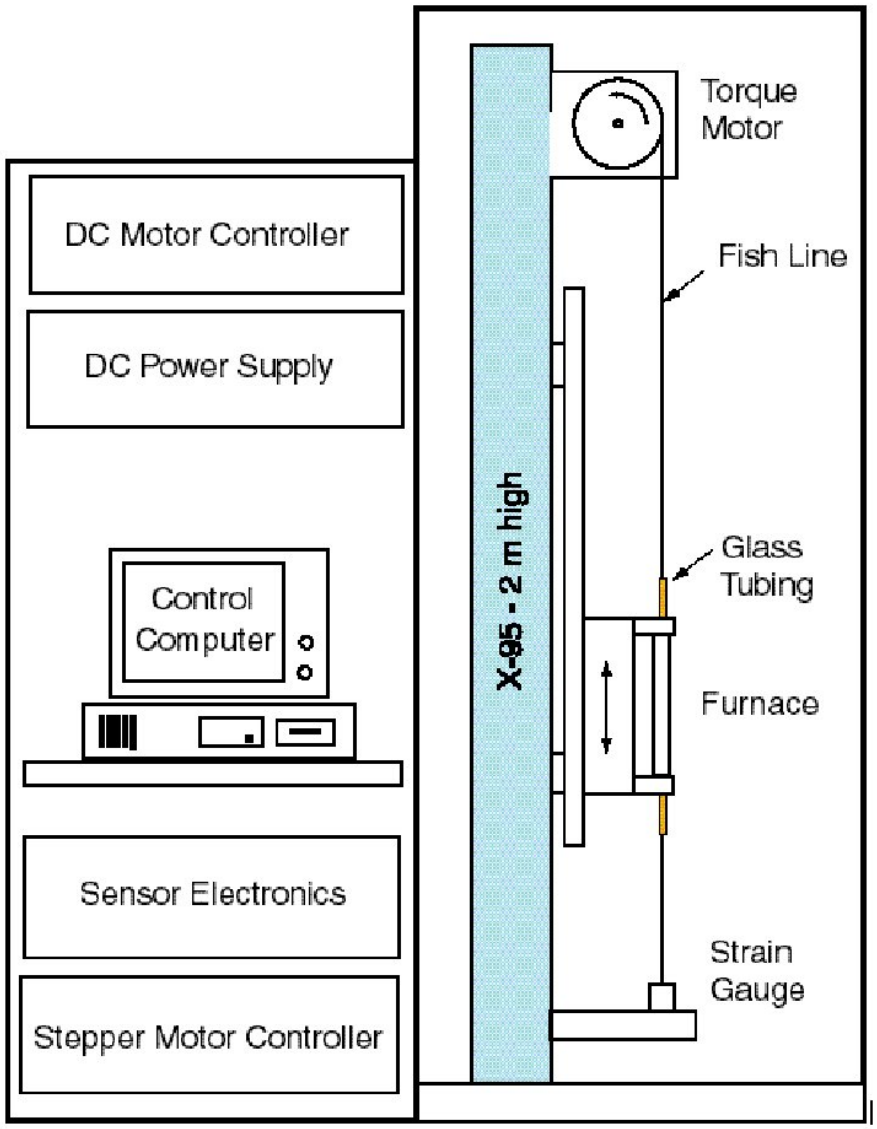

- Glass capillaries of precise shape can be drawn using the DB2 programmable glass puller.
- A lookup table for the furnace position vs amount of glass extension is generated from a conservation of mass equation.
- The LabWindows program then coordinates the movement of the furnace with the extension of the capillary in order to achieve the desired shape with micron accuracy.
- 60 rpm rotation of the glass during drawing is essential to obtaining straight parts.
- Development team over the years: Don Bilderback, Ernie Fontes,Riley Freeland, Steve Hoffman, Dan Thiel, Reinhardt Pahl, Vic Pollock, Rong Huang. Newest team members: Tom Szebenyi, Sterling Cornaby, Aaron Mauer

Brief list of current capillaries

Capillary name	*Type*	*Length (cm)*	*Base/Tip IDs (μm)*	*F (mm)*	*Size (μm)*	*G_{max}*	*Div (mr)*
CH015	concentrating	22.3	25 / 0.8	<0.1	0.8	100	5.8
BSG2, BSG3	focusing	30.0	400/130	30	18	125	<4.0
BSG625 BSG631	focusing	10.5	407/233	55	21	80	<4.0
BSGF413	focusing	10.5	292/162	40	20	10	<4.0
BSG7 BSG301	focusing	5.0	198/125	30	12	75	<4.0
W102	focusing	28	800/300	25	20	150	<12.0
A10	focusing	10.5	211/123	55	14-18	70	2.0
Peb605	focusing	11.5	827/469	55	17-23	455	8.0
SF202	focusing	50	81/44	22	16-18	11	2.0

F(mm) is the working distance: distance from capillary tip to focal point.
Size is the measured FWHM convoluted with 10μm pinhole, except for CH015
G_{max} = (flux through 10 micron pinhole w/capillary)/(flux through 10 micron pinhole, no capillary)
Div(mr) is the designed full divergence confirmed by x-ray tests

High-Pressure Applications to DAC

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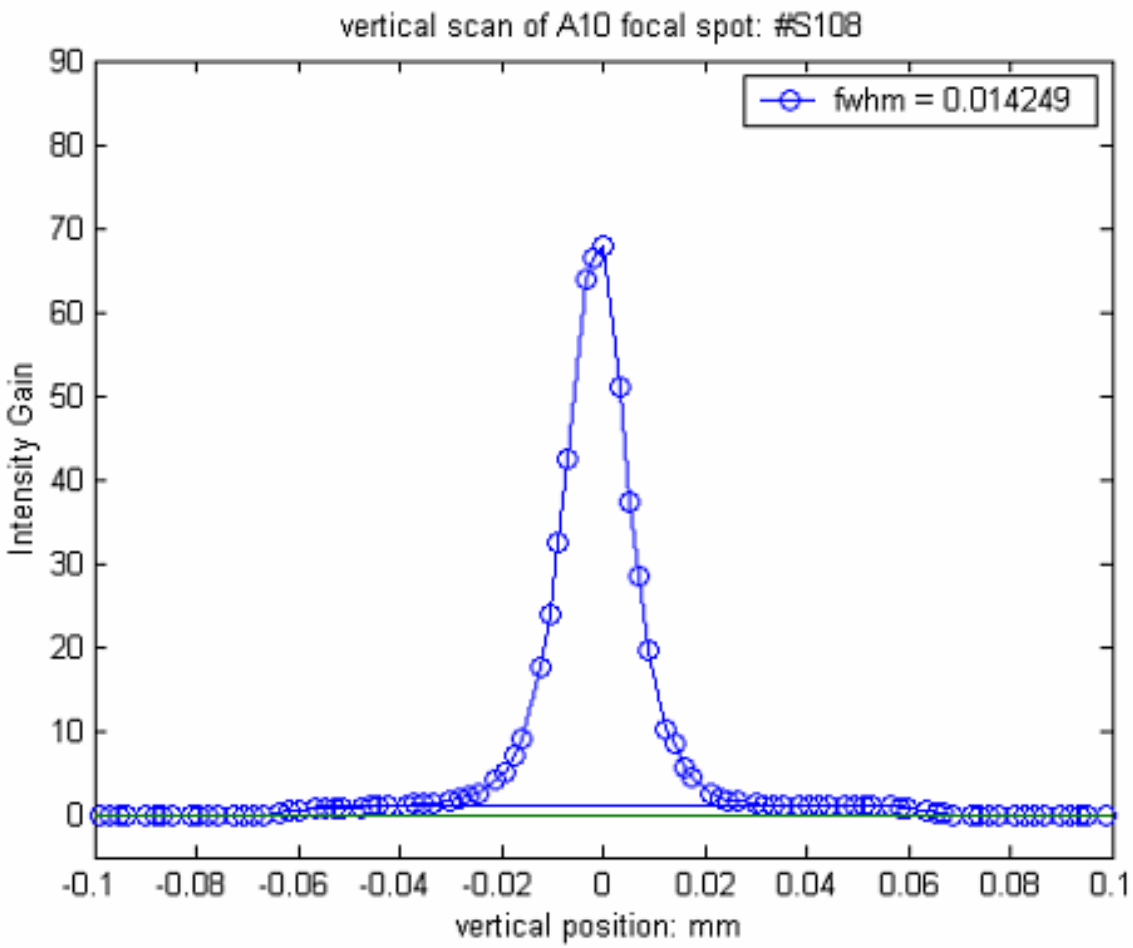
Capillary A10 for HP diffraction for A2 wiggler station (under development)



Capillary A10 pulled by Rong Huang on DB2 puller
Inner diameter of base/tip(μm): 211/123
Capillary length: 105 mm
Shape: Ellipsoidal

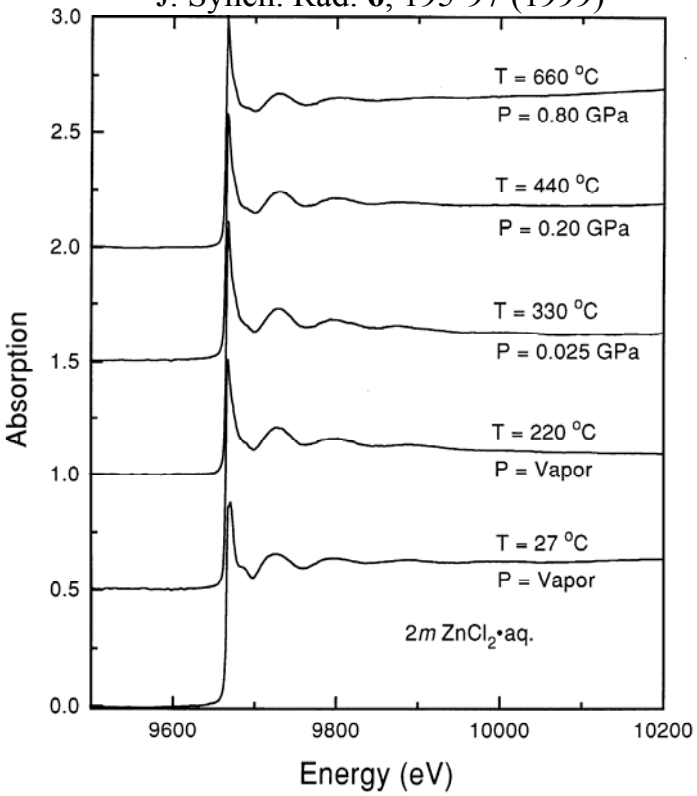
Maximum gain of flux: ~70
Measured at x-ray energy of: 15keV
Maximum image divergence: 2.0 mrad
X-ray spot size: 14 μm (V) by 18 μm (H), fwhm convoluted by 10 μm pinhole)
Working distance beyond tip: 55 mm

Material: borosilicate glass from Pegasus Glassworks
Measured optical figure error (rms): 1.7 μm
Measured slope errors (with A2 x-rays): 70 μrad
Highest energy to focus: 64 keV

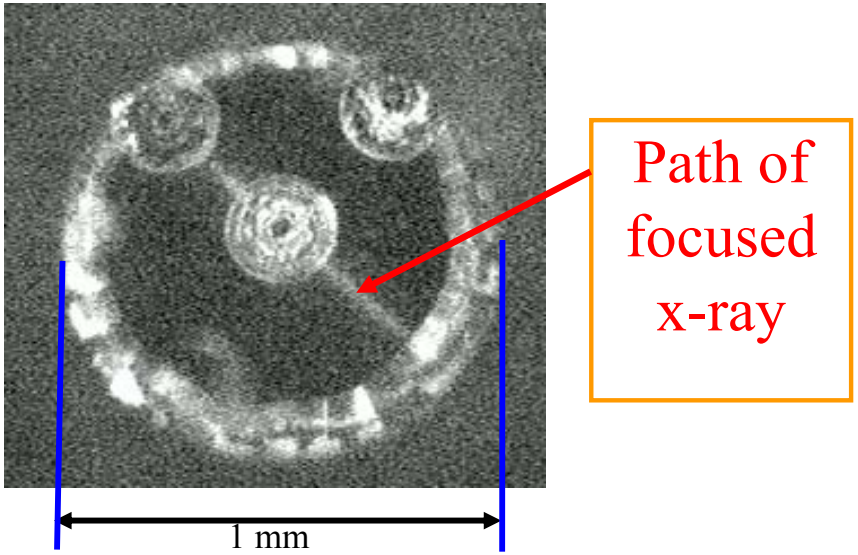
No direct beam block present for profile at left

Exafs & Diffraction at CHESS
B2 with capillary

Mayanovic, R.A.et al.
J. Synch. Rad. **6**, 195-97 (1999)



Bassett, W., et al., Zeitschrift fur Kristallographie **215**, 711-717 (2000)



Focused x-ray path inside a diamond anvil cell at B2 station for XAFS experiment (BSG625 capillary Length of 10.5 cm) with drilled entrance and exit pockets to minimize absorption to and from the sample area.

High-pressure diffraction for Exafs and angle-dispersive work at B2

a) capillary gains ~ 20 over no capillary (source size limited).

b) There is not room along the existing beamline for a KB mirror set

Reducing divergence by 1/4 to 1/2 with slit, the trade-off of divergence vs. resolution

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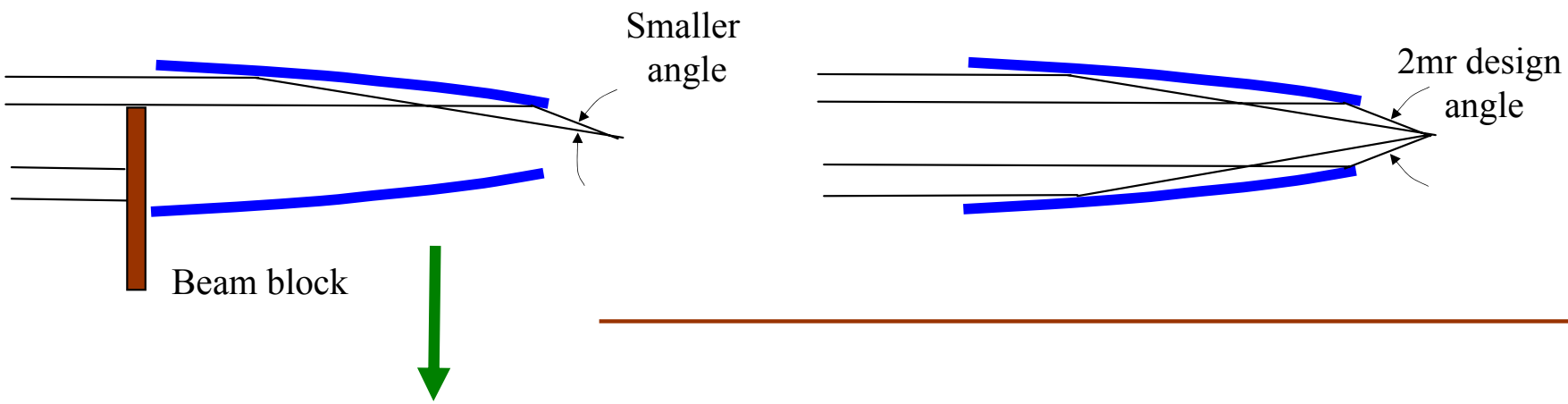
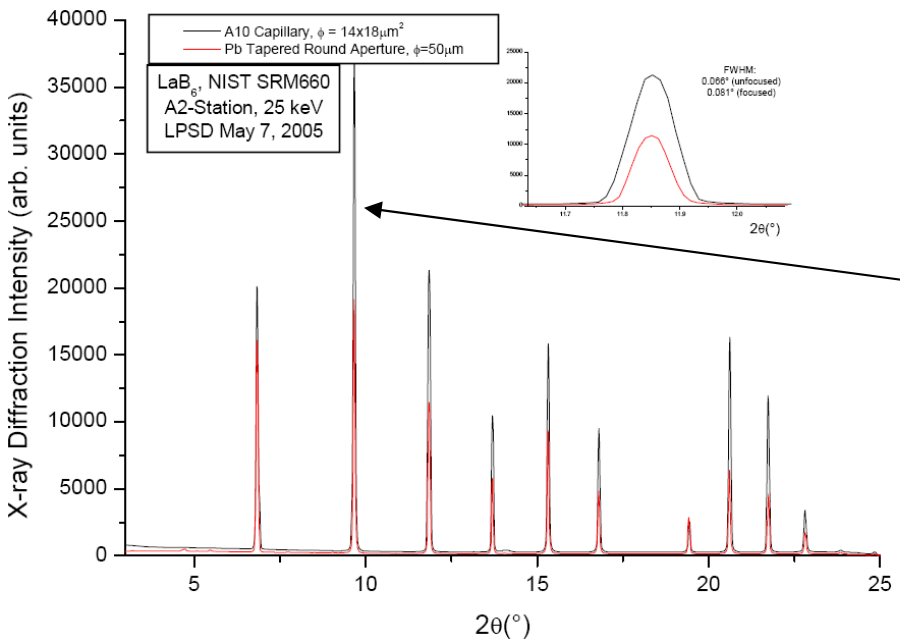
J. Smith & S. Desgreniers, U. of Ottawa; S. Cornaby, A. Kazimirov & D. Bilderback, CHESS; R. Huang, U. Chicago





A10 capillary performance at CHESS A2 at 25 keV with standard powder sample and MAR345 detector

- first quantified experiment with partial capillary beam
- twice the integrated flux of the 50 micron pinhole data in a 14 x 18 micron focused beam spot
- fwhm of diffraction lines increase 10 to 20%
- calculated divergence of 0.5 to 1.2 mr from powder data
- a more optimum capillary would have 35 micron beamsize

Macromolecular Structures

[MacCHESS(Gillilan & others), Bilderback, & Thorne]

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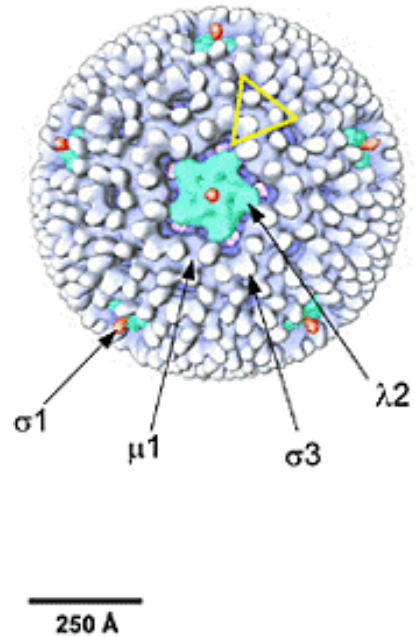
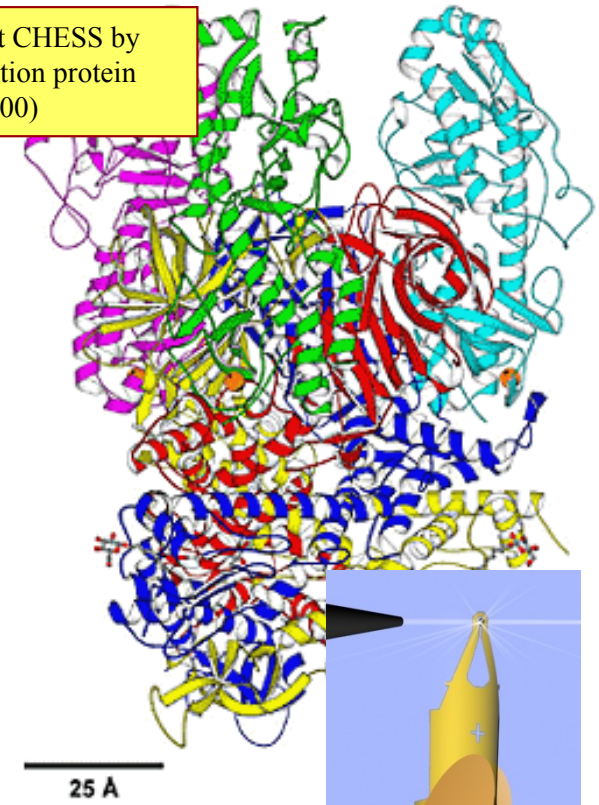
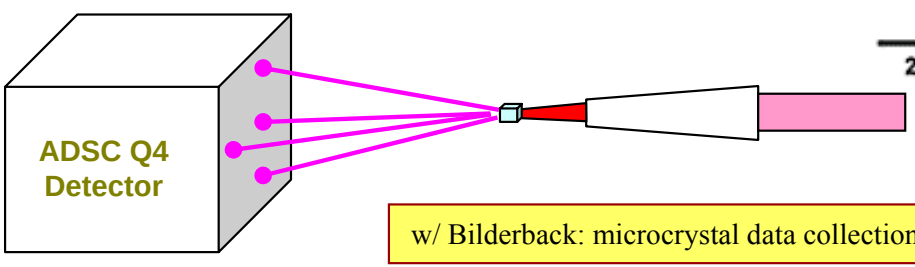
- **MacCHESS group provides user support and pushes frontier of technique development**
  - Microcrystallography
  - Membrane protein crystallization
  - Large unit cells
  - Ultra-high resolution diffraction
  - High throughput crystallography
  - Crystallographic software and phasing
  - New pressure freezing techniques
  - New crystal handling techniques

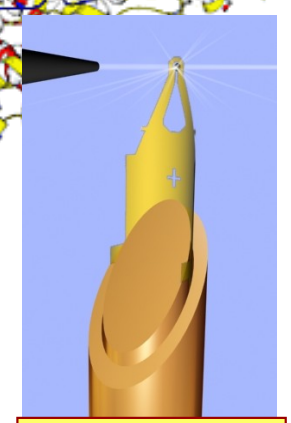
Largest ever x-ray structure solved at CHESS by Harrison (Harvard) Reovirus penetration protein 1255 Angstrom cell, Nature **404** (2000)







w/ Bilderback: microcrystal data collection



Thorne (Physics) improved loop

www.mitegen.com

UNIQUE PROGRAMS → biologists working closely with physicists develop revolutionary techniques

IMPACT → new technologies, new handling, new software for entire SR community

Lysozyme diffraction with capillary

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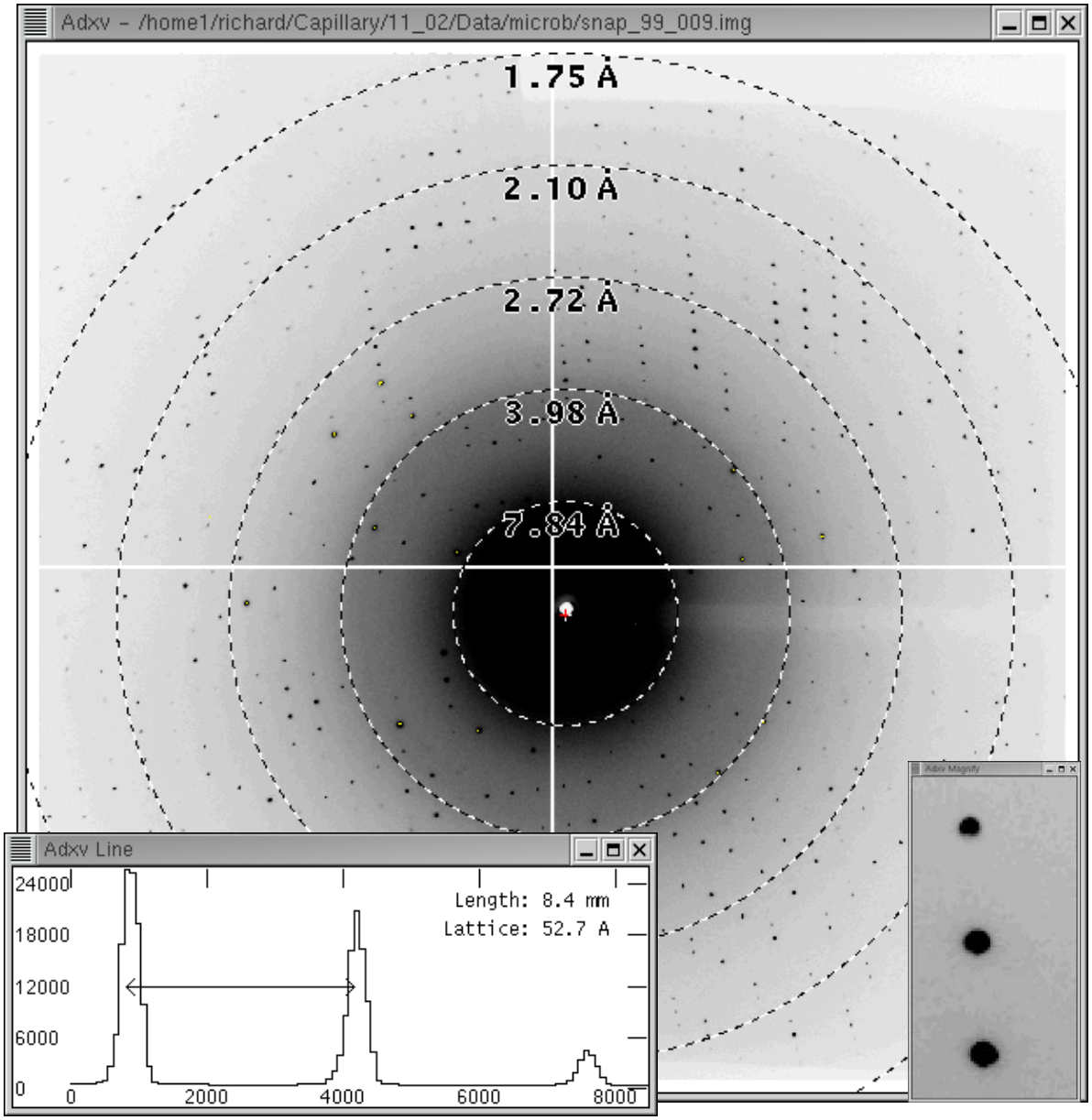
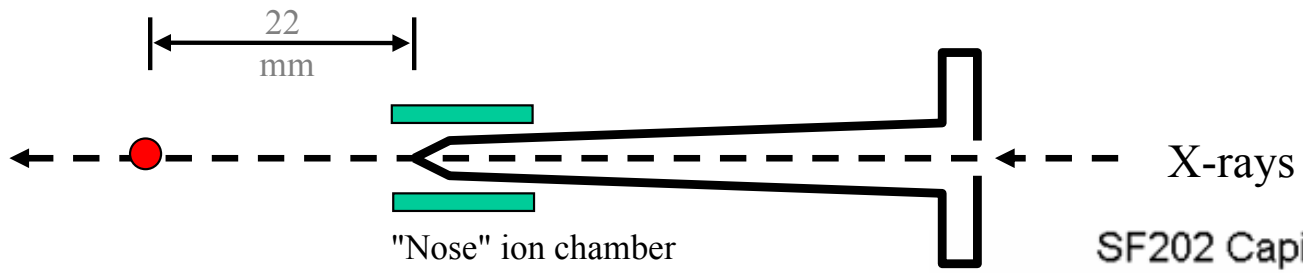


Protein diffraction at F1 station.
Focal size: 14μm(V) by 17μm(H).
E=13.5keV
1.7 mr divergence

Lysozyme crystal
20 seconds exposure,
2 Angstrom resolution

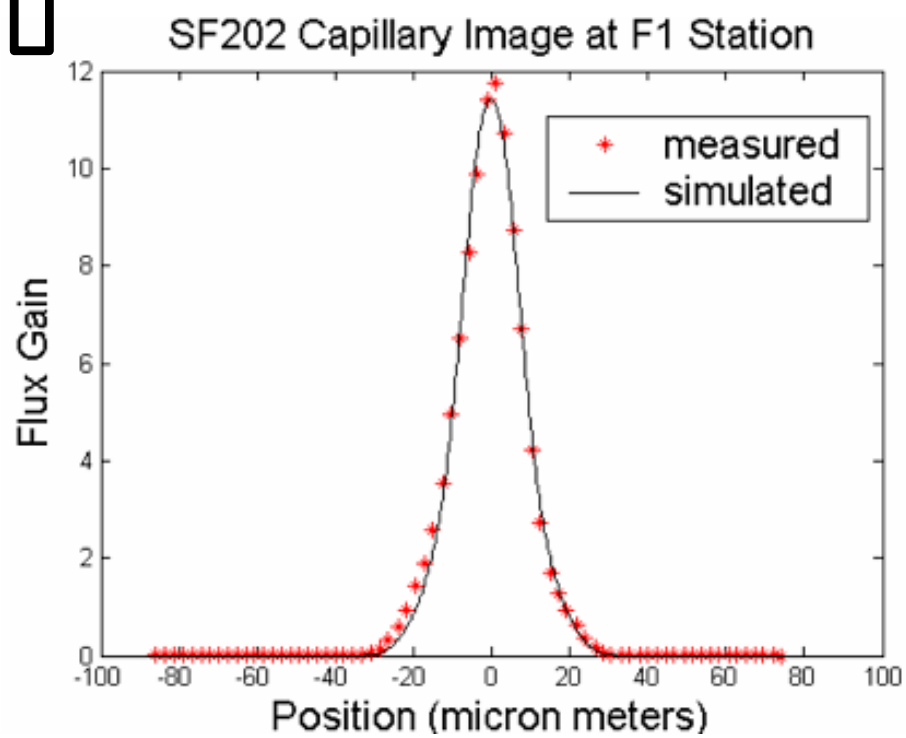
Available for data collection by users!

Protein Crystallography at CHESS F1



Capillary SF202 works at 13.5 keV on double-focusedF1 beamline.
With capillary in "Supper-Collimator-like" holder, we observe x12 flux gain with 1.7 mrad divergence

L=5 cm, Id at base = 81 microns
Id at tip = 44 microns



Life History from Fish Ear Stones

"Fish otolith trace element maps: new approaches with microbeam X-ray fluorescence", K. Limburg, R. Huang, and D. Bilderback submitted to the journal Limnology and Oceanography: Methods (2005).

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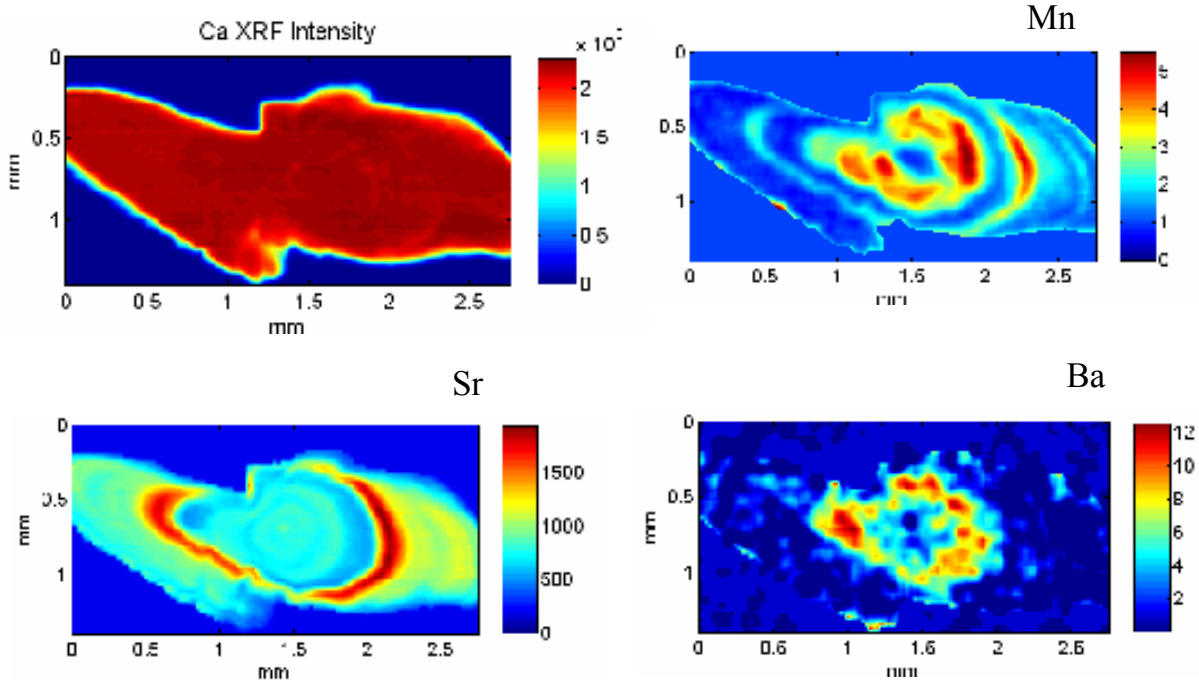
Optical image of an otolith of blueback herring capture in the Mohak River at Rome, NY

Barium is present only in the interior, corresponding to the first season's growth. The high Sr ring is consistent with going out to sea. The three tracers confirm that the fish life began in the Mohawk, spent one winter out at sea and then returned to a lower estuary.



New Tool for Art History

Arthur Woll. D. Bilderback, S. Gruner. CHESS: R. Huang, U. Chicago;
N. Gao, X-ray Optical Systems; C. Bisulca, J. Mass (U. Delaware, Winterthur Museum)

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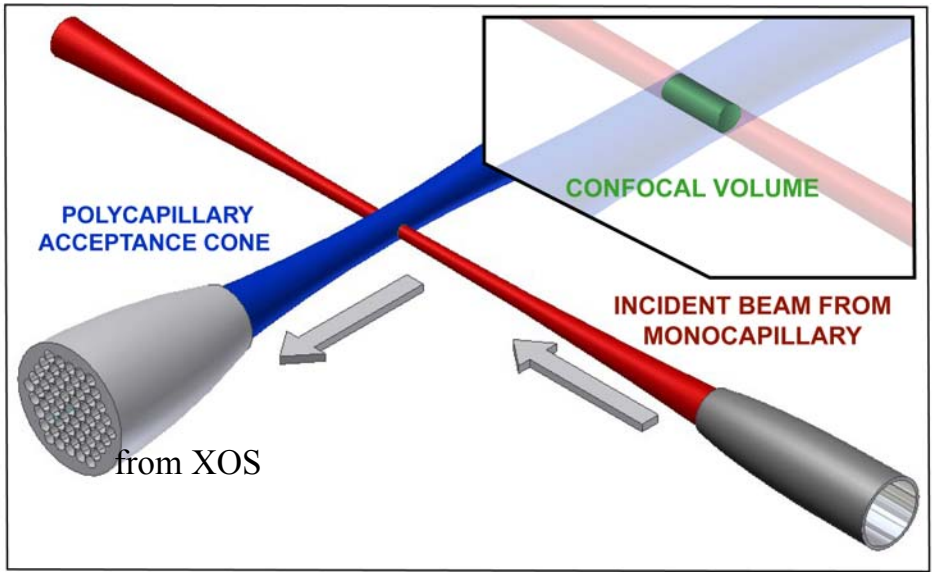
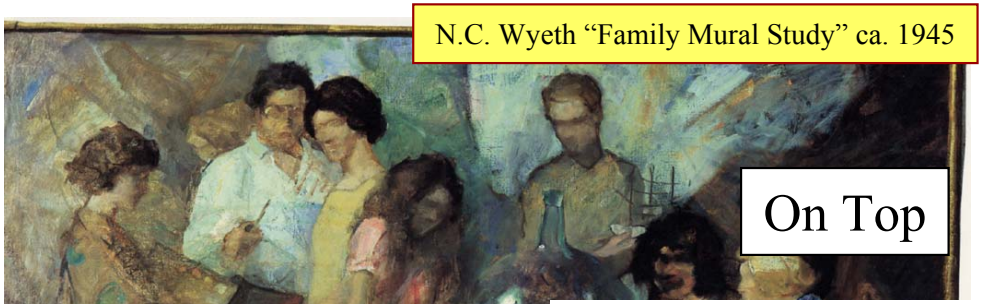
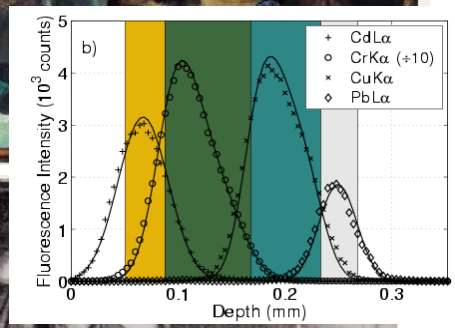
Confocal x-ray fluorescence microscope analyzes buried layers non-destructively

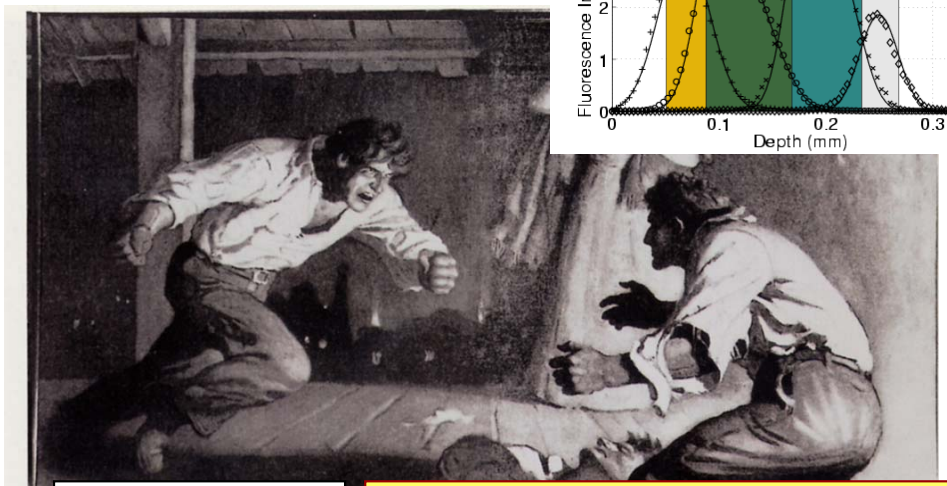




N.C. Wyeth "Family Mural Study" ca. 1945

On Top





Underneath

N.C. Wyeth "Mildest Mannered Man" ca. 1919

UNIQUE PROGRAMS → unique facility combining x-ray and art scientists (NSF IMR grant)

IMPACT → demonstrates state-of-art quantitative analysis

Micro/Optoelectronics Applications

A. Kazimirov, D. Bilderback, R. Huang et al. (CHESS), & A. Sirenko

NJ Institute of Technology, TriQuint Semiconductor

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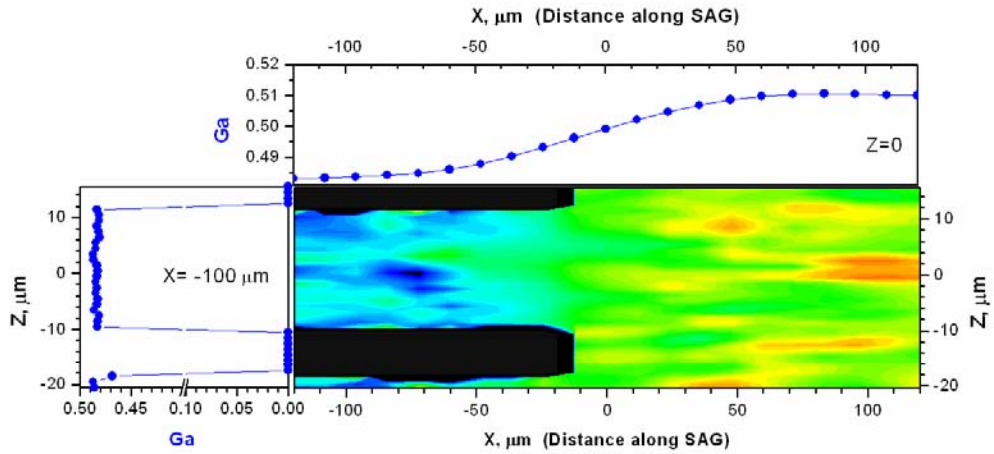
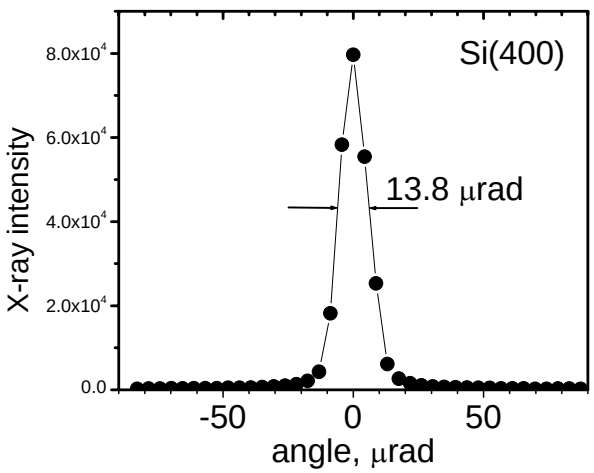
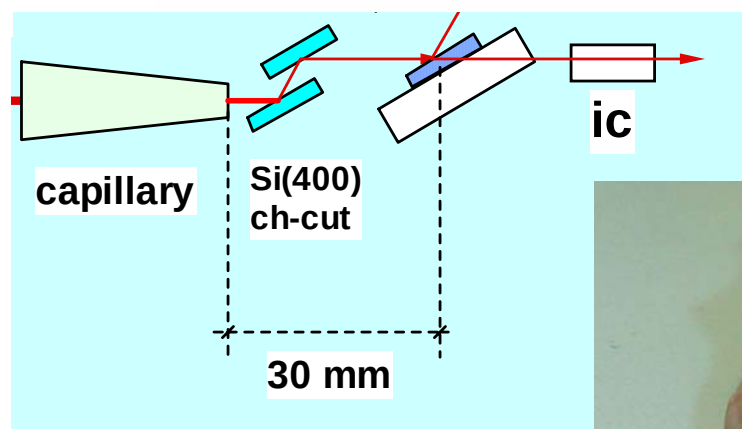
- **High-resolution imaging of semiconduct "selective area growth" materials**
  - **Resolve doping inhomogeneities**
  - **Gallium concentrations measured**
  - **Lattice strains measured**
  - **Focusing and collimation achieved simultaneously**



A. A. Sirenko, et al., J. of Crystal Growth , Vol 253 (2003) 38-45





A. Kazimirov,et al., J. Phys. D: Appl. Phys. **37** (2004) L9-L12.

UNIQUE PROGRAMS → unique combination of strain and compositional analysis

IMPACT → new technique has moved to APS to explore detection limits (zone plate replaces capillary)

A Single-bounce Capillary for Focusing of Hard X-rays at Hasylab Beamline "L"

G. Falkenberg, K. Rickers, D. Bilderback and R. Huang (APS), Hasylab Annual report 2003

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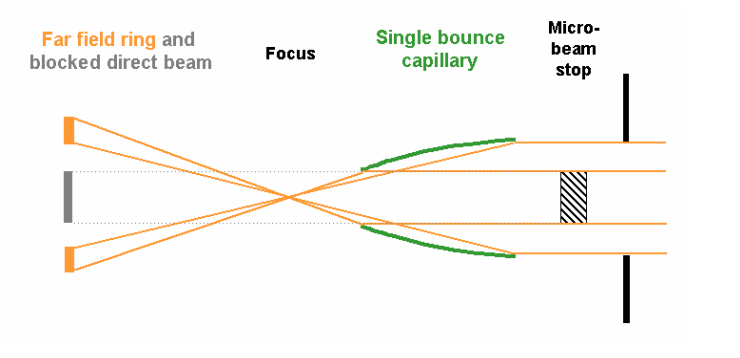
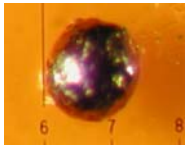
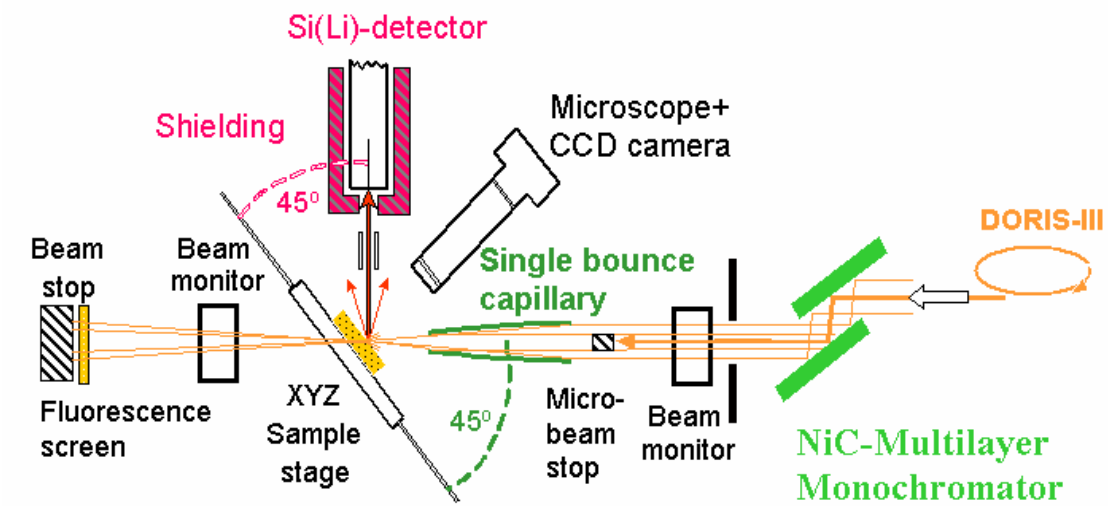
Capillary specs: 4 mr design, 50 mm working length, 103 mm long, 350 microns (base), 200 microns (tip), elliptical taper

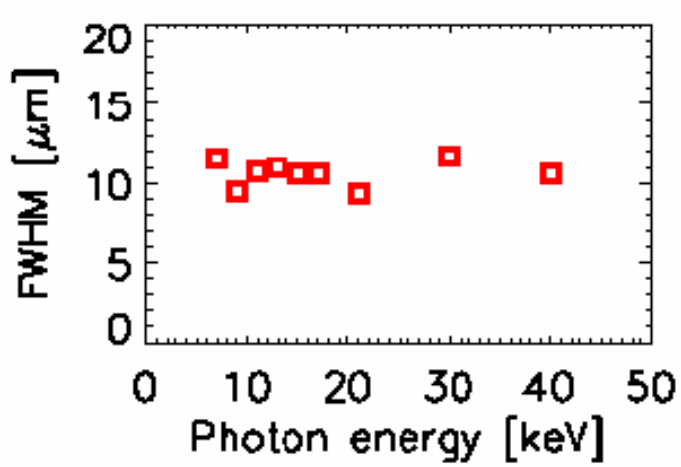


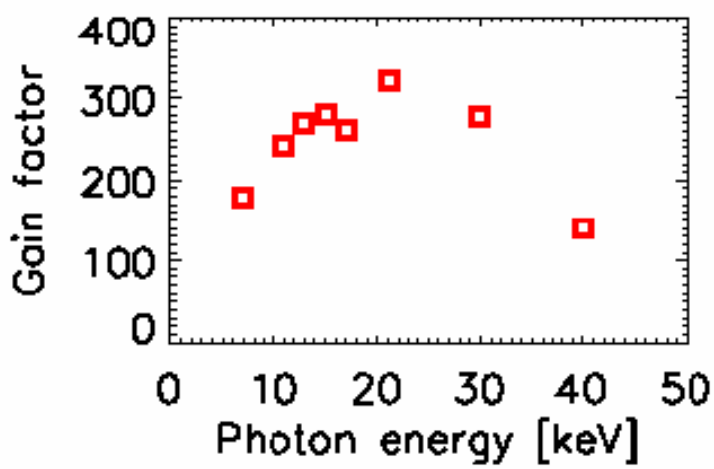


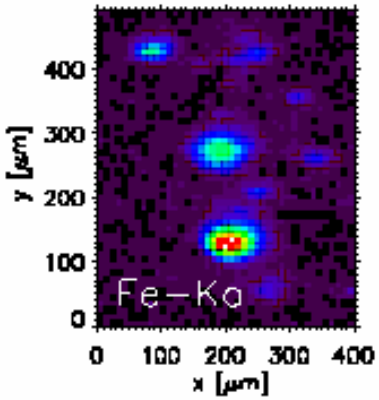
Micro-beam stops, pressed discs of ~ 250 µm diameter from 100 µm T foil



Inclusions in quartz host, Schmidt et al.







Gain of 200-300 observed, 11 micron beam size, all in good agreement with model!

Basic considerations of one-bounce capillary design

(Rong Huang program on-line at capillary.chess.cornell.edu)

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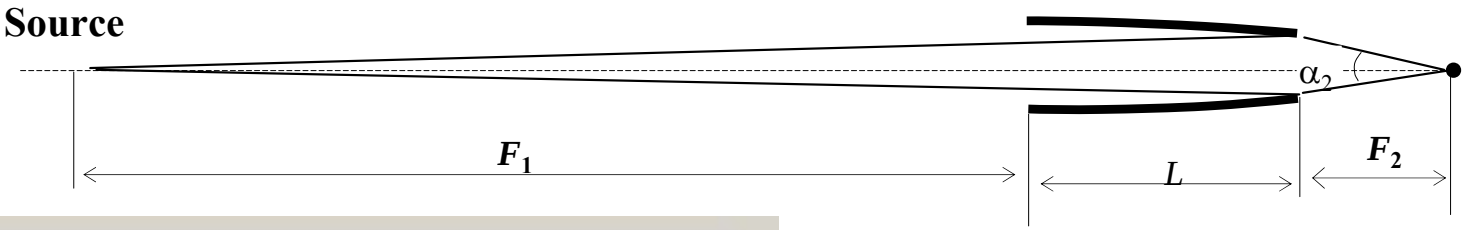
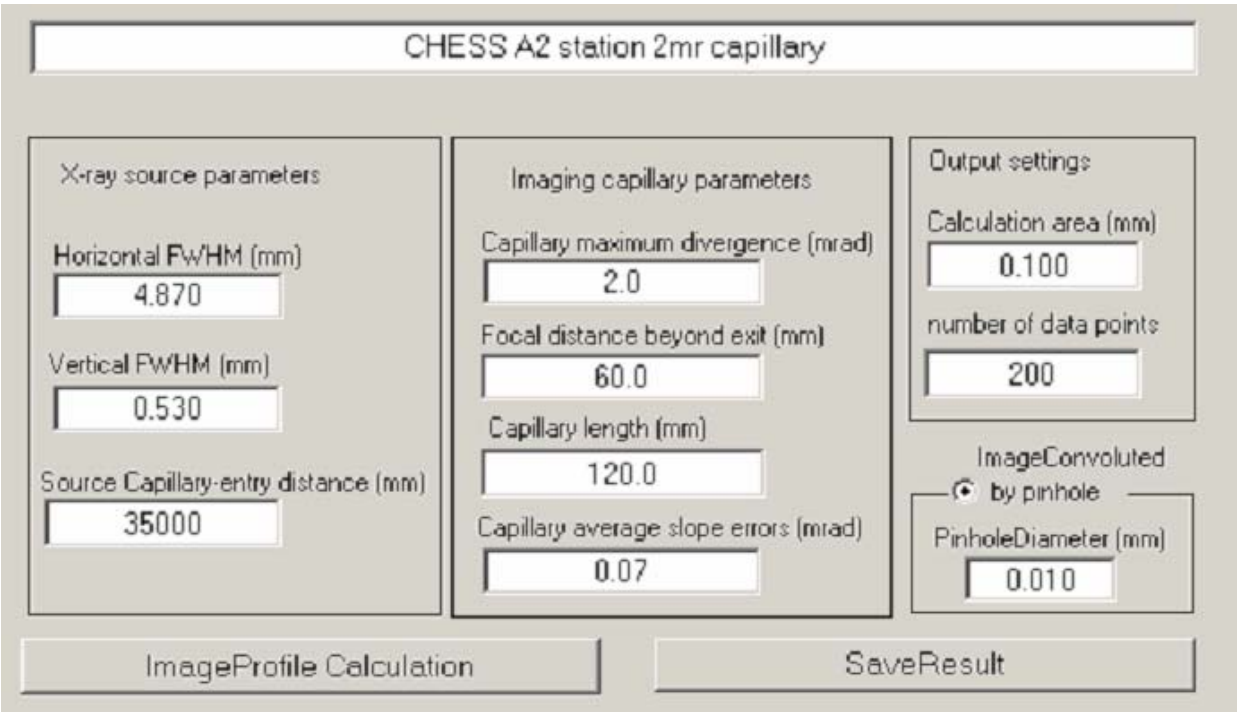
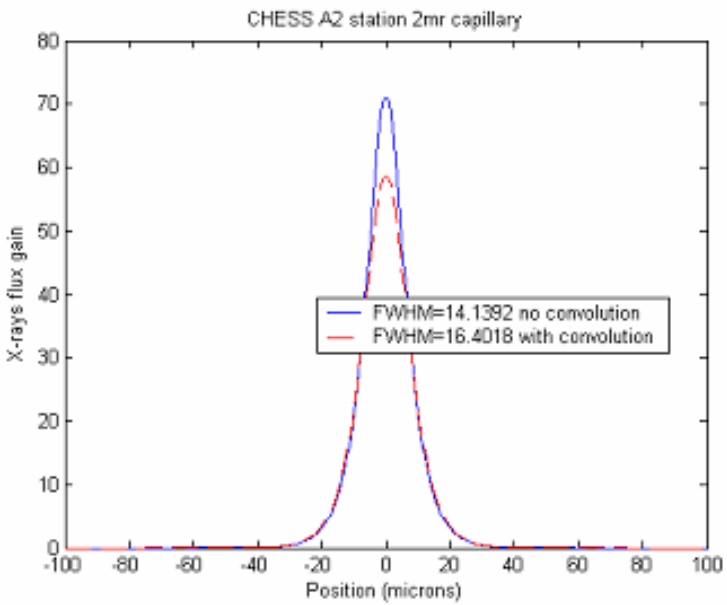
- **Focal distance:** determined by required *focal spot size*, *source size* and *capillary slope errors* or by experiment request (such as high pressure experiment)
- **Tip opening:** determined by *maximum tolerable divergence* and *focal distance*
- **Capillary length:** Compromise between collecting more photons and having good focusing profile
- **Capillary shape:** Ellipsoidal determined by source – image distance







Effect of Capillary Slope Errors on Beam Size & Gain

CHESS

Theoretical Beam Size (fwhm) in microns				
vs. Slope Error	60 microrad	30 microrad	10 microrad	0 microrad
CHESS	13	7	2.8	2.1
APS	9.2	4.8	2	0.13
ERL	9.1	4.6	1.5	0.012 (12 nm)

Theoretical Intensity Gain in Flux/Area Compared to No Capillary Case				
vs. Slope Error	60 microrad	30 microrad	10 microrad	0 microrad
CHESS	85	190	580	890
APS	180	690	3,800	11,500
ERL	184	750	6,700	11,000,000

Perfect optic value (not practical goal)

Assumptions:

- Source to capillary distance: 35 m, maximum capillary divergence=2 mr, focal distance=60 mm, capillary length=120 mm
- Source (fwhm): CHESS [4.87mm (H) x 0.53mm(V)], APS [526 micron (H) x 35 micron (V)], ERL [4.7micron(H or V)]
- Slope error only from "waviness" or "ripple"; capillary centerline is perfectly straight, no scattering or diffraction from real surface

New DB3 glass puller under commissioning

ABTech linear air bearing under test with dual-Keyence profilers for fast figure analysis.

Goal: highly automated puller with fast optical profile analysis to be used in conjunction with web-based capillary design program (developed by Rong Huang)

New puller design team: Don Bilderback, Tom Szebenyi, Sterling Cornaby and Aaron Mauer

Furnace translation: linear air bearing with 0.1 micron readout

Strain gauge/tensioning method with .02 gram resolution at 100g (force) of total tension.

New furnace and temperature controller control to 0.3 C

Velmex tensioning stage pulls upward as glass yields to maintain a constant tension

LabView operating under WinXP controls the equipment

Keyence dual-axis Optical Micrometer measures the profiles either before or after drawing

Conclusions

CHESS

- Lots of applications are taking place with capillaries, particularly at CHESS and Hasylab. They have shown themselves to be very useful in experiments!

- Further development of the pulling process is underway.

- Double-focusing single-bounce optics are really exciting if slope errors can be even better controlled. Capillary optics for 3rd generation applications look very promising in the near-future time frame. First APS tests in Oct, 2005.

- Need to develop high-z coatings for the inside bore to either increase the critical angle about 2x or extend their energy of operation approximately 2x.

- Can we microfabricate even more precise optics?