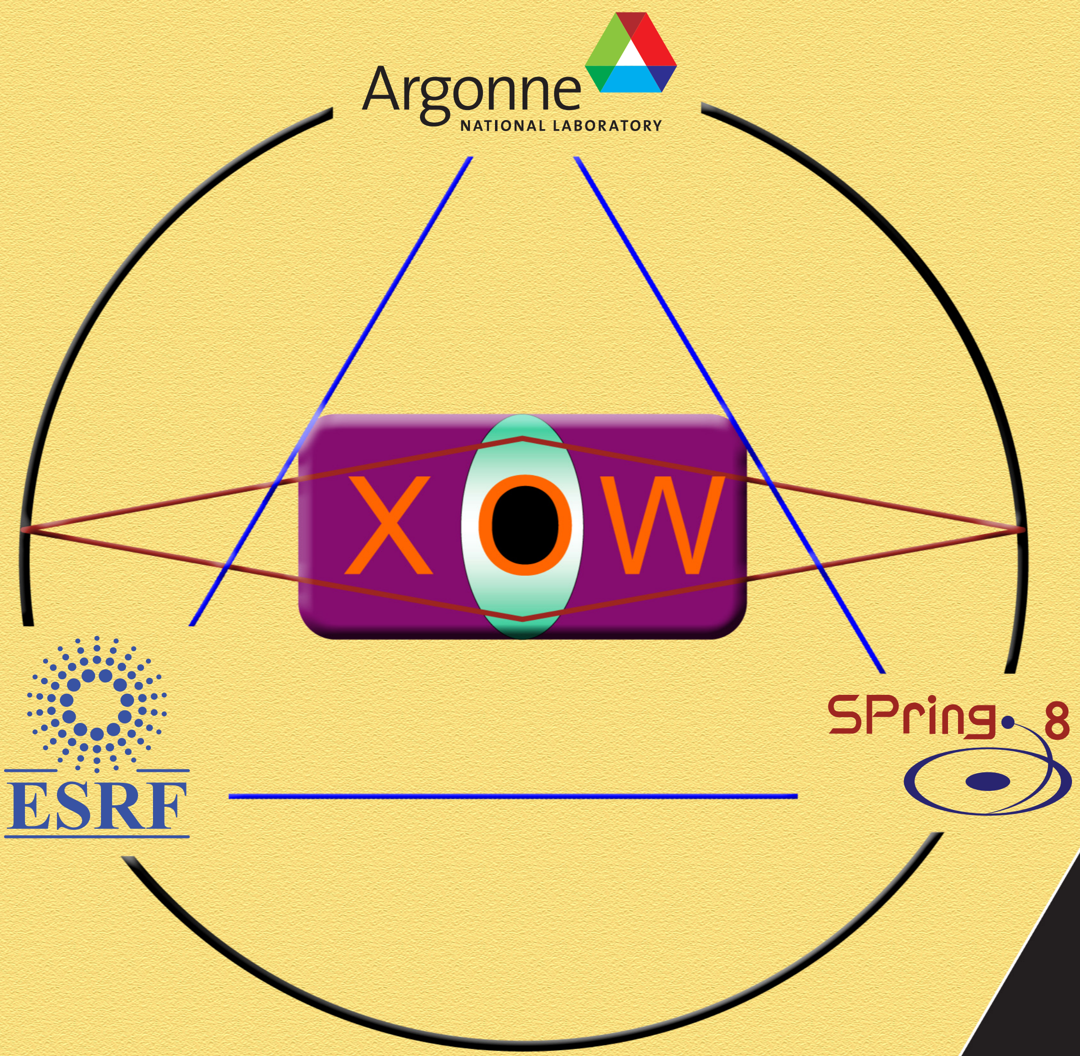
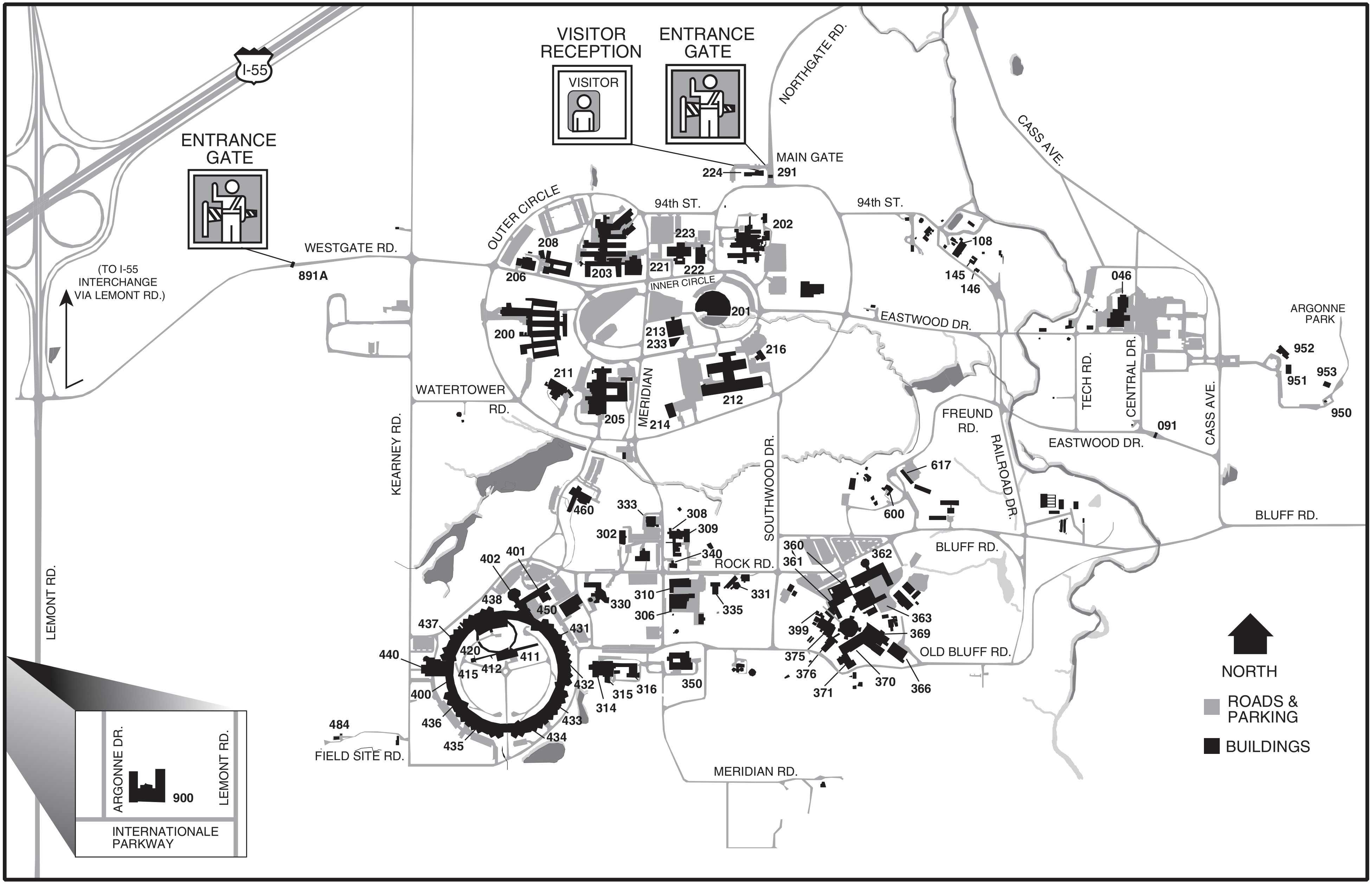
3-Way X-ray Optics Workshop V



Advanced Photon Source
Argonne National Laboratory
March 17, 2008

Map of Argonne National Laboratory and the Advanced Photon Source



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General Information

Location

Talks will be held in conference rooms E1100/E1200 located on the first floor of the APS Conference Center, Building 402, behind the Auditorium. Vendor exhibits will be nearby.

Meals

Coffee and pastries will be provided at the start of the workshop.
A coffee service will be provided during the breaks.
A box lunch will be provided to speakers and exhibitors.

Computer Terminals

Computer terminals are set up in the Building 401 atrium. These will be available to reach the internet.

Messages

Messages to be relayed to attendees during the workshop can be directed to:

Ms. Linda Shoudis
630-252-0160 (office)
630-992-3381 (cell)
ljs@aps.anl.gov

Workshop Dinner

A bus will leave from the APS main entrance after the workshop at 5:30 PM.
Visit to Fermi Laboratory with a lecture by a docent, followed by dinner at the Chez Leon restaurant.

Financial Sponsors

Blake Industries, Inc.
American Institute of Physics
Insync, Inc.
KLA-Tencor/ADE Phase Shift
Rigaku Innovative Technologies

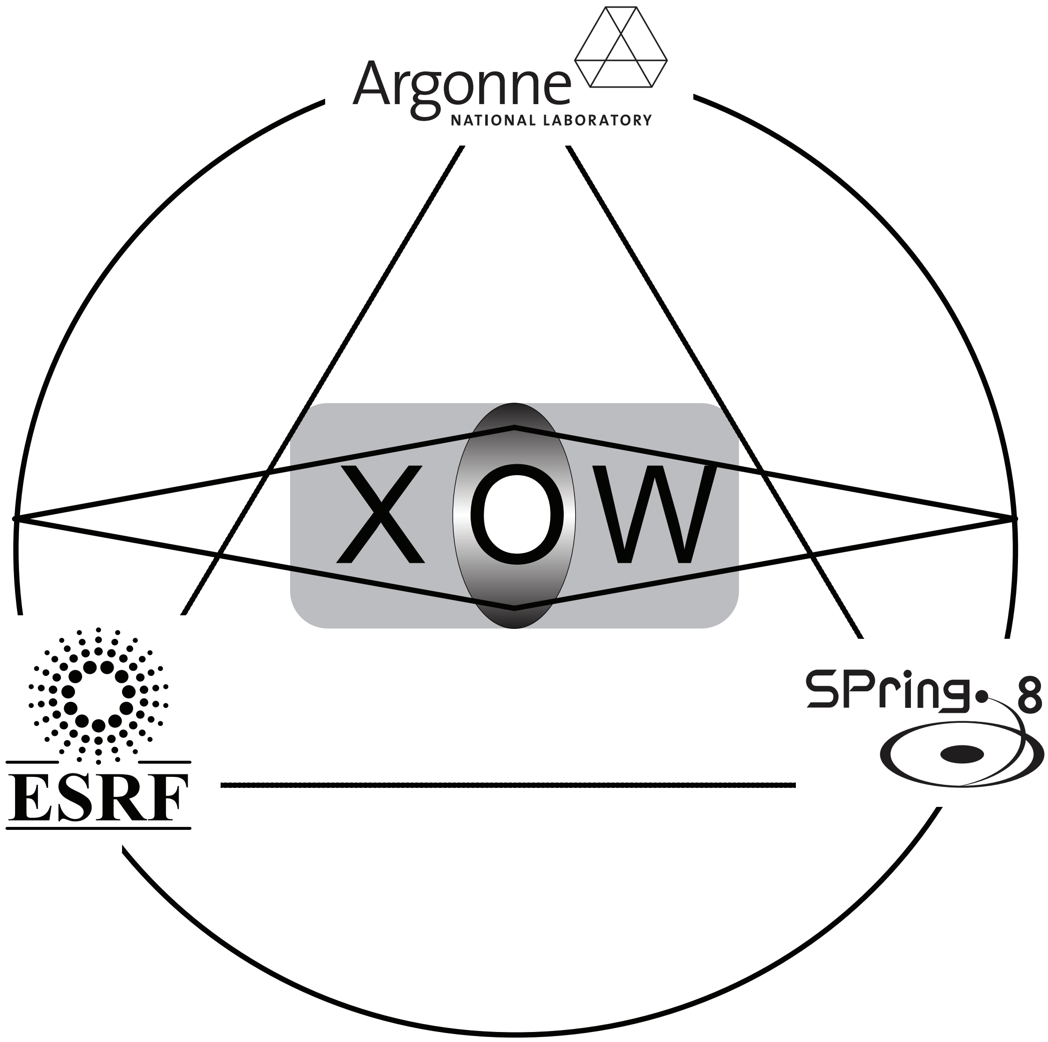
Program Committee:

A.T. Macrander
J. Härtwig
S. Goto

Acknowledgements:

The workshop organizers would like to acknowledge the assistance of
L. Shoudis (XSD), M. Nelson (IPD), and P. Styka (XSD).

Comprehensive Program



March 17, 2008
Room: 401/E1100 & E1200

8:15 **Welcome**
Gabrielle Long

8:30 Overview of Optics at APS
Albert Macrander

9:00 X-ray Optics Related News from the ESRF
Jürgen Härtwig

9:30 Overview of Optics at Spring-8
Shunji Goto

10:00 **Break**

10:30 Focusing hard X-rays to sub-10nm by reflective optics
Kazuto Yamauchi

11:00 Progress and Perspectives at the ESRF Multilayer Facility
Christain Morawe

11:30 Profile Coating for Elliptical KB Mirrors and Wedged Multilayer Laue Lens
Chian Liu

11:45 Wedged MLL Growth in the Rotary Deposition System
Raymond Conley

12:00 **Hot topics/Box lunch**
Albert Macrander

1:00 **Local tours**

2:00 Fabrication of Aspheric Optics for X-ray Applications
Ali Khounsary

2:15 Results of Hard-X-ray Mirror Metrology Round-Robin carried out at the APS, ESRF and SPring-8 Metrology Laboratories
Lahsen Assoufid

2:30 Current Status on the Metrology Laboratory at Spring-8
Haruhiko Ohashi

2:45 Comparison of Slope and Height Profiles for Flat Sycnhrotron X-ray Mirrors Measured with a LTP and a Fizeau Interferometer
Jun Qian

3:00 Diamonds as X-ray Linear and Nonlinear Optical Elements
Kenji Tamasaku

3:15 Focusing Mirror for X-ray Free Electron Laser
H. Mimura

3:30 **Break**

4:00 Monolithic Monochromators at ESRF
Trevor Mairs

4:15 ESRF Nanotechnology Platform
Ray Barrett

4:30 Crystal Surface Optimization for Bonse-Hart USAXS Instrument at APS
Ruben Khachatryan

4:45 Assessing the Quality of X-ray Optic Surfaces of Si Crystals Cut by Diamond-Wire and Rotating Blade Sawing Techniques
Michael Wieczorek

5:00 **APS Reception**

5:30 **Bus to Fermi Lab and workshop dinner at Chez Leon restaurant**

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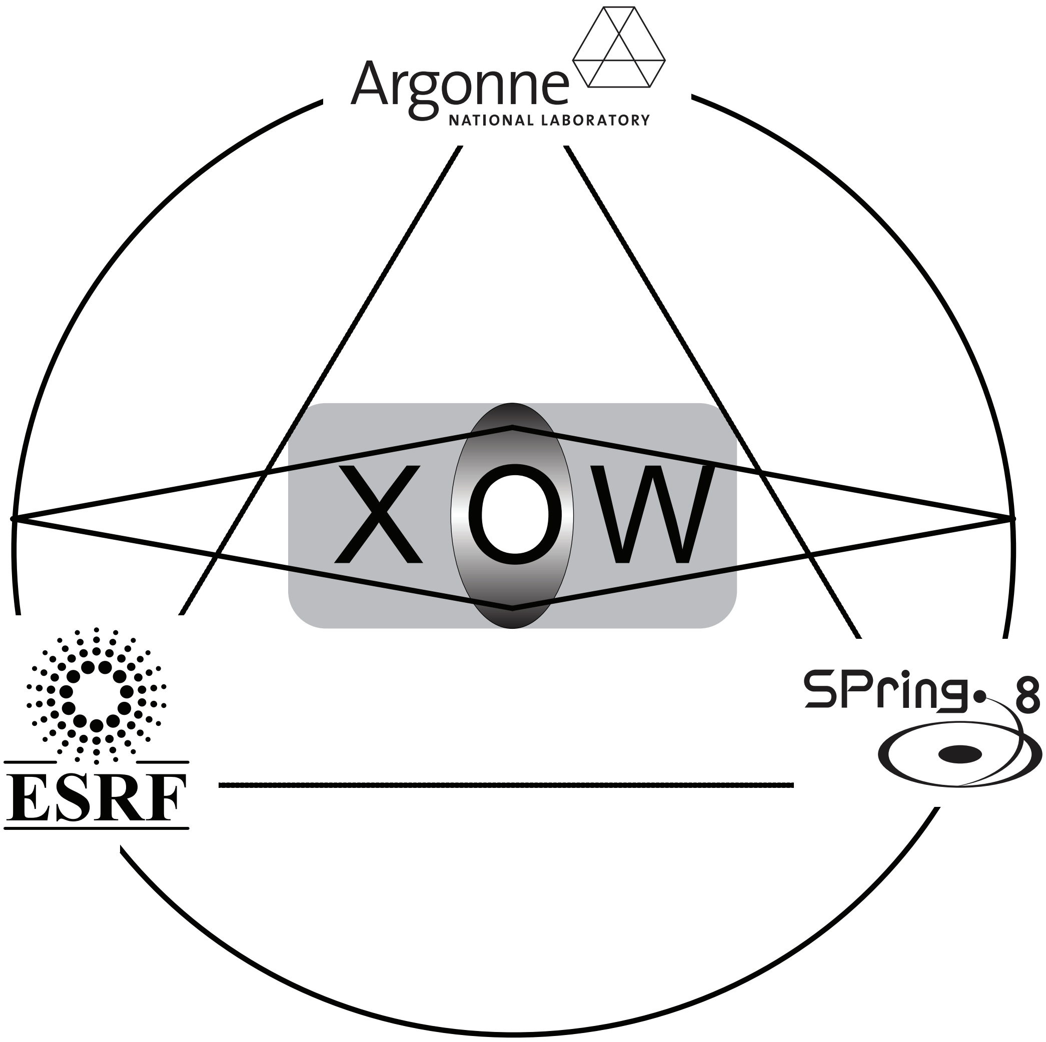
Presentation Abstracts



Overview of Optics at the Advanced Photon Source

A. Macrander

Optics Fabrication and Metrology Group, X-ray Science Division

Abstract

First , a general overview of the present research and development efforts on optics for use at the APS will be presented. Second, recent results for wave optical simulations and for a topography study of type IIa diamonds will presented.

This work supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

X-ray optics related news from the ESRF

Jürgen Härtwig, ESRF Optics Group

A short review over recent X-ray optics developments, plans and problems will be given:
- changes in laboratory equipment,
- test possibilities for cryogenic cooling and high heat load conditions,
- generic variants of high-heat-load channel-cut, multilayer and "pseudo-channel-cut" monochromators as well as mirrors.
- improvement of the coherence properties of high-heat-load double monochromators

In some more detail we will report about the following topics:
- contact less temperature measurement of diamond plates,
- mounting of small single crystal diamonds onto other large area diamonds,
- X-ray topography and high-resolution diffraction on type IIa HPHT diamonds.

Overview of optics at SPring-8

Shunji Goto

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sgoto@spring8.or.jp

Recent topics regarding optics developments at SPring-8 will be presented:
(1) Crystal monochromators including characterization of IIa synthetic diamond for double crystal setting at 1 km beamline,
(2) K-B mirror development toward sub-10 nm focusing and for a large-size elliptical mirror for XFEL application in collaboration with RIKEN and Osaka University,
(3) Characterization PVD beryllium and CVD diamond using coherent x-rays, and
(4) Upgrade of metrology laboratory.

Focusing hard X-rays to sub-10 nm by reflective optics

Kazuto Yamauchi and Hidekazu Mimura

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Hard X-ray focusing system using KB mirrors is a promising device from the viewpoints of high efficiency and large aperture. The collaboration between SPring-8 and Osaka University was started, 7 years ago. We have been developing various technologies regarding KB mirrors as follows;

1) Computer-controlled figuring system (EEM, PCVM)
2) Surface smoothing technique (EEM)
3) Interferometric metrology for surface figure measurement of X-ray mirror optics (MSI, RADSI)
4) Wave-optical simulator
5) Compact mirror positioning device for alignment
6) At-wavelength metrology using phase retrieval method
7) Multilayer deposition system
8) High resolution intensity profile measurement of hard X-ray nanobeam

The fabricated mirrors are evaluated at 1km-long beam line (BL29XUL) of SPring-8. We achieved a focal size of 25 nm at 15 keV, which is the same as diffraction limited focused beam size. High focusing efficiency was also confirmed.

We already started its application to fluorescence X-ray nanoscopy/spectroscopy and transfer of these fabrication technologies to a venture company (JTEC, Japan).

Now, we are promoting sub-10 nm hard X-ray focusing project. At-wavelength metrology is being developed, in which phase-retrieval simulator was coded for determining phase errors on mirror surfaces from only intensity profiles of a focused beam. Multilayer deposition system was also completed.

In our presentation, we will talk about our fabrication technologies and the latest results in this project.

Progress and perspectives at the ESRF multilayer facility

Christian Morawe, ESRF Optics Group

The new ESRF multilayer deposition facility was installed and tested in 2007. Initial results of the tests and of the ongoing commissioning activity will be summarized. A comparison with the performance of the old deposition system will complement the description.

The potential impact of the new equipment on multilayer based x-ray optics will be outlined. Here, KB focusing devices and high heat load double monochromators are key elements and will play a fundamental role in the technical development for the ESRF Upgrade Programme.

Profile Coating for Elliptical KB Mirrors and Wedged Multilayer Laue Lens

Chian Liu

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Profile coating is a coating technique using a mask with a calculated aperture and substrate motion to produce a desired surface profile along the direction perpendicular to the moving direction. The shape of the mask contour depends on the desired profile and the thickness distribution directly above the sputter gun at the substrate level. Profile coating has been successfully applied to produce elliptical Kirkpatrick-Baez (KB) mirrors using both cylindrical and flat super-polished Si substrates [1, 2]. An 85 nm focusing has been achieved using Au-profile-coated KB mirrors with flat Si substrates [3]. Precision elliptical KB mirrors with sub nm figure errors have been routinely produced with both Au and Pt coatings on flat substrates. In this talk I will present our recent results of profile coated KB mirrors and discuss the stability issues of these mirrors.

1. C. Liu, L. Assoufid, R. Conley, A. T. Macrander, G. E. Ice, and J. Z. Tischler, "Profile coatings and its application for KB mirrors," Opt. Eng. 42(12), 3622, 2003.

2. C. Liu, R. Conley, L. Assoufid, Z. Cai, J. Qian, and A. T. Macrander, "From flat substrate to elliptical KB mirror by profile coating," AIP Conf. Proc. 705, 704, 2004.

3. W. Liu, G.E. Ice, J.Z. Tischler, A. Khounsary, C. Liu, L. Assoufid, A.T. Macrander, Rev. Sci. Instrum. 76, 113701 (2005)

This work is supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

Wedged MLL Growth in the Rotary Deposition System

R. Conley[†,1] C. Liu[1], J. Qian[1], C. M. Kewish[1], A. T. Macrander[1], H. Yan[1,2], H. C. Kang[2,3], J. Maser[1,2], and G. B. Stephenson[2,3]

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2) Center for Nanoscale Materials, Argonne National Laboratory, Argonne, Illinois 60439

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ABSTRACT

A Multilayer Laue Lens (MLL) is an X-ray focusing optic fabricated from a multilayer structure consisting of thousands of layers of two different materials produced by thin-film deposition where each layer satisfies the Fresnel zone plate law. An improved MLL geometry can be created by growing each layer with an in-plane thickness gradient to form a wedge, so that every interface makes the correct angle with the incident beam for symmetric Bragg diffraction [1]. The ultimate hard X-ray focusing performance of a wedged MLL has been predicted to be significantly better than that of a non-wedged MLL, giving sub-1 nm resolution with high efficiency. Here we describe a method to deposit the multilayer structure needed for an ideal full-wedged MLL and highlight our latest deposition results on a 40μm thick structure consisting of 6,543 layers with 3nm outermost zones grown in the APS rotary deposition system [2]. Our use of a KSA-MOS in-situ stress monitor for stress mitigation to allow for growth of wedged MLLs with thicknesses in the tens of micrometer range is presented.

† electronic mail: rconley@aps.anl.gov

This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

[1] R. Conley, C. Liu, J. Qian, C.M. Kewish, A.T. Macrander, H. Yan, H.C. Kang, J. Maser, G.B. Stephenson, "Wedged multilayer Laue lens," Rev. Sci. Instrum., submitted.

[2] R. Conley, C. Liu, C.M. Kewish, A.T. Macrander, C. Morawe, "Multilayer Growth in the APS Rotary Deposition System", in Advances in X-Ray/EUV Optics, Components, and Applications, A.M. Khounsary, C. Morawe, eds., *Proc. SPIE* **670404**, (2007).

Fabrication of Aspheric Optics for X-ray Applications

Ali Khounsary
Advanced Photon Source
Argonne National Laboratory
Argonne, IL 60439

Reflective optics provides a flexible and versatile means for spatial and/or spectral conditioning of an incident X-ray beam. One of its prime advantages is achromatic collimation and focusing. A wide variety of coating selections offers a broad range of reflectivity, numerical aperture, and spectral cut-off possibilities. And an almost infinite permutation of possible multilayer coatings allows the generation of quasi-monochromatic beams with tailored spectra and reflectivities. Thin reflective optical substrates allow dynamic bending for a range of focal lengths and focus sizes. Thicker substrates can be polished and configured into various shapes including aspheres.

Yet, for many of the state-of-the-art applications, the fabrication of the substrate remains the critical issue, influenced by material selection, dimensional stability, shape, figure, finish and, of course, cost. This is especially true for aspherical substrates such as elliptical and parabolic substrates, which are used, respectively, for focusing and collimating X-rays. This presentation describes some of the fabrication options available and shows how a combination of the techniques might provide state-of-the-art substrates at a reasonable cost.

Results of hard-x-ray mirror metrology round-robin carried out at the APS, ESRF and SPring-8 metrology laboratories

L. Assoufid[a], A. Rommeveaux[b], H. Ohashi[c], H. Mimura[d], K. Yamauchi[d], J. Qian[a], T. Ishikawa[c,e], C. Morawe[b], A. T. Macrander[a], A. Khounsary[a], S. Goto[c]

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[b] ESRF, 6 rue J. Horowitz, BP 220, 38043 Grenoble, France
[c] JASRI SPring-8, 1-1-1 Kouto Sayo, Hyogo 679-5198, Japan
[d] Department of Precision Science and Technology, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka, 565-0871, Japan
[e] Riken SPring-8 Center, 1-1-1 Kouto Sayo, Hyogo 679-5148, Japan

ABSTRACT

Two series of round-robin (RR) measurements have been carried out at the APS, ESRF, and SPring-8 metrology facilities to compare and improve measurements with their respective long trace profilers (LTPs). The first series of RR measurements, carried out in 2005, focused on large, flat mirrors and showed an excellent agreement amongst the three LTPs [2]. The second RR series of measurements, carried out in 2007, focused on high quality, elliptical and spherical mirrors, and revealed that measurement of mirrors with a strong surface slope variation, such as elliptical mirrors for hard x-ray nanofocusing, is strongly affected by LTP systematic errors [3]. As a result, we have devised a new measurement procedure that significantly mitigates the effect of LTP systematic errors. With the new measurement procedure, the three LTPs agreed within <0.2 µrad for slope error, <0.2 nm for shape error, and <0.3% for radius of curvature measurements. For both series of RR measurements, LTP results were confirmed by microstitching data obtained at Osaka University (Japan). The effectiveness of the elliptical and spherical mirror measurement procedure was further confirmed by measurements carried out using *in situ* x-ray profilometry [3].

ACKNOWLEDGEMENTS

The work at Argonne was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

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[1] L, Assoufid, A. Rommeveaux, H. Ohashi, K. Yamauchi, H. Mimura, J. Qian, O. Hignette, T. Ishikawa, C. Morawe, A. Macrander, A. Khounsary, and S. Goto, Proc. SPIE **5921,** 129-140 (2005).

[2] A. Rommeveaux, L. Assoufid, H. Ohashi, H. Mimura, K. Yamauchi, J. Qian, T. Ishikawa, C. Morawe, A.T. Macrander, A. Khounsary, S. Goto, SPIE Proc. **6704**, 67040B-1(2007).

[3] L. Assoufid, O. Hignette, M. Burghamer, C. Riekel, A. Rommeveaux, J. Qian, C. Liu, R. Conley and A. T. Macrander, unpublished data of measurements carried out using x-ray pencil beam profilometry on the ESRF 13-ID beamline (May 2007).

Current status on metrology laboratory at SPring-8

Haruhiko Ohashi

JASRI/SPring-8, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan,
hohashi@spring8.or.jp

Optical metrology laboratory at SPring-8 has some standard instruments such as a long trace profiler (LTP), an atomic force microcopy (AFM), an optical surface profiler, a stylus surface profiler, and a Fizeau interferometer. In order to meet rising demands for measurement of various optical elements from beamline scientists as well as optics group staffs, technical experts have been trained to operate these instruments for two years.

New instruments for high-quality mirrors are being developed. Two types of the stitching interferometer for a long mirror, the micro stitching interferometer (MSI) and the relative angle determinable stitching interferometer (RADSI) have been developed in collaboration with Osaka University, JTEC Corporation, and RIKEN. These are also used for development of an XFEL mirror.

A long trace profiler (LTP) is being upgraded in order to improve accuracy of slope measurement for a long aspherical mirror. Major parts except optical elements will be changed on this March. A scanning mechanics of pencil beam is replaced with a new air slider of ceramics designed to ensure high repeatability. The aged detector has been replaced with a new CCD camera. We have updated control system and developed control software.

A booth of 15 m (W) × 8 m (D) ×3 m (H) was constructed in experimental hall. It is divided into two rooms. One is a simple clean room for development of new optical instruments. The other is used for test of new vacuum components.

Comparison of slope and height profiles for flat synchrotron x-ray mirrors measured with a long trace profiler and a Fizeau interferometer*

Jun Qian, Lahsen Assoufid and Albert Macrander
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

ABSTRACT

Long trace profilers (LTPs) have been used at many synchrotron radiation laboratories worldwide for over a decade to measure surface slope profiles of long grazing incidence x-ray mirrors. Phase measuring interferometers (PMIs) of the Fizeau type, on the other hand, are being used by most mirror manufacturers to accomplish the same task. However, large mirrors whose dimensions exceed the aperture of the Fizeau interferometer require measurements to be carried out at grazing incidence, and aspheric optics requires the use of a null lens. While an LTP provides a direct measurement of 1D slope profiles, PMIs measure area height profiles from which the slope can be obtained by a differentiation algorithm. Measurements of the two types of instruments have been found by us to be in good agreement. This paper documents that comparison. We measured two different nominally flat mirrors with both the LTP in operation at the Advanced Photon Source (a type-II LTP) and a Fizeau-type PMI interferometer (Wyko model 6000). One mirror was 500 mm long and made of Zerodur, and the other mirror was 350 mm long and made of silicon. Slope error results with these instruments for the Zerodur mirror agree within nearly 100%. A significant difference was observed with the much higher quality silicon mirror.

This work is supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE- AC02-06CH11357.

Diamonds as x-ray linear and nonlinear optical elements

Kenji Tamasaku, Hiroshi Yamazaki*, Motohiro Suzuki*, Yoshinori Nishino, Shunji Goto*, and Tetsuya Ishikawa

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*JASRI , 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan

This talk will overview the recent results using high-quality diamonds, especially, as monochromator crystals, and nonlinear crystals.

The beam image of double crystal setup was measured under highly coherent illumination at the 1-km beamline of SPring-8. The defects produced intensity modulation in the far field image, whereas they were hardly recognized at near field. The observation indicated clearly that defect-free diamonds (or defect-free area with sufficient dimensions) are required to handle coherent x rays.

Diamonds are good nonlinear media to investigate x-ray nonlinear optics, because of higher radiation hardness, lower absorption, and higher crystallinity. Some novel features observed in the parametric down-conversion of x rays into extreme ultraviolet [1] will be presented.

References

[1] K. Tamasaku, and T. Ishikawa, Phys. Rev. Lett. 98, 244801 (2007).

Focusing mirror for X-ray free electron laser

H. Mimura[1], S. Morita[2], T. Kimura[1,] D. Yamakawa[1], W. Lin[2], T. Uehara[2], H. Yumoto[1,3], S. Matsuyama[1], Y. Nishino[4], K. Tamasaku[4], H.Ohashi[3], M. Yabashi[3], T. Ishikawa[3,4], H. Omori[2], K. Yamauchi[1]

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As coming forth generation X-ray sources, X-ray free electron laser (XFEL) facility is being constructed at DESY, LCLS and SPring-8. Focusing XFEL will produce the ultimately high photon density beam.

We present the fabrication of a large-size total reflection mirror for focusing XFEL to nanometer dimension. The focusing mirror has an elliptical curved shape with a length of 400 mm and focal length of 550 mm.

ELectrolytic In-process Dressing Grinding (ELID) is used for rapid rough figuring. Elastic Emission Machining (EEM) is employed for final figuring and surface smoothing. Figure accuracy with peak-to-valley height of 2 nm was achieved. The focusing test was performed at BL29XUL of SPring-8. The focused beam size is approximately 80 nm at 15 keV, which is the almost same as theoretical size.

Monolithic monochromators at ESRF

Trevor Mairs, ESRF, Technical Beamline Support

Over the past 10 years there have been nine monolithic channel-cut monochromators installed at ESRF on undulator beamlines. This appears to be in contradiction to the tendencies at other 3rd generation light sources. The advantages of the channel-cut option are discussed. The obvious disadvantage of non-fixed exit and manual polishing of the crystals is addressed. The status of the development of a Z-shaped channel-cut monochromator is given.

The ESRF nanotechnology platform

Ray Barrett, ESRF X-ray Imaging Group

A significant fraction of the upgrade program of the ESRF involves the development of nanoprobe and nano-imaging beamlines. The experience of the ESRF in developing instruments with micron or sub-micron lateral resolution acquired over the past 10 years will provide an important foundation for these new programs. Furthermore, in the medium-term plans, several projects are currently under development (upgrades of ID11 and ID13, ID22NI…) and are considered here as pilot projects. A nanotechnology platform was created to coordinate the R&D programs related to the development of new nanoprobes. This platform has two primary objectives and aims at being:

▪ A body of technical experts which will provide
- Interface with industrial suppliers
- Assistance for specification and definition of instruments
- Skilled resources for specific instrument developments
- Validation and commissioning of new solutions (standardisation)
- Technical survey and documentation

▪ An advisory structure for management on
- Resource management and budget allocations
- Coordination with other facilities (e.g. X-ray optics)
- Participation in European networks

Crystal surface optimization for Bonse-Hart USAXS instrument at APS

R. Khachatryan, J. Ilavsky, M Wieczorek, and A. Macrander

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Bonse-Hart design of USAXS instrument is highly efficient method to characterize microstructures over extended range - from micron sizes down to nanometers. However, its performance is closely related to the rocking curve profiles of the crystals, which is related to their surface finish. USAXS instrument at APS, currently located on beamline 32ID, has been testing and improving the surface quality of the crystals over last year or so, resulting in reduction of instrumental background by about factor of 5 - 10 in selected scattering angle ranges. This significantly improved the performance and increased the range of applications for which this instrument can be used. The Fabrication steps for optical components and test results will be presented and discussed.

This work was performed under the auspices of the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-AC02-06CH11357.

Assessing the Quality of X-ray Optic Surfaces of Si Crystals Cut by Diamond-Wire and Rotating Blade Sawing Techniques

Michael Wieczorek [a], Xianrong Huang [a], Jozef Maj [a], Ray Conley [a], Jun Qian [a], Albert Macrander [a], Cynthia Christensen [b], and Ruben Khachatryan [a]

[a] X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439
[b] Diamondwire Technology, Colorado Springs, CO 80916

ABSTRACT

The next generation of X-ray diffraction optics will benefit from crystal surfaces with very high quality (extremely flat and strain-free), but knowledge on how to achieve such surfaces and how surface imperfections affect the diffraction properties is sparse in the literature. As a first step to initialize a systematic study on this topic, we evaluate in this paper the surface quality of two Si (111) wafers cut by a diamond-wire saw and a rotating blade saw, respectively. We concentrate on revealing lattice strains induced by the two cutting methods and on strain evolution during three rounds of chemical etching (without polishing). The measurements also provide some important clues as to how surface roughness affects rocking curve widths and other diffraction properties.

This work was performed under the auspices of the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-AC02-06CH11357.

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