better analysis counts

Polycapillary X-ray Optic Fundamentals, Applications and Sub-Systems

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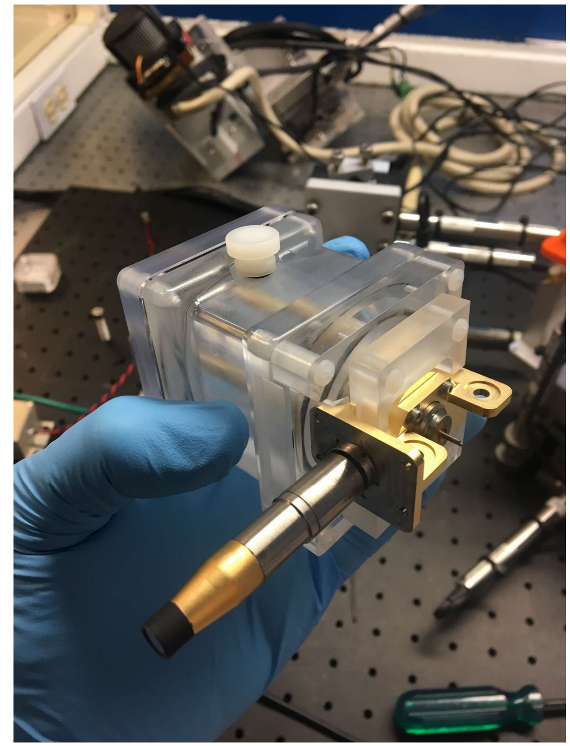
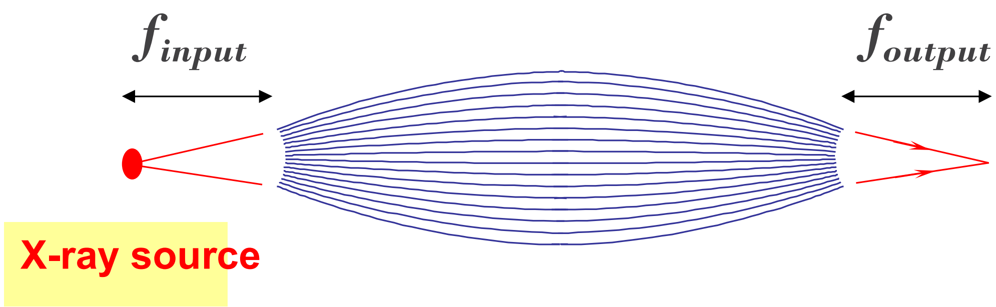
Outline

- Polycapillary Optic Types
- Fundamentals of Polycapillary Optics
- Polycapillary Optic Performance Examples
- Applications
- Success Stories
- Polycapillary enabled sub-systems
- Summary

Raw and Packaged Optics

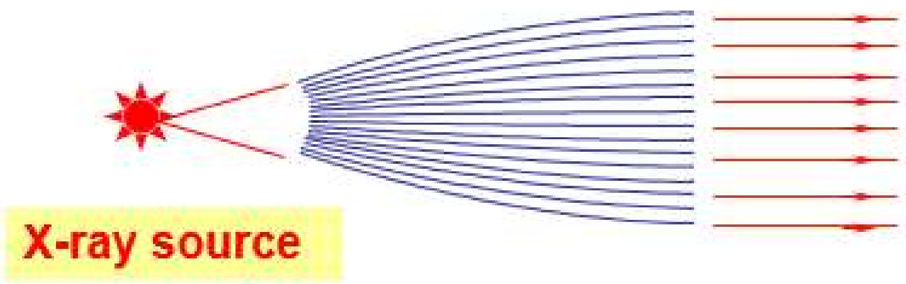


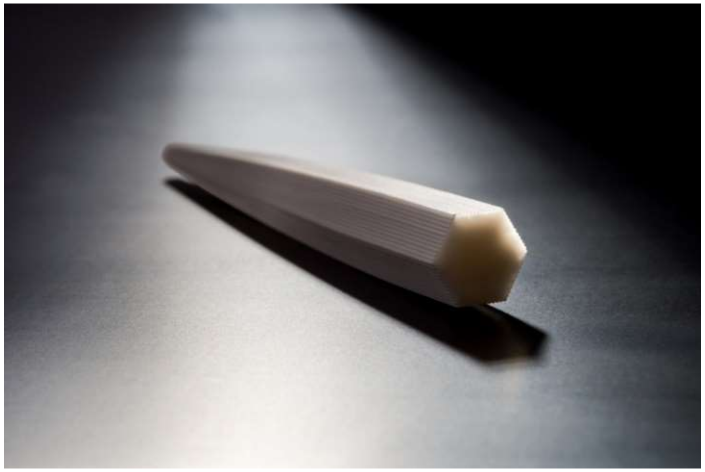
Polycapillary Focusing Optic



- Sub-million to Multi-million capillary channels per optic
- Each individual channel is aligned to the same point, the focal point
- The focal point exists on both the input and output side of the optic
- Optic provides a large collection angle which leads to high output x-ray flux
- A polycapillary optic is not an imaging optic
- Focal spot sizes as small as 5 microns
- Provides flux density up to five orders of magnitude higher than a pinhole collimator.

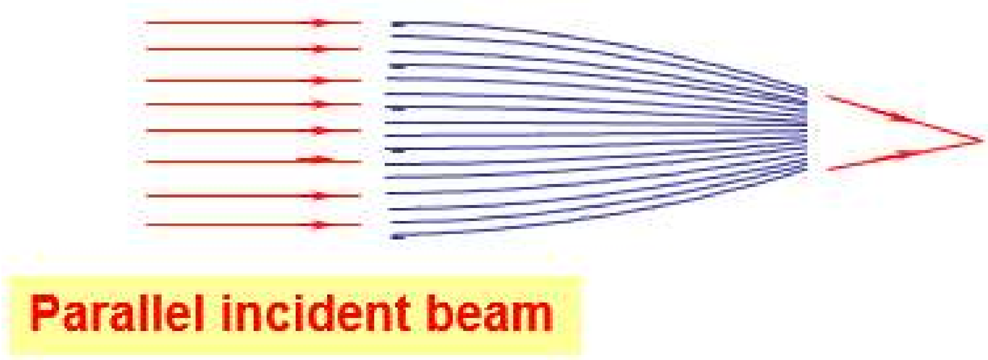
Polycapillary Collimating Optic

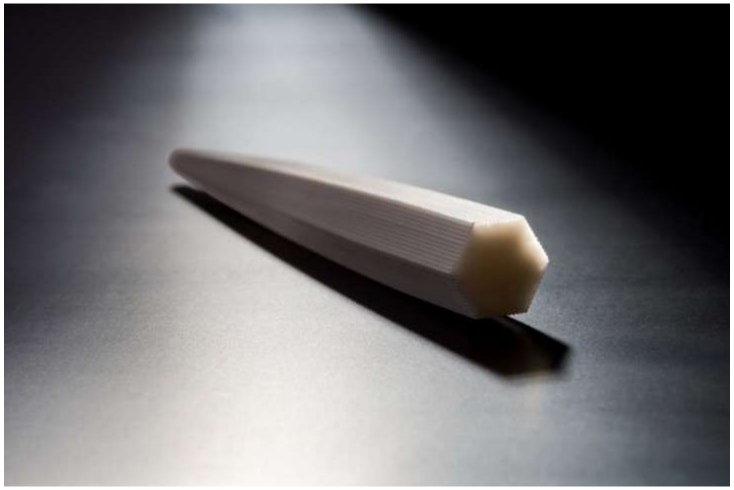




- Quasi parallel output beam with divergent angle equal to the critical angle.
- Output beam diameter can be as large as 25mm
- Output beam brilliance is improved by up to two orders of magnitude compare to a conventional soller slit.

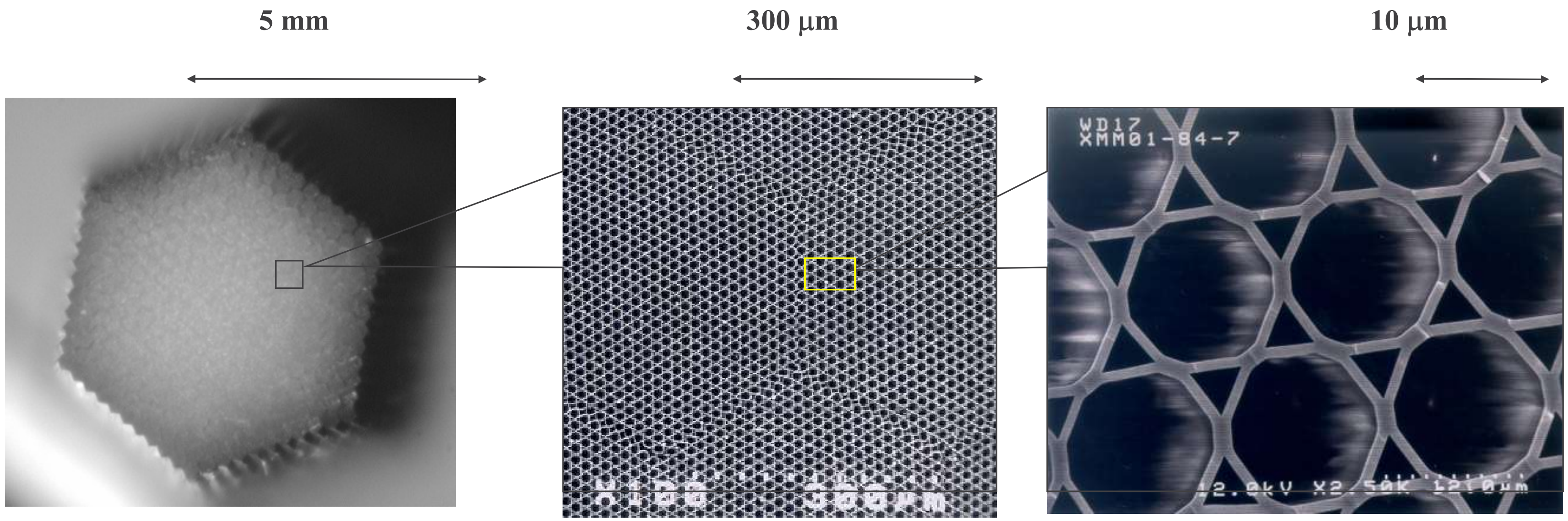
Polycapillary Half Focusing Optic





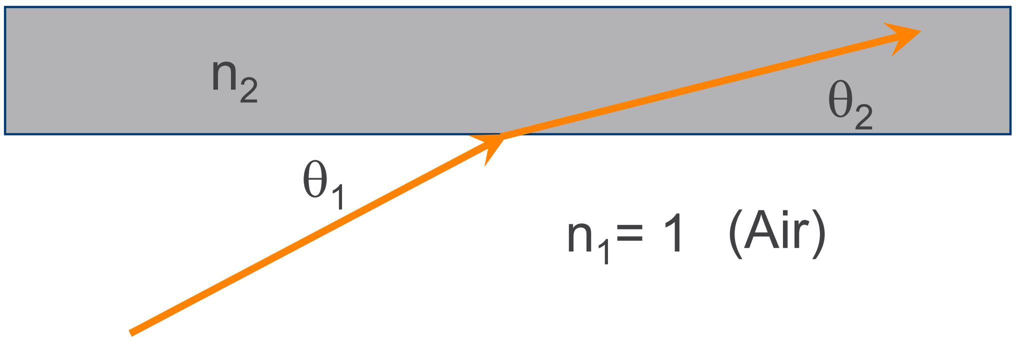
- Focal spot size can be smaller than that obtained with lab sources
- Can be beneficial for special lab applications
- Mainly used for synchrotron applications.

Fundamentals of Polycapillary Optic Structure



- Channel size in single micron range
- Open area up to 80%
- Surface roughness minimized
- Optic shape/profile precisely controlled to single microns

Fundamental- X-ray Propagation



- **Snell's Law: $n_1 \cos(\theta_1) = n_2 \cos(\theta_2)$**
- **For X-rays, the refractive index is complex, expressed as: $n_2 = 1 - \delta + i\beta$**
- **δ and β describe the dispersive and absorptive aspects of the wave-matter interaction. Their values can be calculated from the atomic scattering factors.**
- **Critical angle: $\theta_c = \sqrt{2\delta}$ (when $\theta_2 = 0$)**

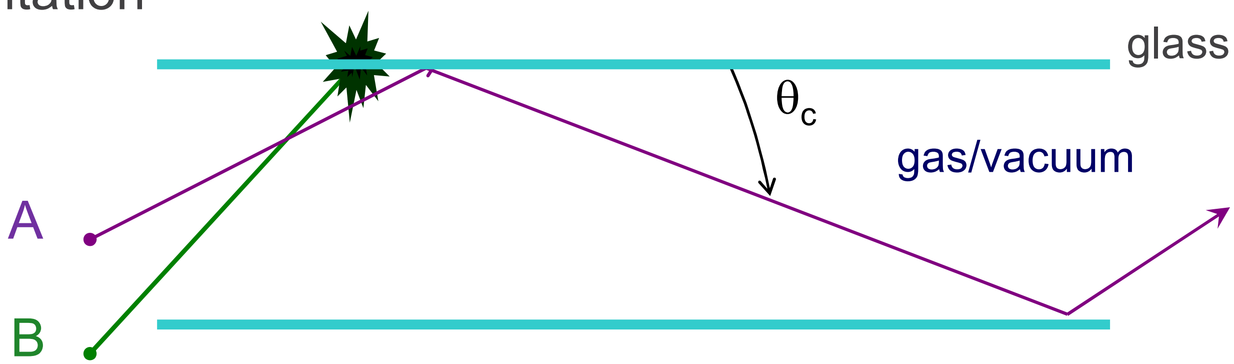
Critical angle examples

Material	X-ray energy (keV)	X-ray wavelength (Å)	critical angle (°)
SiO_2	2	6.2	0.927
	4	3.1	0.48
	8	1.55	0.239
	12	1.03	0.159
	16	0.77	0.119
	24	0.52	0.079

Material	X-ray energy (keV)	X-ray wavelength (Å)	critical angle (°)
Gold	2	6.2	1.83
	4	3.1	1.097
	8	1.55	0.56
	12	1.03	0.348
	16	0.77	0.282
	24	0.52	0.191

Fundamental- X-ray Propagation

- X-ray Propagation by Total external reflection
- Straight Orientation



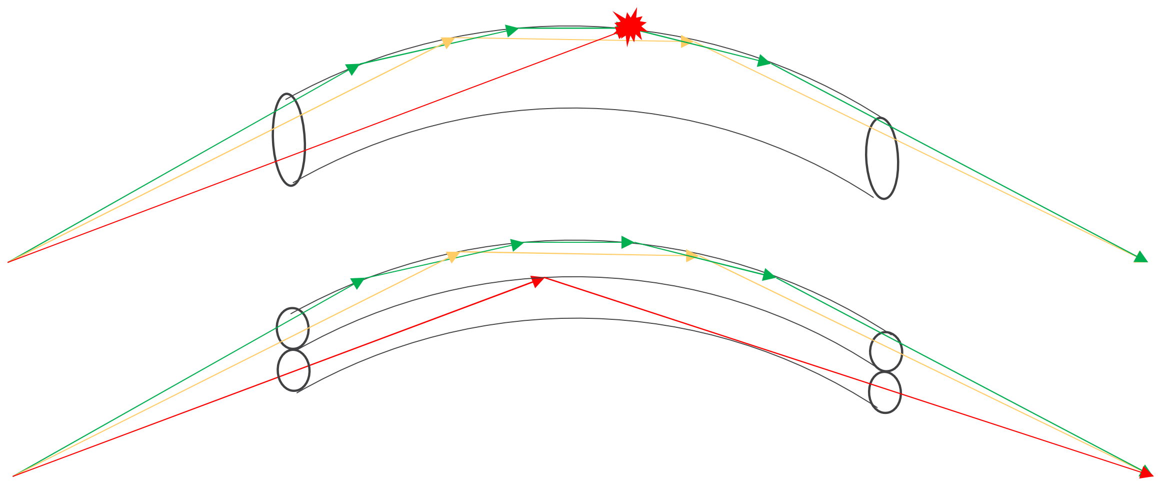
Incident photon "A" angle ≤ critical angle (θ_c)
Incident photon "B" angle > critical angle (θ_c)

- For silicate glass:

$$\theta_c\,(mrad) \cong \frac{30}{energy(keV)}$$

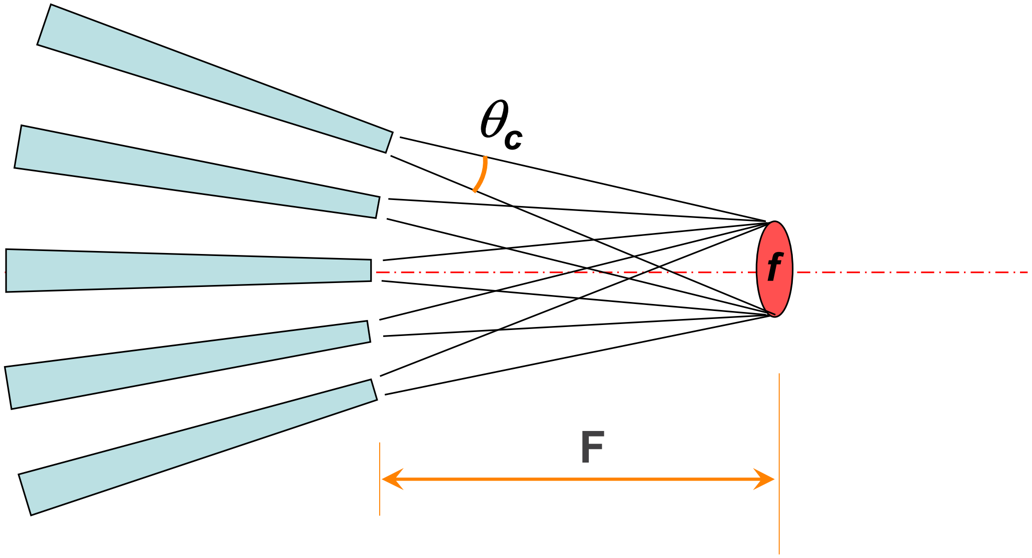
Cu Ka (8.0 keV)
$\theta_c \approx$ 3.8 mrad. or 0.22º
Mo Ka (17.4 keV)
$\theta_c \approx$ 1.7 mrad. or 0.10º

Fundamentals of Polycapillary Optics



- X-rays transmit efficiently through small capillary channels by multiple total reflections.
- Smaller channel size is the key for more efficient transmission
- Maximum curvature is dependent on x-ray energies.

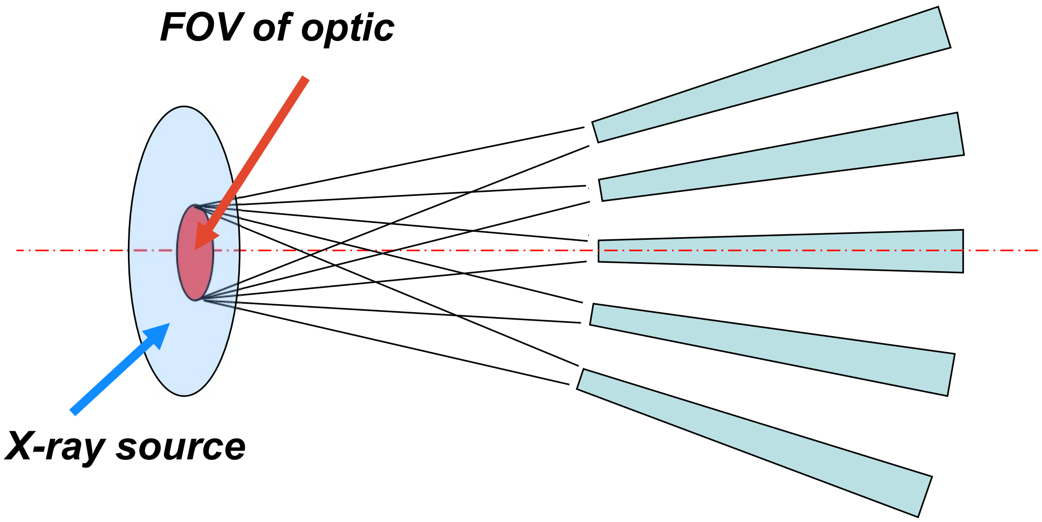
Fundamentals- Spot Size



$$f \approx F \cdot \theta_c$$

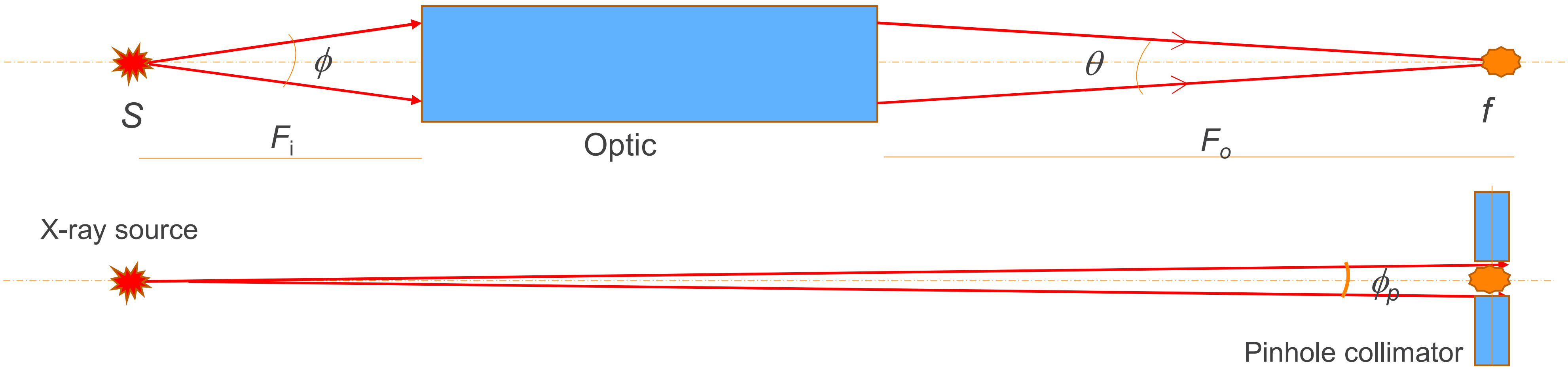
- Below 5keV the divergent angle will stay approximately 6-10mrad but above 5keV will remain the critical angle
- Source size 1-50
- Focal spot size is mainly determined by the focal distance and the energy
- Higher energy provides a smaller θ_c which yields a smaller focal spot with the same optic.
- Spot size is independent of the optic outer diameter!

Fundamentals- Field of View



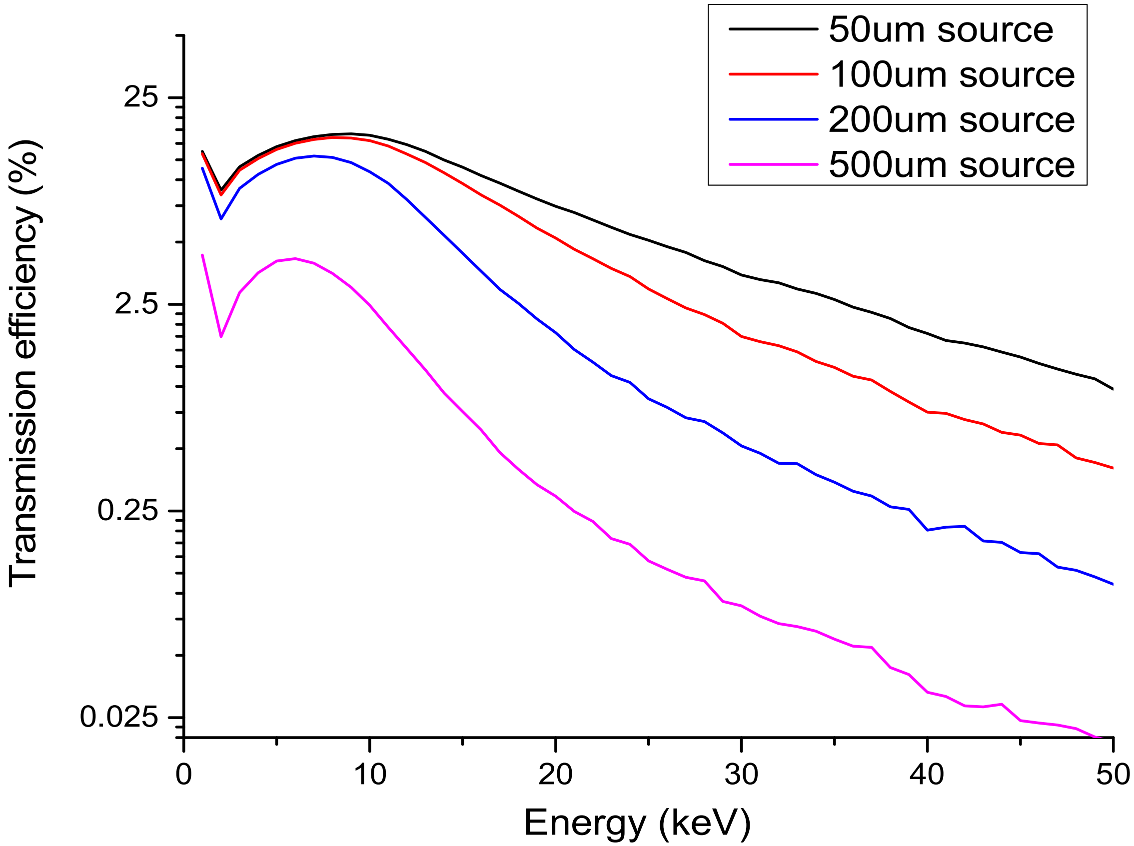
- Field of view predominantly determined by the focal distance and energy
- Independent of entrance diameter or the capture angle
- The feature makes a polycapillary optic a good spatial filter for certain applications including confocal XRF and XRD (for background suppressing).
- The transmission efficiency of optics inversely proportional, approximately, to the source area

Fundamentals- Key Performance Parameters



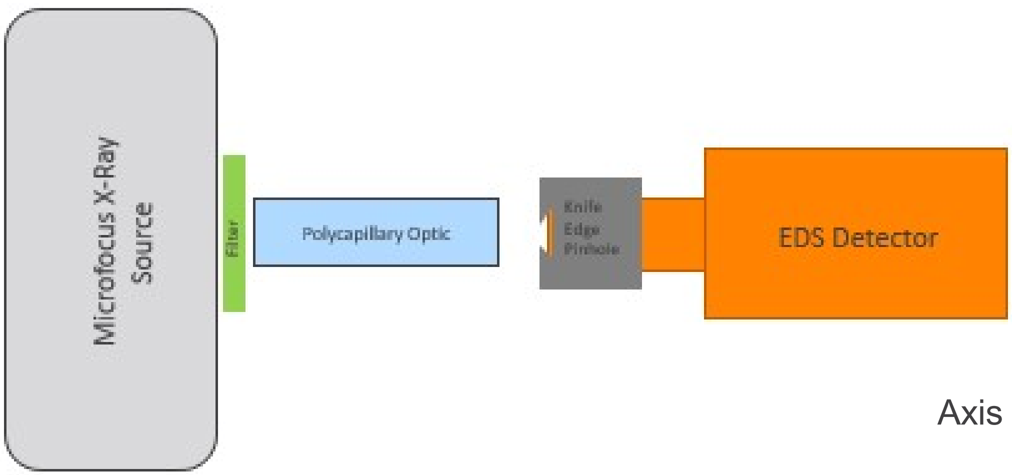
- Focal spot size [FWHM] (f) $\approx F_o \cdot \theta_c$
- Transmission efficiency (T) = ratio of the output x-rays to those captured at the input of the optic
- Effective collection solid angle (ECSA) = $\phi^2 \cdot T$
- Flux density gain = $\phi^2 \cdot T / \phi_p^2$
- Divergent angle (θ)

Fundamentals- Transmission vs. Energy

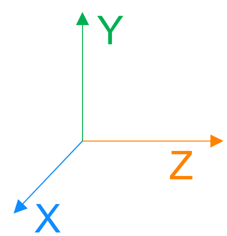


Fundamentals- Optic Characterization

- Characterization with a direct beam with knife edge method is shown in the figure below.
- To align the optic to the source, in both cases, the optic will have to be adjusted in X, Y, Z to maximize the count rate into the detector.
  - X and Y should be adjusted first to optimize the position the optic to the source anode.
  - Z will be moved to optimize the input focal distance.
- The sensitivity of the optic alignment to the source will vary based on source size and optic design, but for some general numbers the optic being off even 200μm will cause a significant impact to the output beam performance.
  - To characterize the focal spot size
    - Direct beam method
      - With a knife edge pinhole on the detector, scan across the focus in X or Y
      - Move the detector along the beam path (Z) and repeat scan across the spot to identify the smallest scan fit.

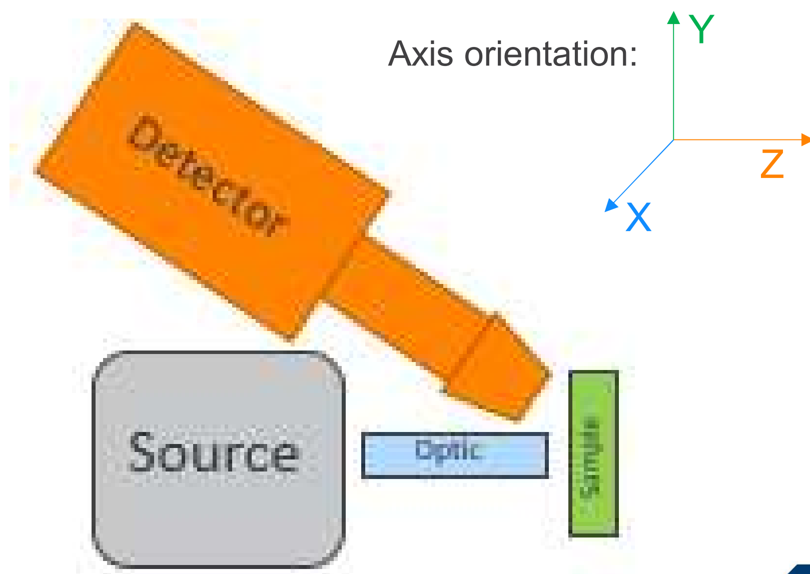


Axis orientation:

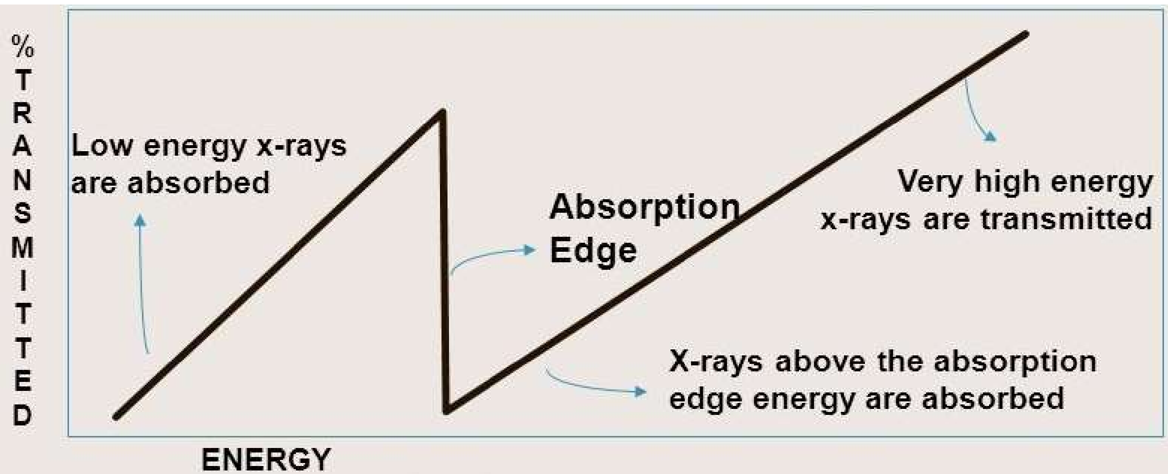


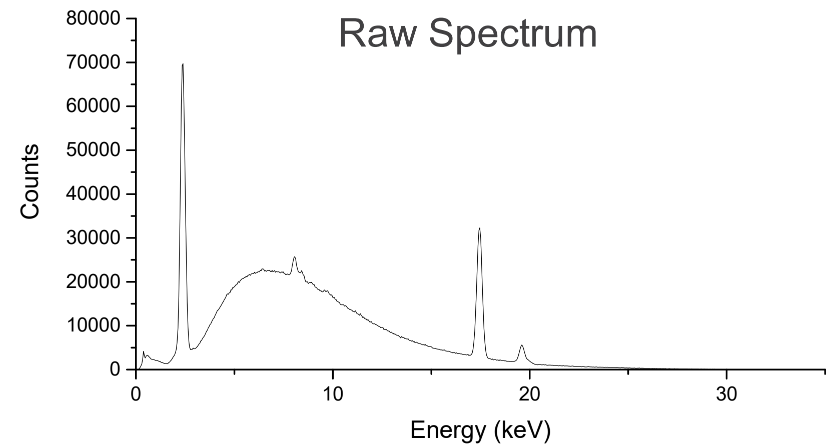
Fundamentals- Optic Characterization

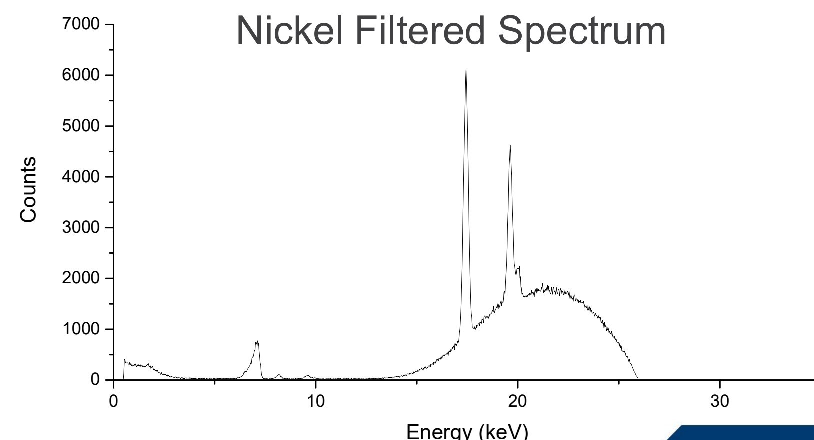
- Characterization with a fluorescence knife edge method is shown in the figure below.
- To align the optic to the source, the optic will have to be adjusted in X, Y, Z to maximize the count rate into the detector.
  - X and Y should be adjusted first to optimize the position the optic to the source anode.
  - Z will be moved to optimize the input focal distance.
- The sensitivity of the optic alignment to the source will vary based on source size and optic design, but for some general numbers the optic being off even 200µm will cause a significant impact to the output beam performance.
- To characterize the focal spot size
  - Fluorescence method
    - Scan a sharp edge sample across the focus, X or Y depending on sample orientation.
    - Move the sample along the beam path (Z) and repeat scan across the spot to identify the smallest scan fit.



Fundamentals- Spectrum Filtration







- The benefit during characterization is to reduce overall intensity which will aid in reducing detector dead time.
- From an application point of view, if a primary filter is to be used for a polycapillary system, it can be placed between the source and optic to obtain a clean output beam. Comparing to a pinhole collimator system, the filter will cause additional fluorescence.

Polycapillary Optic Performance Examples

Focusing Optics							
Working distance (mm)	2	4	9	20	50	100	200
Focal spot size* (μm, FWHM, 17.4keV)	7	15	25	45	100	180	300
Intensity gain* (vs a plnhole collimator of same size, 100mm from the source)	6000	4500	3500	2000	800	300	120

Note: *With a 100μm X-ray source.

Applications include micro-XRF for elemental mapping, plating thickness and fine feature analysis.

Half-focusing Optics (XRF/XAS)					
Working distance (mm)	2	4	9	20	50
Focal spot size* (μm, FWHM, 17.4keV)	7	15	25	45	100
Intensity gain* (vs a pinhole collimator of same size)	850	550	400	200	80

Note: *With an incident beam of 2mm in diameter and a divergent angle of <0.5mrad

Applications include micro XRF, micro XAS, and confocal XRF.

Collimating/Parallel Beam Optics (XRD/WDS/XRF)								
Output beam diameter (mm)	0.5	1	2	3	4	6	10	15
Intensity gain*	12	45	130	250	370	470	680	850

Applications include powder XRD, texture and stress analysis, WDS and confocal XRF.

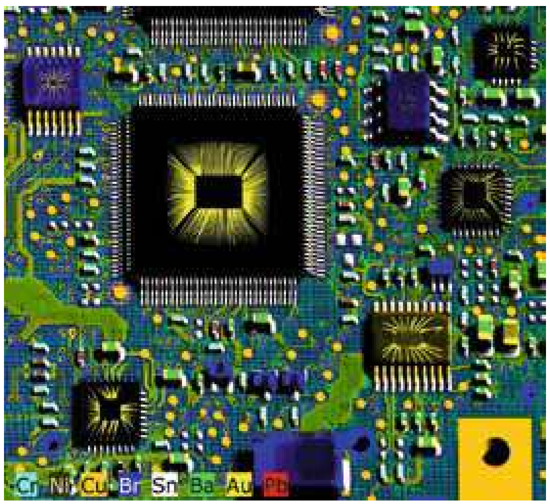
Polycapillary Optic Applications

- Micro XRF
- Micro XRF on SEM
- Synchrotron micro x-ray analysis
- Wavelength-dispersive spectrometer (WDS)
- Microcalorimetry
- Confocal XRF
- Large source optics
- Parallel-beam XRD
- Conical Polycapillary Optic Imaging
- Parallel Beam XRD

Micro XRF Applications

Microelectronics

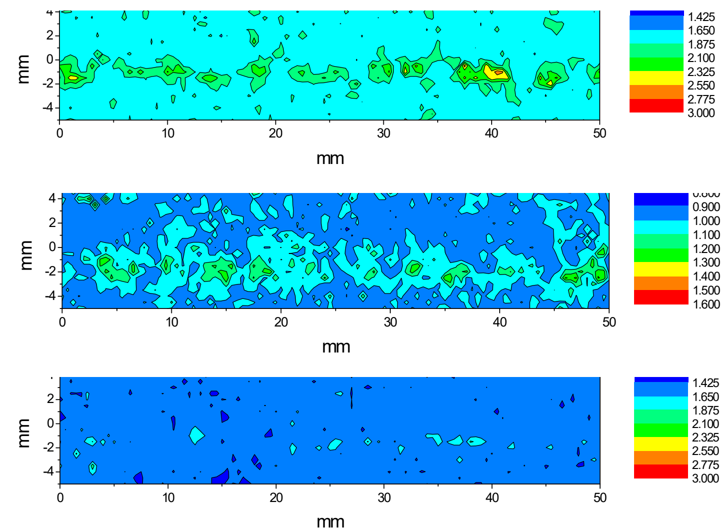
Plating thickness
Connectors
Soldering defects
WEEE / RoHS



Elemental mapping of PCB
courtesy of Bruker

Material research

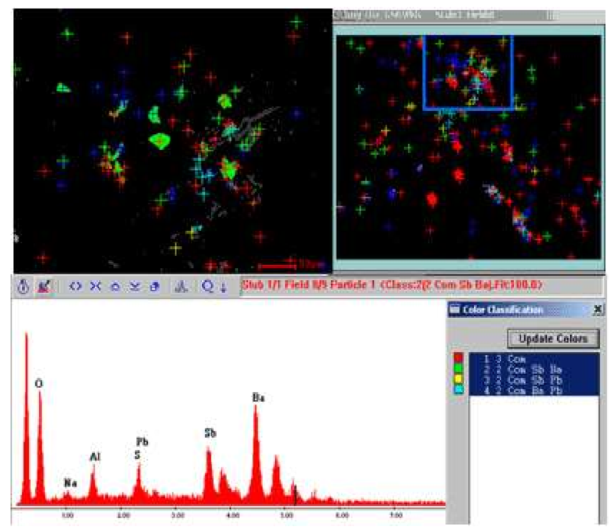
Semiconductor
Solar cells
Concrete
Metals and alloys
Air particulates



Macro-segregation in steel alloy
(Mn concentration mapping)

Forensics

Gun shot residues
Fingerprints
Gems
Pigments



Gunshot residue analysis using µXRF technology
Courtesy of EDAX

Micro XRF Applications

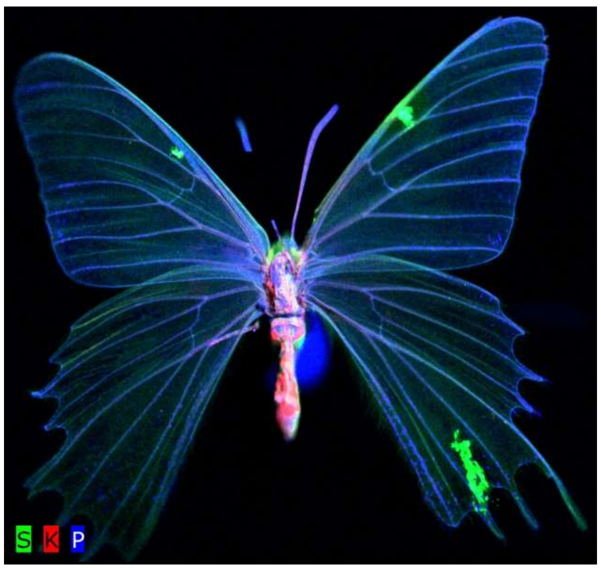
Life science

Tissue mapping

Leaves

Heavy metals

Pharmaceuticals



Elemental maps of a butterfly

courtesy of Bruker

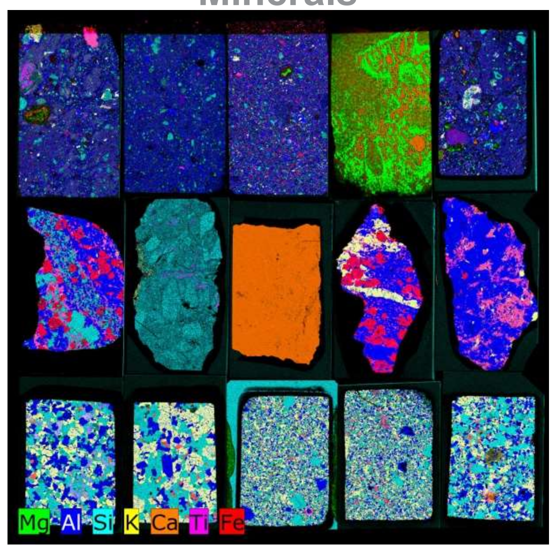
Geoanalysis

Rocks

Fossil

Particles and inclusions

Minerals



Elemental maps of mosaic

courtesy of Bruker

Art conservation

Pigments and inks

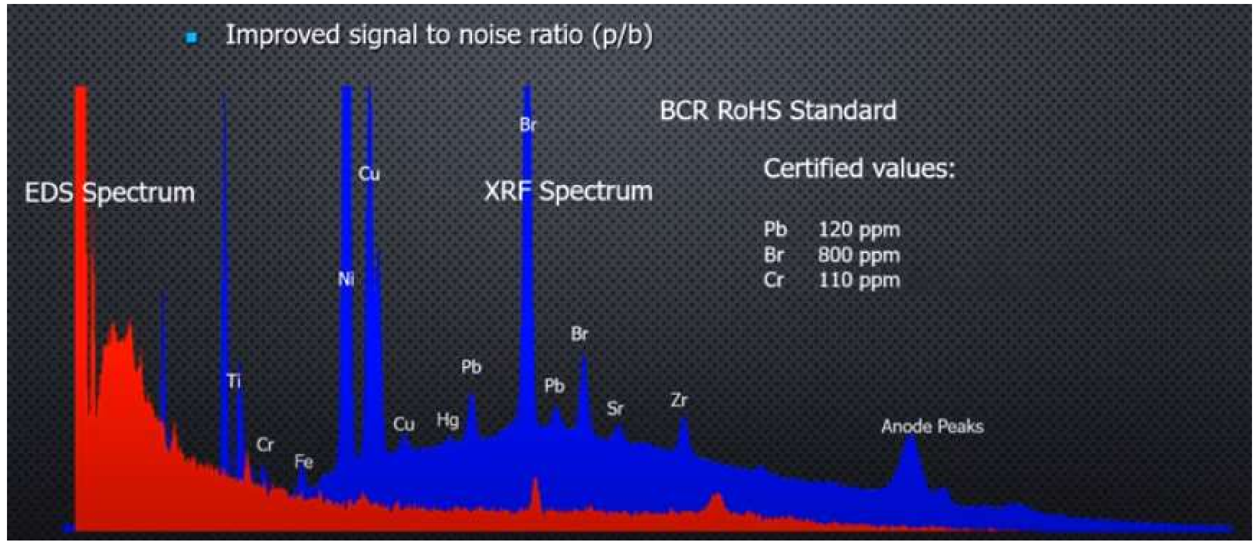
Hidden paintings

Corrosion

Multi-elemental maps of a hidden painting

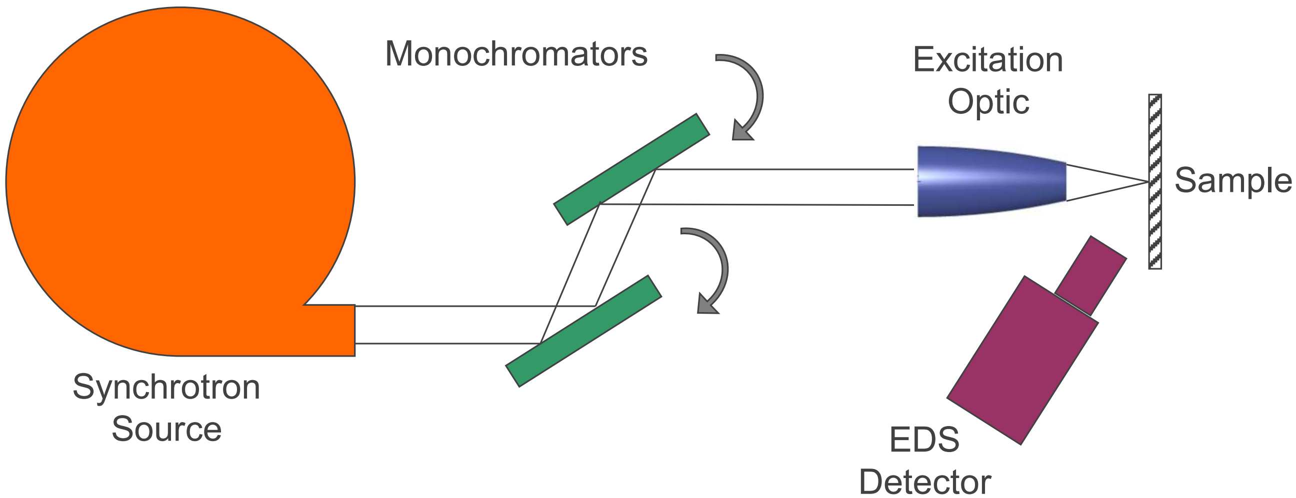
courtesy of Bruker

Applications- uXRF on SEM



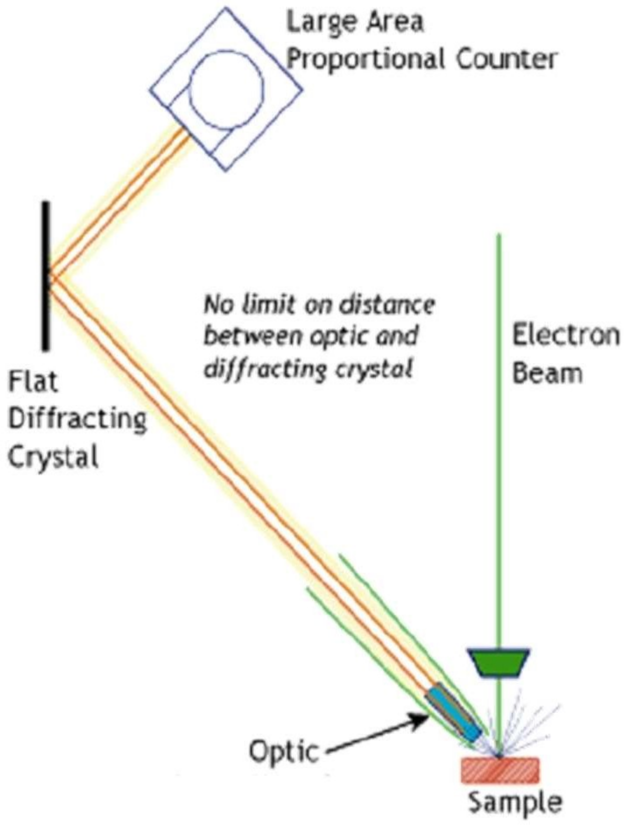
- Better x-ray sensitivity at energies >2keV
- Improved signal to noise ration (p/b)
- Increased peak separation for overlaps and higher energy lines
- Combine EDS light elements with XRF heavier elements
- *In situ* analysis, without sample transfer.
- No specimen charging with XRF
- Small laboratory footprint

Synchrotron Applications



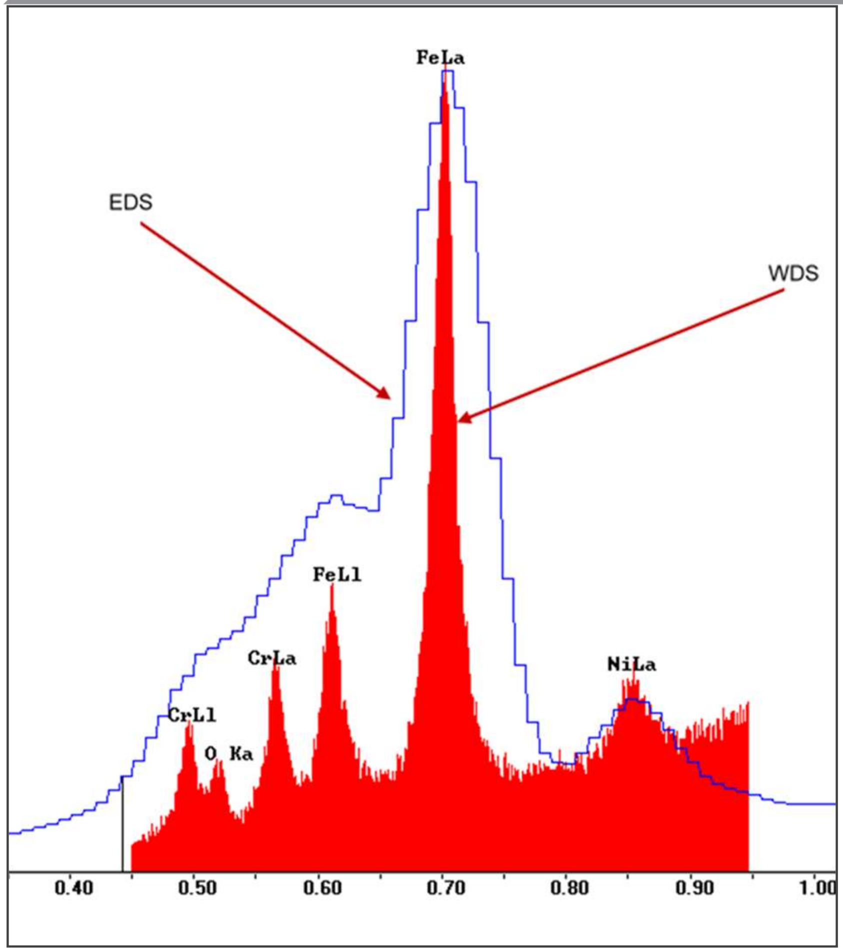
- Polycapillary output focal spot down to 5 microns
- Intensity gain of 3-4 orders of magnitude compared to pinhole apertures
- Output focal spot does not move with small energy tuning (Particularly important for XANES measurements)
- Collection Polycapillary Optic adds confocal measurement capabilities.

Applications- Wavelength Dispersive Spectrometer



- Polycapillary collimating optic coupled with flat crystals
- Multiple crystals to cover wide energy range
- Less complex design than conventional WDS using curved crystals
- Polycapillary optic PBS will cover a broader energy range when compared to a single bounce crystal system.

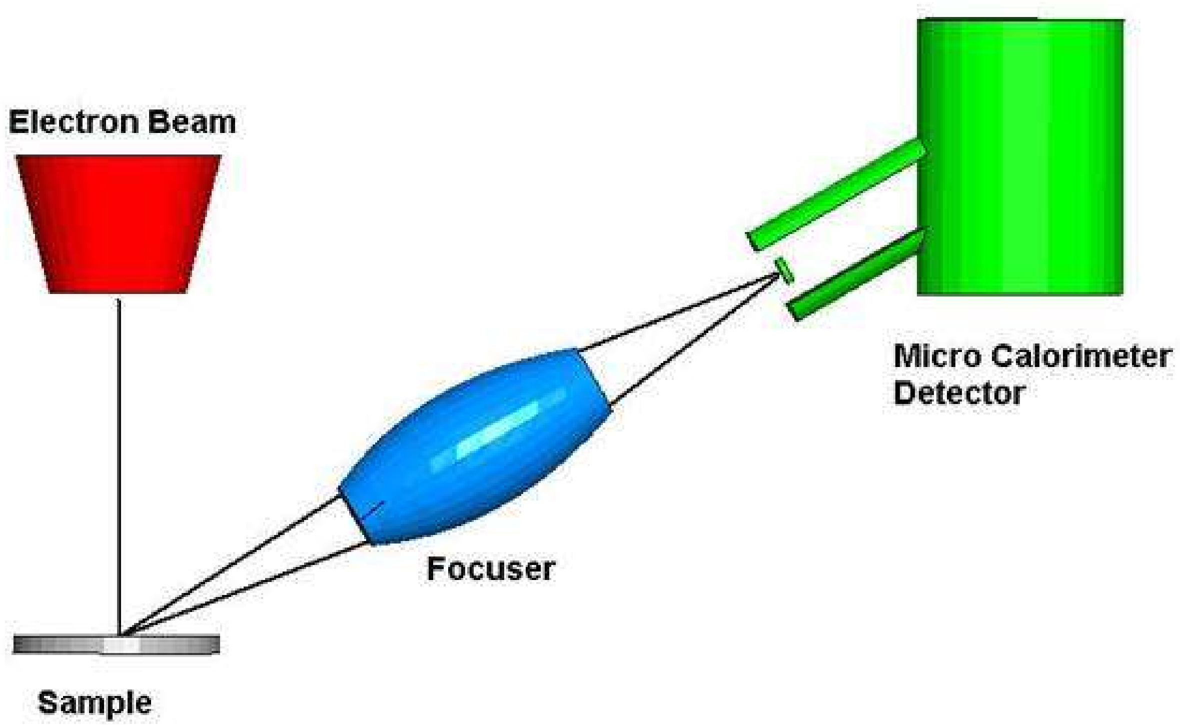
Applications- Wavelength Dispersive Spectrometer



WDS Benefits

- Better Resolution- resolve peak overlaps
- Improved sensitivity, limits of detection
- Higher accuracy in quantitative analysis
- Improve confidence in results

Applications- Microcalorimitry



- The polycapillary optic will collect the signal from the sample and focus into the small active area of the detector to increase the effective collection solid angle.

Applications- Confocal XRF and XAFS in Diamond-Anvil Cells

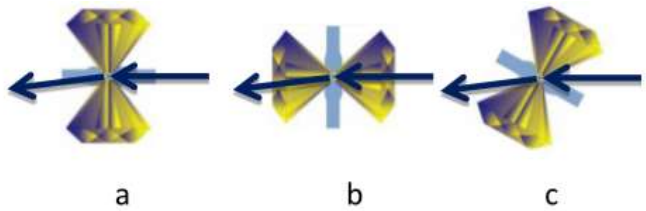


FIG. 1. Schematic view of different scattering geometries. (a) X ray enters into and exits from the gasket; (b) X-ray goes in and out from (perforated) diamonds; (c) X-ray goes in and out from a tilted DAC to reduce the path length in the gasket.

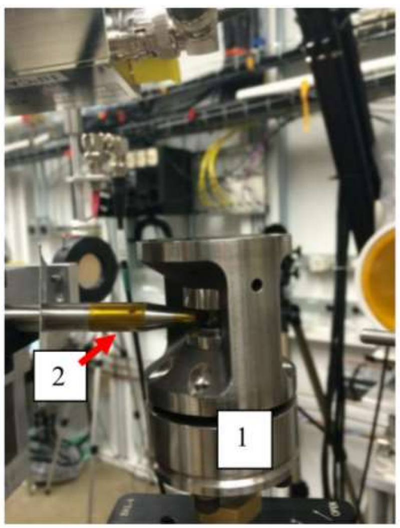


FIG. 3. Photo showing the focusing polycapillary (2) extending into the DAC (1).

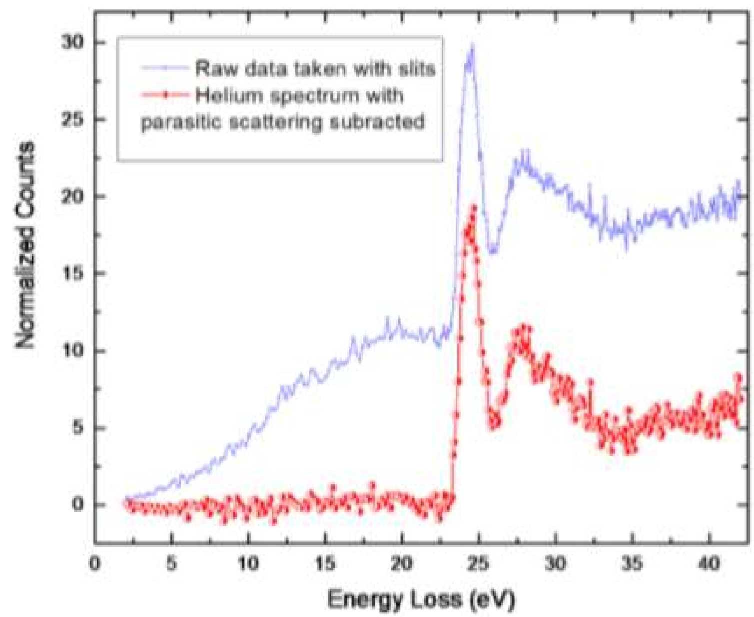
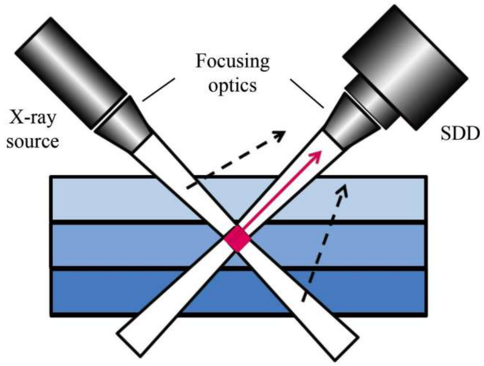


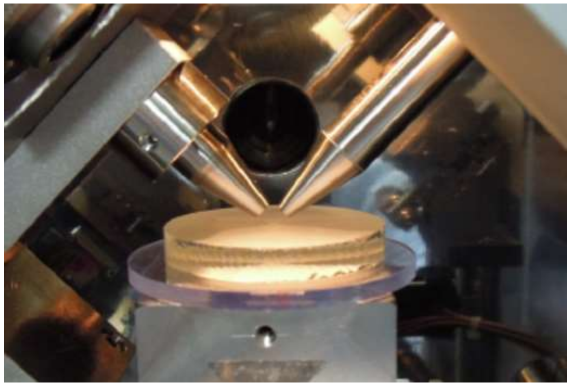
FIG. 4. Combined raw data from several scans taken with conventional post-sample slits used to reduce parasitic scattering from the DAC, along with analyzed data after the parasitic scattering from diamond and the gasket were subtracted. Typical count rate in the main peak of the background subtracted helium spectrum was 220 cts/min.

Courtesy of Paul Chow from APS, Argonne, IL

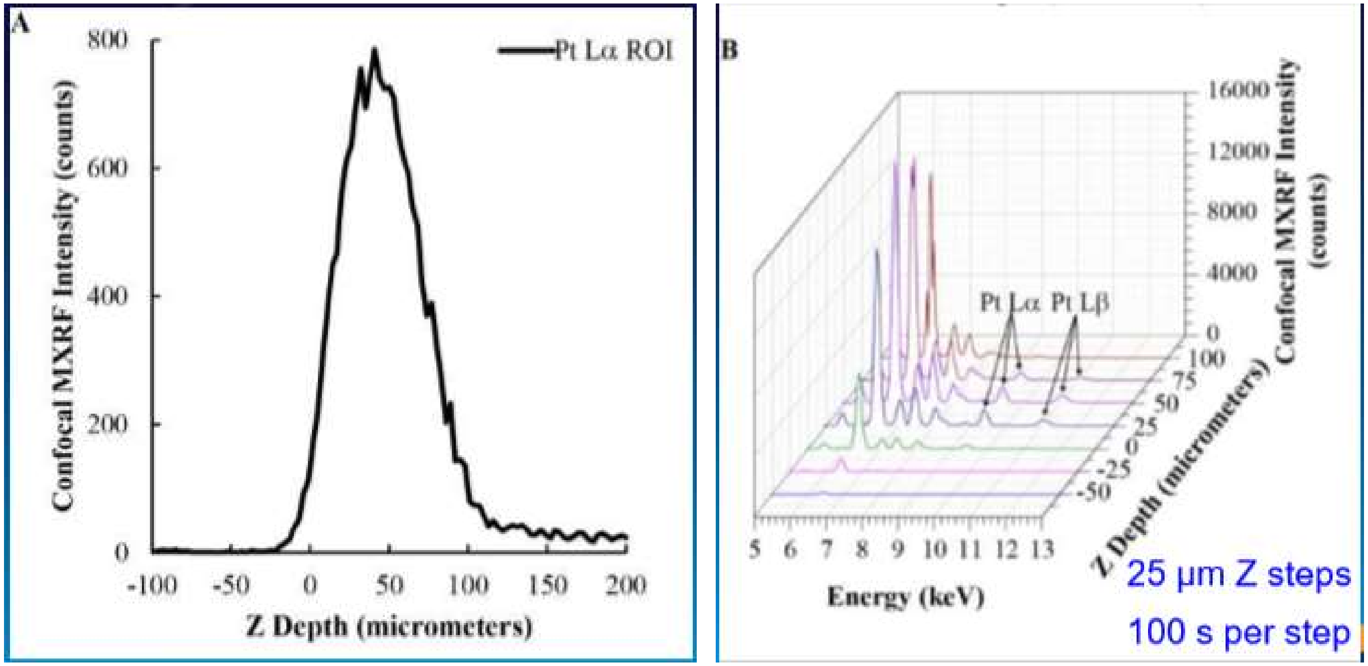
Applications- Confocal XRF



Dual polycapillary optics with 15um spatial resolution



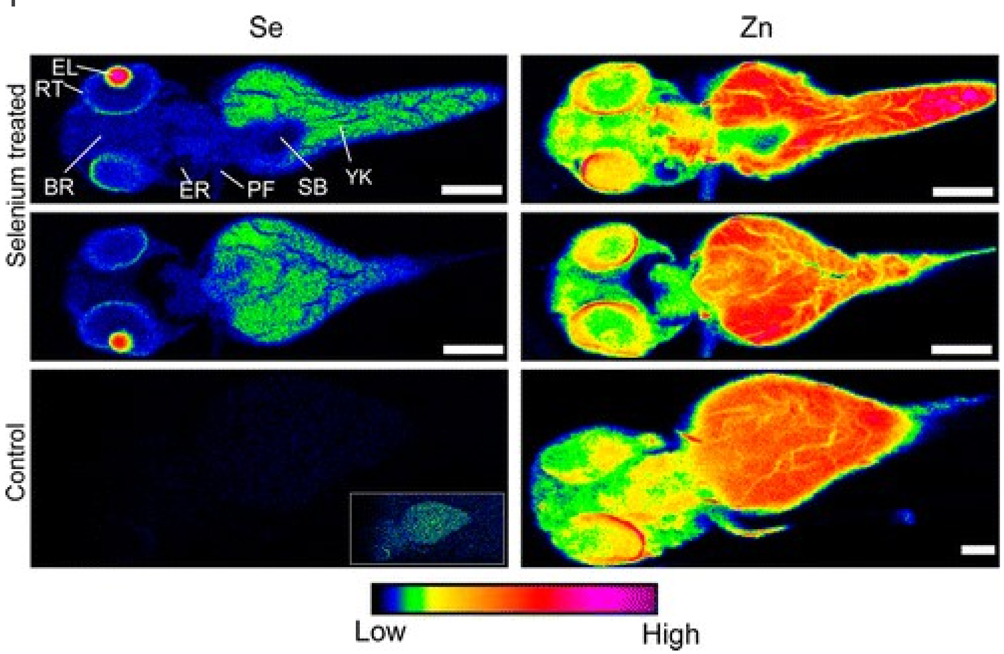
Confocal XRF system built by Osaka City University, Japan; Prof. Kouichi Tsuji



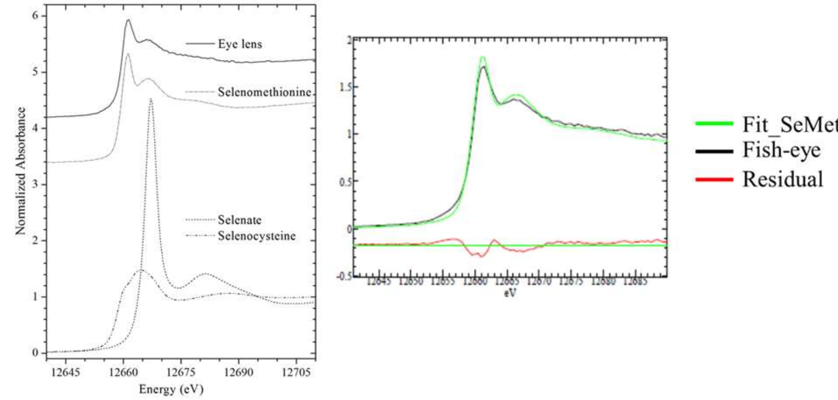
Depth Profile and Spectra of particle A using Confocal XRF from George Havrilla at LANL

Applications- Confocal XRF

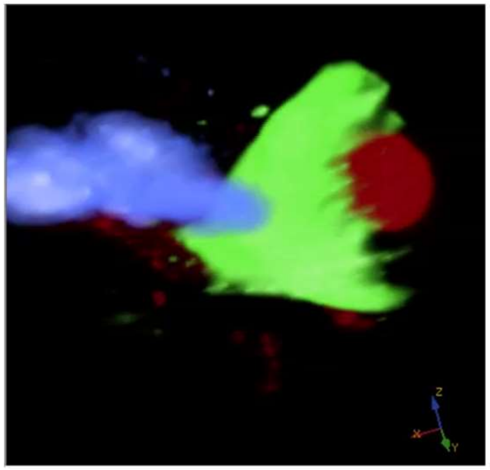
Motivation: understand tissue specific spatial distribution and speciation of Se in the early developmental stage of fish using confocal XRF powered by Kb mirror and polycapillary optic.



Se preferentially accumulated to highest level in the eye-lens, with lower level in the retina, yolk and other tissues.

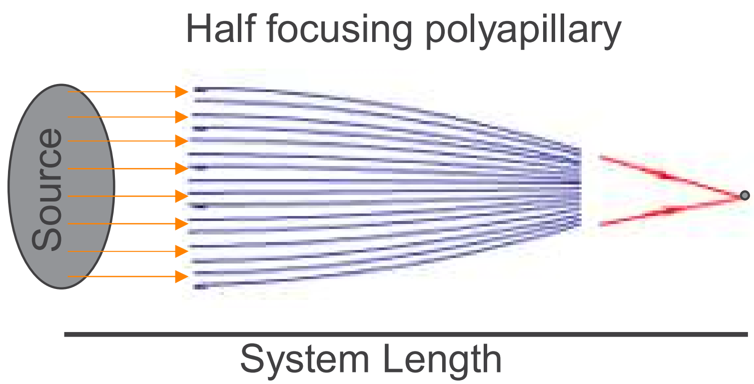


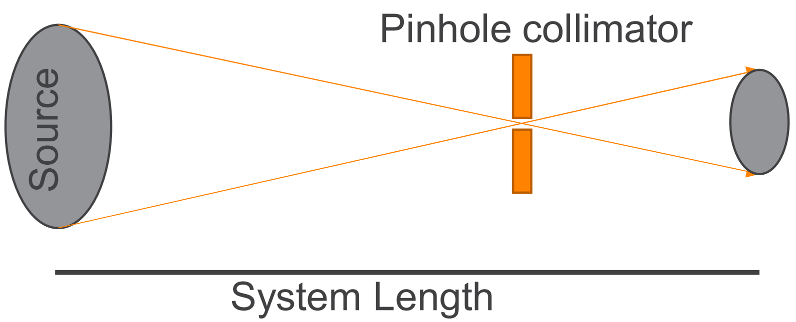
Chemical form of eye-lens selenium is selenomethionine-like.



Courtesy Zou Finfrock, Canadian Light Source

Applications- Large Source Polycapillary Optic





- Small size pinhole (less than 50um) is not easy to make!
- Pinhole needs certain thickness to block the unwanted radiation
- The pinhole becomes a “pipe”
- No working space due to the beam divergence
- Polycapillary optic provides the solution but the efficiency will be very low.

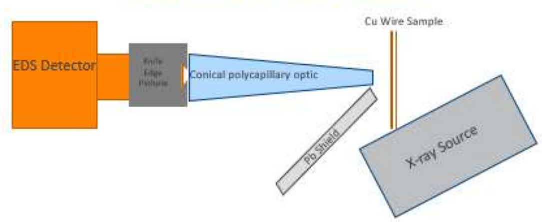
Applications- Conical Optic Imaging

What is a conical optic and why do we care?

- This is a polycapillary optic in the shape of a cone
- These optics offer benefits for imaging applications.
- The significance of this effect depends on the experimental conditions and the optic design
- Application Impact: enhanced intensity and resolution.

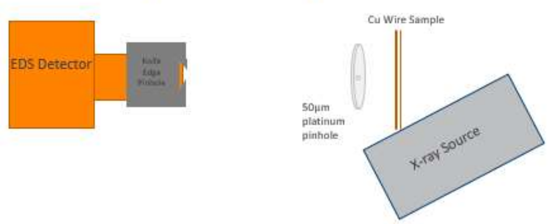
Optic Imaging Experiment

Experiment Setup Sketch

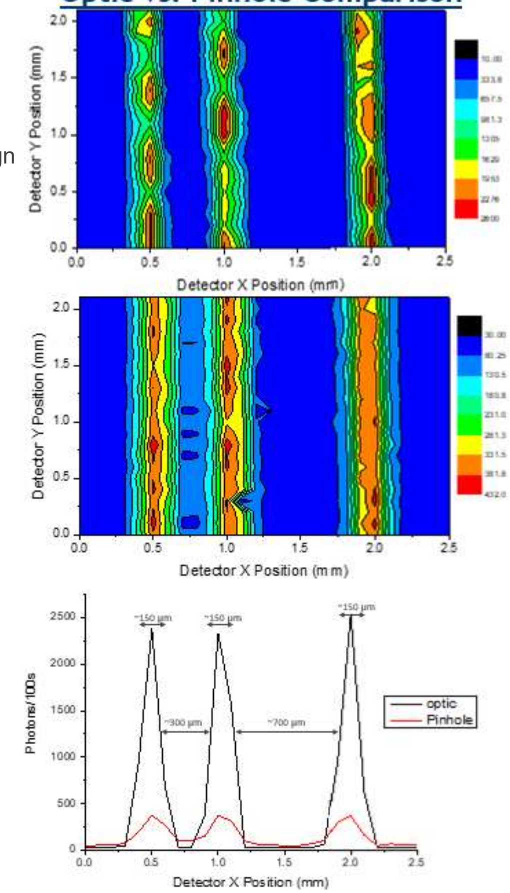


Pinhole Imaging Experiment

Experiment Setup Sketch



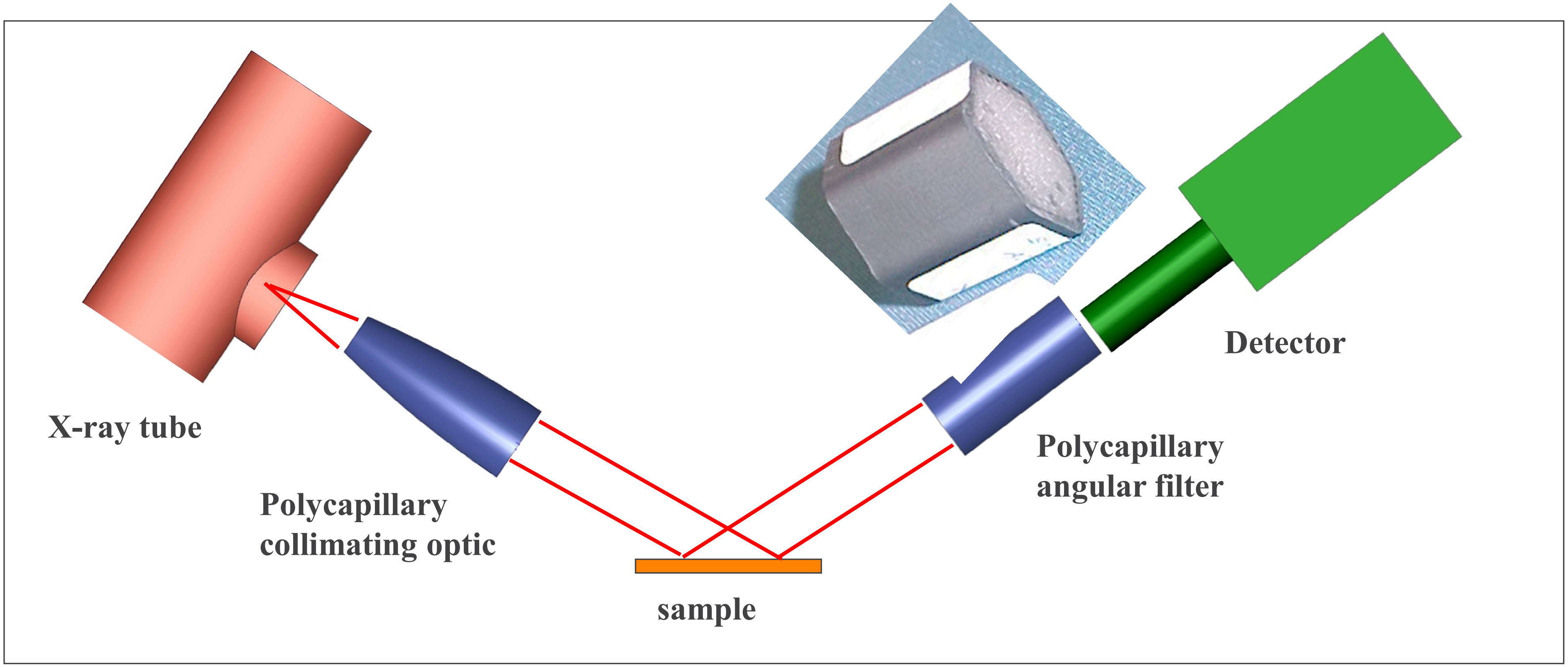
Optic vs. Pinhole Comparison



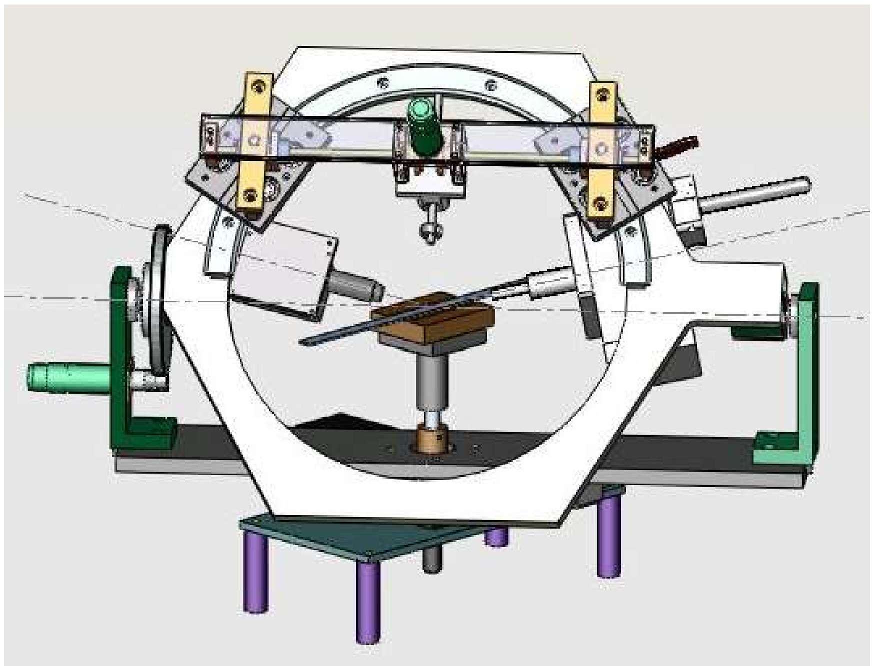
SUMMARY

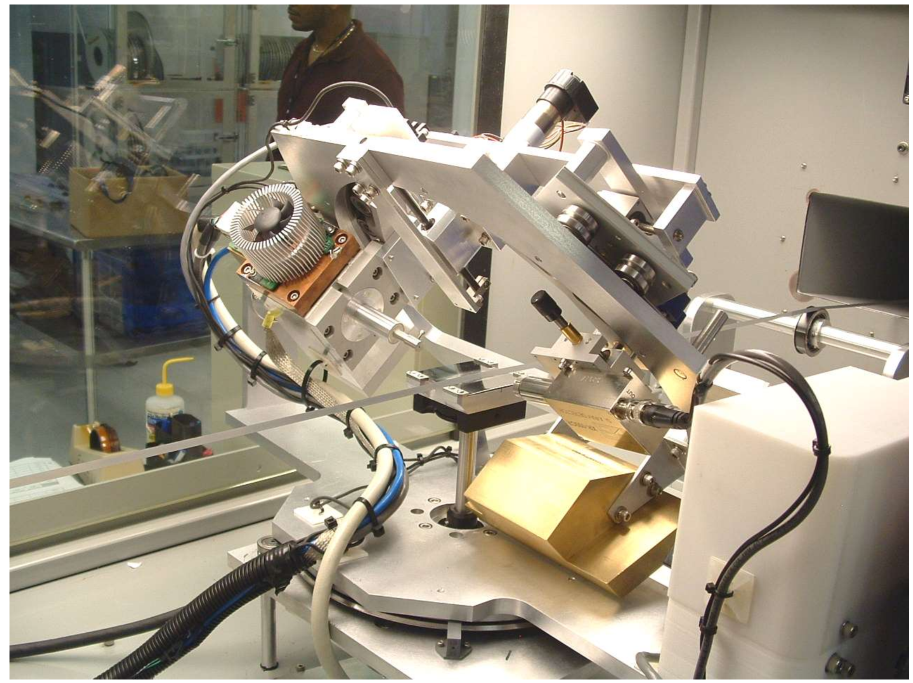
- Shown in the 2D maps and line scan comparison, we can observe:
  - The size of the 50µm Cu wire is now on the order of 3X the size.
  - The size of the spaces between the 50µm Cu wire are also on the order of 3X the size they were originally.
    - Between wire 1 and 2 there was a space of 138µm before the optic → ~300µm after the optic
    - Between wire 2 and 3 there was a space of 234µm before the optic → ~700µm after the optic.
  - Additionally, the comparison of intensity, with the same power parameters, is showing that the optic produces ~6x that of the pinhole.

Applications- Parallel Beam X-Ray Diffraction (PB-XRD)



Applications- PB-XRD





- PB-XRD System for in-situ texture measurement of superconductor tape
- Precise angle adjustment in three orthogonal directions
- Allows Theta-Theta scan, Phi scan and Chi scan
- Can measure texture of different thin films

Success stories of polycapillary optic technology

- Polycapillary optics are used in the majority of high-end, commercial, micro XRF instruments because of the added performance capabilities.
- One of the most successful applications of polycapillary optic enhances systems is Microelectronics.
- Polycapillary based micro XRF instruments are excellent tools for quality and process control
- The plating thickness gauge application provides cost savings and is one of main reasons that the end users seek the technology.
- The fast measurement speed is capable from the polycapillary optic and enabled by the developments of high-speed detector technologies.

Polycapillary X-ray Optic Enabled Sub-System

- Micro focus X-ray source integrated with polycapillary optic.
- Optimal alignment achieved in factory with comprehensive stability test.
  - <0.5% RSD/°C over 8 hours
- Source/optic is capable of easy exchange for service of unit.
- Built in safety shutter, cooling fan and primary 8 position filter wheel.
- PCS50 controller offers precise command of fleX-Beam and custom settings.

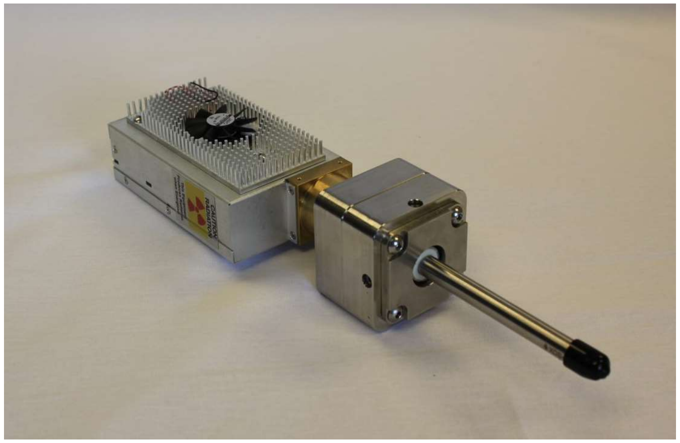
Highly-Focusing Optics						
Working distance (mm)	2	4	9	20	50	100
Focal spot size* (µm, FWHM)	8	15	25	45	100	180
Output beam intensity* (photons/s)	3.5×10^7	7.0×10^7	1.5×10^8	2.0×10^8	3.0×10^8	4.0×10^8

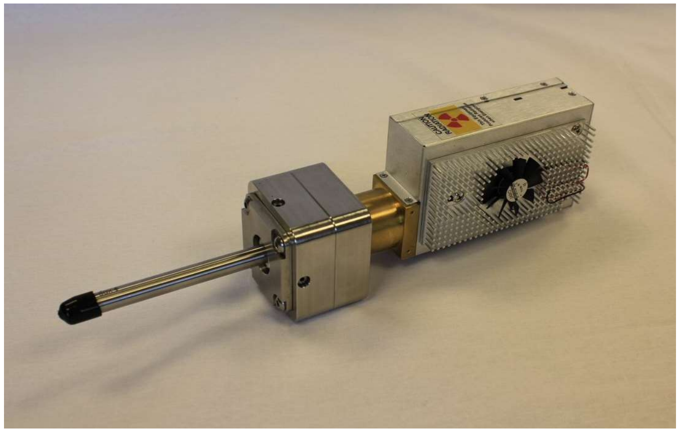
Typical Applications: Micro XRF
- Small Feature Analysis
- Film & Plating Thickness
- High-Resolution Elemental Mapping

** Note: For Mo Kα radiation using a 100µm, Mo-anode x-ray source at 50 kV/1mA*

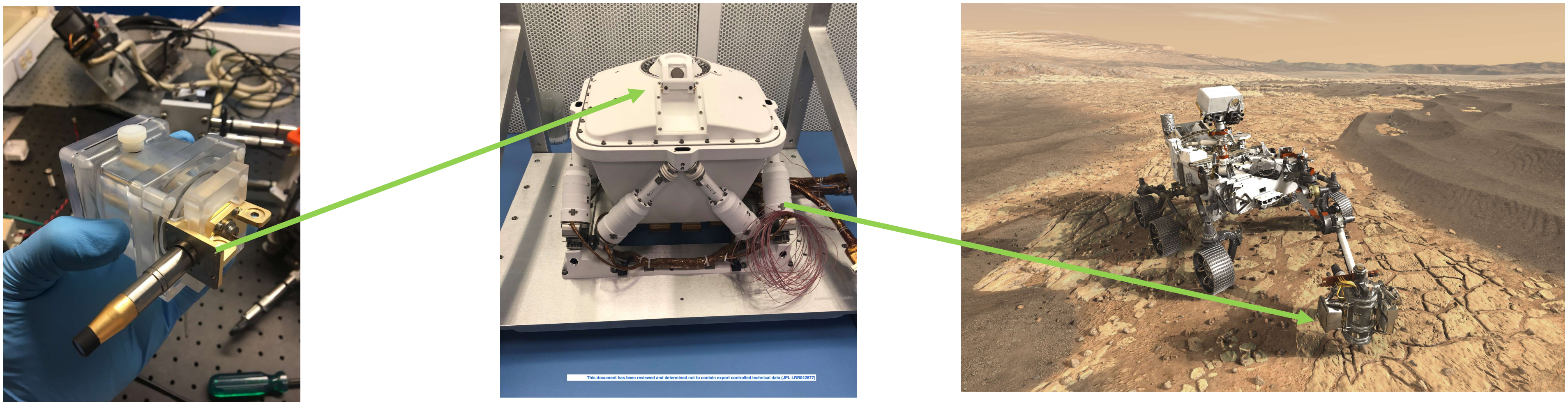
Polycapillary X-ray Optic Enabled Sub-System

- Small package of integrated X-ray source, power supply, controller and polycapillary optic.
- Source is capable of easy exchange for service of unit.
- Optic is capable of easy exchange for multiple applications.
- USB communication with PC interface
- Single primary filter slot integrated
- Optimal alignment achieved in factory with comprehensive stability test
- Originally designed for JPL NASA science team for Mars 2020 Project.
- Commercially available Q3, 2020





XOS Polycapillary Optic on Mars



- A polycapillary focusing optic will be used in the PIXL (Planetary Instrumentation for X-ray Lithochemistry) Sensor, built by JPL
- PIXL is one of seven scientific instruments on the Mars Rover to be launched in 2020
- The use of the polycapillary optic allows the instrument to identify chemical elements at fine scale with a milliwatt x-ray source
- Robust XRF head motion control design to allow precise and reliable long-term operation

Summary

- The polycapillary x-ray optic is an excellent multi-functional device that have been successfully used in a variety of applications.
- fleX-Beam is the solution for easy source/optic integration.
- Having a clear understanding of an x-ray optical system is important for both the system designers and the end users.
- All products are custom designed to provide the most optimized performance for the application of interest.
- If you have any interest in XOS polycapillary x-ray optics or the complete fleX-Beam sub-system, please reach out to info@xos.com.

THANK YOU

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