Today's outline - September 21, 2021

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- Beamtime at MRCAT

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- Writing a General User Proposal

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- Reflectivity research topics

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- Mirrors

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Reading Assignment: Chapter 3.9–3.10

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- Mirrors

Reading Assignment: Chapter 3.9–3.10

Homework Assignment #03:
Chapter 3: 1,3,4,6,8
due Tuesday, October 05, 2021

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- Beamtime at MRCAT
- Writing a General User Proposal
- Reflectivity research topics
- Mirrors

Reading Assignment: Chapter 3.9–3.10

Homework Assignment #03:
Chapter 3: 1,3,4,6,8
due Tuesday, October 05, 2021

Homework Assignment #04:
Chapter 4: 2,4,6,7.10
due Tuesday, October 19, 2021

Beamtime at MRCAT

September 25–28, I will be running the MRCAT bending magnet line for *ex situ* EXAFS measurements of some battery electrodes. My plan is to have an open, recorded session on one of those days where any of you can join to observe. There are two possible options for this session.

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Saturday, September 25, 2021 – my preference

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Saturday, September 25, 2021 – my preference

Monday, September 27, 2021 – alternative choice

Inform me if you intend to come to the session and which day is preferable.

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Monday, September 27, 2021 – alternative choice

Inform me if you intend to come to the session and which day is preferable.

We will do flux measurements, x-ray absorption spectroscopy measurements, use ion chambers and possibly the 4 element SDD detector for fluorescence measurements

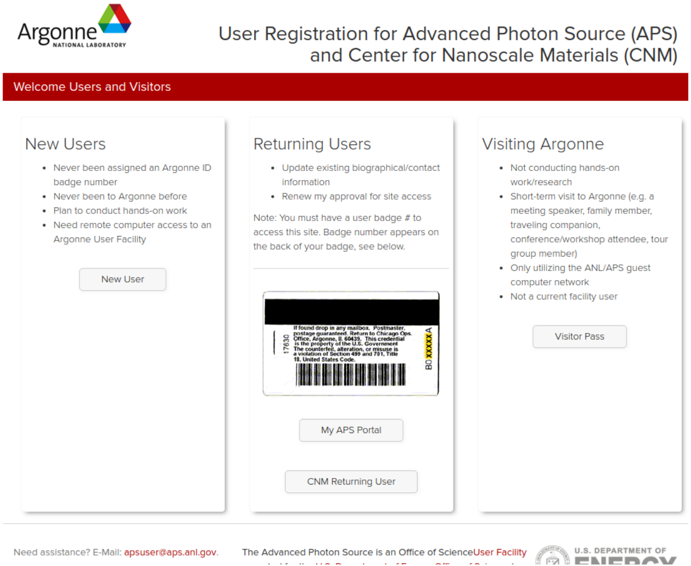
Writing a General User Proposal

1. Log into the APS site
2. Start a general user proposal
3. Add an Abstract
4. Choose a beam line
5. Answer the 6 important questions

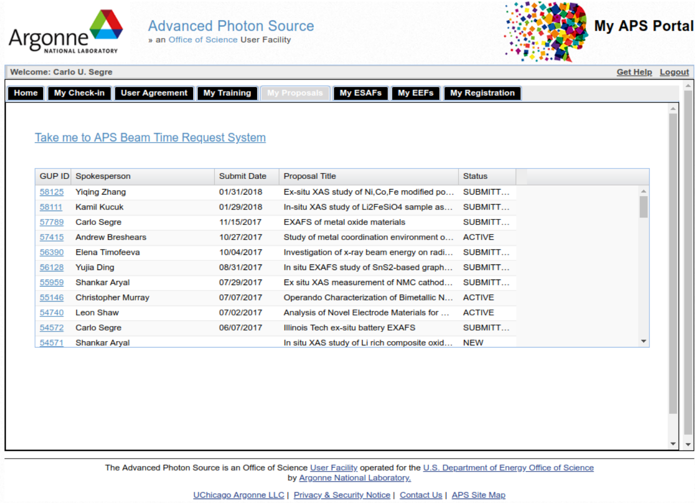
A tutorial can be found on the course home page

http://csrri.iit.edu/∼segre/phys570/21F/gu_proposal.html

Register & log into the APS Portal



APS Portal details



Argonne NATIONAL LABORATORY

Advanced Photon Source

» an Office of Science User Facility

My APS Portal

Welcome: Carlo U. Segre   Get Help Logout

Home | My Check-in | User Agreement | My Training | My Proposals | My ESAFs | My EEFs | My Registration

Take me to APS Beam Time Request System

GUP ID	Spokesperson	Submit Date	Proposal Title	Status
58125	Yiqing Zhang	01/31/2018	Ex-situ XAS study of Ni,Co,Fe modified po…	SUBMITT…
58111	Kamil Kucuk	01/29/2018	In-situ XAS study of Li2FeSiO4 sample as…	SUBMITT…
57789	Carlo Segre	11/15/2017	EXAFS of metal oxide materials	SUBMITT…
57415	Andrew Breshears	10/27/2017	Study of metal coordination environment o…	ACTIVE
56390	Elena Timofeeva	10/04/2017	Investigation of x-ray beam energy on radi…	SUBMITT…
56128	Yujia Ding	08/31/2017	In situ EXAFS study of SnS2-based graph…	SUBMITT…
55959	Shankar Aryal	07/29/2017	Ex situ XAS measurement of NMC cathod…	SUBMITT…
55146	Christopher Murray	07/07/2017	Operando Characterization of Bimetallic N…	ACTIVE
54740	Leon Shaw	07/02/2017	Analysis of Novel Electrode Materials for …	ACTIVE
54572	Carlo Segre	06/07/2017	Illinois Tech ex-situ battery EXAFS	SUBMITT…
54571	Shankar Aryal		In situ XAS study of Li rich composite oxid…	NEW

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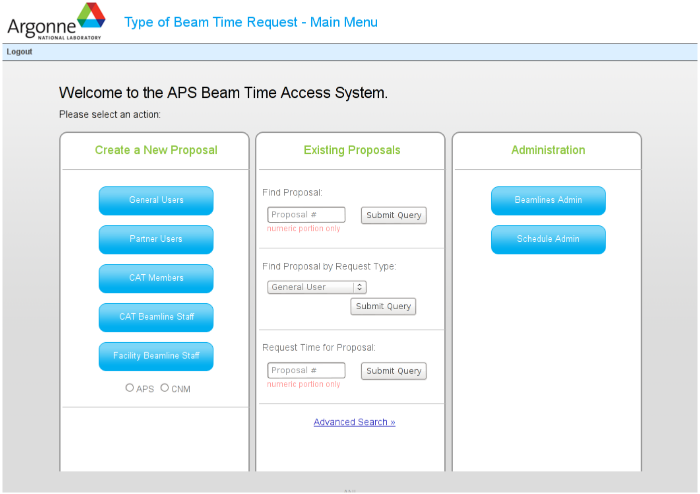
Start a General User Proposal



Add title & answer details

Argonne NATIONAL LABORATORY — APS - General User Proposal

Main Menu | Search Criteria | Instructions | Logout

General

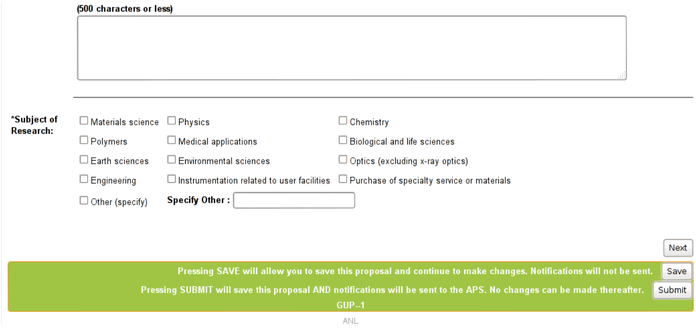
GUP--1
General User Program: gu_program@aps.anl.gov, 630-252-9090
Technical assistance: mis_mgrs@aps.anl.gov

*Proposal Title:

Shifts Recommended by PRP:	not available	Shifts Allocated by BAC or Scheduled by Beamline in current cycle	(0)	Shifts Used to date: (0)	Shifts Remaining: not available

Do you want this proposal to be considered for project status? description	Yes	No
Does this proposal require mail-in service?	Yes	No
*Does this research involve macromolecular crystallography (single crystals) ?	Yes	No
*Will the data collected be considered proprietary ?	Yes	No
*Will the data collected be considered classified ?	Yes	No
Does this research involve human subjects or materials ?	Yes	No
Does this research involve live animals?	Yes	No
*Are there known safety hazards associated with the proposed experimental procedures or your samples ?	Yes	No
*Does this research involve the use of radioactive samples/materials ?	Yes	No
*Does this research involve samples/materials that require a BSL-2 or BSL-3 facility?	Yes	No
Is this research required for a student's thesis ?	Yes	No
Have you spoken to a beamline staff member?	Yes	No
Does this proposal involve the use of a sample from the ATR library?	Yes	No
*Is this proposal related to another general user proposal ? If so, which one(s) and how ?	Yes	No

More details



(500 characters or less)

*Subject of Research:

- Materials science
- Physics
- Chemistry
- Polymers
- Medical applications
- Biological and life sciences
- Earth sciences
- Environmental sciences
- Optics (excluding x-ray optics)
- Engineering
- Instrumentation related to user facilities
- Purchase of specialty service or materials
- Other (specify)

Specify Other :

Next

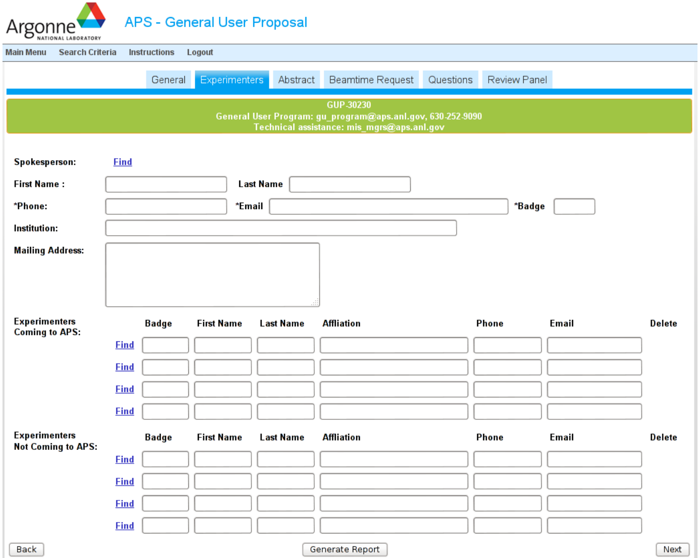
Pressing SAVE will allow you to save this proposal and continue to make changes. Notifications will not be sent. Save

Pressing SUBMIT will save this proposal AND notifications will be sent to the APS. No changes can be made thereafter. Submit

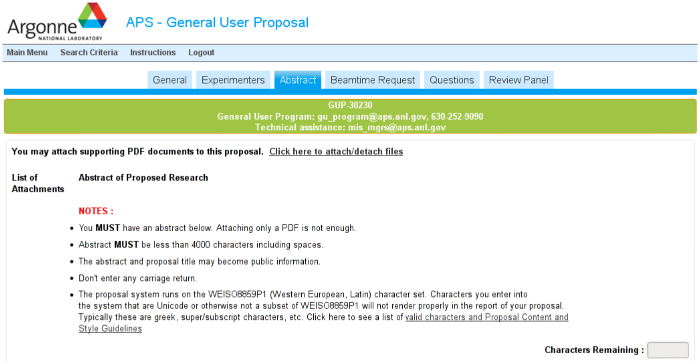
GUP--1

ANL

Select experimenters



Insert abstract


Argonne NATIONAL LABORATORY APS - General User Proposal

Main Menu Search Criteria Instructions Logout

General Experimenters Abstract Beamtime Request Questions Review Panel

GUP-30230
General User Program: gu_program@aps.anl.gov, 630-252-9090
Technical assistance: mis_mgrs@aps.anl.gov

You may attach supporting PDF documents to this proposal. Click here to attach/detach files

List of Attachments

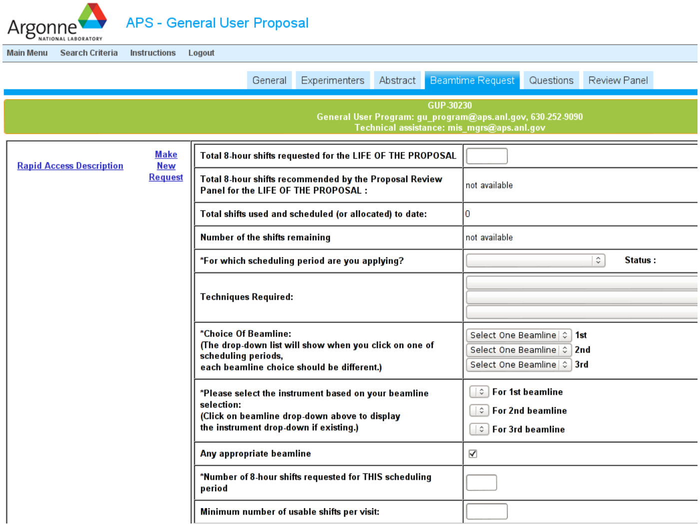
Abstract of Proposed Research

NOTES :

- You **MUST** have an abstract below. Attaching only a PDF is not enough.
- Abstract **MUST** be less than 4000 characters including spaces.
- The abstract and proposal title may become public information.
- Don't enter any carriage return.
- The proposal system runs on the WEISO8859P1 (Western European, Latin) character set. Characters you enter into the system that are Unicode or otherwise not a subset of WEISO8859P1 will not render properly in the report of your proposal. Typically these are greek, super/subscript characters, etc. Click here to see a list of valid characters and Proposal Content and Style Guidelines

Characters Remaining :

Make Beam Time Request



APS - General User Proposal

Main Menu | Search Criteria | Instructions | Logout

General | Experimenters | Abstract | Beamtime Request | Questions | Review Panel

GUP-30230
General User Program: gu_program@aps.anl.gov, 630-252-9090
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Rapid Access Description

Make New Request

Total 8-hour shifts requested for the LIFE OF THE PROPOSAL	
Total 8-hour shifts recommended by the Proposal Review Panel for the LIFE OF THE PROPOSAL :	not available
Total shifts used and scheduled (or allocated) to date:	0
Number of the shifts remaining	not available
*For which scheduling period are you applying?	Status :
Techniques Required:	
*Choice Of Beamline: (The drop-down list will show when you click on one of scheduling periods, each beamline choice should be different.)	Select One Beamline 1st Select One Beamline 2nd Select One Beamline 3rd
*Please select the instrument based on your beamline selection: (Click on beamline drop-down above to display the instrument drop-down if existing.)	For 1st beamline For 2nd beamline For 3rd beamline
Any appropriate beamline	☑
*Number of 8-hour shifts requested for THIS scheduling period	
Minimum number of usable shifts per visit:	

Beam Time Request continued

Do you have specific scheduling requirements ?	
What equipment is required ? What equipment will you bring ?	
Please list any new publications resulting from your work at the APS.	
Describe the progress made during your most recent beamtime. (2000 characters including spaces)	
Prefered Dates (MM/DD/YYYY)	From To to to to to
Unacceptable Dates (MM/DD/YYYY)	From To to to to to

Back Generate Report

Answer the 6 important questions

Argonne NATIONAL LABORATORY

APS - General User Proposal

Main Menu Search Criteria Instructions Logout

General | Experimenters | Abstract | Beamtime Request | Questions | Review Panel

GUP-30230
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Please specify the funding source(s) for your proposed research:

☐ DOD (specify)	☐ DOE, Office of Basic Energy Sciences	☐ DOE, Office of Biological and Environmental Research
☐ DOE, Other (specify)	☐ Foreign (specify)	☐ HHIH
☐ Howard Hughes Medical Institute (HHMI)	☐ Industry	☐ NASA
☐ NIH	☐ NSF	☐ Other U.S. Government
☐ USDA	☐ Other (specify)	**Specify Other:**

What is the scientific or technical purpose and importance of the proposed research? (limit : 500 words)

Answer the 6 important questions

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Why do you need the APS for this research? (limit: 100 words)

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Why do you need the beamline you have chosen? (limit: 100 words)

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Why do you need the beamline you have chosen? (limit: 100 words)

Describe the participants' previous experience with synchrotron radiation and the experimental results obtained. (If you refer to previous publications, be sure to include complete citations.) (limit: 100 words)

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Describe samples and explain the proposed experiment, including procedures. Explain the basis for your estimated beam time needs. (limit: 500 words)

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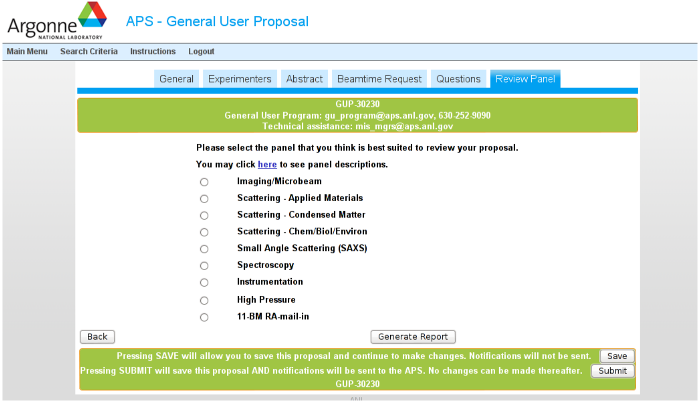
Describe samples and explain the proposed experiment, including procedures. Explain the basis for your estimated beam time needs. (limit: 500 words)

Provide an overall estimate of the amount of beam time you will need to accomplish the goals of your proposed experimental program. How many visits during the two-year proposal period do you expect to need? How many shifts will you need during each visit (approximately)? (limit: 500 words)

Select the review panel



Layering in liquid films

TEHOS, tetrakis–(2-ethylhexoxy)–silane, a non-polar, roughly spherical molecule, was deposited on Si(111) single crystals

C.-J. Yu et al., "Observation of molecular layering in thin liquid films using x-ray reflectivity", *Phys. Rev. Lett.* **82**, 2326–2329 (1999).

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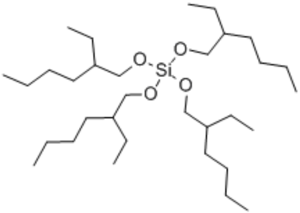

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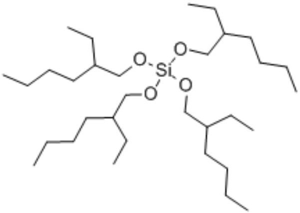

Specular reflection measurements were made at MRCAT (Sector 10 at APS) and at X18A (at NSLS).

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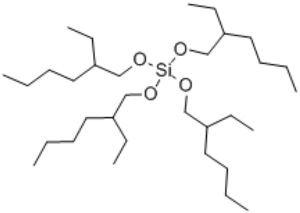

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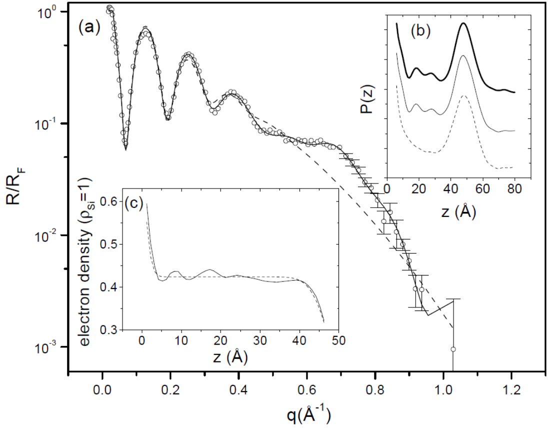

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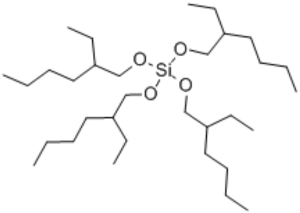

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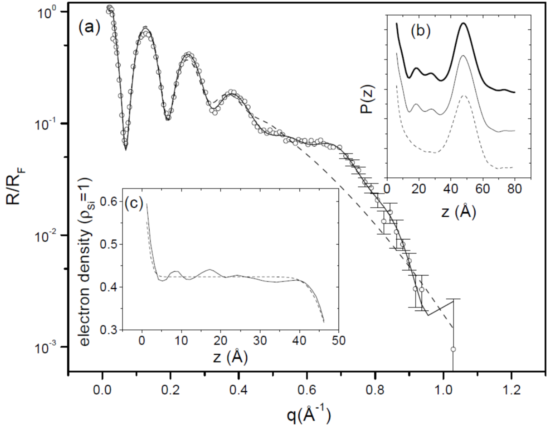

Deviations from uniform density are used to fit experimental reflectivity

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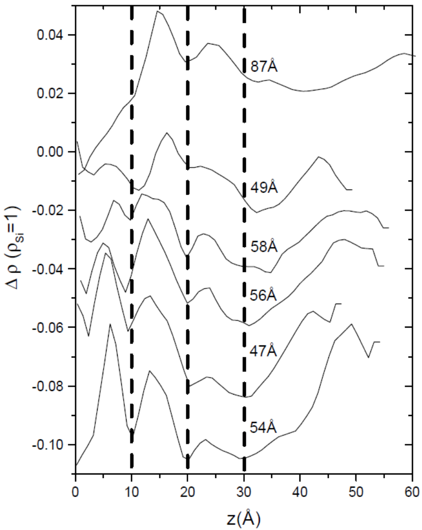
Layering in liquid films



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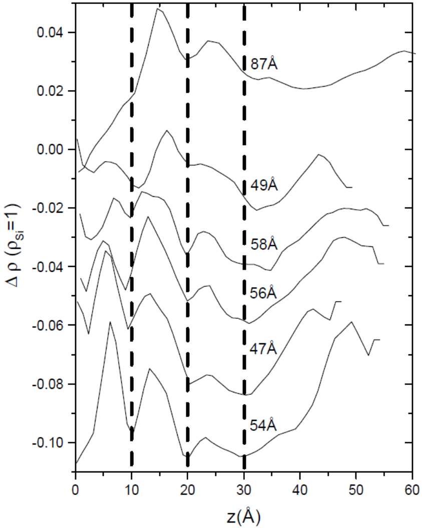

The peak below 10Å appears in all but the thickest film and depends on the interactions between film and substrate.

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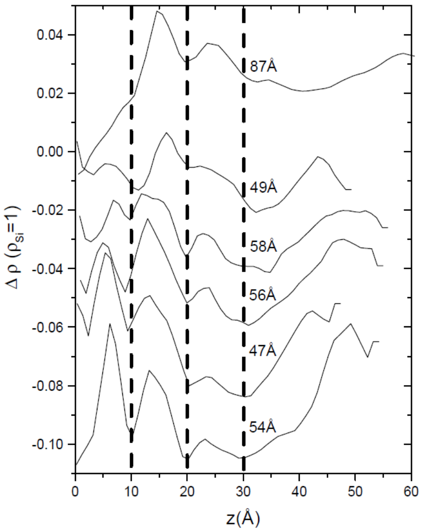

The peak below 10Å appears in all but the thickest film and depends on the interactions between film and substrate.

There are always peaks between 10-20Å and 20-30Å and a broad peak at the free surface showing the presence of ordered layers of molecules.

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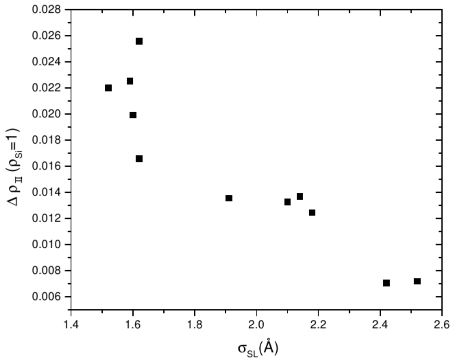

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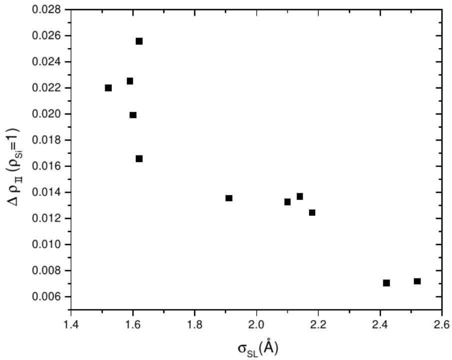

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There are always peaks between 10-20Å and 20-30Å and a broad peak at the free surface showing the presence of ordered layers of molecules.

The authors conclude that the presence of a hard smooth surface is required for ordering and therefore deviations from an ideal, isotropic liquid.

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Film growth kinetics

The goal of this project was to understand the evolution of surface roughness during the growth of a silver thin film.

C. Thompson et al., "X-ray-reflectivity study of the growth kinetics of vapor-deposited silver films," *Phys. Rev. B* **49**, 4902–4907 (1994).

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5 deposition with thicknesses varying from 10 nm to 150 nm were studies

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Gaussian roughness profile with a "roughness" exponent $0 < h < 1$.

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Gaussian roughness profile with a "roughness" exponent $0 < h < 1$. As the film is grown by vapor deposition, the rms width σ, grows with a "growth exponent" β and the correlation length in the plane of the surface, ξ evolves with the "dynamic" scaling exponent, $z_s = h/\beta$.

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$$g(r) \propto r^{2h} \qquad \sigma \propto t^{\beta}$$

$$\xi \propto t^{1/z_s} \qquad \langle h \rangle \propto t$$

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Gaussian roughness profile with a "roughness" exponent $0 < h < 1$. As the film is grown by vapor deposition, the rms width σ, grows with a "growth exponent" β and the correlation length in the plane of the surface, ξ evolves with the "dynamic" scaling exponent, $z_s = h/\beta$.

$$g(r) \propto r^{2h} \qquad \sigma \propto t^{\beta}$$

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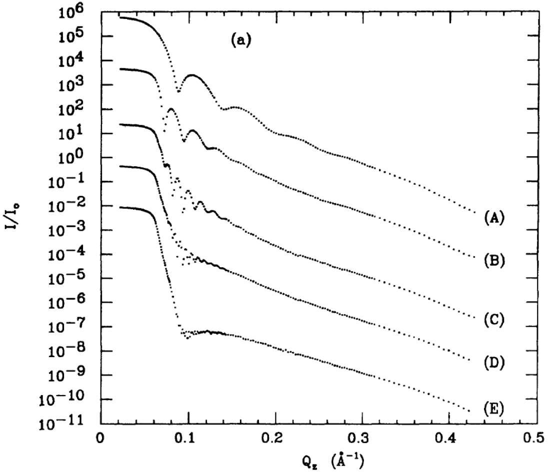

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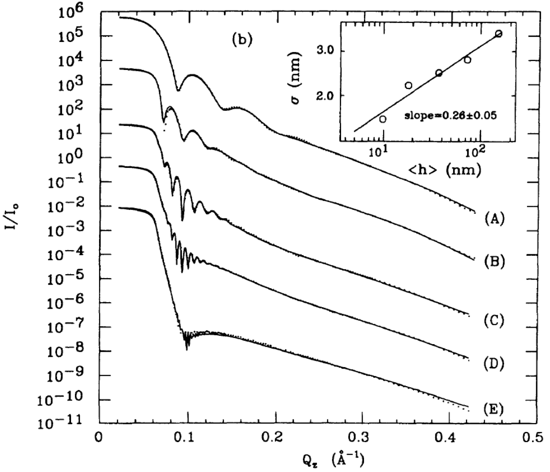

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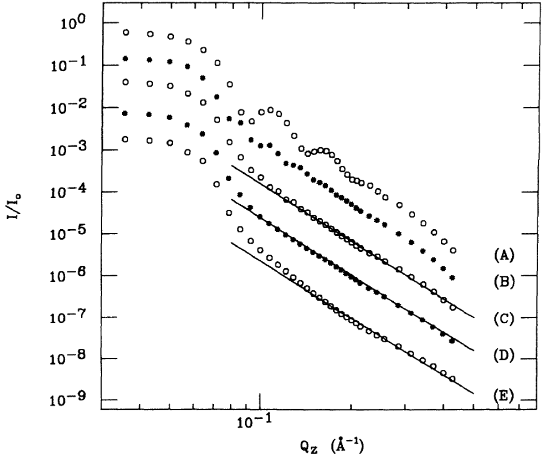

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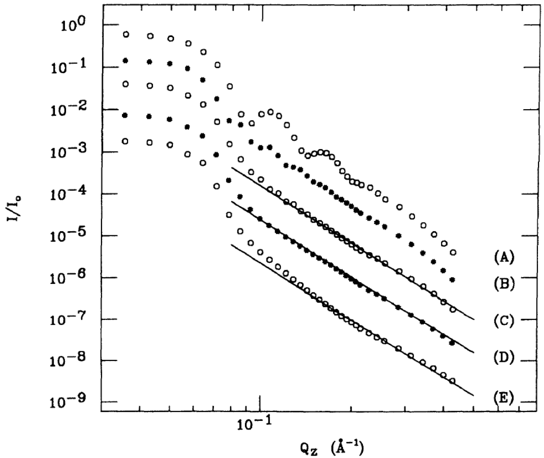

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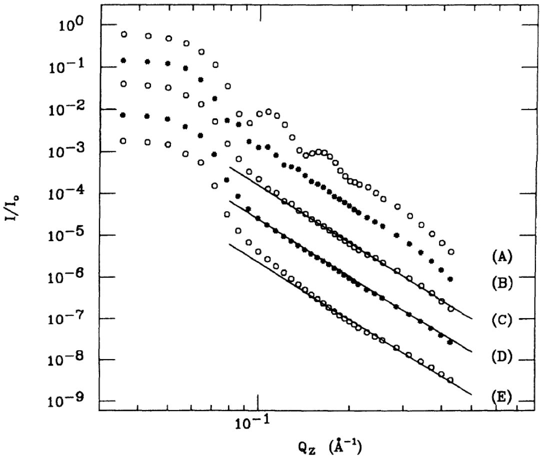

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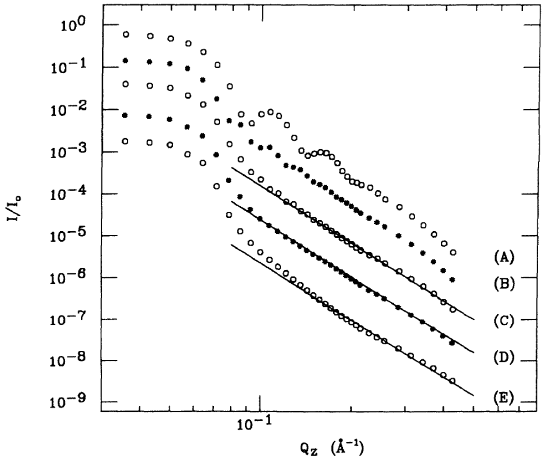

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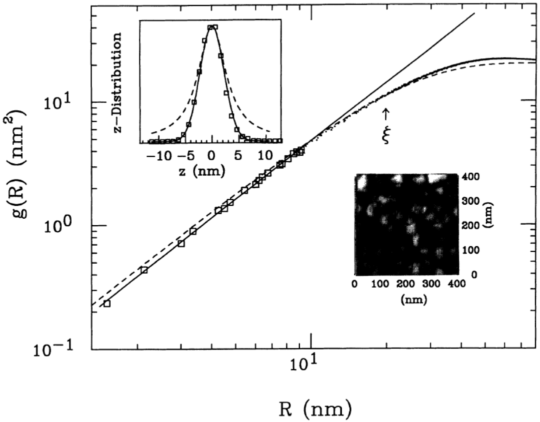

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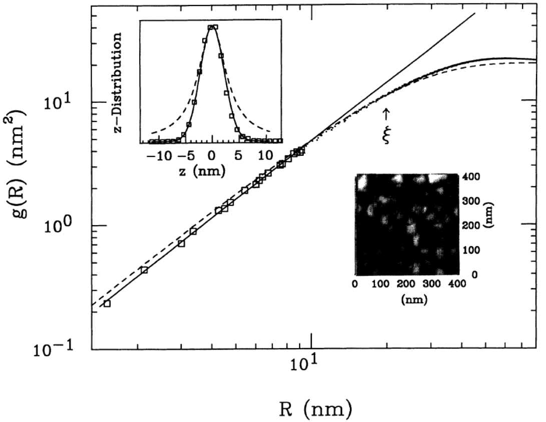

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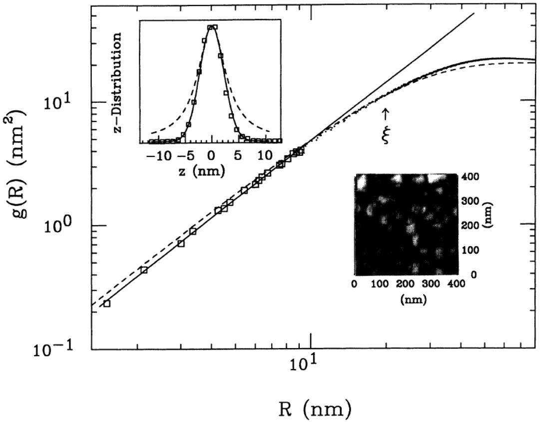

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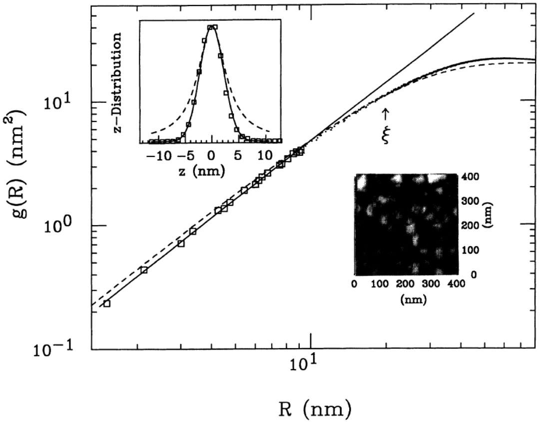

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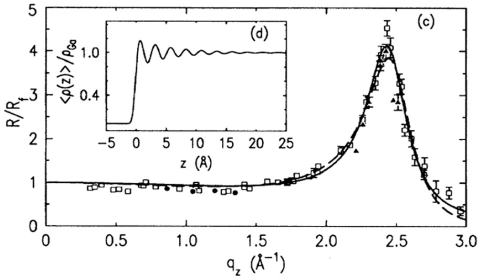

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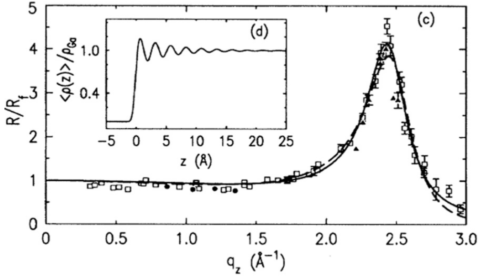

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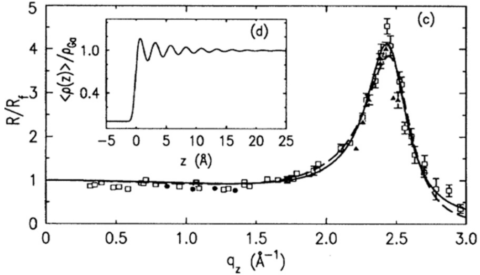

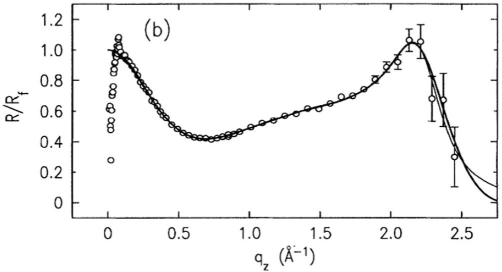

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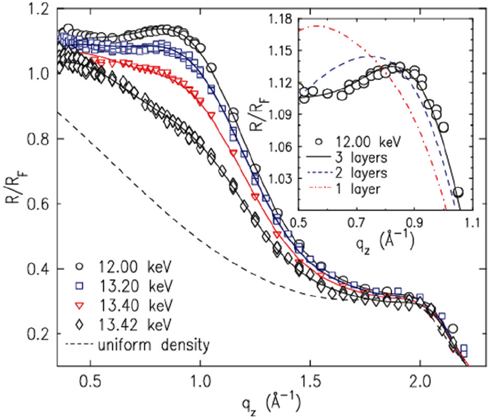

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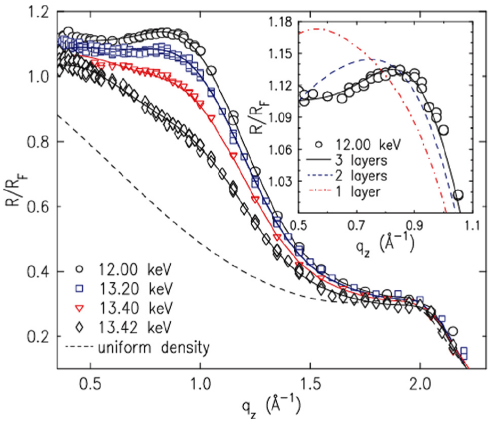
surface layer is rich in Bi (95%), second layer is deficient (25%), and third layer is rich in Bi (53%) once again



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Tangential focusing mirror

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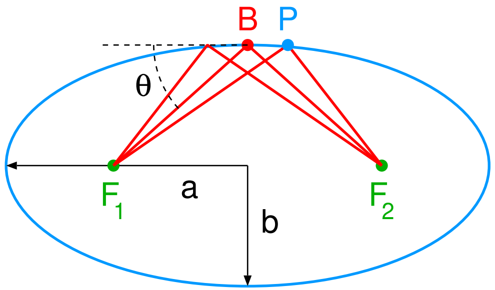

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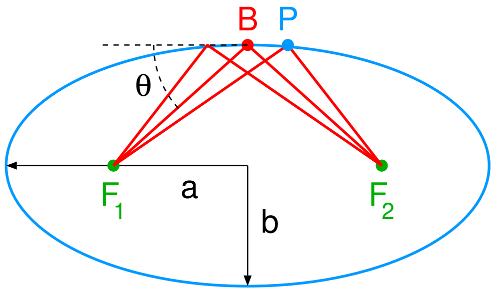
The shape of an ideal mirror is an ellipse, where any ray coming from one focus will be projected to the second focus. Consider a 1:1 focusing mirror. For an ellipse the sum of the distances from any point on the ellipse to the foci is a constant.

$$F_1P + F_2P = 2a$$



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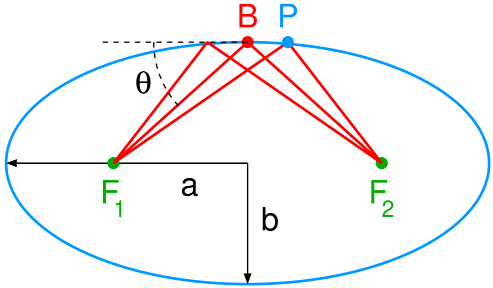

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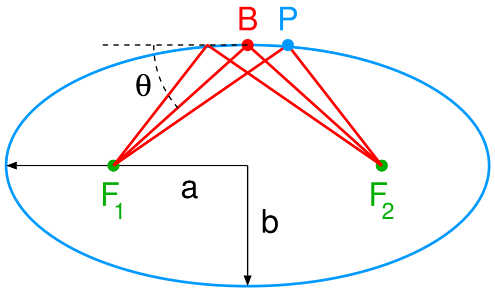

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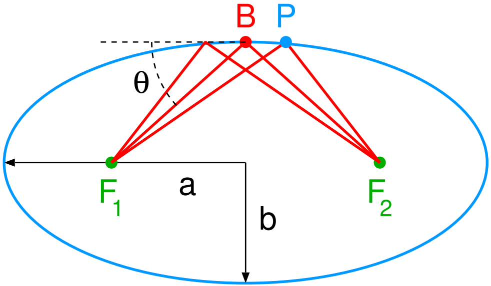

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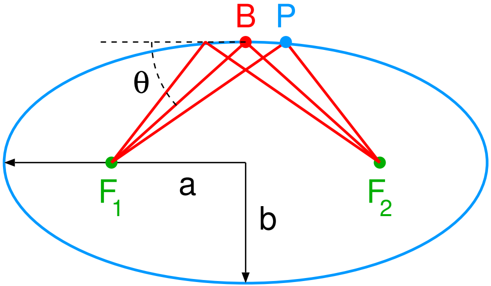

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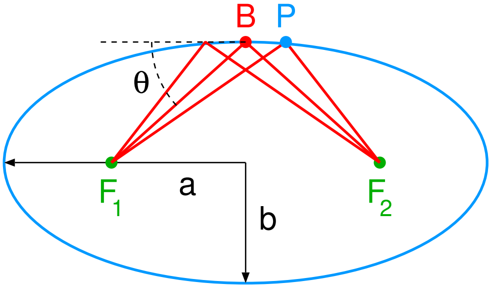

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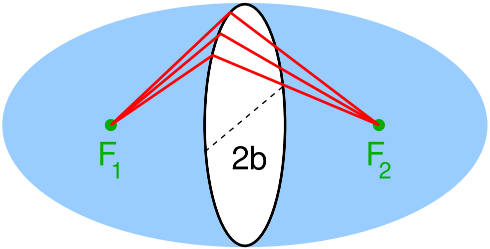
Saggital focusing mirror

Ellipses are hard figures to make, so usually, they are approximated by circles. In the case of saggital focusing, an ellipsoid of revolution with diameter $2b$, is used for focusing.



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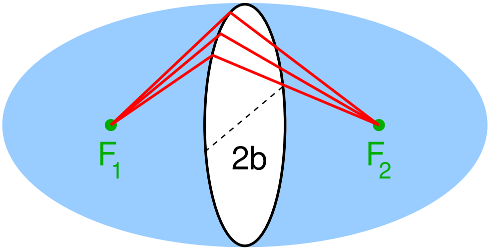

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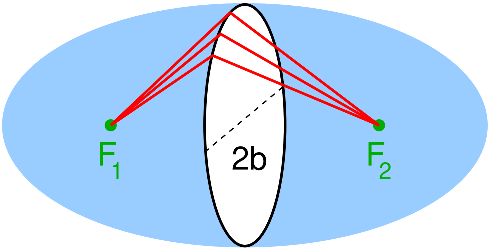

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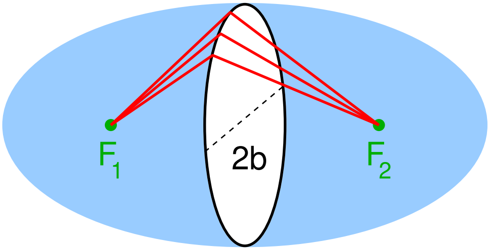

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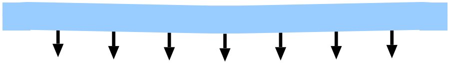

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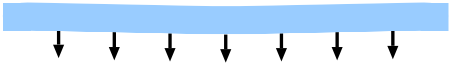

A cost effective way to focus in both directions is a toroidal mirror which has a fixed bend in the transverse direction but which can be bent longitudinally to change the vertical focus.

Dual focusing options

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- Toroidal mirror — simple, moderate focus, good for initial focusing element, easy to distort beam
- Saggittal focusing crystal & vertical focusing mirror — adjustable in both directions, good for initial focusing element

Dual focusing options

- Toroidal mirror — simple, moderate focus, good for initial focusing element, easy to distort beam
- Saggittal focusing crystal & vertical focusing mirror — adjustable in both directions, good for initial focusing element
- Kirkpatrick-Baez mirror pair — in combination with an initial focusing element, good for final small focal spot and variable energy

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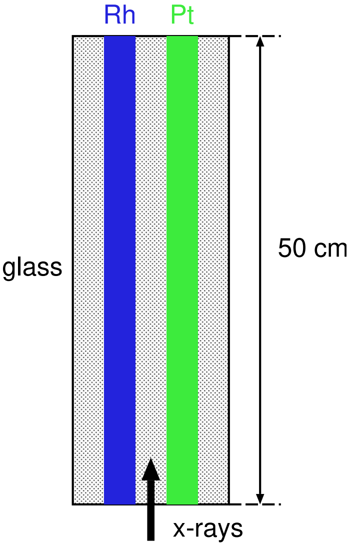
- Zone plates — in combination with an initial focusing element, gives smallest focal spot, but hard to vary energy
- Refractive lenses — good final focus, focus moves with energy, significant attenuation and hard to change focal length

The MRCAT mirror



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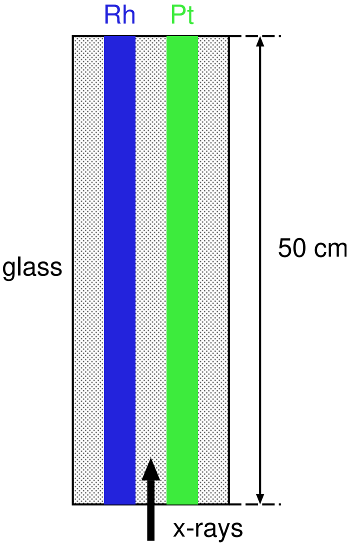

Ultra low expansion glass polished to a few Å roughness

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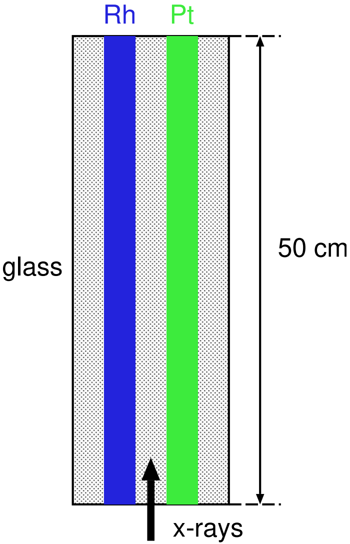

Ultra low expansion glass polished to a few Å roughness

One platinum stripe and one rhodium stripe deposited along the length of the mirror on top of a chromium buffer layer

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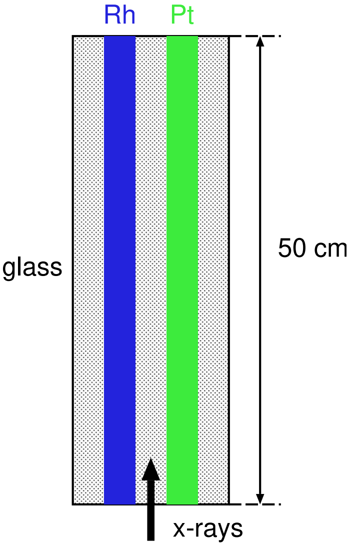

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A mounting system which permits angular positioning to less than 1/100 of a degree as well as horizontal and vertical motions

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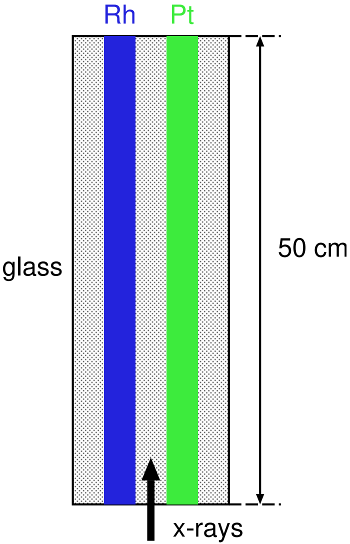

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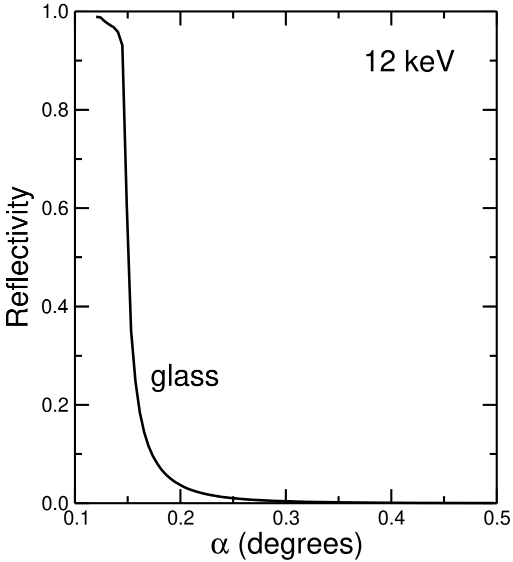
A bending mechanism to permit vertical focusing of the beam to $\sim 60\ \mu\text{m}$

Mirror performance

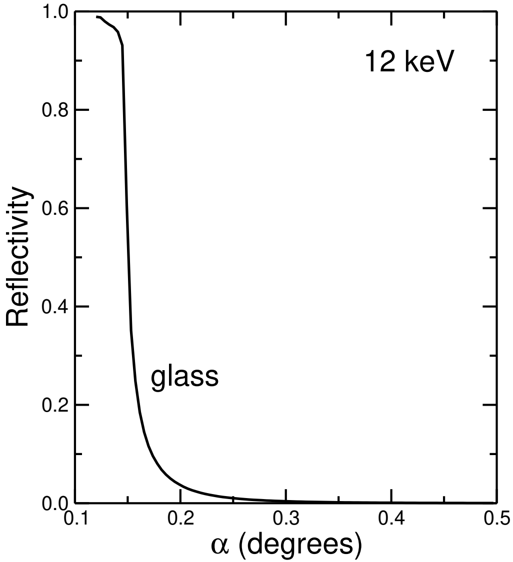
When illuminated with 12 keV x-rays on the glass "stripe", the reflectivity is measured as:



Mirror performance

When illuminated with 12 keV x-rays on the glass "stripe", the reflectivity is measured as:

With the Rh stripe, the thin slab reflection is evident and the critical angle is significantly higher.



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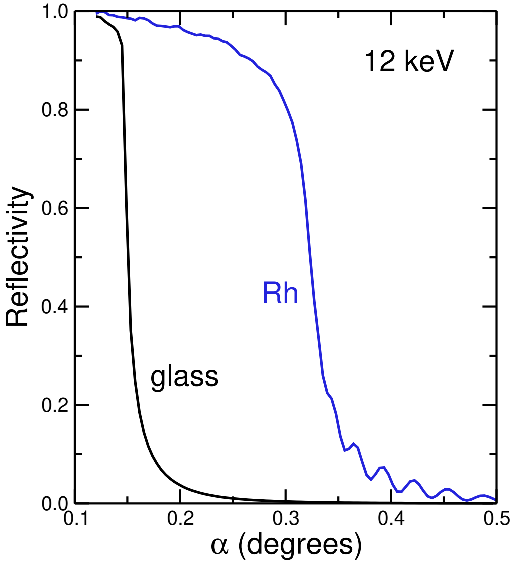

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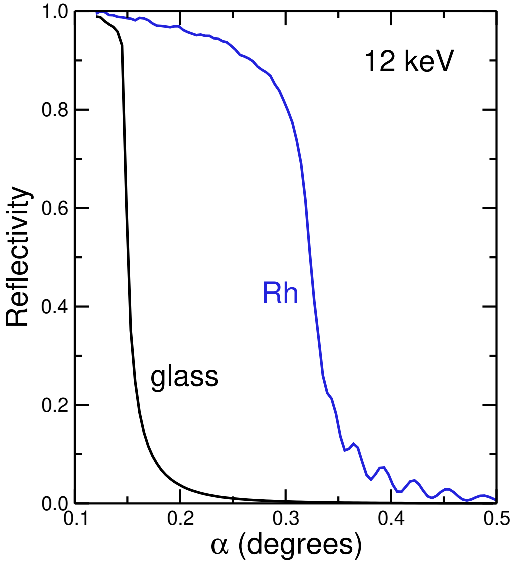
The Pt stripe gives a higher critical angle still but a lower reflectivity and it looks like an infinite slab.



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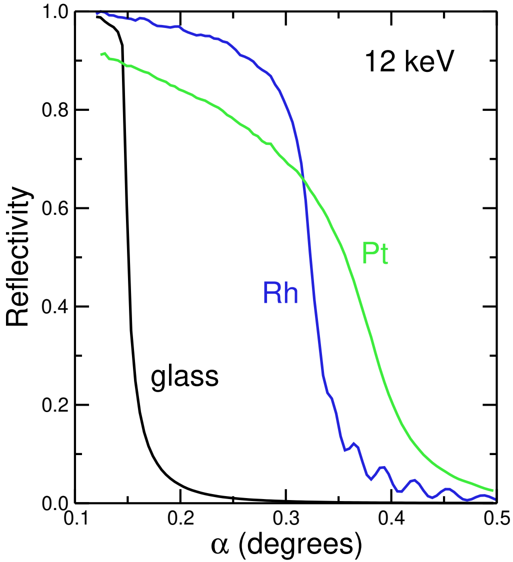

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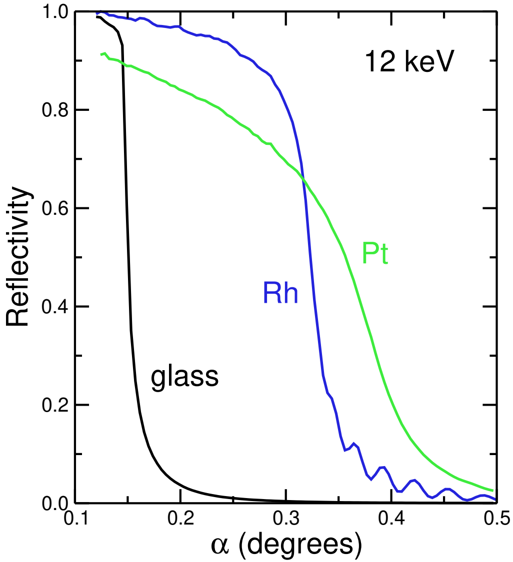
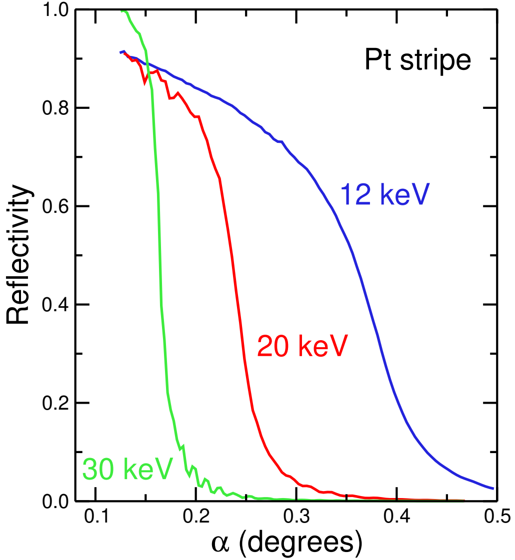
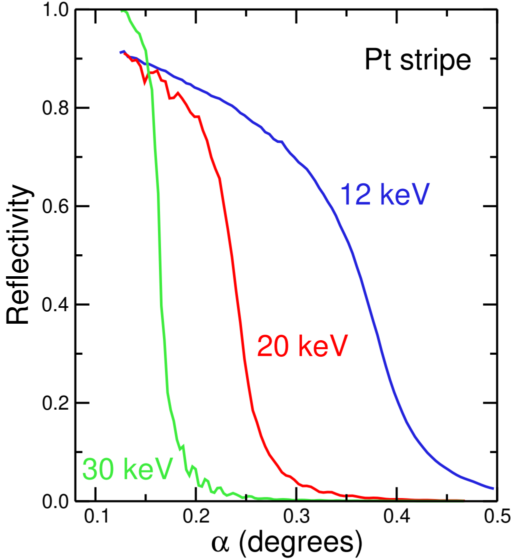
Why?



Mirror performance (cont.)

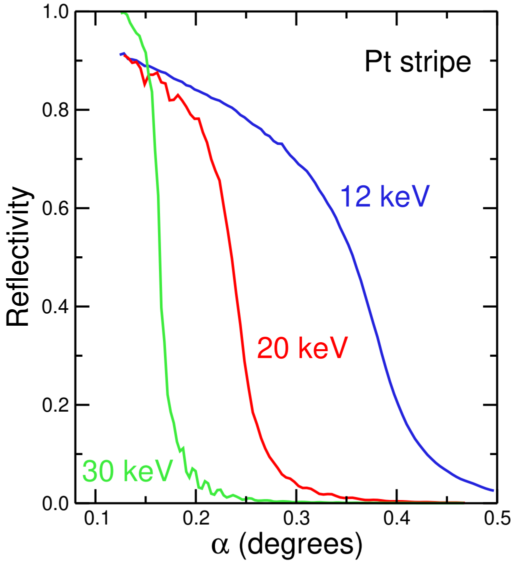


Mirror performance (cont.)



As we move up in energy the critical angle for the Pt stripe drops.

Mirror performance (cont.)



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The reflectivity at low angles improves as we are well away from the Pt absorption edges at 11,565 eV, 13,273 eV, and 13,880 eV.

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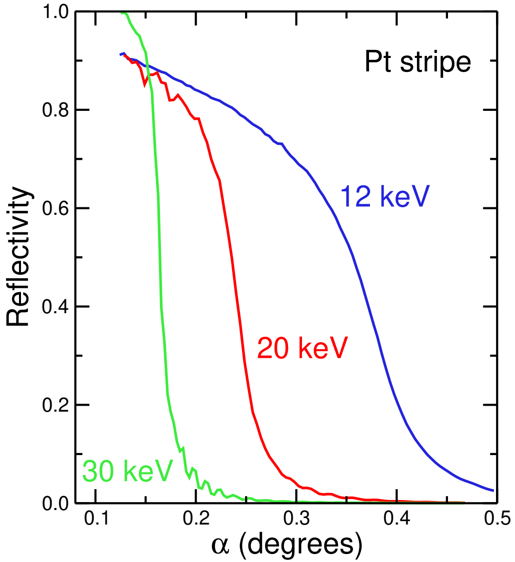

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As energy rises, the Pt layer begins to show the reflectivity of a thin slab.