Physics of X-Ray and Neutron Multilayer Structures

7-9 Nov 2018

Palaiseau (France)

Welcome address

The 2018 Physics of X-Ray and Neutron Multilayer Structures Workshop follows the fruitful PXRNMS 2016 Workshop organized by the colleagues of the Industrial Focus Group XUV at MESA+ at the University of Twente, The Netherlands, as well as the former PXRMS conferences that were successfully organized for many years but ended in 2010.

The Workshop is devoted to the physics of multilayer interference coatings designed for x-ray radiation or neutron beams, from their design and preparation to their characterization and application. Multilayers are nowadays essential components in a multitude of scientific and technological applications and are required to perform increasingly complex functions and to maintain stable performance in various environments. The detailed studies of multilayer structures presented in this Workshop from the materials science, solid state physics, surface and interface physics, x-ray and neutron optics and application points of view, will contribute towards further advancements in multilayer performance.

We would like to thank our institutional and commercial Sponsors for their financial support, as well as the Program Committee and the Local Organizing Committee for their assistance and involvement in the scientific and practical organization of the Workshop.

We wish you a productive Workshop and a pleasant stay in Palaiseau and Paris.

Philippe JONNARD, Franck DELMOTTE

Topics of the Workshop

The Workshop will focus on the physics of nanometer-scale multilayer films optimized for various applications in the Extreme Ultraviolet (EUV) and X-ray domains as well as neutron optics. The main topics that will be discussed include:

Multilayer X-Ray and Neutron Optics
Multilayer Design and Modeling
Film Growth and Microstructure
Roughness and Interface Formation
Surface and Thin-Film Modifications using Ion Beams
Film Removal Techniques
Growth Models and Computer Simulations
X-Ray and Neutron Scattering
Surface and Interface Topography
Layer and Interface Composition
Wavefront Characterization and Correction
Mechanical Properties and Stability
Optical Properties
Novel Characterization Techniques
Polarization Control
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Applications

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- Dr. Eric Ziegler, European Synchrotron Radiation Facility, Grenoble, France

Useful information

Please see the leaflet in your bag.
Do not hesitate to contact any member of the local organizing committee for information.

Invited lectures

Keynote speaker

David Attwood (University of California, Berkeley, USA)
Advances in X-ray and EUV Optics Lead to Broadly Based Scientific and Industrial Opportunities

Invited speakers

Catherine Burcklen (Lawrence Livermore National Laboratory, USA)
Advances in hard x-ray aperiodic multilayers for imaging with large field of view

Ani Chandrasekaran (University of Twente, The Netherlands)
Metal-on-metal interface formation laws of nanoscale thin films

Elena Filatova (Saint-Petersburg State University, Russia)
X-Ray Spectroscopic Methods for Thin Films and Interfaces Study

Qiushi Huang (Tongji University, China)
Multilayer coated nanogratings with sub-50 nm periodicity for ultrahigh resolution soft X-ray spectroscopy

Ryuji Maruyama (Japan Atomic Energy Agency, Japan)
Recent progress in the development of neutron polarizing supermirror at J-PARC

Christian Morawe (European Synchrotron Radiation Facility, France)
Multilayer x-ray optics at the ESRF

Program of the Workshop

Wednesday, November 7, 2018

Time	*Event*
08:00 - 09:10	**Registration and Welcome coffee** (Coffee break and exhibition hall)
09:10 - 09:20	**Welcome speech** (Conference room)
09:20 - 10:40	**Session 1** (Conference room) – Chairwoman: Regina Soufli
09:20 - 10:00	› Advances in X-ray and EUV Optics Lead to Broadly Based Scientific and Industrial Opportunities - *David Attwood, University of California, Berkeley (keynote talk)*
10:00 - 10:20	› Piezoelectric actuators for adaptive multilayer mirrors in XUV lithography systems - *Mohammadreza Nematollahi, University of Twente, The Netherlands*
10:20 - 10:40	› Extreme ultraviolet multilayers for solar physics applications - *Alain Jody Corso, National Research Council of Italy*
10:40 - 11:05	**Coffee break** (Coffee break and exhibition hall)
11:05 - 12:30	**Session 2** (Conference room) – Chairman: Qiushi Huang
11:05 - 11:30	› Recent progress in the development of neutron polarizing supermirror at J-PARC - *Ryuji Maruyama, J-PARC Center, Japan Atomic Energy Agency (invited)*
11:30 - 11:50	› Improved interface widths in Ni/Ti multilayer mirrors by ion-assisted B4C co-deposition - *Sjoerd Broekhuijsen, Thin Film Physics Division, Department of Physics, Chemistry and Biology (IFM), Linköping University, Sweden*
11:50 - 12:10	› Neutron polarizing supermirrors for the WASP instrument at the ILL: the end of a mass production coating project. - *Thierry Bigault, Institut Laue-Langevin*
12:10 - 12:30	› Large area neutron supermirror and X-ray multilayer mirror deposition facility at Bhabha Atomic Research Centre INDIA - *Arup Biswas, Bhabha Atomic Research Centre*
12:30 - 14:00	**Lunch** (Canteen)
14:00 - 15:45	**Session 3** (Conference room) – Chairman: Eric Louis
14:00 - 14:25	› Metal-on-metal interface formation laws of nanoscale thin films - *Anirudhan Chandrasekaran, Industrial Focus Group XUV Optics, MESA+ institute for Nanotechnology, University of Twente, The Netherlands (invited)*
14:25 - 14:45	› Optical constants with improved accuracy in the regions of L-, M- N- and O-absorption edges and their impact in multilayer modeling - *Regina Soufli, Lawrence Livermore National Laboratory / Laboratoire Charles Fabry*
14:45 - 15:05	› Growth of Mo/Si multilayers at oblique deposition induces periodical nano-arrays - *Dmitriy Voronov, Lawrence Berkeley National Laboratory*
15:05 - 15:25	› Atomic oxygen diffusion in ultrathin transition metal oxide films at near room temperature - *Cristiane Stilhano Vilas Boas, MESA+ Institute for Nanotechnology, University of Twente*
15:25 - 15:45	› Manufacturing and characterization of substrates for imaging multilayer X-ray optics - *Nikolai Chkhalo, Institute for Physics of Microstructures of Russian Academy of Sciences*

Time	Event
15:45 - 18:00	**Poster session and coffee break** (Coffee break and exhibition hall)

› Advancing X-ray standing wave data analysis - *Igor Makhotkin, University of Twente*

› Angular distribution of a characteristic x-ray emission transmitted by a periodic multilayer - *Karine Le Guen, Laboratoire de Chimie Physique - Matière et Rayonnement, Sorbonne Université-CNRS*

› ATTOLAB SE10: a versatile and integrated beamline for attosecond physics on gases and surfaces - *David Bresteau, LIDYL, CEA, CNRS, Université Paris-Saclay, CEA Saclay, Gif-sur-Yvette, France*

› Characterization of a monochromatic x-ray imaging using Fresnel zone plates and a multilayer mirror - *Annaig CHALEIL, CEA DAM*

› Characterization of a multilayer x-ray waveguide by x-ray diffuse scattering and grazing incidence x-ray fluorescence - *Philippe Jonnard, Laboratoire de Chimie Physique - Matière et Rayonnement - Jussieu*

› Combined XRR-GIXRF analysis at SOLEIL - *Yves Ménesguen, CEA*

› Dedicated X-ray optics for new generation small X-ray sources - *Markus Krämer, AXO DRESDEN GmbH*

› Derivation of beryllium optical constants from Mo/Be multilayers - *Mewael Giday Sertsu, BESSY II, Helmholtz - Zentrum Berlin*

› Explosive crystallization of Co layers in C/Co/C three-layers - *Oleksandr Devizenko, National Technical University “Kharkiv Polytechnic Institute”*

› Influence of nanometer protective layers on temporal stability and mechanical properties of thin-film aluminium absorption filters - *Nikolay Tsybin, Institute of Applied Physics of RAS*

› Layer structure and phase composition in W/Si multilayer X-ray mirrors - *Vitalii Chumak, National Technical University “Kharkiv Polytechnic Institute”*

› Modeling of EUV multilayers and filters for solar physics and evaluation of tabulated optical constants - *Jennifer Rebellato - Centre National d'Études Spatiales / Laboratoire Charles Fabry*

› Multilayer-based high-precision polarimeter at Diamond Light Source - *Hongchang Wang, Diamond Light Source*

› Observation of Polymer Blend Films on Transmission EUV Microscopy - *Mitsunori Toyoda, Tohoku University [Sendai], Tokyo Polytechnic University*

› Optical properties of graphene at hydrogen Lyman alpha - *Nadeem Ahmed Malik, Department of Information Engineering, University of Padova, Italy / CNR-IFN UOS Padova*

› Progress on Multilayer-coated Optics for High Energy X-ray Imaging Tools at the US National Labs - *Chris Walton,* Lawrence Livermore National Laboratory

› Spatially resolved EUV and XUV reflectometry - *Frank Scholze, Physikalisch-Technische Bundesanstalt*

Time	*Event*
	› Study on propagation of interface imperfections across Co/Ti multilayers with ultra-short perio *Piyali Sarkar, Arup Biswas and Dibyendu Bhattacharyya, Bhabha Atomic Research Centre*
	› Surface and interface observation of Zirconium oxynitride films as well as TiN/ZrON bilayers - *Yanyan Yuan, Jiangsu University of Science and Technology*
	› Trends on Montel X-ray Optics for Inelastic Scattering and Microfocus X-ray Sources for X-ray Diffractometry - *Frank Hertlein, incoatec GmbH*

Thursday, November 8, 2018

Time	*Event*
09:05 - 10:30	**Session 4** (Conference room) – Chairman: Eric Ziegler
09:05 - 09:30	› Advances in hard x-ray aperiodic multilayers for imaging with large field of view - *Catherine Burcklen, Lawrence Livermore National Laboratory (invited)*
09:30 - 09:50	› Multilayer mirrors based on beryllium for an extreme ultraviolet range - *Vladimir Polkovnikov, Institute for Physics of Microstructures of the RAS*
09:50 - 10:10	› Recent advances in development of Cr/Sc based reflective multilayer coatings for x-ray applications - *Evgueni Meltchakov, Laboratoire Charles Fabry*
10:10 - 10:30	› Chromatic Aberration Control on Microscope Imaging System with EUV Multilayer Mirrors - *Mitsunori Toyoda, Tohoku University [Sendai], Tokyo Polytechnic University*
10:30 - 10:55	**Coffee break** (Coffee break and exhibition hall)
10:55 - 12:40	**Session 5** (Conference room) – Chairwoman: Maria Pelizzo
10:55 - 11:20	› X-Ray Spectroscopic Methods for Thin Films and Interfaces Study - *Elena Filatova, Institute of Physics, St-Petersburg State University, St. Petersburg (invited)*
11:20 - 11:40	› Proximity effect in Co/Pt multilayer investigated through X-ray resonant magnetic reflectivity - *Adriano Verna, Università degli Studi Roma Tre*
11:40 - 12:00	› Angle-Resolved XRF for Depth-Resolved Elemental Analysis of Stratified Materials in the Laboratory - *Jonas Baumann, Technical University Berlin*
12:00 - 12:20	› Correlated lateral density fluctuations in the Si layers of a W/Si multilayer revealed by GISAXS - *Igor Makhotkin, University of Twente*
12:20 - 12:40	› Study of Pd/Y multilayers with B4C barrier layers using GIXR and x-ray standing wave enhanced HAXPES - *Meiyi Wu, Sorbonne Université, Faculté des Sciences et Ingénierie, UMR CNRS, Laboratoire de Chimie Physique - Matière et Rayonnement*
12:40 - 14:00	**Lunch** (Canteen)
14:00 - 17:00	**Lab Tour** - SOLEIL SYNCHROTRON or ATTOLAB or LCF (pre-registration required).
19:00 - 22:30	**Conference Dinner** - Train Bleu restaurant in Gare de Lyon, Paris

Friday, November 9, 2018

Time	*Event*
09:05 - 10:30	**Session 6** (Conference room) – Chairman: Thierry Bigault
09:05 - 09:30	› Multilayer coated nanogratings with sub-50 nm periodicity for ultrahigh resolution soft X-ray spectroscopy - *Qiushi Huang, Key Laboratory of Advanced Micro-Structured Materials, Institute of Precision Optical Engineering, Tongji University (invited)*
09:30 - 09:50	› Gratings for VUV to X-ray applications - *François Polack, Synchrotron SOLEIL*
09:50 - 10:10	› Highly efficient multilayer-coated blazed gratings for the tender X-ray energy range - *Andrey Sokolov, Helmholtz-Zentrum Berlin für Materialien und Energie, BESSY-II, Berlin, Germany*
10:10 - 10:30	› Nearly perfect blaze of the X-ray multilayer-coated gratings with non-conformal boundaries - *Maxim Lubov, St. Petersburg Academic University*
10:30 - 10:55	**Coffee break** (Coffee break and exhibition hall)
10:55 - 12:40	**Session 7** (Coffee break and exhibition hall) – Chairman: Nicholai Chkhalo
10:55 - 11:20	› Multilayer x-ray optics at the ESRF - *Christian Morawe, The European Synchrotron (invited)*
11:20 - 11:40	› Research activity of evaluation tools including soft X-ray optics for the research of EUV lithography at University of Hyogo - *Takeo Watanabe, Center for EUVL, Laboratory of Advanced Science and Technology for Industry, University of Hyogo*
11:40 - 12:00	› High reflectance Al/MgF2 mirrors by hot deposition - *Juan Larruquert, Instituto de Optica-Consejo Superior de Investigaciones Científicas*
12:00 - 12:20	› Broad Band EUV/VUV Multilayer Coatings from 16.9 to 130 nm for a Solar Spectrograph Space Mission - *Tobias Fiedler, optiX fab GmbH, Jena*
12:20 - 12:40	› Thin-film preparation and characterization at the HZG - *Michael Störmer, Helmholtz-Zentrum Geesthacht*
12:40 - 14:00	**Lunch** (Canteen)
14:00 - 15:40	**Session 8** (Conference room) – Chairman: Hartmut Enkisch
14:00 - 14:20	› Photon-induced damage processes in Ruthenium thin films developed for Free Electron Laser optics - *Eric Louis, MESA+ Institute for Nanotechnology, University of Twente*
14:20 - 14:40	› Coatings in harsh space environment - *Maria Guglielmina Pelizzo, National Reseach Council of Italy*
14:40 - 15:00	› A compact soft X-ray reflectometer based on a multi-spectral fluorescence point source - *Alexei Erko, Institut für angewandte Photonik (IAP), Berlin*
15:00 - 15:20	› X-EUV/XRay Hartmann wavefront sensing for at-wavelength metrology and correction - *Fabrice Harms, Imagine Optic*
15:20 - 15:40	› Kossel interferences of proton-induced X-ray emission lines to study the interfaces of thin film waveguides - *Jiaping Zhang, Sorbonne Université, State Key Laboratory of Solidification Processing, Northwestern Polytechnical University*
15:40 - 15:50	**Closing speech** (Conference room)

Advances in X-ray and EUV Optics Lead to Broadly Based Scientific and Industrial Opportunities

David Attwood, University of California, Berkeley

In this overview we discuss advances in x-ray and extreme ultraviolet (EUV) optics, including multilayer coatings, which have provided many new science opportunities in the fields of solar physics, coherent sources, microscopy and photolithography.

Examples will include optics for the many synchrotron and free electron laser (FEL) facilities, but also with laser produced plasmas, such as employed in the present march to high volume manufacturing of computer and smart phone chips. Challenges for EUV and X-ray optics in future applications will also be discussed.

Piezoelectric actuators for adaptive multilayer mirrors in XUV lithography systems

Mohammadreza Nematollahi[1], Philip Lucke[1], Muharrem Bayraktar[1], Kerstin Hild[2], Toralf Gruner[2], Andrey Yakshin[1], Guus Rijnders[3], Eric Louis[1], Fred Bijkerk[1]

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[2] *Carl Zeiss SMT GmbH, Rudolf-Eber-Str. 2, 73447 Oberkochen, Germany*

[3] *Inorganic Materials Science Group, MESA+ Institute for Nanotechnology, University of Twente, PO-Box 217, 7500AE, Enschede, The Netherlands*

Multilayer mirrors are key components in extreme ultraviolet (XUV) lithography systems. Despite the high reflection, they absorb some energy leading to heat load and temperature change of the substrate. The temperature change results in wavefront deformation and image distortion. Therefore, adaptive XUV mirrors are required in order to improve the resolving power of the imaging and projection systems [1]. In this work, we propose piezoelectric based adaptive multilayer mirrors (Figure 1a). We present the results for the piezoelectric actuator and demonstrate how the desired graduated profile can be achieved.

The piezoelectric film must be grown on an XUV compatible amorphous substrate. But direct deposition on such a substrate results in low crystalline quality and piezoelectric response. So we used a crystalline nanosheet seed layer that was coated on the substrate by the Langmuir Blodgett method. Consequently, the films grown on the nanosheets had a single out of plane (100) crystalline orientation.

The extension of the piezoelectric film through the thickness causes the film to contract laterally due to Poisson's ratio. This effect which lowers the effective out of plane extension is known as the substrate clamping. To minimize the clamping, the piezoelectric layer was deposited with a columnar microstructure (Figure 1b). The presence of a few nanometer void between the columns allowed a high piezoelectric coefficient above 400 pm/V. The columnar structure was achieved by optimizing the growth of the piezoelectric film and the bottom electrode.

Finally, in order to have a graduated surface profile we used a resistive *mediation layer* on the piezoelectric film (Figure 1c). The sheet resistance of the mediation layer was optimized in order to have a fast response while minimizing the resistive heating. The next step in fabrication of the adaptive mirrors is to add the wiring and isolation layers. It is also essential to reach a sub-nanometer smooth surface by a smoothening layer before the multilayer mirror can be deposited.

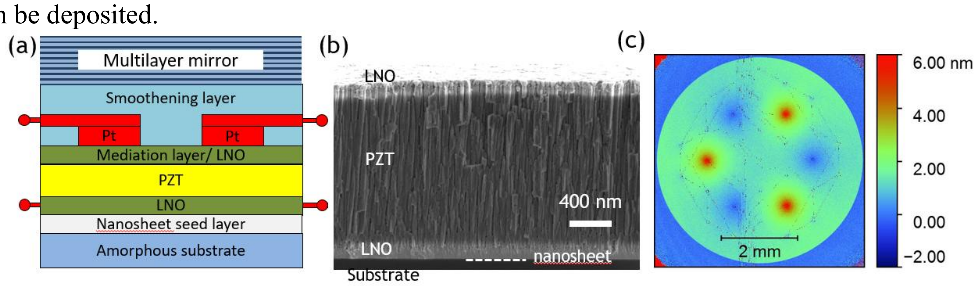

Figure 1: (a) Piezoelectric actuator under the multilayer mirror, (b) textured PZT film with columnar structure with LNO top and bottom electrodes all deposited by pulsed laser deposition, (c) An array of pixels are powered at different voltages to produce the surface profile; the graduated profile is achieved by the mediation layer.

References

[1] Muharrem Bayraktar, "Adaptive multilayer optics for extreme ultraviolet wavelengths", PhD thesis, University of Twente, Enschede, The Netherlands, 2015

Extreme ultraviolet multilayers for solar physics applications

A.J. Corso[1], M.G. Pelizzo[1]

[1] *National Research Council of Italy, Institute for Photonics and Nanotechnologies*

A review of extreme ultraviolet multilayers for solar physics applications is presented. Several promising material couples have been explored to optimize the peak reflectance at different wavelengths [1]. Ad hoc capping layers can be employed both to protect the structure underneath and to enhance the reflectance performance [2]. Such goal is achieved also by introducing an a-periodic design in the top most layers. The results are discussed also considering other features, such as spectral width and purity [3, 4]. Further improvement of related performance can be achieved by a-periodic designs, which provide novel solutions to be considered in view of future space missions. The proposed designs are discussed considering their stability in time and along space missions.

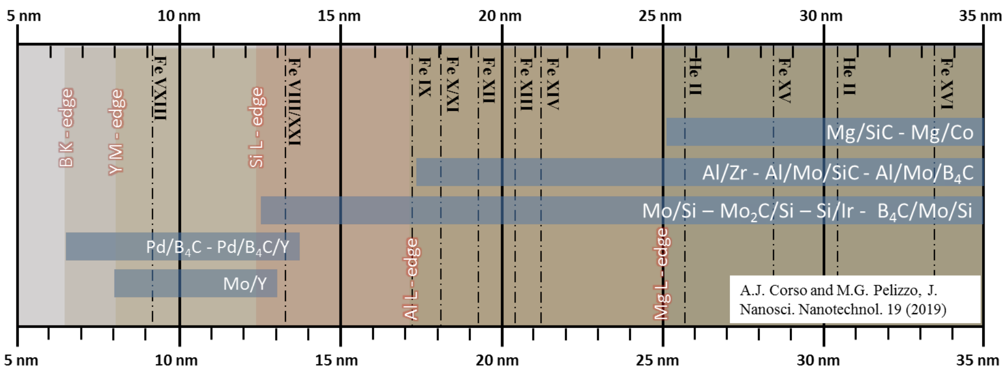

Figure 1: Chart of the most common materials employed for EUV multilayer stacks in the 8-35 nm spectral range.

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[2] A.J. Corso, P. Zuppella, P. Nicolosi, D.L. Windt, E. Gullikson, and M.G. Pelizzo, "Capped Mo/Si multilayers with improved performance at 30.4 nm for future solar missions", *Optics Express* 19(15), 13963–13973 (2011).

[3] A.J. Corso, P. Zuppella, D.L. Windt, M. Zangrando, and M.G. Pelizzo, "Extreme ultraviolet multilayer for the FERMI@ Elettra free electron laser beam transport system", *Optics Express* 20(7), 8006–8014 (2012).

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Recent progress in the development of neutron polarizing supermirror at J-PARC

R. Maruyama

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Neutron polarizing supermirror is one of the most important neutron optical elements for spin-polarization of neutron beams. It is a stack of alternating layers of ferromagnetic and non-magnetic materials with a variation in bilayer thickness to extend the bandwidth of the neutron spin-polarization. Polarizing supermirror needs to display high polarization efficiency over a wide bandwidth under low external field when it is used for time-of-flight neutron scattering instruments installed at spallation neutron sources such as the J-PARC. In this talk, the following topics on the development of the polarizing supermirror will be presented.

1) Possibility of fine-tuning of the scattering length density (SLD) for the ion beam sputtering (IBS) was explored to achieve higher polarization. The multilayer structure of Fe/Ge system used for the polarizing supermirror fabricated by the IBS was investigated using polarized neutron reflectivity and scanning transmission electron microscopy with energy-dispersive X-ray analysis. The obtained result revealed that the incorporation of sputtering gas particles (Ar) in the Ge layer gives rise to a marked reduction in the SLD and contributes to the SLD contrast between the Fe and Ge layers almost vanishing for spin-down neutrons. Bundesmann et al. have shown that the implantation of primary Ar ions backscattered at the target is responsible for the incorporation of Ar particles and that the fraction increases with increasing ion incidence angle and increasing polar emission angle [1]. Fe/Ge polarizing supermirror of which the critical angle is five times greater than Ni, fabricated under the same sputtering condition, showed a spin-up reflectivity of 0.70 at the critical angle. The polarization was higher than 0.97 over the entire bandwidth [2].

2) Dependence of the in-plane magnetic structure of the magnetic multilayer on the field strength was investigated by using polarized neutron off-specular scattering and grazing incidence small angle scattering measurements. The observation of the in-plane magnetic structure during the process of magnetization is important to gain insight into the mechanism that controls the magnetic properties which considerably differ from the bulk and to improve the magnetic properties in terms of softness and squareness. The magnetic properties of the multilayers with a nanocrystalline grain size less than the ferromagnetic exchange length can be understood by the random anisotropy model (RAM). The result showed that the in-plane length of the area with uniform orientation of spins does not increase with increasing field strength. The obtained magnetic behavior is in good agreement with the RAM. Since this model predicts that the reduction in the grain size and uniform uniaxial anisotropy will result in soft magnetic properties, this offers a possibility of further improvement in the magnetic properties.

References

[1] C. Bundesmann, R. Feder, R. Wunderich, U. Teschner, M. Grundmann, and H. Neumann, Thin Solid Films **589**, 487 (2015).

[2] R. Maruyama, D. Yamazaki, K. Akutsu, T. Hanashima, N. Miyata, H. Aoki, M. Takeda, and K. Soyama, Nucl. Instrum. Methods Phys. Res. A **888**, 70 (2018).

Improved interface widths in Ni/Ti multilayer mirrors by ion-assisted B_4C co-deposition

Sjoerd Broekhuijsen,[1] Fredrik Eriksson,[1] Naureen Ghafoor,[1] Daniel Ostach,[2] Norbert Schell,[2] and Jens Birch[1]

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The reflected absolute intensity as well as neutron energy range of state-of-the-art Ni/Ti multilayer neutron supermirrors are hampered by Ni/Ti interface widths. Typically, the interfaces are rather wide, about 0.7 nm, which is attributed to nanocrystallites, interdiffusion, and/or intermixing, thus limiting the optical contrast across the interface as well as the minimum possible layer thickness that can be used in a supermirror [1].

In this work we employ boron-carbon doping, in combination with modulation of ion-assistance during B_4C magnetron sputter co-deposition of the individual Ni and Ti layers, in order to eliminate nanocrystallites and minimizing kinetic roughening This approach has previously been shown to be a successful route for Cr/Sc X-ray multilayer mirrors [2].

In situ high-energy synchrotron wide angle X-ray scattering (WAXS) revealed an effective hindering of Ni/Ti crystallization through a minute concurrent B_4C flux during growth. Post-growth X-ray reflectivity (XRR) confirmed the incorporation of B_4C but also showed that interface widths deteriorated when a constant substrate bias of -30 V was used. However, by employing a two-stage substrate bias, where the initial 1 nm of each layer was grown with -30 V, followed by -100 V bias for the remaining of the layer, XRR showed that interface widths in B_4C-doped multilayers in the order of <0.45 nm were obtained. Cross-sectional transmission electron microscopy revealed smooth amorphous layers with no accumulated roughness.

The present results show that B-C doping of Ni/Ti multilayers leads to significantly smaller interface widths, compared to state-of-the-art, when combined with engineered interfaces through temporal control of the ion assistance. Simulations indicate significant improvements of neutron supermirror performance by employing this technique using ^{11}B isotope-enriched B_4C source material. Proof of concept is tested using neutron reflectivity at ILL.

References

[1] T. Verez, S. Sajti, L. Cser, S. Bálint, L. Bottyán, *J. Appl. Cryst.* **50**, 184 (2017)
[2] N. Ghafoor, F. Eriksson, A. Aquila, E. Gullikson, F. Schafers, G. Greczynski, J. Birch, *Optics Express* **25** 18274 (2017)

Neutron polarizing supermirrors for the WASP instrument at the ILL: the end of a mass production coating project.

Thierry BIGAULT[1*], Guillaume DELPHIN[1], Florian PHILIT[1], Vincent GAIGNON[1], Amandine VITTOZ[1], Pierre COURTOIS[1]

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The neutron instrument WASP (Wide Angle Spin Echo) at the ILL [1] is entering in commissioning phase, and will soon accept its first users. This instrument will be unique worldwide, and its unprecedented performances are expected to open new horizons in the investigations using the neutron spin echo technique. One critical optical component of this instrument is its wide-solid angle spin-polarization analyzer [2]. It consists in 90 benders like illustrated on Figure 1b, each of them covering an angle of 1 degree 3 meters away from the sample. Each bender contains 36 polarizing channels made out of double-side magnetic multilayer coated mirrors. The coating design is a Co/Ti/Gd 541-layer supermirror with anti-reflecting/absorbing layer. The production of these supermirror coatings, covering a total area of 239 m^2, spanned about 10 years and is now over. The principle of this wavelength and angular broadband spin analyser will be briefly given, and then some intermediate characterisation of the benders will be presented. Preliminary measurements performed with the first available neutrons on WASP will also be given.

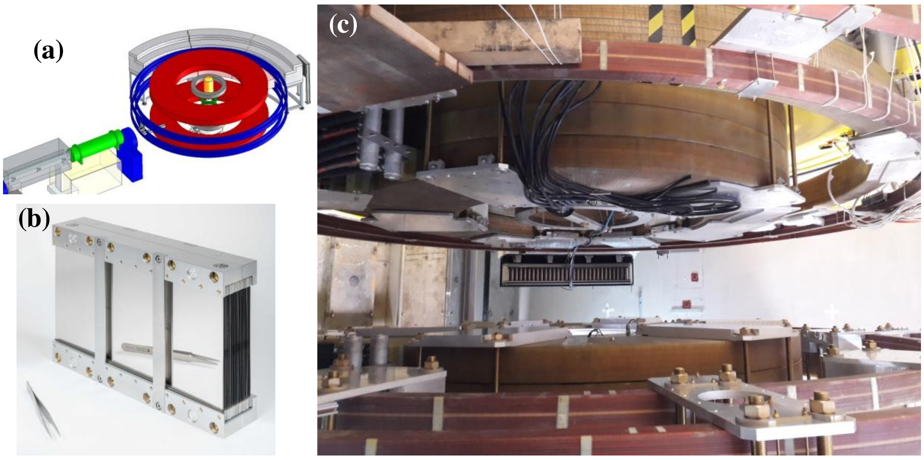

Figure 1: (a) General scheme of the WASP instrument. (b) Analyzer bender covering horizontally 1° from the sample. (c) Photograph of the instrument during final assembly.

References

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Oral Presentation

Large area neutron supermirror and X-ray multilayer mirror deposition facility at Bhabha Atomic Research Centre INDIA

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A 9 m long DC/RF in-line magnetron sputtering system has been developed in our laboratory for deposition of periodic and non-periodic multilayer films on large substrate (150 mm x 1500 mm) with thickness non-uniformity less than 3%. Using this system m=2.0 (100 layer), m=2.25 (204 layer) and m=2.5 (312 layer) Co/Ti supermirror polarizers have been deposited, and characterized at DHRUVA, Trombay [1] by PNR measurement. The desired up and down polaraization neutron reflectivities have been achieved in the supermirror polarisers when all Co layers of the multilayer structure are deposited under mixed ambience of argon and dry air [2]. It has been found that following this recipe roughness propagation in the multilayer structure is decreased. The measured PNR spectrum of a m=2.8 supermirror fabricated following the above recipe is shown in FIG 1. Further chemical, structural and magnetic investigations have been done on Co/Ti multilayers to find the cause behind this observation. The m=2.5 (312 layer) Co/Ti supermirrors are characterized by XTEM measurement and a distinct decrease has been observed in the interface roughness in case of the sample deposited with air + argon compared to that deposited under only argon ambience, as shown in FIG 2 (a&b). Also 20-layer Co/Ti periodic multilayers and Co/Ti/Co trilayer of different bilayer thickness are prepared with only argon and with argon + air and these samples have been characterized by XRD, GIXR, PNR and magnetic measurements [3]. Chemical state is characterized by XPS and XANES measurement and it shows there is no signature of oxide or nitride formation when the samples are deposited under mixed ambience of argon and air. Specular GIXR measurement shows that there is a decrease in interface width and the rocking scan measurement confirms further that this decrease is mainly in physical roughness (σ_r), as shown in FIG 2 (c). A drastic change has been observed in the XRD measurement as shown in FIG 2 (d), which shows that the Co film deposited with argon + air is amorphous. This proves that the Co crystal structure is completely destroyed when oxygen or nitrogen atoms are entering into the Co crystal. This amorphisation is smoothening the interface and a better Co/Ti multilayer structure is formed. The PNR and magnetic measurements support each other, PNR also confirms decrease of magnetic dead layer at interface when Co layer is deposited with air.

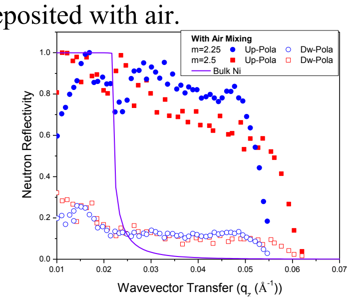

Figure 1: PNR of m=2.25 and m=2.5 Co/Ti supermirror deposited with Argon + dry Air

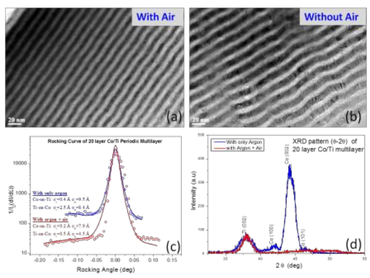

Figure 2: Characterization of samples deposited with air and without air (a)-(b)XTEM measurement of supermirror. (c) Rocking scan of periodic multilayer (d) XRD of periodic multilayer

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Metal-on-metal interface formation laws of nanoscale thin films

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In thin film-based devices, there is a constant effort to prevent or at least limit interlayer formation between the layers. Interface effects become much more critical especially in the case of nanoscale thin film systems. Although there has been a tremendous development in post-deposition characterization of thin film structures, there is currently a limited capability to accurately predict the interface formation between two layers without having to deposit test structures. In this work, we use low energy ion scattering (LEIS) to systematically measure the interface thickness for several transition metal-on-transition metal (TM-on-TM) sputter deposited thin films and identify a scaling law for interface formation between any two TM layers. We find that there is a clear trend in interface thickness with respect to difference in surface energies of the layers and there is a subtrend based on the crystal structure of the metal layers. Based on these two materials properties, the interface thickness for any given TM-on-TM combination can be predicted with sub-nanometer level accuracy. This ability improves the possibility to control interface properties and thus, achieve the best possible thin film structure for the desired application.

Optical constants with improved accuracy in the regions of L-, M-N- and O-absorption edges and their impact in multilayer modeling

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We are presenting a new set of measurements and a self-consistent determination of the optical constants (refractive index) of Cr, W and Pt thin films in the EUV/soft x-ray range. We use a combination of photoabsorption and reflectance data in the photon energy range 25 to 800 eV which include the L- and M-shell absorption edges of Cr and the N- and O-shell absorption edges of W and Pt. Our experimental data demonstrate for the first time highly resolved fine structure in the region of the Cr $M_{2,3}$ and $L_{2,3}$ edges, the W $N_{4,5}$ and $N_{2,3}$ edges and the Pt $O_{2,3,}$ $N_{6,7}$ and $N_{2,3}$ edges in both the absorptive and dispersive portions of the refractive index, resulting in differences of up to a factor of 3 compared to optical constant values published earlier, for these materials. The improved accuracy of the new optical constants is validated via the f-sum rule test and by simulating experimental reflectance data of single-layer and multilayer coatings containing these materials [1-3]. The implications of these new results, as well as plans for future work, will also be discussed.

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Growth of Mo/Si multilayers at oblique deposition induces periodical nano-arrays

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We report on a new mode of thin film growth which results in formation of highly periodic ripple patterns on a surface of the growing film. Mo/Si multilayers (ML) consisted of 50 pairs of Mo and Si layers with d-spacing of 6.7 nm were deposited at the angles of 0° - 65° using the ion-beam sputtering method. The deposition setup provided a highly collimated deposition flux required for precise control of deposition angles, and relatively high energy of the deposited particles (about 10 eV). The top surface of the MLs was examined with AFM and an internal structure of the ML stack was investigated by cross-sectional TEM. The multilayers deposited at the angles of 0° - 45° with respect to the surface normal exhibited a very smooth interfaces. The first signs of surface roughening are observed at the deposition angle of 45°. At the angle of 55°, the ML interfaces undergo progressive roughening which escalates from the substrate to the top of the ML stack. The roughening occurs in the direction parallel to the projection of the deposition flux with no substantial roughening in the transverse direction, leading to formation of surface ripples perpendicular to the flux direction. Deposition of the multilayer at an angle of 65° results in a vigorous interface roughening and formation of highly periodic lateral ripple structure with a period of 10 nm. A mature ripple pattern forms during growth of a few first layers and then stabilizes due to shadowing effects. The ripples propagate through the whole ML stack with almost no changes in frequency and amplitude, resulting in a highly periodic bulk array composed of silicon and molybdenum nano-rods closely packed in a 6-fold symmetric lattice [1].

Formation of the ripples for the oblique deposition of energetic particles is attributed to momentum transfer effects. For the normal deposition geometry the transfer of the momentum of deposited particles to a wavy surface is known to result in smoothing of the surface due to induced downhill currents [2]. Extension of the Moseler's downhill current mechanism towards the incline deposition leads to Carter-Vishnyakov-type effects when a tangential component of the momentum of the landing atoms induces a directed surface current [3]. The directed current provides smoothing of a surface for small deposition angles but results in enhancement of stochastic roughness at the angles higher than 45°. Under these conditions an amplitude of a wavy surface will grow with a growth rate increasing with the spatial frequency. At the same time growth of very high frequency modes is suppressed by isotropic surface diffusion induced by the thermalized component of the momentum of the arriving atoms. A Fourier surface mode with the highest growth rate dominates the surface morphology exhibiting a highly periodic ripple pattern.

The surface current mechanism is essential for Multilayer Blazed Grating applications. Normal deposition of multilayers on a highly corrugated surface of a blazed grating results in degradation of the saw-tooth groove shape due to smoothing effects [4], while smoothing and roughening are expected to be balanced at the angles around 45°. The simulations show almost zero net impact for all frequency modes of a surface, hence the saw-tooth profile of the grating is to be preserved during the ML growth.

The observed film growth mode can be used far beyond the x-ray optics field, for example, for making periodical 3D nanostructures for optical, magnetic and other applications.

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Atomic oxygen diffusion in ultrathin transition metal oxide films at near room temperature

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Experimental determination of oxygen diffusion kinetic parameters in oxides is a challenging task. Regarding materials for applications in systems such as electronic devices and protective layers of extreme ultraviolet optics, usually deposited as ultrathin films, this process becomes even more complex. The surface/interface regions of thin films may be significant compared to the diffusion zone. This may lead to different diffusion energies than the ones obtained for bulk. Several techniques have been proposed to derive these data, of which isotope exchange depth profile (IEDP) coupled with secondary ion mass spectroscopy (SIMS) is the most used to directly obtain kinetic parameters. Nevertheless, as SIMS is based on the analysis of secondary ions, the surface disturbance during measurement may add further uncertainties that influence on the precise characterization of nanometric layers. In this work, we analyse oxygen interaction with oxide ultrathin films at the near room temperature regime through IEDP coupled with low energy ion scattering (LEIS). The high sensitivity of LEIS allows quasi-static measurements, where there is no influence of ion damage. Furthermore, LEIS has a good sensitivity to mass differences, which enables the correlation of isotope exchange kinetics at surfaces and in-depth penetration with high accuracy of atomic concentrations, allowing the analysis of oxygen interaction in oxide layers. Films of transition metal oxides, such as tantalum (Ta_2O_5), molybdenum (MoO_3) and zirconium (ZrO_2), of 2 nm to 10 nm deposited via DC magnetron sputtering were analysed. After deposition, the samples were transferred in vacuum and analysed prior and after exposure to atomic O-16 and O-18 generated by a plasma gun, at a pressure of 10^{-4} mbar at room temperature. The evolution of surface and in-depth oxygen concentration was tracked for different exposure times. With the diffusion profiles obtained, diffusion modes, surface kinetics and diffusion parameters were determined based on models stated in literature. Due to the high sensitivity of LEIS, oxygen surface exchange and penetration could be observed even at/near room temperature, where the total penetration depth does not exceed more than 3 nm for any of the analysed oxides. It was verified that oxide films present an isotope diffusion profile that indicates a higher diffusion constant in the outermost few monolayers at the surface. This may indicate the presence of a space-charge layer due to oxygen vacancies. The oxygen penetration is shown to be strongly influenced by the presence of metallic and sub-stoichiometric oxide species, but not by the total thickness of the layer.

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Manufacturing and characterization of substrates for imaging multilayer X-ray optics

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Thanks to the successes of recent years, the technology of depositing multilayer mirrors (MLM) has made it possible to create a normal incidence optics for extreme ultraviolet and soft X-ray (XEUV) wavelength ranges (λ~2-60 nm). This potentially allows one to achieve nanometer scale resolution in microscopy and lithography, and ultra-high angular resolution of XEUV telescopes. The main factor limiting progress in this direction is the complexity of manufacturing substrates with the required quality. For one or two orders, shorter wavelengths in respect to the optical range proportionally increased the requirements for the roughness and the accuracy of the shape of the substrates for multilayer mirrors, reducing them to sub-nanometer and even sub-angstrom levels. The solution of the problem of creating diffraction quality optics required a revision of the possibilities of traditional methods of manufacturing and characterizing the surfaces in the entire range of spatial frequencies of roughnesses affecting the imaging and, if necessary, the development of new methods both for measurements and fabrications. A feature of the imaging optics for XEUV is the fact that in most cases it is aspherical. This is due to the need to minimize the number of elements in the optical scheme due to the relatively large power loss of radiation for each reflection. For the characterization of the substrate surface the conventionally used methods are an interferometry with a diffraction reference wave, white light interferometry and atomic force microscopy (AFM). The main method of correcting local errors and "weak" aspherization of the surface shape of substrates is the ion beam processing of the surface, the so-called ion-beam figuring (IBF). Despite the established approaches to the characterization of the surfaces, only the PDI can be attributed to the "first-principle" measurement method.
The report describes the equipment and methods by which the above-mentioned metrological problems are solved at the Institute fof Physics of Microstructures of the Russian Academy of Sciences. In conclusion, as a demonstration of the possibilities of the methods, the result of the ion beam correction of the shape of a spherical substrate with a numerical aperture NA=0.3 will be shown.

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Advances in hard x-ray aperiodic multilayers for imaging with large field of view

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Aperiodic multilayer interference coatings are of particular interest for a variety of hard x-ray applications, including target diagnostics, astrophysics, high energy physics and free-electron lasers. Such applications require large field of view along with the highest achievable photon efficiency and tailored spectral shapes for their optical components, pushing reflective multilayer coatings to their limits. In non-periodic multilayers, the thickness, morphology and composition of each interface may depend on the constituent layer thicknesses. This leads to a multilayer structure with interfacial effects varying across its depth, which need to be understood a priori and taken into account in the coating design. Higher photon energies require multilayer coatings with thinner layers and the above-mentioned interfacial effects become increasingly pronounced.

We have developed a generalized protocol to efficiently design and deposit aperiodic multilayers for the hard x-ray range. As a first step, the analytical solution of the inverse problem is calculated [1]. Interfacial effects (roughness, interdiffusion) are then included in the model for better prediction of the performance, based on results from independent studies of each material system. The deposition recipe, built on a detailed model, is then further optimized to obtain the desired shape and to further increase reflectivity. This approach was tested on several material systems designed to operate in the 17.4 – 35 keV range, at grazing incidence angles of up to 0.6 degrees [2].

This work demonstrates how precise the calibration procedure needs to be and how well the multilayer structure needs to be understood, in order to fabricate advanced aperiodic coatings with experimental performance as close as possible to the target design. The interfacial physics and the influence of barrier layers will also be discussed, for selected aperiodic multilayer material systems.

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Multilayer mirrors based on beryllium for an extreme ultraviolet range

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The development of reflective optics is subject to continuous improvement of the optical properties of the multilayer mirrors (MLMs). Typically, the transition to a qualitatively new level occurs when using new materials. This work presents results of systematic studies of beryllium as a material for MLMs for the EUV spectral range.

The main results are the following. Firstly, the technique of sputtering of Mo/Be MLS was developed providing record, up to 71%, reflection coefficients at a wavelength of 11.4 nm. It is shown that the main cause of the smaller, compared with the theoretical value (77%), reflectivity at 11 nm is "bad" boundary Mo-on-Be - its width was about 0.6 nm. The second boundary, Be-on-Mo, has a transition area width of 0.3 nm only.

The influence of barrier (amorphous) layers on the dielectric permittivity profile and interlayer roughness in thin-film Mo-Be and Be-Mo systems was studied. it was shown that the introduction of Si interlayer decreases peak reflectance at a wavelength $\lambda = 11.36$ nm from 70% to 64% due to strong silicon absorption in this spectral region, but on the contrary at hard X-ray wavelength range the reflection peaks of high order, until the sliding angles up to 6°, became prominent, that clearly indicates the increasing of interface sharpness. The model of the MLMs reconstruction gave the following values of the interface widths: 0.45 nm on Si-on-Be interface and 0.27 nm on the Be-on-Mo interface, against 0.6 and 0.3 nm, interface widths in Mo/Be MLMs, respectively.

The observed effect of smoothing the boundaries in Mo/Be MLMs with Si interlayers led us to study Mo/Be/Si mirrors optimized for a maximum of the reflection coefficient in the region of 13.5 nm. The optimum Mo/Be/Si composition was found, which ideally has a reflection coefficient higher than Mo/Si. A series of samples of various compositions was made. At angles close to the normal, almost all samples have reflection coefficients above 71%. The highest reflection coefficient at the wavelength of 13.5 nm was 71.9% and almost 73% at a wavelength of 12.9 nm.

During the experiments, the reflective characteristics of the Be/Al and Be/Si/Al MLMs optimized for the spectral range near 17.1 nm were studied. It was found that silicon has an amorphous effect, reducing the interlayer roughness from 1.3 nm to 0.6 nm. The peak value of the reflection coefficient of Be/Si/Al structures reaches 61% with a FWHM of about 0.4 nm.

It has been experimentally shown that structures of the Zr/Be/Si/Al, optimized at 17.1 nm, have an even higher value of the peak reflection coefficient of 67%.

On the basis of the same composition, Al/Be/Si, it was possible to create stable reflective characteristics of MLMs for operation at a wavelength of 30.4 nm. They outperform traditional MLMs based on the Si/Mg pair, not only in stability, but also in terms of reflection (34% vs. 30%), and also in the FWHM (1 nm versus 1.2 nm).

Be/Mg structures have been studied. If the maximum reflection coefficient of Al/Be/Si is 34% with FWHM of 1 nm, then for Be/Mg these values are 56% and 1.6 nm, respectively.

Further studies were carried out to determine the temporal stability of the reflective characteristics of these mirrors when stored in room conditions. It is shown that no changes are observed for MLMs Al/Be/Si and Mg/Be + Alcap for a long time (about a year).

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Recent advances in development of Cr/Sc based reflective multilayer coatings for x-ray applications

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Many scientific and industrial applications in x-ray range require the use of reflective multilayer coatings with a good efficiency and stability and often of a particular spectral band width. Here we report on recent advances in optimization of Cr/Sc-based multilayer systems for various x-ray applications. We have investigated and developed several approaches to design periodic and aperiodic multilayer mirrors using as a base the Cr/Sc pair of materials. A method to characterize sub-nanometric layers based on grazing incidence X-ray reflectometry has been proposed and tested experimentally. We have elaborated a certain number of periodic and large-band aperiodic multilayer mirrors for plasma diagnostics at grazing incidence in the hard x-ray range between 2 to 10 keV [1].
The development of efficient normal incidence optics in the soft x-ray range remains quite a challenge. Proposed more than 20 years ago as a most promising system for the water-window range, the Cr/Sc multilayer could provide about 60% peak theoretical reflectance at near-normal incidence around the Sc $L_{2,3}$ edge at 397 eV. However, the values of measured reflectance of Cr/Sc multilayers achieved so far are much lower than predicted because of a number of reasons. Apart of the problem of stability while depositing several hundreds of sub-nanometer layers, the most significant reflectivity loss is caused by formation of a rough interface as a result of the material interdiffusion.
We studied an influence of boron carbide (B_4C) introduced in the multilayer structure as a barrier layer or a third material and an effect of nitridation of chrome layers during deposition on optical and structural properties of the Cr/Sc reflective coatings with the aim to improve the performance of multilayer mirrors in the water-window range. Several sets of Cr/Sc-based multilayer mirrors were deposited by magnetron sputtering and characterized by using both soft and hard x-ray reflectivity techniques. Complementary analysis of the structural parameters was performed by using the x-ray reflectivity and standing waves enhanced x-ray fluorescence techniques later on [2]. The normal incidence reflectivity was measured at the metrology beamline of Soleil synchrotron [3]. We will demonstrate that combining both effects (the nitridation of Cr and the use of B_4C barrier layers), we have been able to produce a new multilayer system ($CrN_x/B_4C/Sc$) with the peak reflectance as high as 23% measured at near normal incidence of 85.7° around the photon energy of 397 eV.

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Chromatic Aberration Control on Microscope Imaging System with EUV Multilayer Mirrors

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When we apply extreme ultraviolet (EUV), which has wavelength between 3 to 30 nm, to optical microscopy, high spatial resolution of a few-tens nanometres can be expected in diffraction limited imaging. We have reported an EUV microscope for lithography mask inspection [1-2], where we applied a novel high-magnification objective with reflective-multilayer mirrors [3]. To achieve the high spatial resolution, reduction of the wave aberration on the objective is the key technical challenge. The wave aberration would come from two-main effects. Primary contribution relates to geometrical optics; figure errors and misalignments of the mirrors tend to yield large wave aberrations. These effects, however, can be eliminated by applying the deformable multilayer mirrors, where these geometrical aberrations can be reduced to 1/10 or less of the allowable value [4].

Wave-optical effect on the multilayer coating is another contribution degrading resolution on the EUV microscope. The wave aberrations would come from multi-beam interference on the multilayer structures. In this paper, firstly, we investigated through numerical study on the Schwarzschild objective (operating wavelength: 13.5 nm, numerical aperture: 0.25) and show that the wave aberration resulting from the Mo/Si multilayer structure has chromatic dispersion, and this chromatic aberration disturbs spatial resolution of the diffraction-limited system operated with a light source of wide-spectral band, i.e., laser-produced plasma. To reduce the wave aberration, then, the multi-beam interference on the curved multilayer mirrors was analytically evaluated, and we proposed novel design method of the multilayer structures to correct the chromatic aberration. We found that the aberration can be reduced by modifying period length of the multilayer structure, as a function of the radial coordinate on the mirror substrate. This novel technique was applied to design an aberration-free imaging objective for EUV microscopy. We successfully demonstrated that, the chromatic aberration can be reduced within 0.1 nm rms., which is 1/10 of the allowable value given with the Maréchal condition for diffraction-limited imaging.

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X-Ray Spectroscopic Methods for Thin Films and Interfaces Study

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Today thin films and nano-structured layer systems find a wide range of applications in materials science due to their tunable optical, structural, electronic, magnetic and superconducting properties. Often the quality of films is a governing factor that determines the critical parameters of these devices. Any deviation of their physical, chemical and geometrical parameters from desirable ones causes fluctuations in their properties; for example, the complete disappearance of quantum effects in nano-electronic devices or the catastrophic drop of reflectance of ultra-short-period x-ray multilayer structures (MLS). Increase of MLS reflectivity and progress toward the shorter wavelengths is closely connected with a problem of quality of the interfaces.The problem becomes more complicated due to the formation of interlayers, owing to atomic migration, chemical reactions or implantations in metal-oxide-semiconductor gate stacks and MLS, which may impact the functionality of the devices by for examples, affecting the effective work function of electrodes, the optical contrast or their magnetization.

All of these, in turn, stipulate the higher requirements on the technology of thin film synthesis and quality control. One of the key issues is the precise determination of atomic and chemical composition profiles at various interfaces in layered structures with an in-depth resolution approaching the scale of interatomic distances (~1 Å). X-ray photoelectron spectroscopy with high kinetic energies (HAXPES) is one of such methods. Due to a large value of inelastic mean free path, HAXPES is a very effective non-destructive depth sensitivity technique for chemical analysis of nano-layered systems, which can be realized by changing the photoelectron's emission angle or/and kinetic energy of photoelectrons.The implementation of HAXPES allows establishing as the chemical composition and relative content of atoms in different chemical states as the thickness of all layers constituted the system. The near-edge X-ray absorption fine structure spectroscopy (NEXAFS) with differently polarized radiation, which makes it possible to detect polarization layers on interfaces, is a complementary method and will be also discussed. In the talk the technologically important systems like TiN/γ-Al_2O_3/Si, TiN/HfO_2/B/TiN/Si (B: Al_2O_3 or TiO_2), TiN/Al_2O_3(0.5 – 3 nm)/SiO_2/Si will be presented. Also our first results of applying the approach to MLS structures like Be/Mo will be discussed.

Proximity effect in Co/Pt multilayer investigated through X-ray resonant magnetic reflectivity

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Magnetic multilayers (MLs) presenting a strong perpendicular anisotropy like $(Co/Pt)_n$ or $(Co/Pd)_n$ have attracted a considerable interest over the last two decades due to their potential application in many fields including information storage, sensing and biomedicine [1-4]. Along with perpendicular magnetic anisotropy, a peculiar characteristic of these system is the significative magnetic moment induced in the 5d metal, experimentally demonstrated thanks to the element selectivity of the X-ray magnetic circular dichroism (XMCD) technique [5]. The induced magnetic moment has a pivotal role in determining the properties of these systems. X-ray Resonant Magnetic Reflectivity (XRMR) is a powerful technique that gives depth-sensitive information on the magnetic properties of the investigated systems with sub-nanometer resolution [6]. The distribution in depth of the induced magnetic moments in Pt has been extensively studied by XMCD [7] and XRMR [8] and in a recent paper [9] reflectivity measurements on a $Pd_{bottom}/Co/Pd_{top}$ trilayer system demonstrated the presence of a different induced moment between the bottom and top Pd layers. Despite these interesting results, the characterization of the distribution of magnetic moments in a real $n>1$ $(Co/5d)_n$ multilayer with repeated 5d/Co/5d interfaces, the constitutive part of an effective device, is still lacking. In our work, combined XMCD and XMRM measurements were used to obtain the magnetization depth profile of a complete $[Pt/Co]_4/Pt$ multilayer. The main goal of this project is to verify if the induced magnetization in the Pt layers is constant across the $2n$ Pt/Co and Co/Pt interfaces or a dependence along the growth direction is present. A simpler Pt/Co/Pt trilayer was also characterized as a control sample and for comparison with the existing literature. Both samples were grown by sputtering on a thermally oxidized Si substrate with a Ta buffer layer. XMCD and XMRM scans were carried out across the Co $L_{2,3}$ and the Pt M_3 edge in order to characterize the magnetic properties of both the elements. We will present the results on the chemical and magnetic profile of the investigated samples obtained from the fitting of the XRMR curves.

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Angle-Resolved XRF for Depth-Resolved Elemental Analysis of Stratified Materials in the Laboratory

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X-ray reflectometry (XRR) is a well-established tool to investigate layer thicknesses, roughnesses and densities of multilayer systems. However, when targeting the analysis of boundaries with respect to diffusion or contamination, the pure structural information gained from XRR might be insufficient.

In the case of angle-resolved X-ray fluorescence (ARXRF), depth-resolved elemental information can be gained by rocking the angle of incidence with respect to the sample surface (grazing incidence: GIXRF) or the angle of detection (grazing emission: GEXRF) and performing an XRF measurement for each angle. In special cases, e.g. for multilayer samples, the reflectivity behaviour of the sample modifies the measured signals and provides additional information on the sample structure similar to XRR. The sample structure, but also information about contaminations or diffusion at boundaries can be retrieved from the measured ARXRF profiles via sample modelling and back-calculation of the fluorescence intensity in a fitting approach.

ARXRF measurements are mainly applied at synchrotron radiation facilities, where especially GIXRF profits from a high brilliance of the incident radiation. However, to increase the accessibility of those analytical methods, the Berlin Laboratory for innovative X-ray Technologies (BLiX) pursues further methodological development to make ARXRF an efficient tool in the laboratory. In this scope, several setups were realized, working either in the soft[1] or hard[2,3] X-ray range, in GI- or GEXRF geometry and with different optimizations regarding angular resolution or efficiency.

Here, we present the available setups, discuss the various characteristics and show first proof-of-principle measurements on a well-known C/Ni multilayer sample.

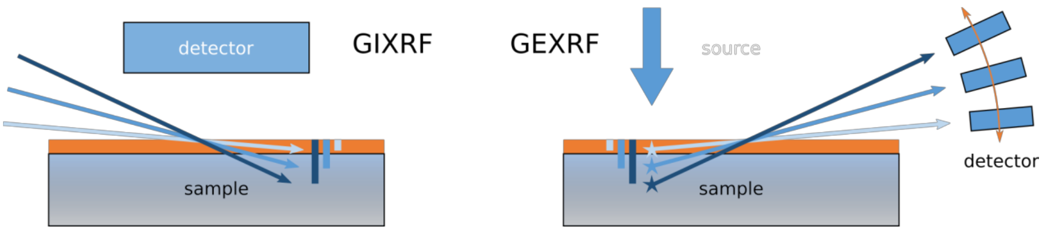

Figure 1: Measurement geometry for GIXRF and GEXRF.

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Correlated lateral density fluctuations in the Si layers of a W/Si multilayer revealed by GISAXS

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A comprehensive structural characterization of W/Si multilayers using X-Ray reflectivity (XRR), Transmission Electron Microscopy (TEM) and Grazing Incidence Small Angle X-ray Scattering (GISAXS) is presented. TEM images revealed density fluctuations in the Si layers, which were further analyzed using GISAXS. Characteristic parameters of the fluctuations were obtained by fitting numerical simulations to the measured scattering pattern. For the numerical simulations the density fluctuations were approximated as a set of spheroids distributed inside the Si layers with a density 8% lower than the bulk Si value. The distribution of these spheroids was described using 3D para-crystal model with cumulative position errors. The results provide additional detail with respect to the data obtained from TEM analysis.

Study of Pd/Y multilayers with B_4C barrier layers using GIXR and x-ray standing wave enhanced HAXPES

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Pd/Y multilayers are high reflectance mirrors designed to work in the 7.5-11 nm wavelength range. Samples, prepared by magnetron sputtering, are deposited with or without B_4C barrier layers located at the interfaces of Pd and Y layers to reduce interdiffusion, which is expected by calculating mixing enthalpy of Pd and Y. Grazing incident x-ray reflectometry is used to characterize these multilayers. B_4C barrier layers are found effective on reducing the Pd-Y interdiffusion. Details of the composition of the multilayers are revealed by hard x-ray photoemission spectroscopy under x-ray standing waves effect. It consists in measuring the photoemission intensity from samples that perform an angular scan in the region corresponding to the multilayer period and the incident photon energy according to the Bragg law. The experimental result indicates that Pd does not chemically react with B nor C at the Pd-B_4C interfaces while Y does at the Y-B_4C interfaces. The formation of Y-B or Y-C chemical compound can be the reason why the interfaces are stabilized. By comparing the experimentally obtained angular variation of the characteristic photoemission with the theoretical calculation, the depth distribution of each component element can be interpreted.

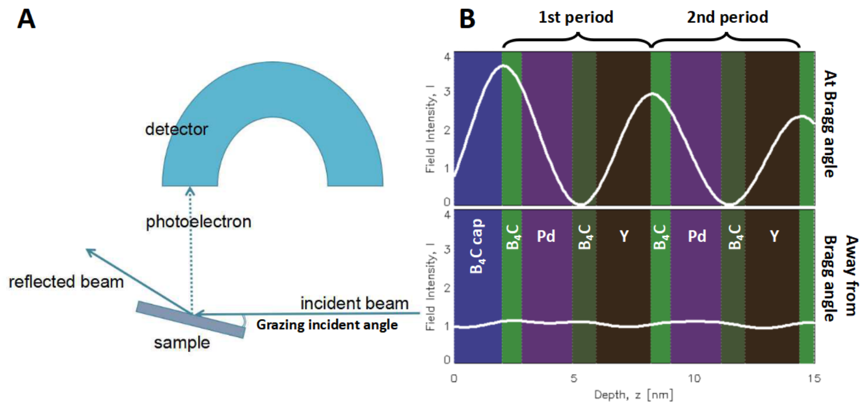

Figure 1: (A) Experimental setup and (B) depth distribution of the x-ray standing wave electric field within a $B_4C/Pd/B_4C/Y$ multilayer irradiated by a 10 keV photon beam.

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Multilayer coated nanogratings with sub-50 nm periodicity for ultrahigh resolution soft X-ray spectroscopy

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Soft X-ray (SXR) spectroscopy has been widely used to study the atomic and electronic structures of materials and biological samples. Grating-based dispersion optics lies at the heart of a SXR spectrometer which determines the spectral resolution and signal intensity. To resolve the fine emission lines and their shapes so as to reveal the complex structure and understand some fundamental process of a solid-state system, high resolution grating optics are demanded [1]. For the conventional gratings, the resolution is limited by the line density and diffraction orders used. To overcome these limitations, an ultrahigh line density multilayer grating is developed. Based on a new nanofabrication technique named "vacancy epitaxy", nanogratings with over 20,000l/mm line density can be fabricated over wafer-scale substrate. The grating has a triangular groove profile with symmetric facets [2]. To make it work at the SXR region, we combine it with multilayer structure and use the Bragg diffraction effect to enhance the efficiency. Moreover, the spectral resolution can be further improved by using a high diffraction order enabled by the Bragg diffraction. A schematic drawing of the novel multilayer nanograting is shown in Fig. 1. A first demonstration has been made at 92eV using a Mo/Si multilayer which exhibited 11% efficiency and extremely large angular dispersion. To work at a higher energy, a Cr/C multilayer grating using the 4th diffraction order is fabricated and worked at close to the C-K edge. The efficiency is lower with 1.2% at 271 eV while the angular dispersion is even larger. Assuming an 11 meters long scattering arm in the beamline, a spectral resolution (E/ΔE) of over 90,000 can be expected at the SXR region. The design, fabrication and characterization of such multilayer nanogratings will be presented.

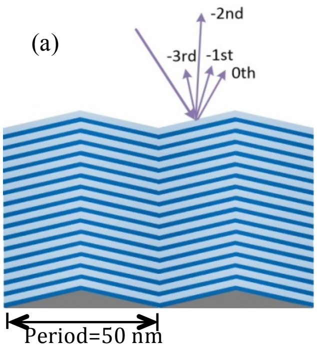

Figure 1: A schematic drawing of the 20,000l/mm multilayer nanograting.

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Gratings for VUV to X-ray applications

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Gratings are widely used in an energy range going from VUV to soft X-rays to analyze or select the photon energy. These gratings typically have line densities ranging from ~200 l/mm to 2000 l/mm. A first revolution came around 1970 where mechanical ruling was replaced by holographic printing and lithography. Holographic gratings are more regular, free of "ghost" and show less scatter. However, only rectangular profiles (lamellar) are easy to produce with this technique, though asymmetric etching in silicon may provide blazed gratings. There is also a limit in the line period can be printed, somewhat over half the holographic wavelength, ie ~250 nm.
Classical metal coated gratings have decreasing performances with higher energies. Multilayer (ML) coatings offer the significant advantage of providing good reflectivity at much larger grazing incidence angle than simple metal coating at the same photon energy. However the incidence angle must be matched to the photon energy and multilayer period through the Bragg law. It implies that the deviation angle varies with the wavelength. However this deviation can be easily compensated by ML coated mirror of matched period. On the other hand, the fact that matching condition should be satisfied for both the grating and the multilayer period channels the diffraction into a single order the efficiency of which can reach high values. Use of ML gratings will be illustrated by the example of SOLEIL Sirius beamline
Classically, gratings are oriented so that grating line direction is perpendicular to the incidence plane. In such case all diffracted orders stay in the incidence plane. When the projection of the incident wave vector on the surface is parallel to the line directions, diffraction orders are distributed on a cone the axis of which is the line direction. The aperture angle of this cone is the incident glancing angle and can be freely chosen. Small glancing angles, combined with blazed profiles or ML coating, allow high efficiencies since there is no screening effect. However, the dispersion given by such a conical geometry is small. Improving it requires to increase the line density. Nano-fabrication techniques could offer an alternative to holography for manufacturing very dense grating. Properties of conical are used for instance in our design of the FAB10 beamline at ATTOLAB behind the HHG* source. They are also considered for spatial X-ray spectrometers.
Designing grating based instruments for the X-ray domain requires to be able to model the diffraction properties of simple and ML coated gratings. Since the considered photon frequencies are well above plasma frequencies, the grating is model as a thick structure with a periodic transverse modulation of the dielectric constant varying with the depth. Computations are somewhat similar to ML reflectivity ones, but propagating a vector of diffracted waves rather than a single one. Examples of computation will be given.

*High Harmonic Generation in Gas

Highly efficient multilayer-coated blazed gratings for the tender X-ray energy range

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The development of efficient optical elements suitable for the tender x-ray range between 1 keV and 5 keV will open interesting opportunities, e.g. for X-ray microscopy and spectroscopic techniques. This energy range covers characteristic emission lines of many important elements like phosphorous, sulfur, chlorine as well as many of the 3d-transition metals, which are essential constituents of organic samples and catalytic materials. The energy range from 1.5 keV to 2.5 keV is especially difficult to cover today as it is a “grey zone” for both grating and crystal monochromator systems. In this range plane grating monochromators have a low transmission and a relatively large amount of stray light, while Si crystal monochromators suffer from high heat load in the near-normal incidence operation required for these energies. We present an alternative by employing a multilayer coating optimized for a high line-density grating which significantly improves its efficiency.
The first successful prototype of a multilayer-coated blazed grating (MLBG) with line density of 2000 lines/mm achieved an efficiency of 35 % at 2 keV with a maximum efficiency of 55 % at 4 keV [1].
In this contribution we present the next achievement for such a MLBG which provides high efficiency of up to 60% in the energy range 3 keV – 5 keV. Aiming to go for even higher resolution, a 2400 lines/mm blazed grating has been manufactured at HZB using a mechanical ruling in combination with ion etching technique [2]. The grating aperture was divided on three sections of different blazed angle, produced by separate etching, to investigate tolerance between ML period and blaze angle values. Multilayer coating of Cr/C with a period of 6.3 nm was deposited at Tongji University. The particular choice of the ML-coating allows to efficiently use the grating in the energy range between 600 eV and 5900 eV. In the given energy range, accurate measurements were carried out using reflectometers at the soft X-ray Optics Beamline [3] and the tender X-ray beamline KMC-1 at BESSY-II. The new grating compares favorably with the first prototype with measured efficiencies of 40 % at 2 keV and 60 % at 4 keV on sections with blaze angle 0.98 and 0.8 degrees. This correlate well with simulations, which were carried out before, and points on new approach for MLBG parameters optimization different from standard idea connected with grating operation in on-blaze mode.

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Nearly-perfect blaze of X-ray multilayer-coated gratings with non-conformal boundaries

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A further progress in the development of multilayer-coated blazed gratings (MBGs), which are considered as the most promising optical elements for many hard and soft x-ray applications, requires improvements of the grating design and controlled fabrication of the multilayer structure. Therefore theoretical investigation of the MBGs design should include both simulations of grating growth and rigorous numerical calculations of light diffraction by gratings with realistic boundary profiles. Using this integrated approach we have studied deformation of the MBGs boundary profiles during growth. Growth simulations were carried out within the continuous approach, in which boundary profile height h is represented as a function of coordinate x and time t [1]. Commercial code PCGrate®-SX v.6.7 [2] was used to calculate diffraction efficiency. Simulations of the grating growth and calculations of the diffraction efficiency η were carried out for the W/B_4C MBG working at a blaze wavelength of 1.3 nm. Detailed study of the grating growth shows that boundary profiles shift relative to each other in the horizontal direction. Values of the boundary profile shifts Δd strongly depends on the incidence angle of deposition flux α. Formation of the horizontally shifted (non-conformal) boundaries is also accompanied by the profiles height reductions. Calculations of the light diffraction by the gratings with realistic, ideal conformal and ideal shifted (values of the shifts were taken from growth simulation) boundary profiles demonstrate that (i) the highest efficiency is achieved when grating layers are highly shifted toward the direction of the incidence radiation, i.e. along the blaze facet of the profile; (ii) the reduction of the profile height during growth leads to the rapid decrease in the efficiency; (iii) efficiency depends in a complex way on profile shifts and height reductions. Thus based on the obtained results a new design of the MBG with the horizontally shifted (non-conformal) boundaries and nearly perfect blaze could be proposed.

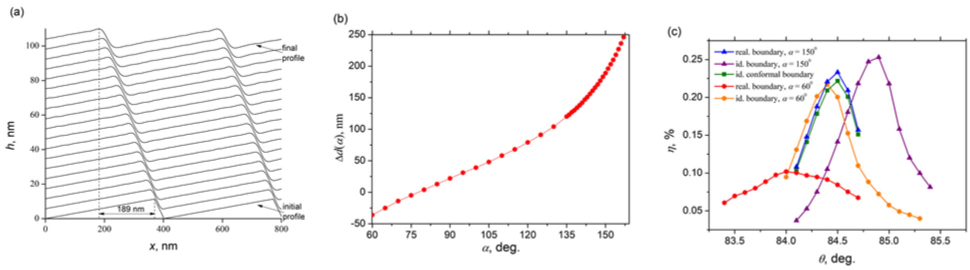

Figure 1: (a) Simulated 18-period W/B_4C MBG with non-conformal realistic boundaries; (b) shifts Δd of the topmost boundary profile vs. deposition flux incidence angle calculated for the W/B_4C MBG; (c) dependence of η vs. 1.3 nm radiation incidence angle θ calculated for W/B4C MBGs with realistic and ideal boundary profiles;

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Multilayer x-ray optics at the ESRF

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The ESRF Multilayer Laboratory is in charge of the development and fabrication of multilayer coatings for x-ray optical applications. The ESRF has been operating a multilayer deposition facility for more than 20 years. Since then, more than 200 optical devices have been delivered to ESRF beamlines.

Multilayer interference mirrors as optical elements on 3rd generation synchrotron sources are an interesting complement to the use of crystals, mirrors, lenses, or other components. Their principal assets are high flux and strong focusing capabilities. By selecting the most appropriate materials and fabrication parameters they can be tailored to the experimental conditions on a given beamline.

The principal applications of multilayers on ESRF beamlines are focusing in the Kirkpatrick-Baez geometry and high-flux monochromators with tailored bandwidths. As of today, about half of all ESRF beamlines are equipped with multilayer based optical elements. Several examples will illustrate their practical use.

The ESRF is in the middle of a major facility upgrade implying the entire replacement of the storage ring and the construction of new beamlines. The new Extremely Brilliant Source (EBS) will offer a significantly smaller source and an increase of the coherent photon flux – requiring new and improved optics. Consequently, the X-ray Optics Group is upgrading its instrumentation to better comply with evolving requirements.

Research activity of evaluation tools including soft X-ray optics for the research of EUV lithography at University of Hyogo

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Extreme ultraviolet (EUV) lithography will be used for the semiconductor electronic devices in high volume manufacturing (HVM) around 2019. The technical issues at 1st phase of EUV lithography are 1) EUV source with high power and high stability, 2) EUV resist which has high sensitivity, high resolution, low LER, and low outgassing properties in simultaneous achievement, 3) pellicle for EUV mask with high transmittance and high stability, and 4) defect free EUV mask and its metrology. In the technical issues at 2nd phase of EUV lithography, EUV resist development and light source appear to be the 1st and 2nd issues, respectively. In these situations, the fundamental studies are still required to resolve these technical issues.

Thus, at NewSUBARU synchrotron light facility of University of Hyogo, many kinds of evaluation and metrology tools has been developed since 1996, including fundamental studies using aspherical mirrors for the imaging optics[1]. And many kinds of research contributions have been carried out.This paper describes 1) the large reflectometer[2] for the evaluation of large Mo/Si multilayers (MLs) mirror, 2) natural defects inspection tools for the EUV mask on a basis of EUV bright field microscope[3] and coherent EUV microscope[4,5], 3) EUV resist evaluation tools for sensitivity[6], outgassing[7], resolution evaluations[8], and absorption coefficients measurement[9], 4) EUV resist chemical reaction analysis using soft-X-ray absorption spectroscopy[10], and 5) chemical structure evaluation of EUV resist and other organic materials using soft X-ray resonance spectroscopy[11]. In these methods many kinds of optics for EUV and soft X-ray regions were developed on a basis of the on the technologies of MLs deposition and semiconductor process such as thin layer deposition, lithograph, and dry etching.

In NewSUBARU, three beamlines such as BL3, BL9, and BL10 beamlines are now operating for EUV lithography development for EUV resist and mask. And these beamlines are opened for the internal and external use of companies, institutes, university and college. Using these beamlines, we have contributed to the fundamental research for the EUVL technology development toward the usage for the HVM.

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High reflectance Al/MgF_2 mirrors by hot deposition

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Al mirrors protected with MgF_2 has been the standard broadband coating with unmatched reflectance in the far ultraviolet (FUV, 100-200 nm) for the last 60 years. They cover a broad spectral range from the far infrared to the FUV down to ~115 nm. Yet, their FUV reflectance is not as high as intrinsic Al reflectance. Al slightly oxidizes in normal atmosphere or even under vacuum, and the thin natural Al oxide fully degrades Al FUV reflectance. That is why Al is typically protected with a thin film of MgF_2. The reflectance of an Al mirror protected with MgF_2 ranges 80%-85% at the important spectral line for astrophysics of H Lyman α at 121.6 nm, whereas intrinsic Al reflectance is ~92%. When one models the reflectance of an Al/MgF_2 mirror with the optical constants of a pure MgF_2 crystal, a reflectance even larger than the one of pure Al is obtained. The reason for the mismatch between such model and experimental measurements on Al/MgF_2 mirrors is that FUV absorption of the protective MgF_2 film, which is classically deposited at room temperature (RT), is orders of magnitude larger than the absorption in the crystal. MgF_2 absorption can be reduced by depositing on a hot substrate; however, old experiments showed that depositing Al on a hot substrate resulted in larger surface roughness and scattering, which reduces the overall reflectance.

A procedure to enhance Al/MgF_2 FUV reflectance was devised by Quijada et al. [1]. It consists in: 1) depositing the Al film over a RT-substrate, 2) protect it with an ultrathin film of MgF_2 at RT, and 3) heat the coating to ~220°C and complete the MgF_2 film deposition. With this procedure, mirrors with a reflectance of ~90% at Lyman α were obtained.

A research has been followed at GOLD (Grupo de Optica de Láminas Delgadas) to reproduce these results and to optimize the deposition temperature for largest FUV reflectance. A reflectance enhancement was measured for increasing temperatures up to an optimum range of 200-250°C. Temperatures of 300°C and higher resulted in large reflectance degradation. The dependence of the outer surface and interface roughness with temperature was measured with AFM and x-ray reflectometry. The growth of pinholes in the hot-deposited coated was investigated.

A summary of this research will be presented in the workshop.

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Broad Band EUV/VUV Multilayer Coatings from 16.9 to 130 nm for a Solar Spectrograph Space Mission

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A new broad band multilayer coating for normal incidence optics has been designed, fabricated and characterized to enable a possible spectrograph for the study of the solar atmosphere in the vacuum ultraviolet (VUV) wavelength range between 17 nm and 200 nm. We present Mo/Si based multilayer coatings with simultaneous broad band reflectance in the two spectral ranges of 16.9 nm to 21.5 nm and 46.3 nm to 127.5 nm. In order to achieve the challenging demands on the wide spectral coverage at the shorter wavelengths (< 22 nm) a special stochastic broad band multilayer design with more than 80 different layers was calculated and successfully realized by DC magnetron sputtering technology. Furthermore, we introduced and investigated different interdiffusion barrier materials for enhanced thermal stability up to 200 °C. Finally, a B_4C capping layer was added and the capping layer thickness was tested and optimized in order to increase the reflectance for longer wavelengths (> 45 nm) while maintaining the short-wavelength performance.

Thin-film preparation and characterization at the HZG

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Magnetron sputtering is a versatile tool to synthesize nanocrystalline or amorphous phases with tailored material properties. This thin-film method enables us to coat highly precise, uniform and flexible. Extraordinary surface quality is required for high-precision x-ray mirrors. This presentation will focus on thin-film preparation of up to 1 m long x-ray mirrors and their thin-film properties. Large x-ray mirrors are required for beam transport at free-electron lasers (FELs) and synchrotron sources worldwide. The demand for large mirrors with lengths up to 1 m single layers and multilayers consisting of light or heavy elements has increased during the last few decades [1-3]. At the Helmholtz-Zentrum Geesthacht (HZG), a 4.5 m-long sputtering facility enables us to deposit a desired single-layer material some tens of nanometers thick. For the European XFEL project, the shape error should be below 2 nm over the whole x-ray mirror length to ensure the safe and efficient delivery of x-ray beams to the scientific instruments. The experimental results achieved will be discussed with regard to current restrictions and future developments.

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Photon-induced damage processes in Ruthenium thin films developed for Free Electron Laser optics

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Radiation tolerance of optical elements exposed to a high dose of laser radiation is an important issue in developing optical elements for x-ray free-electron laser light sources. In order to prevent optics from being damaged, the fundamental mechanisms governing the material response to ultrashort high peak power XFEL pulses must be identified and studied.

In this paper we present experimental results on the damage of a 50 nm thick Ru layer caused by single XUV light pulses of various fluences. With increase of the fluence, we observed initially roughening and eventually ablation of the top part of the irradiated layer [1], [2].

To understand the damage mechanisms we performed a computational study of the entire process, starting with the interaction of the femtosecond XUV (13.5 nm) laser pulse with the electrons in the Ru film, using an event-by-event Monte Carlo code XCASCADE(3D) [3]. It provides us with the cascading range and the ballistic transport of the non-thermalized electrons. Secondly, the evolution of the electron and the lattice temperatures out of thermal equilibrium, together with the atomic motion in the irradiated Ru, were modelled with a combination of two temperature hydrodynamics [3] and molecular dynamics simulation schemes [4]. Our calculations show that the mechanism responsible for the ablation of Ru observed in the experiment is spallation in the stress confinement regime. Processes as melting, cavitation, spallation and recrystallization were modelled as well. The results show a good agreement with the experimental observations.

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Coatings in harsh space environment

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In recent years, the study of space agent effects on optical coatings has become priority in view of future selected missions which will explore increasingly hostile environments. The impact on the morphology, on the structure and on the performance of coatings and materials due to ion and electron irradiation has been studied through various investigative techniques [1,2]. The irradiation sessions have been carried out at accelerators adopting different experimental regimes to reproduce space conditions in laboratory. A predictive model of the optical performance based on of the damage induced by protons and alpha particles has been developed [3]. Changes in the reflectance and transmittance properties have been attributed to density and refraction index variations due to implantation of low energetic particles. Bubble formation has been observed in metals, while delamination occurs when particles accumulated at interfaces, such as those in metal-protected thin films. Blistering of top layers has been observed in oxide-protected metal coatings (Fig.1). Impact on the performance of the coatings in various spectral ranges including extreme ultraviolet is discussed.

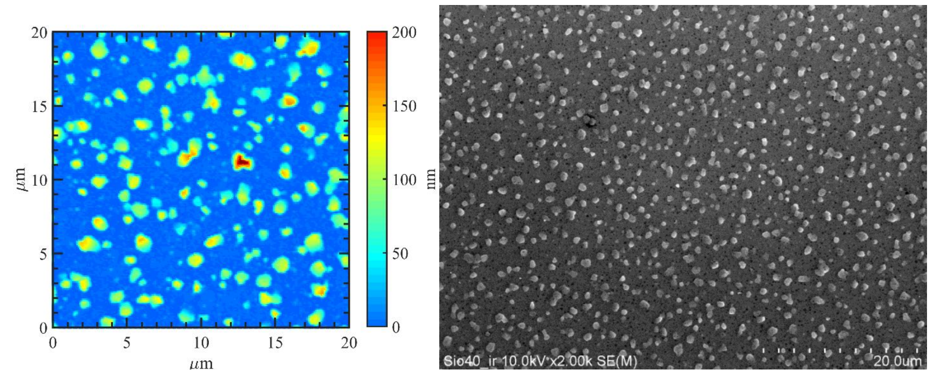

Figure 1 AFM and SEM of SiO_2(32 nm)/Al sample irradiated with 4keV protons at a fluence of $2.7\cdot10^{17}$ cm^{-2}.

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A compact soft X-ray reflectometer based on a multi-spectral fluorescence micro-source

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We report on the status of the soft X-ray reflectometer design and construction within the framework of the Pro Fit project "REFLEX" (10162889) at IAP e.V. The instrument exploits a micron-sized X-ray source generated by an electron gun in conjunction with various target materials in the range from (0.1 – 10) keV. A collimating poly-capillary half lens (PCL) or toroidal mirror is used with optional slits for beam shaping, respectively. The sample is mounted on a six-axis goniometer and both a photodiode and a CCD camera are alternatively available for angular resolved detection. With its size of 1.5 m, the system fits in an ordinary laboratory.

The reflectometer is exclusively designed for absolute efficiency measurements of multilayer mirrors and 2-dimentional variable space gratings used in novel spectrometers and monochromators developed by IAP e.V.

X-EUV/XRay Hartmann wavefront sensing for at-wavelength metrology and correction

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Since more than 20 years Imagine Optic has designed and manufactured Shack-Hartmann wavefront sensors (SHWFS) for many kinds of applications such as telescope alignment, laser characterization, optics qualification or adaptive optics, in various fields such as space optics, microscopy, high power lasers or lithography. Transferring Imaging Optic's expertise gained in the visible range, Hartmann wavefront sensors have been built and used at shorter and shorter wavelengths to measure wavefronts from synchrotron radiation beamlines (SR), High Harmonic Generation (HHG)-based sources, plasma-based soft x-ray lasers, or Free Electrons Lasers (FEL) [1,2].

Many experiments have already been done demonstrating a clear advantage of actually measuring the wavefront at the used wavelength, for example for advanced metrology, alignment of optics at SRs or FELs, and WF correction using active mirror (such as bender) optimization within a few minutes [3]. Detailed WF knowledge is the key in all XUV diffraction [4], physics experiments including vortex beam manipulation [5], or high-accuracy metrology of X-Ray optics [6]. We will present latest developments of EUV (10eV-1keV) and X-Ray (>1keV) WFS, including high accuracy of λ/120 rms at 13 nm, and customized EUV sensors from very large band-pass to high numerical aperture (NA=0.1). Examples of high accuracy metrology of multilayer optics using Hartmann sensing in a lab environment will be reported.

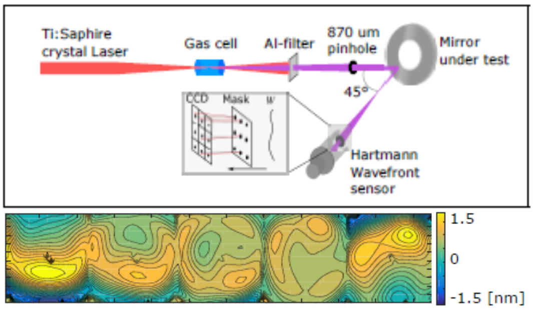

Figure 1: *Experimental setup (top) and WF maps (bottom) at various locations on a multilayer mirror (from [6])*

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Kossel interferences of proton-induced X-ray emission lines to study the interfaces of thin film waveguides

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While waveguide optic is ubiquitous in the visible and infrared spectral range, it is still much less developed in the X-ray regime. X-ray waveguides can be used as spatial and coherence filters for X-ray coherent imaging. They are usually designed and fabricated with a low density guiding layer embedded in high density cladding layers. The interfaces of the thin film waveguides play a crucial role in their service reliabilities. In this study, Pt/Fe/Pt and Ta/Cr/Pt thin film waveguides are firstly designed by using the IMD software, and then deposited on Si substrates by magnetron sputtering. Grazing incident x-ray reflectometry is performed to obtain the geometrical information of the designed thin films waveguides. The Kossel interference of proton-induced X-ray emission lines (PIXE) [1], as a non-destructive way, is used to analyze the interfaces of the thin films, as shown in Fig.1. PIXE is an X-ray spectrographic technique, which can measure the characteristic radiations emitted by electron transitions and identify each element. It is used in Kossel geometry to obtain what we call Kossel curves, i.e. the angular distributions of the intensity of the characteristics emissions. In our experimental conditions, we use an energy dispersive CCD camera, which enables performing the experiment without any rotation of the sample or camera. By comparing the experimentally obtained Kossel curves of the characteristic emissions and the simulated ones, the interfacial environments of the studied stacks can be explained.

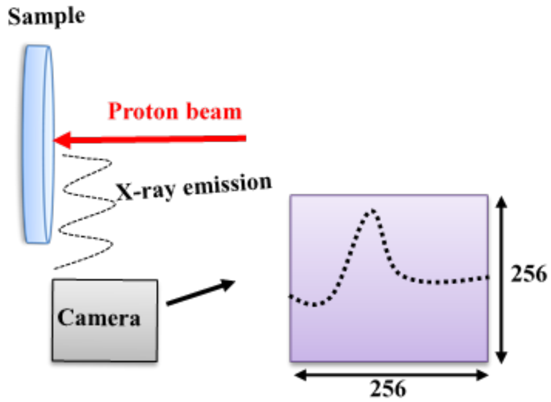

Fig. 1. Experimental setup of the PIXE-Kossel experiment. A column of pixels of the CCD camera measures the emitted intensity at one angle. The 256 columns enables scanning a 3° angular range without moving any part of the experiment.

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Advancing X-ray standing wave data analysis

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The growing complexity of multilayer mirrors (MLMs) calls for the development of accurate and non-destructive characterization techniques suitable for routine characterization of as-deposited MLMs with sub-nm accuracy. The classical X-ray reflectivity technique shows high sensitivity to the smallest details of a multilayer structure, but the interpretation of the measured data remains complex and in some cases non-unique, even if advanced model independent data analysis algorithms are used[1]. To reconstruct the structure of a multilayer mirror reliably a combination of X-ray reflectivity and angular dependent X-ray fluorescence data is easy to use, especially because both data sets can be measured using the same laboratory X-ray setup. The angular dependent fluorescence signal adds the information about the mean positions of distributions of individual elements and the width of their distribution. This information presents natural regularization of electron density profile that filters number of unphysical solutions that can be obtained from GIXR only data analysis. We will present the reconstruction of multilayer structures from combined X-ray reflectivity and X-ray fluorescence measurements. The strategies for optimal measurements, the value of additional data and the challenges of data analysis will be discussed.

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Angular distribution of a characteristic x-ray emission transmitted by a periodic multilayer

Karine Le Guen[1], Meiyi Wu[1], Jean-Michel André[1], Zhanshan Wang[2], Qiushi Huang[2], Ian Vickridge[3], Didier Schmaus[3,4], Emrick Briand[3], Sébastien Steydli[3], Philippe Walter[5] and Philippe Jonnard[1]

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About 20 years ago, André suggested to use a multilayer interferential transmission mirror as a soft x-ray fluorescence spectrometer by placing the multilayer between a sample emitting some characteristic radiation to be analyzed and an x-ray detector [1]. In the transmittance curve of the multilayer measured as a function of the detection angle, the presence of a dip indicates the occurrence of a fluorescence emission. From the Bragg law the fluorescence wavelength can be determined allowing the identification of the chemical element.

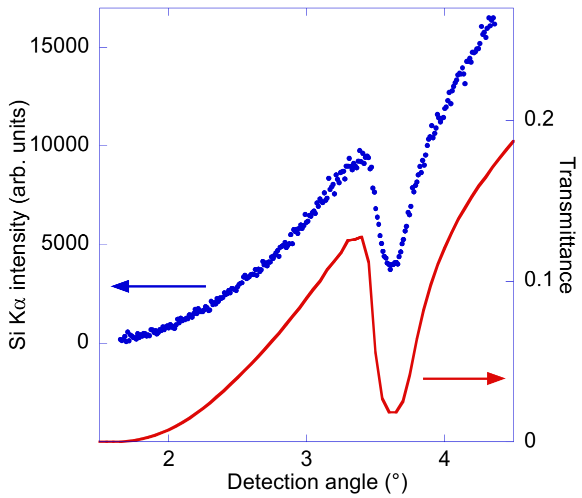

Figure 1: measured and calculated angular distribution of the transmittance of a $[B_4C/Pd/B_4C/Y]_{x20}$ multilayer at the wavelength of the Si Kα emission from the Si substrate.

The dip in the angular distribution of the Si Kα characteristic emission, originating from the substrate and transmitted through the $B_4C/Pd/B_4C/Y$ periodic multilayer, in agreement with simulations, is clearly observed in Figure 1. The emission is induced upon proton irradiation through the multilayer and detected with an energy-sensitive CCD camera. Such a device could be envisaged as a spectrometer without mechanical displacement and using various ionizing sources (electrons, x-rays, ions).

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ATTOLAB SE10: a versatile and integrated beamline for attosecond physics on gases and surfaces

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In recent years, increased reliability and stability of ultrafast energetic lasers based on Ti:Sapphire technology has raised the interest of a broad community of users appealed by ultrashort Extreme Ultra Violet (XUV) sources. In particular, focusing these lasers on gas targets, the process of High order Harmonic Generation (HHG) supplies an XUV spectrum that shows unique specificities as for its high degree of coherence, its low duration, which lies in the attosecond to femtosecond range, and its excellent synchronization to a Visible-IR laser that may be used for pump-probe experiments. Starting from a λ = 800 nm wavelength, typical HHG spectra lie in the 10 eV-120 eV range, which can address a large variety of applications from solid state physics (e.g. spin dynamics, dynamics of the so-called Dirac fermions in topological insulators, multi-ferroic materials, e.g., oxides) and gas phase chemical physics (e.g. time-resolved photoionization in the core- and valence shell of atoms/molecules, highly non-linear harmonic spectroscopy…).

Based on this scientific landscape, we designed an ultrafast XUV facility which offers free-ports to users from solid state and chemical physics backgrounds. The laser system is a Ti:Sapphire system which delivers 23 fs CEP stabilized pulses of 2 mJ at a 10 kHz repetition rate. The beam is focused in a continuous gas jet to produce HHG spectra. A first XUV beamline, currently under commissioning, has been designed by consensus among potential users and coupled to this HHG source. It finally offers three kinds of XUV light beams that can be commuted within 15 min without changing any other experimental parameter: a very broadband, broadband, and narrowband operating points. The spectral bands extend over the full 10-100 eV range, with respectively 20 eV, 1 to 5 eV and 100 meV FWHM, corresponding to pulse durations in the 100 as, 1 fs and 10 fs ranges. All are synchronized, down to attosecond precision with laser pump beams. Extreme care has been taken to provide a very stable and reproducible beamline which meets the needs of the scientific community. In this communication, the performance and technological choices for this beamline will be presented.

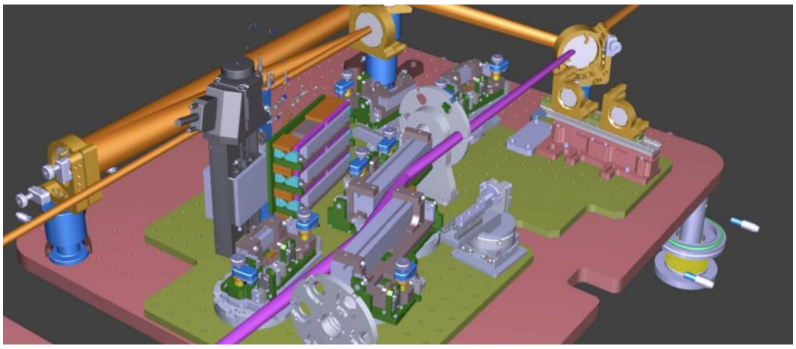

Figure 1: Drawing of the optomechanics mounted on the SE10 beamline for XUV/IR pulses recombination. It implements a monochromator and spectral selection by reflexion on multilayer coatings.

Characterization of a monochromatic x-ray imaging using Fresnel zone plates and a multilayer mirror

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Monochromatic X-ray imaging at micron scale is a convenient tool for studying the dense plasma produced by laser facilities. We used a two channels microscope made of two gold transmission Fresnel Phase Zone Plates (FPZPs) providing a high spatial resolution capability (3 μm) and their high efficiency. Each monochromatic channel consists in a FPZP associated to a multilayer mirror (ML) for spectral selection. The device allows choosing the imaging wavelengths by modifying the focal length and the angle of the ML.

In the latest experimental study, we used two high quality gold FPZPs developed and fabricated at the Paul Scherrer Institut (PSI). The metrology of FPZPs and ML was done at PTB laboratory on the Four-Crystal Monochromatic (FCM) beamline. A specific method using a moving sharp edge was implemented to determine the spatial resolution of the FPZPs.

The characterization of the device was completed by a resolution measurement on LULI2000 facility thanks to grid radiography on a polychromatic plasma source.

Characterization of a multilayer x-ray waveguide by x-ray diffuse scattering and grazing incidence x-ray fluorescence

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A four-layer stack, Al/ZrC/Al/W/Si substrate, designed as a planar x-ray waveguide, is characterized by combining grazing-incidence x-ray reflectivity (GIXRF) and x-ray diffuse scattering (XDS). The sample is prepared by magnetron sputtering and the aimed thicknesses of the different layers are in the order of 10 nm. The experiments are performed on the Métrologie beam line of the SOLEIL synchrotron facility by using the CASTOR setup.

The incident photon energy was chosen to be 10.25 keV. GIXRF experiment is done by measuring the intensity of the characteristic W Lα and Zr Lα emission lines as a function of the glancing angle between the incident hard x-ray beam and the surface of the sample. The variation of the angle is obtained through rotation of the sample. The XDS curves are obtained at fixed detector and source positions. In this case, rocking or ω scan, the sample is rotated while there is a fixed 2° angle between the incident and detected beams. A guided mode feature is present in each experiment, see Figure 1 around 0.22° in both GIXRF and XDS curves. This demonstrates the concentration of the electric field of the incident radiation inside the buried Al layer.

The simulations should enable obtaining a description of the stack, particularly of the interdiffusion zones present at the interfaces. The combination of both GIXRF and XDS results puts constraints on the fit process and makes us expecting a precise description of this Al/ZrC/Al/W planar waveguide.

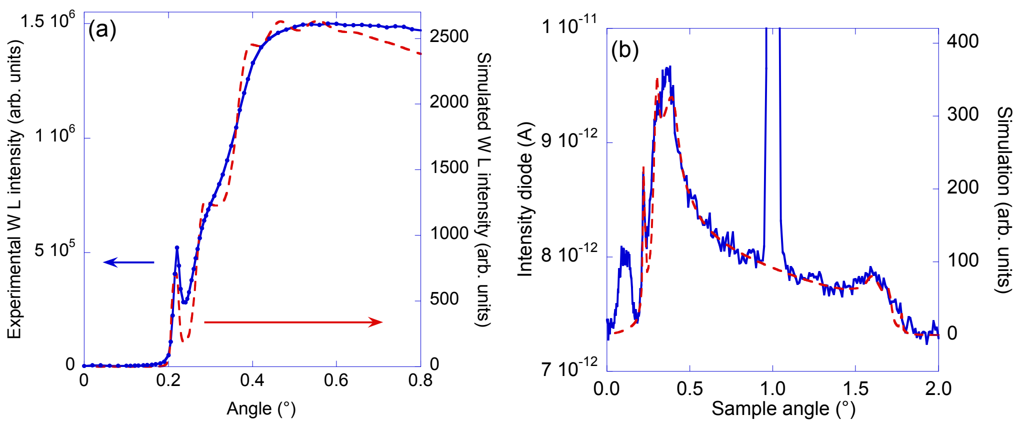

Figure 1: Al/ZrC/Al/W/Si substrate system. (a) Experimental (solid-dotted line) and simulated (dashed line) GIXRF curve of the W Lα emission;(b) Experimental (solid line) and simulated (dashed line) diffuse scattering curves at 10.25 keV.

Combined XRR-GIXRF analysis at SOLEIL

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Combining Grazing Incidence X-Ray Fluorescence (GIXRF) and X-Ray Reflectometry (XRR) is gaining increasing interest for the accurate and precise characterization of innovative materials with structures at the nanometric scale. Different experimental setups dedicated to this techniques are already installed on synchrotron beamlines [1, 2, 3] or on laboratories, based on X-ray tube [4].

CASTOR is a new goniometer designed upon the model developed at Physikalisch-Technische Bundesanstalt (PTB), which is currently used on the METROLOGIE beamline of the SOLEIL Synchrotron [5]. CASTOR can be installed on each branch of the beamline: the XUV branch (30 eV – 1.9 keV) and the hard X-ray branch (3 – 38 keV) giving access to analyses over a large energy range.

The goniometer is composed of a vacuum chamber and a 7-axis manipulator (four translation and three rotation movements), making it a perfect tool for performing characterization methods. It is equipped with photodiodes to acquire the reflected (or transmitted) X-ray beam and silicon drift detector (SDD) to record the fluorescence spectra. The photodiodes were accurately calibrated using an electrical substitution cryogenic radiometer [6] and the SDD was calibrated using the SOLEX lab-source [7]. CASTOR is also equipped with a heating module, allowing to perform combined analysis of thin films under temperature change to provide deeper understanding of the structure and properties of these materials.

The GIXRF data are analyzed using the PyMCA software [8] and the XRR data are simulated with the PyXCEL software developed at CEA-LETI. Different examples will be presented, from single layer to test and optimize the experimental setup, to practical examples on innovative materials, such as transparent conducting oxides or chalcogenides [9, 10]

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Dedicated X-ray optics for new generation small X-ray sources

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Recent state-of-the-art laboratory X-ray sources such as MetalJet sources or Laser Pulsed Plasma sources aim for increasingly high brilliance and flux at small sizes of the focal spot in the range of 10-20 µm or less. Further, the optics are typically placed closer to the anode than “standard” versions. Thus, demands to X-ray optics increase while most tolerances become tighter. If the spot on the anode shrinks, previously unobserved effects can occur in the beam profile such as significant local and global brightness variations. On the other hand, e.g. “shadow images” from laterally splitting $K\alpha_1$ and $K\alpha_2$ parts of the emission spectrum are less pronounced. Mentioned brightness variations are partly caused by imperfections of the mirror substrate like slope errors that can often only be reduced by very extensive (and expensive) super-polishing but also only up to certain limits. Here, dedicated multilayer mirrors for small sources can reduce those unfavourable effects.

Key to a successful design of efficient X-ray optics at small sources is a comprehensive knowledge of the entire system from source via mirror to sample as well as the possibility to simulate and optimize as many of its parameters as possible. Thus, we integrated source parameters (geometrical as well as spectral) into our in-house system simulation software to evaluate various properties in relation to the final X-ray beam at the sample position.

Cooperation with manufacturers of different types of X-ray sources gives wide possibilities to optimize mirrors but also the source geometry for improved performance of the system. The spectral behaviour of the multilayer mirror is then simulated and optimized for all lateral positions to determine the best mirror geometry and coating in terms of overall performance as well as fabrication feasibility and price. Thus, optimal solutions can be provided for industrial applications as well as for high-end research projects.

Derivation of beryllium EUV optical constants from Mo/Be multilayers

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Beryllium (Be) has been known for decades as an important material for applications in extreme ultraviolet and X-rays. A promising application of beryllium mirrors in the EUV lithography around its K1-edge (111.7 eV) has got great attention. It has also gained promising applications as diffusion barrier and spacer layers in multi-element coatings of mirrors for space applications at 17.1 and 30.4 nm wavelengths. Here, we report a record high reflectivity performance of beryllium-containing multilayers aimed for the EUV lithography applications. Besides, experimentally derived optical constants of beryllium in EUV near its absorption edge using combination of at-wavelength metrology techniques are reported. Results are compared to Henke's table. The derived optical constants are indispensable for modeling of beryllium-containing optics in EUV at large and around the absorption edge of beryllium in particular [1, 2, 3].

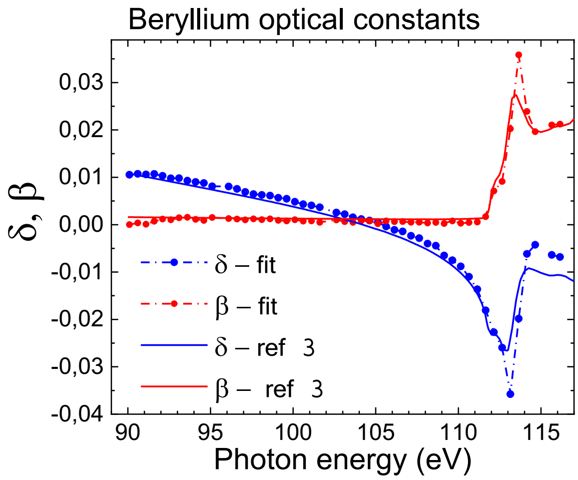

Figure 1: Derived optical constants of Be, in the energy range of 90-116 eV using at-wavelength reflectometry techniques in EUV and X-rays

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Explosive crystallization of Co layers in C/Co/C three-layers

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It is known that in metal/silicon multilayer mirrors there observed a transition from an amorphous to crystalline state in metal-containing layers as their thickness increases. We studied the transition of Co layers from amorphous to crystalline state in C/Co/C three-layers by transmission electron microscopy. Coatings were formed by dc magnetron sputtering with alternative deposition of layers. Two types of structural transition for Co layers were observed: 1) ordinary crystallization with the gradual nucleation and growth of individual Co crystallites (the carbon magnetron was switched off during deposition of Co-layers); 2) explosive crystallization which starts from one or few centers and spreads along the whole layer (the carbon magnetron was switched on during deposition of Co-layers). Thin Co layers (t_{Co}<2 nm) are amorphous. Ordinary crystallization process begins as the metal thickness reaches ~3 nm. In this case Co-layers are in general amorphous with inclusions of clusters (1-2 nm) and crystallites (~9 nm). Crystallization is almost complete at thickness of t_{Co}~4 nm. Here Co-layers are polycrystalline with grains up to 70 nm in the layer plane and have HCP structure. Before explosive crystallization Co layers have similar structure consisting in amorphous matrix with clusters and crystallites (2<t_{Co}<6 nm). After crystallization (t_{Co}>6 nm) Co layers are textured with HCP structure and the texture axis [0001] is directed perpendicular to the layer plane. Further increase of Co thickness (t_{Co}~20 nm) results to a decrease of the crystallite size and the nucleation of crystallites with other orientation. Reduction of Co deposition rate shifts the onset of explosive crystallization to higher thickness (up to t_{Co}~17 nm). We suppose that the carbon in the Co layer has a significant effect on the type of crystallization and the thickness of the Co layers at which the Co crystallization occurs.

Influence of nanometer protective layers on temporal stability and mechanical properties of thin-film aluminium absorption filters

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Aluminium (Al) filters of the extreme ultraviolet (EUV) wavelength range are the most commonly used in solar astronomy due to the fact that they have a wide (17-70 nm) bandwidth and a high degree of blocking of UV, visible and infrared radiation. The disadvantage of the Al filter is that, when stored in air, its transmission in the EUV range is reduced due to oxidation. In addition, Al freestanding films are relatively fragile.

To overcome the disadvantages of Al monolayer film filters, we have previously developed multilayer Al/Si spectral filters [1]. The multilayer structure gives the filter a higher corrosion resistance, which provides high temporal stability of the optical properties of the filter. However, additional absorption in EUV wavelength range introduced by Si interlayers imposes restrictions on the possibility of using Al/Si filters in telescopes operating in the long-wavelength part of the EUV range (silicon appreciably absorbs at wavelengths > 50 nm), as well as in ultra-high resolution telescopes.

The new projects of solar EUV of telescopes under development require a high transmission in the EUV range and an increase in the mesh size of the supporting metal mesh in the entrance filters to reduce the diffraction effect and increase the total transmission of the whole filter. The larger the size of the mesh cell, the more robust the film must be to withstand the vibration and acoustic loads during the spacecraft launch.

In this report, Al filters with thin (nanometer size) $MoSi_2$ or AlN cap layers have been investigated. Such three-layer filters are offered as an alternative to the traditionally used filters in the form of monolayer Al and multilayer Al/Si filters. Thin cap layers somewhat reduce the transmission of the filter in the EUV wavelength range, but not so much as in the case of a multilayer structure. However, in the presence of cap layers, as we have shown, it is possible to significantly increase both the temporal stability and the mechanical strength of Al-based filters. Thus, the ultimate strength of Al film with $MoSi_2$-2.5 nm films was 1.5 times higher than that of a Al monolayer film of the same thickness [2]. And as acoustic tests show (Fig. 1), this allows the use of films of such a structure in entrance filters on a supporting mesh with mesh size of up to 10 mm.

Layer structure and phase composition in W/Si multilayer X-ray mirrors

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X-ray diffractometry in the hard region (λ~0.154 nm) and low-angle reflectometry were used to study the phase structure and composition of tungsten layers with thicknesses up to t_W~10 nm in W/Si multilayer X-ray mirrors (MXMs) deposited by direct-current magnetron sputtering. Depending on the layer thickness they have amorphous (a-W) or polycrystalline (c-W, BCC) structure. The $\sin^2\Psi$-method allowed finding that tensile stresses in the c-W layers do not exceed 1.1 GPa. Tungsten crystallites ($2.7<t_W<10$ nm) contains at least 3 at.% Si. The radial distribution functions of atoms show that a-W layers have an atom arrangement close to β-W. Due to the interface interaction a formation of amorphous interlayers containing a-WSi_2 is observed in all samples. Depending on the deposition rate, disilicide can have an arrangement of atoms close to either the tetragonal phase or the hexagonal phase. An effective surface temperature of depositing layers can be at least 250°C above the substrate temperature. A model for the construction of amorphous W/Si MXMs is presented. Mechanisms for the formation of silicide layers are proposed. The ways of reducing the interface interaction are suggested.

Modeling of EUV multilayers and filters for solar physics and evaluation of tabulated optical constants

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Solar observation missions are essential as they provide data to enrich predictive models about flares, coronal mass ejections (CMEs), solar wind acceleration and heating mechanisms of the solar corona. Such coronal events are characterized by energies corresponding to the emission of extreme ultraviolet (EUV) rays, making them the most relevant to image and analyze [1], [2].

Multilayer mirrors for the EUV (wavelengths from 10 to 100 nm) enabled the imaging and analysis of the solar corona and related phenomena. Since the first EUV telescope aboard a satellite (SoHO mission, 1995 [3]) using Mo/Si periodic multilayers, new interference coatings with higher efficiency have been developed with more than two different materials repeated periodically or aperiodically. Advanced coatings made of three-material multilayers can be found in the EUV telescope of the Solar Orbiter satellite (to be launched in 2020) [4], [5].

More complex multilayers and filters with higher efficiencies require an increased knowledge of EUV optical constants for materials, to carry out optics simulations. The determination of material optical constants in the EUV range is not straightforward and different optical constant values can be found in the literature. Measurement methods of optical constants are sensitive to material absorption edges in this region, as well as surface and interface defects like roughness, diffusion and oxidation. As a result, uncertainties in optical constant values may induce unreliable simulations and by extension, compromise the experimental performance of the final coatings.

In the following work, we are bringing into focus several discrepancies in optical constant values between 10 nm and 80 nm wavelengths, as well as their consequences in simulations. Investigated materials include Al, B_4C, Mg, Mo, SiC and Zr. Experimental spectra are compared using the IMD software, including transmittance data of two filters and reflectivity data of tri-material multilayers developed for the Solar Orbiter EUV telescope [6]. Surprisingly, we determine that there are several important materials / energy ranges where the optical constants are still poorly known. These on-going analyses lead us to select the most accurate optical constants values in the EUV range for a few interesting materials. Such results are promising for the development of a new generation of high-efficiency instruments for heliophysics.

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Multilayer-based high-precision polarimeter at Diamond Light Source

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Knowledge of the degree of polarization of synchrotron radiation light is essential, not only for understanding modern undulator performance, but also to carry out a precise analysis of dichroic and chiral experiments. Multilayer polarizing elements are key optics that can be used to perform a complete polarization analysis of light in the soft X-ray region. The development and performance of a multilayer-based high-precision polarimeter at Diamond Light Source will be presented[1]. The polarimeter has been extensively used on Diamond soft X-ray beamlines for complete polarization analysis of light emitted by APPLE II undulators[2-4]. The capability of the polarimeter has been extended by using W/B_4C multilayer phase retarders and crystal analyser combination for use in the challenging operating range above 1 keV. The polarimeter can also be used for the characterization of reflection and transmission properties of optical elements.

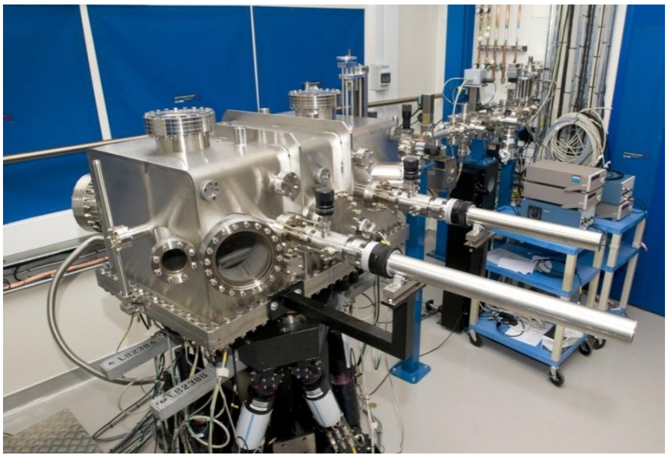

Figure 1: The multilayer-based high precision polarimeter on beamline I06 at Diamond.

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Observation of Polymer Blend Films on Transmission EUV Microscopy

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A transmission EUV microscope operated on photon energy between 90 to 500 eV can visualize two-dimensional distribution of light elements by using their inner shell absorptions. Recently, we are investigating polymer blend samples by applying the elemental contrast inherent on the EUV microscopy, in which at least two kinds of polymers with different composition are separated on sub-μm spatial scale. At the same time, little study related to sample preparations has been reported on observation of polymer samples on EUV region. In this study, we aimed to clarify preparation and observation conditions, i.e., an operating wavelength and sample thickness, for the polymer blend samples on the transmission EUV microscopy. First, we discuss image quality by introducing a signal to noise ratio (*S/N*) on the transmission images. The difference in transmittance of the two kinds of polymers is considered as a signal *S*, while photon noise (or shot noise) represents in the noise factor *N*. The transmittance of polymer films were computed by using the atomic scattering factor database [1]. The *S/N* spectra for the typical polymer blend, i.e., PS (polystyrene) / PMMA (acrylic), are shown in Fig. 1. Each curve shows the spectrum for sample thickness between 100 to 500 nm. The maximum *S/N* was observed around the photon energy of 100 eV, since the polymers have large different transmittance yielding good contrast. The *S/N* was fairly reduced on the higher energy region, i.e., carbon and water windows, where the sample becomes more transparent and it has small contrast, as results. It was also found that, the *S/N* improves as the sample thickness increases, and the maximum was expected when the sample thickness is between 400 to 500 nm. To confirm this theoretical model, two samples (thicknesses t= 290, 360 nm) were prepared, and experimentally observed by using the lab-scale EUV microscope [2]. Then, the S/N was calculated with the transmission EUV images. As a result, we successfully confirmed that, two-times larger S/N was obtained on the thicker samples. In the presentation, we also report details of transmission EUV images of the polymer blend samples.

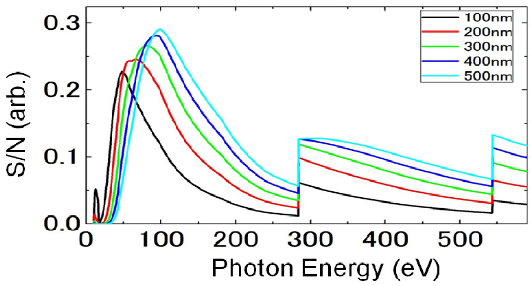

Fig. 1. S/N spectra for PS/PMMA polymer blend film.

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Optical properties of graphene at hydrogen Lyman alpha

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Studied for its unique electrical properties, e.g. high conductivity, zero band gap and semi metallic behavior, massless Dirac fermions, ballistic transport [1, 2], Graphene is the best known among the 2D materials. Furthermore, the chemical inertness, thermal and chemical stability in harsh environments, mechanical strength and impermeability to ion diffusion promote graphene as very strong candidate for corrosion resistance and protective layer on metals and optical coatings [3, 4].

To our knowledge, the optical properties of graphene have been already studied in the infrared (IR), visible (VIS) and ultraviolet (UV) [5-7] regions, but no systematic analysis have been performed at the extreme ultraviolet (EUV) and vacuum ultraviolet (VUV) spectral bands where mainly the photo reduction induced by the radiation have been investigated [8] We recently approached the study of the optical properties of monolayer and tri-layer commercial graphene on SiO_2/Si at hydrogen Lyman alpha spectral line. The measurements of monolayer samples were performed by using the EUV-VUV polarimetry facility available at CNR-IFN-Padova Italy [9], the tri-layers specimens were characterized at the BEAR beamline ELETTRA synchrotron–Trieste. We observed the effects of mono and tri-layer graphene on the angular reflectivity, pseudo Brewster angle and optical contrast of the samples. An increase in the reflectivity and downshift of the pseudo Brewster angle in case of both monolayer and tri-layer graphene was observed.

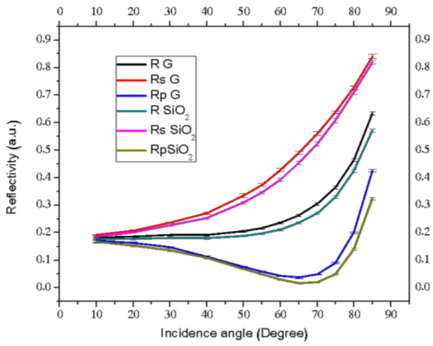

Figure 1: Comparison of s-, p- and average components of reflectivity of graphene/SiO_2/Si & SiO_2/Si.

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Progress on Multilayer-coated Optics for High Energy X-ray Imaging Tools at the US National Labs

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Since 2013, two multilayer-coated x-ray microscopes have been built and fielded at two US National Laboratories, with collaboration by NASA and university partners. Their purpose is to capture 9-17keV x-ray images of confined plasmas over very short times (< 1 ns) to study implosion dynamics. Imaging at high energies is necessary to see through surrounding lower-density plasma and capture behavior of the densest areas of the plasma. The Kirkpatrick-Baez Microscope[1] at Lawrence Livermore National Laboratory (LLNL) images an area of ~ 300 μm x 300 μm with a resolution of ~6 μm, at x-ray energies of 8.8 to 11.8 keV. The mirrors are coated with Pt/C multilayers of variable d-spacing 41-65 Å. The microscope is used to study inertial-confinement fusion (ICF) experiments at the National Ignition Facility (NIF) at LLNL. The Wolter Imager[2] at Sandia National Laboratories (SNL) images an area ~ 5 mm x 5 mm with a resolution of 60-300 μm in the on-axis region. The mirrors are coated with W/Si multilayers of constant d ~ 33.3Å. The imager is dedicated to studying Z-pinch plasmas at the Z-machine at Sandia National Laboratories (SNL).

This paper will discuss different coating-design decisions for the priorities of the different experiments (spatial resolution vs field size, energy selection vs background rejection), and present imaging results and plans for next-generation imaging tools.

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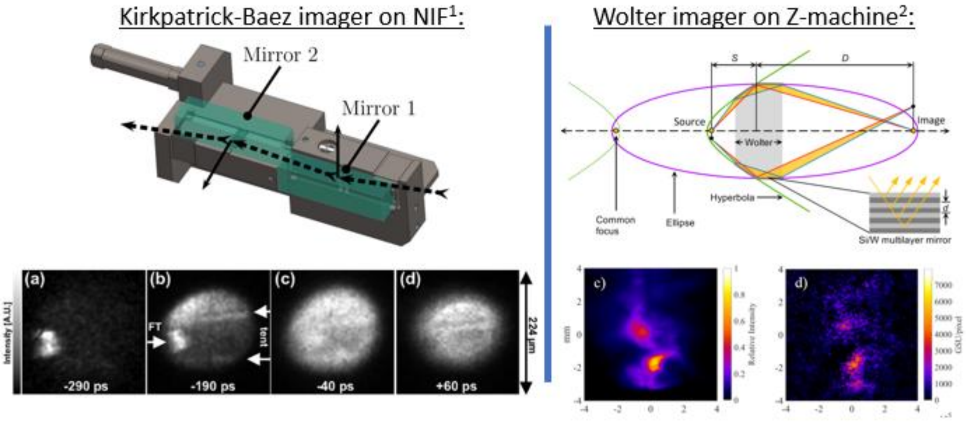

Fig. 1. **(left)**: Optics schematic of Kirkpatrick-Baez imager, top, and time-series images of NIF plasma through implosion and rebound, bottom. The microscope resolves plasma features that are artifacts of the fill tube (FT) and tent that contained the capsule before the shot. **(Right):** Optics schematic of Wolter imager, top, and images of Z-machine plasma, bottom (left and right images are Wolter optic vs. earlier pinhole imager). Contributions by other authors to the imaging results are detailed in the references.

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Spatially resolved EUV and XUV reflectometry

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The development of EUV and XUV technology, particularly the introduction of EUV lithography into HVM poses new challenges on the characterization of optical components. Particularly their homogeneity is of importance. The present measurement capabilities of PTB using synchrotron radiation are based on a monochromatized, focused beam with a typical footprint on the sample in the order of 1 mm². This, however, is not sufficient to detect small defects of optical coatings, local effects of irradiation and lifetime experiments or the like.
We present our new set-ups for the spatially resolved measurement of reflectance and transmittance of optical elements. For the transmittance measurement we prepare a homogeneous almost parallel beam and place a CCD detector close behind the sample. For a sufficiently parallel beam and short enough distance, the resulting resolution is defined by the CCD pixel size, 13.5 µm square.
For the spatial resolved reflectance measurement, the illuminated spot on the mirror under investigation is imaged using a modified Wolter-III-type optic onto a CCD with 10x magnification. The grazing incidence optic provides a broad-band reflectance from 6 nm upwards and thus allows measuring the full reflectance curve of EUV Mo/Si mirrors and also 6.x-nm-optics for beyond-EUV applications, spectrally resolved. We present first application examples for both measurement set-ups.

Study on propagation of interface imperfections across Co/Ti multilayers with ultra-short period

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The most important components of water window (23 Å–44 Å) soft x-ray microscopes are the Schwarzschild objectives coated with thin film multilayer soft X-ray mirrors, fabricated by sequential deposition of alternate absorber and spacer layers. Theoretical calculations using the "IMD" code available under XOP package shows that Co/Ti multilayer is one of the important options as material pair near Ti 2p absorption edge at wavelength of 27.3 Å. However, realization of high-reflectance multilayer coatings designed for this wavelength region is extremely challenging because refractive index in this X-ray range approaches 1 for all materials and therefore reflection coefficient at each interface between two materials is negligibly small; moreover for normal incidence optics each layer thickness should be in sub-nm range. Besides these theoretical constraints, practically high interface roughness and the inter-diffusion between absorber and spacer atoms, causes reflectivity intensity reduction drastically by scattering. Therefore characterization of interface imperfections is most important to understand the reflectivity properties of such short-period multilayer thin films. In this communication, we report on the interface roughness evolution in short period (~ 42Å) Co/Ti multilayers with increased number of periods, investigated by using Grazing Incidence specular as well as non-specular X-ray reflectivity (GIXR) and Cross-sectional Transmission electron microscopy (XTEM) measurements. Three Co/Ti multilayer samples of 10, 20, and 40 numbers of bi-layers have been deposited using a 9 meter long indigenously developed in-line magnetron sputtering system, with $\Gamma = t_{Co}/t_{Co} + t_{Ti}$ =0.4. The interface width (σ), determined from the specular GIXR fitting (inset:Fig1(a)), increases with increase in bi-layer thickness for both Co-on-Ti and Ti-on-Co interface. Initially the rate of change is very slow, however after the deposition of certain number of layers the slope increases sharply. Since inter-diffusion (σ_d) cannot increase with an increase in no. of bi-layers, it is the cumulative accumulation of interface roughness (σ_r), which limits the maximum reflectivity observed in the multilayer devices. However σ_d and σ_r which are the two components of the interface width (σ) cannot be separated out by specular X-ray reflectivity. σ_d and σ_r values, were determined separately from the non-specular X-ray reflectivity fitting, following Distorted Born Wave Approximation and it has been observed that, with increase in bi-layer thickness, σ_r increases for both Co-on-Ti and Ti-on-Co interfaces Also, it has been observed that the σ_r value is always greater than corresponding σ_d value. This result corroborates with our speculation[1] made from specular reflectivity result that for such ultra-low thickness multilayers not inter-diffusion, rather interface roughness plays dominant role in determining performance of the multilayers and accumulation of interface roughness limits the maximum reflectivity observed in such multilayer devices.

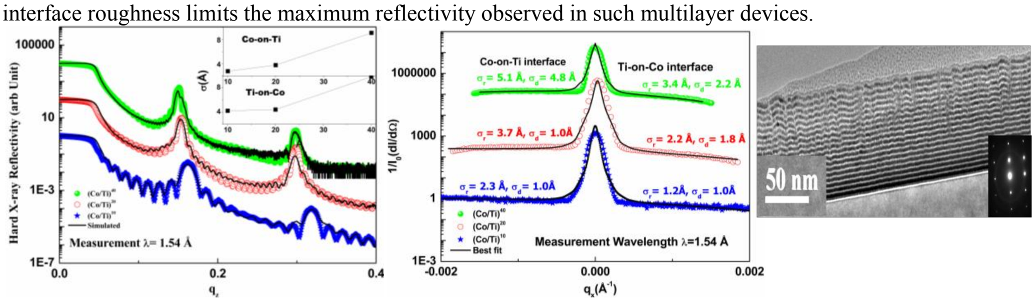

Figure 1: (a) Specular & (b) Non-specular reflectivity data (scatter points) of Co/Ti multilayer with increasing number of periods alongwith respective theoretical fits (line); (c) Cross-sectional TEM micrograph of 20-bilayer (Co/Ti) multilayer

High resolution bright field TEM micrographs of the 20 bi-layer Co/Ti sample (fig. 2) clearly depict that as the growth of the multilayer proceeds and number of bi-layer increases, interface roughness increases and wavy pattern in the growth becomes more and more prominent. Thus there is an accumulation of interface roughness in these ultra-thin Co/Ti multilayers, which propagates across the interface as more number of layers are being added. It should be mentioned here that contrast between Co and Ti layer remains almost same throughout the multilayer, which again confirms our speculation that interface diffusion remains the same. Thus for such short-period Co/Ti multilayer "restart of growth" model, characteristics of the layer-by-layer growth is not followed exactly, rather an island-type

Surface and interface observation of Zirconium oxynitride films as well as TiN/ZrON bilayers

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Abstract

Zirconium oxynitrides films possess potential excellent physical, chemical and mechanical properties by tuning the O/N ratio in ZrO_xN_y films during processing. In this paper, zirconium oxynitrides films were prepared by using reactive magnetron sputtering technique. In order to understand the effect of a small amount of N or O on the properties of the ZrO_xN_y films, this research proposes to prepare ZrO_xN_y thin films by RF magnetron sputtering using ZrO_2 and ZrN as target as well as nitrogen and oxygen as a reactive gas, respectively. In addition, interface chemical environment between ZrO_xN_y/TiN bilayers is vital in the period multilayer structure application. Therefore surface composition of the ZrO_xN_y films and the interface interaction between ZrO_xN_y/TiN bilayers with both reverse interface structures are investigated by XPS and XAS technologies, respectively. XPS results show that the concentration of N is strongly affected by the target selection. When the ZrO_2 material is used as target is highly dependence on the flows of N_2. However, when the ZrN material is used as target, no N element was detected for the fresh film sample only having O and Zr constituent. But when the N_2 was introduced as reactive gas with ZrN target, ZrO_xN_y film with a certain content of N are formed. XAS presented that the bilayer with different deposition order present a different spectral changes indicating different interface behaviors. It means that interface atoms environment is affected by the different deposition order of layers. These results provide the excellent suggestions for the preparation of zirconium oxynitrides films or super-lattice films applied in the aspect of mechanical and optical properties.

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Trends on Montel X-ray Optics for Inelastic Scattering and Microfocus X-ray Sources for X-ray Diffractometry

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Many applications in the field of X-ray analytics require an X-ray beam with high flux density at the sample position. Examples for these applications are single crystal diffraction, small angle scattering or microdiffraction to name but a few. The ideal source for diffractometry combines a device, that produces a microfocus X-ray beam of a size of below 50 µm, with a perfectly shaped high-reflectivity X-ray optics like multilayer mirrors, that are able to focus or collimate the beam to the sample or detector position.
In this contribution we will be presenting the latest developments of this kind of solution. Our family of air-cooled Incoatec Microfocus Sources deliver collimated beams with a divergence of below 0.5 mrad or focused beams with sizes down to about 100 µm with a flux of up to 1E+09 ph/s and a peak flux density of up to 8E+10 ph/s mm².
Most of these sources are integrated in standard instruments. More and more they are also successfully integrated in customized set ups for in-situ measurements of crystal and thin film growth. We will be showing some examples in the field of X-ray diffractometry and SAXS, that were not long ago only possible with synchrotron or rotating anode sources.

Different kinds of X-ray mirrors are required for beam alignment, guidance or monochromator applications. Therefore various types of optics with coatings for energy ranges between 100 eV and 80 keV have been investigated.
We will present new developments of Montel Optics for synchrotron applications. Different types of these two-dimensional optics are used at DLS, NSLS and APS and Ricken, for example in an analyzer system for inelastic scattering. One new 230 mm Montel Optic for 11215 eV was delivered these days to ESRF for BL ID20.

Figure 1: Montel Optics **-** 100-250 mm in length
Different cross sections from 40 x 40 mm to 10 x 10 mm. Optics, with slope errors < 1 arcsec, were sold to NSLS, DLS, APS and ESRF. They are used at inelastic scattering beamlines.

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