Thin Films for X-ray Optics

Ray Conley
June 2017
Synchrotron Optics

Two basic goals: focusing
Synchrotron Optics

Two basic goals: and energy selection

Visible Light

X-rays
Interaction of X rays with matter

Optical index \( n = 1 - \delta + i\beta \leq 1 \)

Snell’s law \( \frac{n_1}{n_2} = \frac{\cos \theta_2}{\cos \theta_1} \)

Critical angle of total reflection \( \cos \theta_C = \frac{n_2}{n_1} \)

→ Total reflection x-ray mirrors (TRM)

But: @ \( E = 10 \) keV: \( \theta_C \approx 5\text{mrad} \approx 0.3^\circ \)

→ Very long mirrors at high energies!
Reflective Multilayers
Multilayers rely on optical interference between many interfaces

\[ d = n\lambda/2 \sin(\theta) \]
Target Erosion

400 bilayers

WSi$_2$ = 7.1 Å to 7.2 Å

Si = 29.4 Å to 31 Å

After growth rate decay compensation.

400 bilayers

WSi$_2$ = 7.2 Å, Si = 30.8 Å

1st order R = 70.6 %, BW = 1.34
Depth-graded wide bandpass multilayer

• Computational method employed for multilayer design * can be used to calculate the required multilayer stack parameters for arbitrary reflectivity profiles, within reason.

• 77 bilayers of WSi$_2$ and Si

• Bilayer spacing from 2.2 nm to 5.5 nm

• Relatively flat-topped reflectivity = 9.5%, Bandpass = 48%

* Erko Et. Al., JSR 1998
Kozhevnikov Et. Al., NIMA 2001
Morawe Et. Al., NIMA 2002
# Three Stripe Double Multilayer Monochromator

<table>
<thead>
<tr>
<th>D</th>
<th>D</th>
<th>21 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>A</td>
<td>25 Å</td>
</tr>
<tr>
<td>C</td>
<td>C</td>
<td>33.5 Å</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Energy Range</th>
<th>Material System</th>
<th>D-spacing</th>
<th>M-silicide Thickness</th>
<th># Bilayers</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;20keV</td>
<td>WSi2/Si</td>
<td>21 Å</td>
<td>8.4 Å</td>
<td>250</td>
</tr>
<tr>
<td>&lt;20keV</td>
<td>MoSi2/Si</td>
<td>25 Å</td>
<td>10.0 Å</td>
<td>250</td>
</tr>
<tr>
<td>&lt;20keV</td>
<td>MoSi2/Si</td>
<td>33.5 Å</td>
<td>11.7 Å</td>
<td>140</td>
</tr>
</tbody>
</table>

Surface, Si #2 | Surface, Si #1
Profile Coating System

Sources
Substrate Tray

Coated substrate
Flux shroud
Uniform Mask
Magnetron Source
Profile Coating System

a) Coated substrate
b) Uniform Mask

Argonne NATIONAL LABORATORY
Coated substrates behind 10mm wide slit.

Residual shadowing of the uniform masks can be seen on the right-hand flux shield.
Three Stripe Double Multilayer Monochromator

Reflectance measurements at 8 keV taken at 1BM.

The inset schematic illustrates measurement orientation. Small peaks around 0.5° are from harmonic contamination.
Three Stripe Double Multilayer Monochromator

33.5 Å Multilayer
Measured at 1-BM @ 18keV

Measurement at APS beamline 1-BM of the 33.5 Å multilayer stripe.

1st order peak detail illustrates good thickness uniformity through the multilayer stack.

<table>
<thead>
<tr>
<th>Energy</th>
<th>D-spacing</th>
<th>2mm wide slit</th>
<th>0.5mm wide slit</th>
</tr>
</thead>
<tbody>
<tr>
<td>22keV</td>
<td>21 Å</td>
<td>0.374</td>
<td>0.6</td>
</tr>
<tr>
<td>18keV</td>
<td>25 Å</td>
<td>0.778</td>
<td>0.784</td>
</tr>
<tr>
<td>18keV</td>
<td>33.5 Å</td>
<td>0.807</td>
<td>0.802</td>
</tr>
</tbody>
</table>

1BM Efficiency measurements of three stripes at two slit widths.
Thickness Gradient Control – What’s the difference?

Profile Coating
- Efficient figure changes
- Universal masking technique

Differential Deposition
- Precision thickness-graded multilayers
- Final phase correcting layer
Thickness Gradient Control – What’s the difference?

Profile Coating
- Efficient figure changes
- Universal masking technique

Differential Deposition
- Precision thickness-graded multilayers
- Final phase correcting layer
Profile Coating – Film Thickness Distribution

\[ t = m \cdot h^2 (h^2 + r^2 + a^2)/[\rho/[\rho^2 + r^2 + a^2 + 2ar]^{1.5} (h^2 + r^2 + a^2 - 2ar)^{1.5}] \]

Direction of substrate translation

Pt Profile-Coated Elliptical Mirror

 indiscriminate of source distance: 36 m
Focal length: 83.62 mm
Grazing angle: 3.97 millirad

Focus simulation of the 40 mm long mirror.

Pt-KB height profile.

This 40 mm long mirror started out as a spherically polished substrate. The total sag of the mirror is almost 4 μm. The RMS residual height error is 0.5 nm.
Thickness Gradient Control – What’s the difference?

Profile Coating
- Efficient figure changes
- Universal masking technique

Differential Deposition
- Precision thickness-graded multilayers
- Final phase correcting layer
200mm Laterally-Graded ML KB

Design for 80 KeV
W/B₄C, d=1.8 to 2.2 nm
Deposited with velocity profiling
Individual erosion rate compensation

θ-2θ measurements at 8 keV and 32 keV of laterally-graded W/B₄C multilayer.

The sharp, featureless Bragg reflections at 8 keV indicate proper layer periodicity. Measurements at 32 keV include beamline impurities, nonetheless, reflectance is over 50%.
Laterally-graded W/B₄C multilayer d-spacing.

The gradient along the length of a 200 mm long substrate is shown in (a) along with the design requirement (solid line). The corresponding stage velocity varies by ~40%, proportional to the variation in B₄C. Error is only +/-0.15% over the central 80% of the mirror.
Rotary Deposition System

Basic System Attributes:
Cryopumped, base pressure < 10^{-8} torr
Upstream and downstream process gas control capability
Single-process gas (no reactive sputtering)
Horizontal and up-sputtering geometry
Substrates are rotated on a drum
PLC interlocked, PC controlled
No bake-out, no load-lock
For small MLL and ML
Also radially-symmetric multilayers

APS Rotary Deposition System Upgrade Design
APS Rotary Deposition System Upgrade Design

Multi-port rotary union

Two hollow-shaft ferrofluidic feedthroughs
APS Modular Deposition System

- Advanced multilayer optics and supermirrors such as
  - Multilayer monochromators for high-energy (>30 keV) x-rays
  - Fabrication of 3-D graded multilayer optics for focusing and collimation
  - Supermirrors of x-ray energies up 100 keV (Depth-graded)
  - High-aspect-ratio ML grating structures for interferometry, spectroscopy with high energy x-rays
  - Low-stress multilayer coatings for nanofocusing KB mirrors
- Multilayer Laue lens R&D
- **Mirror figure correction (up to 1.5 meters in length)**

![APS Modular Deposition System Diagram](image-url)
Modular Deposition System Scope (extra)

Can impact all 4 of the “Grand Challenges” outlined in the DOE x-ray optics workshop thin-film chapter

- R&D on film damage origins, mitigation, and lifetime
- Experimental verification of film growth computational modeling
- Development of methods for true 3-D film thickness gradients
- MLL R&D on larger apertures and materials for expanded energy range


https://science.energy.gov/~media/bes/pdf/reports/files/BES_XRay_Optics_rpt_print.pdf
Precision Linear Motion

- In-vacuum brushless DC linear motor
- Velocity error <0.0025%
- Stage slope error \( \sim 1.5 \, \mu \text{rad} \) rms over 4.5 meter travel length
- 5nm position resolution, +/-670nm PV accuracy
- 5nm homing resolution
Deposition Sources

Five 75mm diameter round cathodes for routine user coatings
Three 250 mm x 90 mm cathodes for multilayer deposition

Flexible source accommodation
Cylindrical rotating cathodes shown

Factor of ~100 lower target erosion rate
Larger magnets-lower pressure/smooth films
Higher vertical uniformity without masking

Eliminating target erosion compensation with planar cathodes
- significant production rate gains
Possible route:
Modulated ion-assisted deposition

Low energy leads to low intermixing but high surface roughness

High ion energy leads to more intermixing but lower surface roughness.

Combine the two!

Requires magnetically guiding secondary electrons to the sample

Feature: Modulated negative bias at the sample

Atomic scale interface engineering by modulated ion-assisted deposition applied to soft x-ray multilayer optics

Fredrik Eriksson,¹,* Naureen Ghafoor,¹ Franz Schäfers,² Eric M. Gullikson,³ Samir Aouadi,⁴ Susanne Rohde,⁵ Lars Hultman,¹ and Jens Birch¹

¹Thin Film Physics Division, Department of Physics, Linköping University, S-58183 Linköping, Sweden
In-situ Metrology + Ion Beam Figuring

15 APS beamlines have identified mirrors that would benefit from figure correction

Inexpensive alternative to complete mirror replacement

Potential to utilize equipment for other applications

In-situ metrology + ion milling offers fast turn-around (1 week vs. 6 months) and ability to utilize reactive materials

Challenges:

mirror surface measurement + mirror position targeting
Ion Beam Figuring

Central Line Profile (~3mm wide)
before IBF, PV=30nm, RMS=10.2nm
after IBF, PV=8.7nm, RMS=1.47nm

before IBF: RMS 11.5nm; PV 52.6nm
after one IBF pass: RMS 1.7nm; PV 15.5nm

Example mirror figure correction courtesy ZEISS
Ion Source Chamber

Sputtering cathode for Ion Assisted Sputtering

RFICP mill

Focused DC mill on z-axis translator

Focused ion mill for ion beam deposition

Target
Ion Beam Figuring

Major components and status:
• Strip 100mm RFICP ion mill for large area correction
  • Installed
• 6mm focusing ion mill for deterministic correction
  • Translator designed
• Precision gas mixing and metering for reactive gas use
  • Designed, all components ordered, build ongoing
• Controls
  • Electronics installed, awaiting controls integration (BCDA)
In-situ UHV Mirror Figure Measurement

Basic idea:
Attach a Fizeau interferometer to a viewport, which is then attached to a bellows
Track stage trajectory during stitching
### In-situ Surface Measurement Using Interferometry

<table>
<thead>
<tr>
<th>Pros</th>
<th>Cons</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Keeps the mirror under vacuum</td>
<td>• Must dedicate the instrument</td>
</tr>
<tr>
<td>• No air turbulence</td>
<td>• Viewport deflection &amp; internal stress</td>
</tr>
<tr>
<td>• No humidity effects</td>
<td></td>
</tr>
<tr>
<td>• Fast iteration rate</td>
<td></td>
</tr>
<tr>
<td>• Avoid oxidation (metals, etc)</td>
<td></td>
</tr>
<tr>
<td>• Accurate registration between measurement &amp; ion mills</td>
<td></td>
</tr>
</tbody>
</table>

#### Deflection at Atmosphere

![Radius=33 km](image1)

#### Deflection Under Vacuum

![Radius=1.46 km](image2)

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**Note:**
- In-situ measurement techniques are crucial for precise surface analysis in various applications.
- Interferometry, a key method, offers advantages like maintaining vacuum and avoiding oxidation effects.
- Challenges include dedicating the instrument and managing internal stress.

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**Interferometry Methods**

- Advantage for using the wavelength-tuned PSI
  - Reference flat can be set inside of the VC without requiring a PZT tuning system.
  - Use spatial phase shifting technique which makes fast data acquisition possible. Offers both vibration immunity and the ability for dynamic measurement.
  - On-axis geometry reduces re-tracing errors (only refraction must be accounted for)

**mechanically tuned Fizeau PSI**

**wavelength tuned PSI**
“Normal” Fizeau Interferometry

Fringe pattern of a flat mirror.

Fringe pattern of a flat mirror measured with a curved mirror inserted in between the 2-surface cavity.

Fringe pattern of a flat mirror measured with a curved mirror inserted outside the 2-surface cavity.
Interferometry measurement of a Si substrate

A)

Reference  SUT
Flat

B)

Reference  SUT
Flat

A curved TF simulating as the VC window-

- The difference of the surface figures from the measurements of the A and B. RMS=1.58 nm and PV=8.06 nm.
Stability Measurements

A series of 5 static measurements were taken at 1 minute intervals. 10 difference subtractions of each series are plotted. Overall magnitude of the measurements was very similar. Variation is likely dominated by environmental turbulence such as thermal drift.

### Schematic of stationary setup with and without VCW inserted in the optical path.

<table>
<thead>
<tr>
<th>PV (nm)</th>
<th>RMS (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avg</td>
<td>3.866</td>
</tr>
<tr>
<td>σ</td>
<td>0.874</td>
</tr>
</tbody>
</table>

**With VCW**

**Without VCW**
Tilt Measurements

Axis of rotation schematic and test result.

Difference subtraction measurements were taken when the VCW is facing the FizCam and also facing the TF.
Implementation
Optical Column Angular Motion
Optical Column Angular Motion
Optical Column Angular Motion
Vertical Translation

Two Purposes:
1. Precise alignment of translation axis and substrate plane
2. Service/maintenance
Vertical Translation

Two Purposes:
1. Precise alignment of translation axis and substrate plane
2. Service/maintenance
In-situ Interferometry Gimbal

Transmission flat supported by steel band during measurement
Wheels engage to lift and then rotate the t-flat for calibration
Both axes rotate on c-flex bearings for zero backlash and drag
Transmission Flat Gimbal

- UHV compatible
- Precision tip/tilt
- Both axes rotate on c-flex bearings for zero backlash and drag
- Rotates 360 degrees for three-flat reference nulling
- Transmission flat supported by steel band during measurement
- Wheels engage to lift and then rotate the t-flat for calibration
Gimbal

Remote transmission mount

UHV compatible

Precision tip/tilt

Rotates 360 degrees for three-flat reference nulling
Gimbal Chamber
Alternative ex-situ design (Backup plan)

In-vacuum cart is used ex-situ for best possible position registration.

Dual ion mills
one on a z-axis translator

Ex-situ Interferometer

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Modular Deposition System Scope

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- Experimental verification of film growth computational modeling
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https://science.energy.gov/~media/bes/pdf/reports/files/BES_XRay_Optics_rpt_print.pdf
Dynamic Aperture

Array of dynamically-actuated fingers for real-time control of thin film deposition

Deposition mask

Dynamic aperture

Array of dynamically-actuated fingers

Cathode
Material target
Dynamic aperture
Thin film
Substrate
Surface correction techniques:

- Present: two reflections
  - Differential deposition
  - Ion-beam figuring

- Future: single-reflection
  - Combination of differential deposition and ion-beam figuring of over-coating
Baffle fingers
Cathodes

Brushless DC micromotors and other miniature components for a 5mm wide finger array

Dynamic Aperture
Dynamic Aperture

Two designs
Design 3.0 – Ready for UHV testing

Design 3.0
Dynamic Aperture

Design 4.0 – Ready for controller integration
In-situ deposition flux density monitoring

*Atomic Absorption Spectroscopy*

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**Spring 2016**

**TWO Phase II awarded**
- K-Space, Inc.
- Accustrata

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**5. OPTICS DEVICES FOR LIGHT SOURCE FACILITIES**

<table>
<thead>
<tr>
<th>Maximum Phase I Award Amount: $150,000</th>
<th>Maximum Phase II Award Amount: $1,000,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accepting SBIR Phase I Applications: YES</td>
<td>Accepting SBIR Fast-Track Applications: YES</td>
</tr>
<tr>
<td>Accepting STTR Phase I Applications: YES</td>
<td>Accepting STTR Fast-Track Applications: YES</td>
</tr>
</tbody>
</table>

The Office of Basic Energy Sciences, within the DOE’s Office of Science, is responsible for current and future synchrotron radiation light sources, free electron lasers, and spallation neutron source user facilities. This topic seeks the development of x-ray optics devices to support the light source user facilities.

Grant applications are sought in the following subtopics:

1. Advanced In Situ Thin Film Growth Monitors
MDS-2017

Machine has been “commissioned”

- Controls are generally functioning properly. Some bugs remain
- Automation is gradually being added/integrated
- All subsystems have been tested
- Multilayers fabricated and delivered (4ID-C, 12BM)
- Source optimization, deposition parameter tuning is ongoing
- Ready for velocity profiling (for lateral gradients)
Acknowledgements

- Mark Erdmann
- Jason Carter
- Dan Nocher
- Scott Izzo
- Jun Qian
- Bing Shi
- Elina Kasman
- Lahsen Assoufid
- Josh Abraham
- Felix Roman & Scheck electricians
- Oliver Schmidt
- Weasel

- APS Optics, MOM, drafting groups
- NSLS-II XHN, Optics R&D groups
- APS machine shop
- CVD Equipment
- APS Management
- NSLS-II Management
Thank you for your time