

### A new state of the art instrument for x-ray optics fabrication: scope, capabilities, and design of the Modular Deposition System

Ray Conley TWG Meeting 2016



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- APS Optics Group
- APS MOM Group
- APS Drafting Group
- APS machine shop
- CVD Equipment
- APS Management
- NSLS-II Staff

### Modular Deposition System Goals and Scope

- Figure correction of large flat mirrors up to 1.5 m
- Figure correction of aspherical mirrors up to 1.5 m (metrology limited)
- Thin-film recoating and figure correction for dynamically bent K-B mirror systems
- Multilayer monochromators, energies from ~250eV up to 100's keV
- Fabrication of 3-D graded multilayer optics for focusing and collimation
- Fabrication of fixed-geometry K-B mirrors
- Advanced multilayer optics and supermirrors such as
  - Multilayer monochromators for high-energy (>30 keV) x-rays
  - Supermirrors of x-ray energies up 100 keV (Depth-graded)
  - Advanced high-aspect-ratio ML grating structures for interferometry and other spectroscopy with high energy x-rays
  - Low-stress multilayer coatings for nanofocusing KB mirrors
- Multilayer Laue lens R&D
  - Grow thicker than the cathode limitation with cylindrical cathodes (1mm apertures or greater-film stress limited)
  - Deposit "Jelly Roll" zone plates
- Deposit crystallographically-oriented thin films for user requirements
  - Ion beam deposition, Ion Assisted Sputtering

### **Modular Deposition System Specifications**

- 22 feet long, 16" dia. Main chamber size
- Loadlocked
- Five 3" dia. Round cathodes, three 250mm planar cathodes
- In-vacuum direct drive brushless DC linear servo
- Precision in-vacuum direct drive brushless linear DC motor
  - Velocity stability better than 0.0025%
  - Homing precision of 50nm or better
  - Slope error ~1.5 mrad
- ~5x10-8 torr base pressure
- Multi-gas capable for reactive sputtering
- Reconfigurable deposition source ports for flexibility:
  - Cylindrical (rotating) cathodes
  - Atomic Absorption Flux Monitoring
  - Dual ion-milling, both an RF-ICP 100mm vertical source and a 6mm focused DC mill
  - In-situ surface measurement
  - Ability to add automated ex-situ surface measurement with the *same* servo drive system (best method for mirror-position registration)
  - Ion beam deposition
  - Confocal ion-assisted sputtering (one target only)
  - Dynamic Aperture slit masking apparatus
    - Marriage of deformable masking with velocity profiling
- Mirrors up to 1.5 meters/ 80Kg
- Substrate biasing (electrical signal lines available at the mirror)
- Will sit in the OCR400 cleanroom on the experimental floor

#### Modular Deposition System Scope (Phase 1)

- Thin-film recoating for dynamically bent K-B mirror systems
- Multilayer monochromators (double or single)
- Fabrication of 1-D graded multilayer optics for focusing and collimation
- Advanced multilayer optics and supermirrors such as
  - Multilayer monochromators
  - Supermirrors of x-ray energies up 100 keV (Depth-graded)
  - Advanced high-aspect-ratio ML grating structures for interferometry and other spectroscopy with high energy xrays
  - Low-stress multilayer coatings for bendable nanofocusing KB mirrors
- Multilayer Laue lens R&D



# **Precision Linear Motion**

- In-vacuum brushless DC linear motor
- Velocity error <0.0025%
- Stage slope error ~1.5 µrad rms over 4.5 meter travel length
- 10nm position resolution, +/-670nm PV accuracy
- 10nm homing resolution



Commercially Available Linear Motor









#### **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/smoother films Higher vertical uniformity without masking



Eliminating target erosion compensation with planar cathodes - significant production rate gains

Cathode Selections The new machine will be able to use any combination of sources (MODULAR)



Feature:

Flexible acceptance of many deposition sources



Considerations:

Higher strength magnetic field – lower discharge voltage

Target erosion-> growth rate change

Necessitates correction for ML

Node formation on targets

-Caused by chamber impurities





# Can also be used for MLL

#### Status

- 105µm thick deposition (AI/WSi2)
- Routine RIE/FIB sectioning
- Absolute layer placement is highly accurate
- Arbitrary number of layers possible (15,170 is current record)
- Zero roughness propagation
- Significant decrease in film stress TXM DATA

Normal growth - stress defects





### Latest Results: Phase Reconstruction





#### Mirror Equivalent: 0.015nm RMS!



# **Reactive Sputtering Growth Rate**

#### Why reactive sputtering?

Film stress reduction, nitrides, oxides

However:

Reactive sputtering growth rate very sensitive to pressure/temperature

Requirement:

Equipment Temperature Stability







### Multilayer Interface Engineering, Growth Dynamics

Issues: Material chemistry, intermixing

Extreme environments Temperature, corrosion MoMoMobarrier<br/>layerB4CB4CSiSiSi

Dr. F. Tichelaar, Delft University of Technology

Uses: Polarizers, Analyzers Narrow bandpass MLs Conformal deposition Highly-corrugated surfaces

Feature: Multiple cathodes or sources



### Multilayer Interface Engineering, Growth Dynamics

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

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### **Thickness Gradient Control**

#### **Profile Deposition (or profile coating)**

- · Linear substrate motion with respect to source
- Sagittal thickness control via masks
- Uniform meridional thickness via constant velocity



### **Thickness Gradient Control**

#### Differential deposition (Slide from C. Morawe)

- · Linear substrate motion with respect to source
- Meridional thickness control via speed variation
- Sagittal thickness tuning via masks





### **Thickness Gradient Control - What's the difference?**

Differential Deposition -Precision thickness-graded multilayers -Final phase correcting layer



Profile Coating -Efficient figure changes -Universal masking technique



#### **Profile Coating**



Direction of substrate translation

#### Profile coating

Coat up to 22x10x2.5 cm

Two 3"  $\phi$ sputtering guns DC



Pt-profiled KB on spherical Si RMS figure error=0.5nm Average roughness=**2.3 Å** 



- Small deposition system (APS) dedicated to mirror fabrication
  - Routine elliptical Pt coating up to ~10µm thick
  - Single-process gas only
  - No monitoring or feedback system
  - Translation stage is failing
  - Sputters up
  - No provision for velocity profiling, erosion compensation, depth-graded MLs, etc

### 200mm Laterally-Graded ML KB

For 80 KeV focusing at NSLS

 $W/B_4C$ , d=1.8 to 2.2 nm

Deposited with velocity profiling

Individual erosion rate compensation





### 200mm Laterally-Graded ML





(picometers) error ſ d-spacing -2 -3 20 -60 -20 -80 -40 0 40 60 80 100 Position (mm)

### Modular Deposition System Impact (Phase 2)

- Figure correction of large flat mirrors up 1.5 m
- Figure correction of aspherical mirrors up 1.5 m (metrology limited)
- Thin-film recoating and figure correction for dynamically bent K-B mirror systems
- Multilayer monochromators
- Fabrication of 3-D graded multilayer optics for focusing and collimation
- Fabrication of fixed-geometry K-B mirrors
- Advanced multilayer optics and supermirrors such as
  - Multilayer monochromators for high-energy (>30 keV) x-rays
  - Supermirrors of x-ray energies up 100 keV (Depth-graded)
  - Advanced high-aspect-ratio ML grating structures for interferometry and other spectroscopy with high energy x-rays
  - Three-dimensional multilayer structures for focusing, beam conditioning, and beam shaping.
  - Low-stress multilayer coatings for nanofocusing KB mirrors
- Multilayer Laue lens R&D
  - Grow thicker than the cathode limitation with cylindrical cathodes (1mm apertures or greater-film stress limited)
  - Deposit "Jelly Roll" zone plates
- Deposit crystallographically-oriented thin films for user requirements
  - Ion beam deposition, Ion Assisted Sputtering



### Ion Source Chamber





#### RF-ICP 100mm broad-beam source

Product	RFICP 100		
Discharge	Inductively coupled		
RF Discharge Power (max)	800W		
Beam current (max)	>400mA		
Voltage range	100-1200V		
Gases	Inert (Ar,Xe, etc) & reactive (O2, N2, others)		
Typical flows	5-30sccm		
Ion optics (self-align)	OptiBeam™		
Beam size @ grids	10cm Φ (typical)		
Grids	Molybdenum & Graphite		
Beam Shape	Collimated, convergent, divergent		
Neutralizer	Non-immersed		
Height (nominal)	9.25" (23.5cm)		
Diameter (nominal)	7.525" (19.1cm)		
Feedthrough direct:	10" CF		







#### KDC 10mm focused source



Models	KDC10
Discharge	DC magnetic confinement
Filament cathodes	One
Anode voltage	0-100V VDC
Ion optics	OptiBeam <sup>™</sup>
Grids	Application specific
Alignment	Self-aligned
Beam size @ grid	1cm (typical)
Neutralizer (std)	Filament
Power controller	KSC 1202
Options	
Ion optics	Collimated, focused, defocused
Controller options	4 gas channels, recipe storage
Neutralizer	Sidewinder or LFN 2000
Mount	Remote or direct flange



Fig. 4-5 Ion-beam profiles for focused and collimated two-grid graphite ion optics. The ion-beam current is 10 mA, the beam voltage is 600 V, the accel voltage is 90 V, and the argon flow is 4 sccm.

### Ion-Beam Polishing with Various Noble Gases



JOURNAL OF APPLIED PHYSICS 112, 123502 (2012)



#### Influence of noble gas ion polishing species on extreme ultraviolet mirrors

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# Ion Milling

- 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



Example mirror figure correction courtesy ZEISS

#### **Real challenges:**

mirror surface measurement + mirror position targeting

### In-situ UHV Mirror Figure Measurement

Basic idea:

Attach a Fizeau interferometer to a viewport, which is then attached to a bellows Use autocollimator to track stage trajectory during stitching

Measurement of flats:

Tip-tilt on the interferometer is required No reference sphere required

Measurement of aspheres:

Tip-tilt on the interferometer is required

Reference sphere required

May need to track angular alignment of Fizeau during stitching



#### Vacuum Window Issues

#### Experiment by Jun Qian







Interference pattern



Interference pattern





Interference pattern

# In-situ Fizeau Measurement

Pros:

Keeps the mirror under vacuum Fast iteration rate Ability to avoid oxidation (metals, etc) Accurate registration between measurement and ion mills

Cons:

Cannot place the mirror in vertical deflection mode Need to consider viewport deflection Cannot use the instrument for normal metrology usage

Impact of barometric pressure variation in viewport:





# Viewport radius of curvature vs pressure and thickness

	0.25"	0.5"
772.16 Torr	63.2 m	1597 m
744.22 Torr	60.9 m	1660 m



### In-situ Surface Measurement Using Interferometry

#### Pros

- Keeps the mirror under vacuum
- No air turbulence
- No humidity effects
- Fast iteration rate
- Avoid oxidation (metals, etc)
- Accurate registration between measurement & ion mills

#### Deflection at Atmosphere



#### Cons

- Must dedicate the instrument
- Viewport deflection & internal stress









**UHV** gimbal

#### **Measurements**





\*Wavelength tunable on-axis dynamic interferometer (Not a Fizeau)

### **UHV 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



#### 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

### Alternative automated ex-situ design (backup plan)



### Alternative automated ex-situ design What's required of the "base system"?



A spool piece 1 spring-loaded car stop (not shown)

1 set of extra car rails for the ex-situ table (also not shown)

# Differential profiling can be used for ion milling

RF required for processes involving insulating materials/gases

Filamentless – source lasts for a very long time between rebuilds

Mo optics for reactive gases (O2, H2, N2, + exotics, SF6, C4F8, etc...)









### **Deterministic Deposition/ Ion Beam Figuring**



Surface correction techniques: (a) differential deposition, (b) ionbeam figuring, (c) combination, (d) ion-beam figuring of over-coating.



Ion source or sputtering source inside a chimney with dynamic apertures

# **Conformal Coatings-Extreme Figure ML Mirrors**

• Mini-Montel ML mirrors:



- KB mirrors (V,H) KB mirrors
- One multi-crystal analyzer per edge
- Flat crystal technology and expertise sufficient
- Incident side optics proven, in existence
- Performance dependent on collimating Montel mirror

Slide from DOE Lehman CD-2 Review of the APS Upgrade Project 4-6 December 2012

Challenge: (Ignoring the difficult substrate) Graded-d ML within the interior of the steep concave surface. Requires flux change in both axes simultaneously, and off-normal growth + low roughness.

Solutions? -Differential Deposition and simultaneous controllable slit aperture (perhaps)

True three-dimensional flux gradients may be possible!

- Single-reflection toroid mirrors:
- Convert divergent beam from a target into a parallel beam



#### **Dynamic Aperture**

Currently, transverse thin-film/ion milling profile is defined by a static aperture Flux gradients possible along both X and Y axes For figure correction or 3-dimensional multilayer profiles

Cathodes

Baffle fingers

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





### Modular Deposition System Considerations -Feedthroughs! Lots of feedthroughs! (and space)



#### Dynamic Aperture/Actuated Baffle Design Underway





#### In-situ deposition flux density monitoring Atomic Absorption Spectroscopy





APL 2014, Du, Et. Al.

5. OPTICS DEVICES FOR LIGHT SOURCE FACILITIES				
Maximum Phase I Award Amount: \$150,000	Maximum Phase II Award Amount: \$1,000,000			
Accepting SBIR Phase I Applications: YES	Accepting SBIR Fast-Track Applications: YES			
Accepting STTR Phase I Applications: YES	Accepting STTR Fast-Track Applications: YES			
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:				
a. Advanced In Situ Thin Film Growth Monitors				













#### The End



### March 2013 DOE Optics Workshop

Thin Film Summary (Four bullets, no particular order) •R&D on damage origins, mitigation, recovery, and long lifetime of coatings is crucial.

•Comprehensive investigation of the physics of thin-film growth, interfaces and atomistic modeling is necessary to advance performance of all thinfilm optics, including structured coatings, ultra short d-spacing multilayers, gratings, Laue lenses etc.

•Multilayer Laue lens research, including stress reduction, larger thicknesses, manual thinning, focused ion-beam milling, and mounting needs to continue and the results of this effort will be applicable towards other thick or diffractive multilayer optics.

 Investigation of methods for 3-dimensional multilayer deposition on highly profiled surfaces will enable new science (such as IXS and small angle scattering).

#### Focusing/Collimating Multilayer Mirrors Three basic types -Depth periodic, laterally graded

Highest integrated reflectance for single energy

#### -Depth graded, laterally uniform

Lowest integrated reflectance for single energy Allows wavelength changes w/o optics adjustments



#### -Depth graded + laterally graded

Can be matched to source Slight energy tunability w/o flux loss



#### Double thickness gradient multilayer calculations

#### Coded entirely by Ken Lauer

The below graph is layer thickness through the stack, but only at the center position of the mirror. The materials are W/B4C



Office of Science

"Supermirror" Design – both lateral and depth gradients Figure of merit – highest integrated reflectivity across the entire stack Genetic evolution algorithm

Reflectance vs. energy at the central point in the mirror for 5 different stacks.



Reflectance vs. angle for the central energy (39.1KeV).



# Multilayer Optimization Algorithms

# What is missing/What needs more work? -Extensive deposition process modeling

- -Experimental verification
- -Investigation of new deposition techniques
- -Environment modification
- -Explore new materials
- -Flux collimation
- -Geometry

What would Benefit -Narrow-BP MLs -Gratings/structured coatings -MLs in extreme environments -Cryo-cooled MLM -Interface engineering ...The entire field!



IBS growth – change in energy altered mobility + performance



### Conformal growth of Mo/Si multilayers on grating substrates using collimated ion beam sputtering

D. L. Voronov,<sup>1,a)</sup> P. Gawlitza,<sup>2</sup> R. Cambie,<sup>1</sup> S. Dhuey,<sup>1</sup> E. M. Gullikson,<sup>1</sup> T. Warwick,<sup>1</sup> S. Braun,<sup>2</sup> V. V. Yashchuk,<sup>1</sup> and H. A. Padmore<sup>1</sup> <sup>1</sup>Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, California 94720, USA <sup>2</sup>Fraunhofer Institute for Material and Beam Technology, Winterbergstraße 28, 01277 Dresden, Germany

# Thin Film Damage

- Thin-films often have very different properties from the bulk
- Peak power, environment
- Radiation damage, thermal damage
- Chemical excitation



Corrosion barrier

SEM images of SiC film exposed to single LCLS FEL pulses at 0.83 keV and peak fluences of (a) 1.0, (b) 1.6, (c) 2.9, (d) 5.8, (e) 14.8, and (f) 57.5 J/cm<sup>2</sup>.



S. P. Hau-Riege et al, Opt. Express 18, 23933-23938 (2010).

# **Another Issue: Coherence Preservation**

Quantitatively characterize and ultimately control wavefront disturbances introduced by optic elements (mirrors, multilayers, focusing devices, windows)



Effect of a  $WSi_2/Si ML$  on the fringes created by a well-defined object (100µm diameter B fiber) A. Fluerasu, L. Berman, R. Conley, A. Snigirev

### Worldwide MLL efforts

# Three groups: German, Japanese, and Chinese

TABLE (1). Parameters of MLLs for KB configuration.				
	MLL-1	MLL-2		
Layer Thickness (nm)	8.5~11.4	7.3 ~ 9.8		
Total Thickness (µm)	6.6	5.6		
No. of Layers	670	670		
Focal Length @ 20 keV (mm)	9.5	6.3		



#### Development of Multilayer Laue Lenses; (1) Linear Type

T. Koyama<sup>a</sup>, H. Takenaka<sup>b</sup>, S. Ichimaru<sup>b</sup>, T. Ohchi<sup>b</sup>, T. Tsuji<sup>a</sup>, H. Takano<sup>a</sup>, and Y. Kagoshima<sup>a</sup>

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#### W/Si or Ti/ZrO2 PLD, 400nm thick.



AIP ADVANCES 2, 012175 (2012)

#### A combined Kirkpatrick-Baez mirror and multilayer lens for sub-10 nm x-ray focusing

A. Ruhlandt,<sup>1</sup> T. Liese,<sup>2</sup> V. Radisch,<sup>2</sup> S. P. Krüger,<sup>1</sup> M. Osterhoff,<sup>1</sup> K. Giewekemeyer,<sup>1</sup> H. U. Krebs,<sup>2</sup> and T. Salditt<sup>1,a</sup> <sup>1</sup>Institut für Röntgenphysik, Universität Göttingen, Friedrich-Hund-Platz 1, 37077 Göttingen, Germany

### Jelly-Roll Multilayer Zone Plates



#### **Summary**

Some needs for thin-film based optics at APS met currently

All needs for thin-film based optics at APS will be met soon, given minor metrology equipment upgrades

New deposition equipment, existing staff expertise puts APS at the forefront for the foreseeable future

The APS upgrade only puts further emphasis on existing R&D topics

APS contributes to the synchrotron community with optics, and expertise

### Metrology equipment

1.5 KW maximum power

 $E = 8.048 \text{ keV} (CuK\alpha_1)$ 

Ge <111> symmetrically-cut monochromator

5 slit sets

Custom sample-tilt stage

Essential tool for multilayer fabrication





And... WYKO interferometer AFM MicroXAM Michelson interferometer KSA MOS in-situ stress monitor SEM, EDX available at CNM New APS LTP **1BM** 

### APS Thin Film Laboratories-Current Coating systems

#### **Profile coating**

Coat up to 22x10x2.5 cm

Two 3"  $\phi$ sputtering guns

DC only



#### General purpose

Coat up 150x15x14 cm

Four 3"  $\phi$  Sputtering guns

DC & RF

Two evaporators, one ion mill

Three cryopumps –

base pressure <  $1x10^8$  Torr



#### Specialty multilayers & MLL

Coat up 12x5x4 cm

Two 3" dia. and two 2"x6" sputtering guns

At present, DC only

One cryopump –

base pressure  $< 3x10^9$  Torr



### **Existing Large Deposition System**

- Delivered 1000's of coatings over nearly 20 years!!!
- -Cryopumped, with fixed upstream gas flow control.
- -Currently, four 3" dia. round MAK cathodes.
  - Sputter up (debris disruption for long depositions)
- 10 to 15 year old gas delivery and monitoring (prone to drift)
- Cable-based linear translation stage which is intrinsically unstable and unreliable.
- Single-process gas system.
- -The machine is adequate for some (large d-spacing multilayer deposition, certain thick coatings, flat MLL deposition, and metallization of large mirrors.
- -No velocity profiling, data logging, etc.



### Where will we be soon?

#### Goals

- Support all coating needs for the APS community. Support other laboratories and SR facilities
  - Precision multilayers up to ~1.5 meters long
  - Profile coated KB micro and nanofocusing mirrors
  - Mirror metallization, user science sample coatings
  - Figure correction with in-situ metrology and ion beam figuring
  - Existing, long term collaborations with all USA lightsources
    - LNLS, NIF and other high-energy laser labs
  - Use 1BM for more diverse investigation –
- Contribute to the x-ray optics scientific community
- Explore R&D topics highlighted by DOE Funding Agency



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1BM MLL Characterization
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#### DOE Optics Workshop Thin Film R&D Directions:

•R&D on damage origins, mitigation, recovery, and long lifetime of coatings is crucial.

• Comprehensive investigation of the physics of thin-film growth, interfaces and atomistic modeling is necessary to advance performance of all thin-film optics, including structured coatings, ultra short d-spacing multilayers, gratings, Laue lenses etc.

•Multilayer Laue lens research, including stress reduction, larger thicknesses, manual thinning, focused ion-beam milling, and mounting needs to continue and the results of this effort will be applicable towards other thick or diffractive multilayer optics.

•Investigation of methods for 3-dimensional multilayer deposition on highly profiled surfaces will enable new science (such as IXS and small angle scattering).

### **250mm Planar Cathodes**

- Factor of ~100 lower target erosion rate
- Larger magnets-lower pressure
- Higher intrinsic vertical uniformity w/o masking





~0.5% thickness variation through the stack



