Cr/C multilayer mirror for Ni-like Ta X-ray laser application

Mingqi Cui^{1*}, Jingtao Zhu², Haochuan Li²

Abstract: Soft X-ray laser working in or near the "water window" (22.8 - 43.6 Å) region enables many fundamental and applied researches. The working wavelength of Ni-like Ta soft X-ray laser is 44.83 Å, just near the "water window". High reflection multilayers are required for this application in China. In this work, we design and fabricate carbon-based multilayer reflective mirrors. Cr/C multilayer was selected from proposed candidates such as Co/C, Ni/C, and CoCr/C material combinations. The period thickness of bi-layer is only 22.6 Å. Therefore, the interface roughness and diffusion will strongly reduce the multilayer reflectivity. Moreover, the bi-layer number is much larger than several hundreds. Cr/C multilayer was deposited by magnetron sputtering technique. Multilayers with bi-layer numbers of 150, 200, 250 and 300 were deposited onto super polished silicon substrate with surface roughness 3 Å. All multilayers have been characterized by grazing incident X-ray reflectance (XRR). Then, the near-normal incidence reflectance measurements were performed at beamline 3W1B, Beijing synchrotron radiation (BSRF). Transmission electron microscopy measurement also clearly shows the sharp Cr-C interface in multilayer. All measured results show well defined multilayer structure and marvelous optical performance. Figure 1 shows that the highest reflectance of 13.2% for the 300-bilayer Cr/C multilayer mirror at near normal incidence.

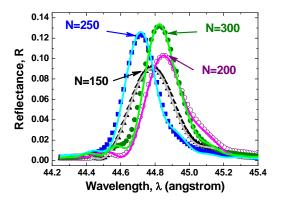


Fig. 1 Near-normal reflectance of Cr/C multilayers measured at 3W1B, Beijing synchrotron radiation facility

(dots for measured data, solid line for fitted ones)

Keywords: multilayer; X-ray laser; reflectance mirror; synchrotron radiation

¹⁾ Beijing Synchrotron Radiation Facility, Institute of High Energy Physics, Chinese Academy of Science, Beijing 100049, China

²⁾ School of Physics Science and Engineering, Tongji University, Shanghai 200092, China

^{*}Corresponding author's E-mail address: cuimg@ihep.ac.cn

Testing of Reflective Quarter-Wave Retarder in EUV Range

A.E.H Gaballah*1,2,3, P. Zuppella², A.J. Corso², P. Nicolosi^{1,2}

1 University of Padova, Department of Information Engineering, via Gradenigo 6B, 35131 Padova, Italy.

2 CNR-IFN UOS Padova, Via Trasea 7, 35131 Padova, Italy. 3 National Institute of Standards (NIS) Tersa St. 12211 Giza, Egypt. *Corresponding author: gaballah@dei.unipd.it

Abstract

There has been an increasing demand to understand the optical, electronic, and structure properties of materials at shorter wavelengths in the far ultraviolet (FUV) and extreme ultraviolet (EUV) range. This has pushed researchers to examine and outline new optical devices as quarter wave retarder (QWR) which, coupled with other techniques, can provide valuable information about physical and optical properties of materials[1].

Aluminum is a decent hopeful material that has been used for quite a while for multilayer optical tools in the FUV-EUV range because of its high reflectivity performances. Several studies have been done to use quarter-wave retarders based on a single layer of aluminum reflective coatings[2]. However, for wavelengths shorter than 160 nm the presence of aluminum oxide Al_2O_3 due to the strong reaction with air strongly influences the optical properties of the film. The oxide layer has very high absorption in vacuum ultraviolet range reducing the reflectance of the aluminum in this region. [3] Then some protective cap layer structure is required to avoid oxidation without affecting the resulting performances.

For that purpose, we investigated the polarization properties of reflective EUV polarimetric apparatus based on Al layer structure as a quarter-wave retarder (QWR) with a protective capping layer to avoid oxidation and contamination to improve stability and reflectivity efficiency. This device works within a suitably wide spectral range (88-160 nm) where some important spectral emission lines are as the hydrogen Lyman alpha 121.6 and Oxygen VI (103.2 nm) lines. The structure was characterized by performing reflectivity measurements with s- and p-polarized light as a function of the wavelengths for different incidence angles. In addition, the phase retardance on reflection was determined however, it is not an easy task to characterize the phase delay. Previously, common methods for measuring the phase shift of an optical tools are using interferometric techniques [4]. But in the EUV, these procedures are constrained frequently to work at small grazing incidence or a narrow bandwidth. At this time, a commonly used method in the EUV ML characterization is based on the photoemission analyses obtained via Total Electrons Yield (TEY) measurements combined to reflectance estimations to test the standing wave on the surface of a multilayer film. [5].

Such design could be particularly useful as diagnostic tools in EUV-ellipsometry field. The system can be a relatively simple alternative to Large Scale Facilities and can be applied to test optical components by deriving their efficiency and their phase effect, i.e. determining the Mueller Matrix terms, and even to the analysis of optical surface and interface properties of thin films. In addition, the phase retarder element could be used in other experimental applications for generating EUV radiation beams of suitable polarization or for their characterization.

REFERENCES

- [1] M. F. Tesch, M. C. Gilbert, H.-Ch. Mertins, D. E. Bürgler, U. Berges, and C. M. Schneider, "X-ray magneto-optical polarization spectroscopy: an analysis from the visible region to the x-ray regime" Appl. Opt. 52(18), 4294-4310 (2013).
- [2] T. Saito, A Ejiri, and H. Onuki, "polarization properties of an evaporated Aluminum mirror in the VUV region", App. Opt., 22 pp. 4538, (1990).
- [3] F.Gervais "Aluminum Oxid" [Hand Book of Optical constant of solids II E.D], Palik ed., Academic Press, Orlando, (1991).
- [4] I. Walmsleya, L. Waxer, and C. Dorrer, Rev. Sci. Instrum. 72, 1 (2001).
 [5] A. Aquila, F. Salmassi, and E. Gullikson, "Metrologies for the phase characterization of attosecond extreme ultraviolet optics," Opt. Lett., vol. 33, no. 5, pp. 455-457, 2008.

In-house X-ray Standing Wave study of LaN/B multilayer mirrors.

PXRNMS, University of Twente, Enschede, 10-11 November 2016

C.P. Hendrikx, I. A. Makhotkin, R.W.E. van de Kruijs and Fred Bijkerk

Industrial Focus Group XUV Optics, MESA+ Institute for Nanotechnology, University of Twente, Drienerlolaan 5, Enschede, 7522 NB, The Netherlands

Development of the state-of-the-art periodic multilayer structures requires implementation of ultrathin anti diffusion and/or contrast enhancing layers. The study of these structures by regular X-ray reflectivity techniques is limited by the lack of optical contrast between the layers. To study this type of objects with a higher precision an atom-sensitive technique is required. The ideal candidate is the X-Ray standing waves (XSW) technique.

The X-rays wave, reflected from a multilayer structure in the Bragg reflection conditions, forms the strong XSW with period equal to the period of this multilayer. By tilting the angle of incidence in the vicinity of the first Bragg angle, the phase of the standing wave is changing and the electric field maximum shifts trough the period. As a consequence atom-specific fluorescent yield form atoms placed at different locations in the multilayer period will be excited at different angles of incidence. The angular dependent intercity of fluorescence measured from the multilayer gives information about the atomic distributions in various regions in the multilayer period.

To get an exact description of the multilayer period an electric field description is calculated¹ inside the multilayer by using an optical constant profile obtained from a GIXR analysis².

The XSW measurements from LaN/B multilayer mirrors were performed on in-house PANalytical Empyrean diffractometer equipped with AMPTEC Peltier cooled XRF detector. We will show results of XSW measurements and data analysis from periodic LaN/B multilayer structures.

- 1. S. N. Yakunin, I. A. Makhotkin, et. al., J. Appl. Phys. 115 (13), (2014).
- 2. A. Zameshin, I. A. Makhotkin, et. al., Journal of Applied Crystallography 49 (4), 1300-1307 (2016).

Development of high reflectance Cr/V multilayer mirror for water window applications

Qiushi Huang¹, Jiani Fei¹, Yang Liu¹, ZhongZhang¹, Philippe Jonnard^{2,3}, Angelo Giglia⁴, Zhanshan Wang¹*

1 Key Laboratory of Advanced Micro-Structured Materials MOE, Institute of Precision Optical Engineering, School of Physics Science and Engineering, Tongji University, Shanghai 200092, China 2 Sorbonne Universités, UPMC Univ Paris 06, Laboratoire de Chimie Physique-Matière et Rayonnement, 11 Rue Pierre et Marie Curie, F-75231 Paris Cedex 05, France 3 CNRS UMR 7614, Laboratoire de Chimie Physique-Matière et Rayonnement, 11 Rue Pierre et Marie Curie, F-75231 Paris Cedex 05, France 4 CNR Istituto Officina Materiali, 34149 Trieste, Italy wangzs @tongii.edu.cn

Abstract

Imaging and spectroscopy in the "water window" (λ =2.3-4.4nm) has been long pursued in the fields of biology and material science, driven by the natural contrast between carbon and oxygen, and the high spatial resolution provided by the short wavelength. Besides the high quality sources, multilayer mirror is another key component for the water window microscope. Due to the short working wavelength, the d-spacing of the multilayer is only 1-2nm. This imposes a severe challenge for the fabrication of such multilayer mirrors. Cr/V multilayer is one of the promising candidate working near the V-L edge (λ =2.4nm) []. To develop high reflectance multilayer mirror for this region, the physical structure inside the multilayer with different layer thicknesses were investigated which indicate an obvious polycrystallization of the structure. Interface engineering was further applied to the Cr/V system which produced a maximum reflectance of 24% near the V-L edge at 42° grazing incidence [2,3].

References

- [1] E. M. Gullikson, F. Salmassi, A. L. Aquila, and F. Dollar, Progress in short period multilayer coatings for water window applications, Lawrence Berkeley National Laboratory, 2008, http://escholarship.org/uc/item/8hv7q0hj.
- [2] Q. Huang, J. Fei, Y. Liu, P. Li, M. Wen, C. Xie, P. Jonnard, A. Giglia, Z. Zhang, K. Wang, Z. Wang, High reflectance Cr/V multilayer with B4C barrier layer for water window wavelength region, Opt. Lett. 41(4), 701 (2016).
- [3] Pin Li, Qiushi Huang, Li Jiang, Wenbin Li, Jiani Fei, Jie Zhu, Zhong Zhang, Zhanshan Wang, Structure and depth profile composition analysis of Cr/(B4C)/V/(B4C) multilayer for water window application, Vacuum, 128, 85-90 (2016).

Interface Growth in FeCo-Si Multilayers determined with atomic resolution

Th. Krist, S.-J. Cho, J. Hoffmann Helmholtz-Zentrum Berlin, Hahn-Meitner-Platz 1, 14109 Berlin, Germany

FeCo-Si multilayer systems can be used as neutron polarisers due to the matching of their scattering length densities for one spin component. To optimise their performance, the thickness of interface layers should be as small as possible. For this purpose a model system was built and characterised by several methods.

Polarized neutron reflectometry is used to characterise a model system of a monochromator with 5 bilayers of nominally 100 Å Si and 100 Å Fe89Co11 covered with a final layer of 100 Å Si to prevent oxidation of the Fe89Co11 layer. From these data it was possible to determine the thickness and composition of the interface layers.

The values of these properties depend on the sputtering parameters. Under the conditions of minimal thickness of the interface layers neutron reflectometry showed on top of the silicon layer 15 Å $(Fe_{89}Co_{11})_{0.51}Si_{0.49}$ and on top of the iron cobalt layer 18 Å $(Fe_{89}Co_{11})_{0.67}Si_{0.33}$. The errors are in the thickness ± 2 Å and in the composition $\pm 2\%$.

Additional measurements with in situ fast kinetic ellipsometry and x-ray scattering agree with the neutron results within the error limits.

In situ fast kinetic ellipsometry allows investigating the growth of sputtered $Fe_{89}Co_{11}$ -Si multilayers, revealing that during growth the iron cobalt layer undergoes a phase transition. After the formation of the interface layer on top of the silicon layer the iron cobalt alloy starts to grow in an amorphous phase until a thickness of 25 Å is reached. Then it crystallizes down to the interface layer whereby it shrinks to 18.5 Å. The atomic density changes nearly proportionally to the imaginary part of the index of refraction.

Using all these data it was possible to describe the growth of the multilayer system with atomic resolution.

Email: krist@helmholtz-berlin.de

Grazing incidence EUV surface metrology: Benchmarking of DPP source table-top scatterometry versus PTB synchrotron based EUV-Radiometry

Oleksiy Maryasov^{1,2}, Larissa Juschkin¹, Christian Laubis², Frank Scholze²

Optical scatterometry is a powerful technique for surface roughness metrology and profile characterization of nano-structured layered surfaces. Besides being a fast, non-contact and non-destructive method, it provides spectrally resolved data on the roughness power spectral density (PSD). Several mirror samples with a multilayer coating (ML) with different periods were produced on substrates of different roughness. The samples are characterized in terms of surface roughness by both atomic force microscopy and EUV scatterometry. The EUV scattering patterns where recorded first using a table-top scatterometry setup [1] based on a discharge-produced plasma (DPP) source [2] with a CCD camera and second using the PTB EUV-reflectometer at the EUV-Radiometry beamline at the Metrology Light Source (MLS) [3]. The latter data were recorded by scanning the angular range of the scattered radiation with a photodiode detector of known solid angle of detection. Figure 1 shows typical grazing incidence scattering patterns as recorded using the CCD at the laboratory set-up (a) and using the photodiode detector at the PTB EUV beamline (b).

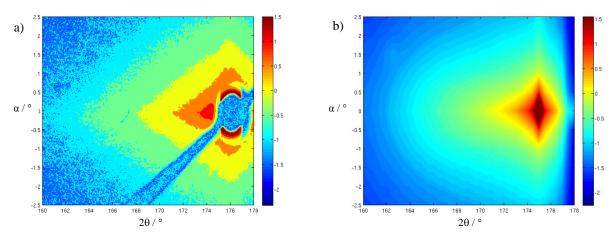


Figure 1. Example of scattering patterns from a test sample with CeO_2/B_4C multilayer coating (N = 10, d = 20 nm), measured with the DPP source with CCD detector (a) and measured at the synchrotron with diode detector (b), both under $AOI = 87.5^{\circ}$ @ 13.5 nm wavelength.

The laboratory DPP source provides enough photons for recording scattering patterns in reasonably short times from 5 to 10 min. The CCD scattering patterns provide principally higher angular resolution, but due to the lower photon flux from the DPP source the data are effectively limited by signal to noise ratio. The CCD also requires a beamstop (Figure 1a) for blocking the specular beam. At grazing incidence angles the EUV light does not penetrate into the ML-stack and hence does not reflect internal ML interface roughness. We only observe scatter related to the surface roughness which can therefore be directly compared to AFM data. The quality of the PSD as obtained using a laboratory source will be discussed and compared to results derived from synchrotron radiation based measurements.

References:

- [1] O. Maryasov et al. "Table-top EUV scatterometer MARYS with high-brightness discharge plasma source", JARA-FIT Annual Report (2015)
- [2] F. Kuepper, K. Bergmann et al. "Source operation manual", Fraunhofer ILT (2014)
- [3] C. Laubis, et al. "Update on EUV-Radiometry at PTB", Proc. SPIE, 977627 (2016)

¹ – RWTH Aachen University, Steinbachstraße 15, 52074 Aachen, Germany

² – Physikalisch-Technische Bundesanstalt (PTB), Abbestraße 2-12, 10587 Berlin, Germany

In-situ stress measurement of thin film and multilayer deposition

Johan Reinink, Robbert van de Kruijs, and Fred Bijkerk

Industrial Focus Group XUV Optics, MESA Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

The growth of thin films is often accompanied by a strain induced surface stress. This strain depends on the growth mode (e.g. amorphous, crystalline), and modifies the electrical, mechanical and optical properties of the thin film, down to atomic film thickness. We measure the stress development during deposition of Mo/Si based multilayers, used as optical coatings in EUV photolithography. We use a laser deflection based measurement setup. The measurements reveal the stress development of the crystallization of the Mo layer and the interface formation of Si on Mo in a real-time mode during growth. The effect of ion treatment of layers on stress is also investigated. Several deposition conditions are varied and barrier layers are added to determine their influence. Measurement results show several possibilities for stress engineering.

Development of multilayer coated replicated neutron focusing optics

S. Romaine¹, R. Bruni¹, M. Gubarev², D. Hussey³, B. Khaykovich⁴

- ¹Smithsonian Astrophysical Observatory, Cambridge, MA 02138, USA;
- ² National Space Sciences and Technology Center, Huntsville, AL USA
- ³ National Institute of Standards and Technology, Gaithersburg, MD, USA
- ⁴Nuclear Reactor Laboratory, Massachusetts Institute of Technology, Cambridge, MA 02139, USA;

Abstract

We have been developing multilayer coated X-ray optics for future X-ray missions using an electroformed nickel replication process to fabricate complete shells which are then coated with multilayers using DC magnetron sputtering. However, for small diameter optics the cathodes used to deposit the coatings can no longer fit inside the optic for direct deposition. Therefore we have developed an indirect coating process which allows us to achieve multilayer coatings on the inside surface of arbitrarily small diameter integral optics. The multilayer coating is applied to the mandrel and is released with the optic as part of the electroformed nickel replication process. We are now applying this same technology to fabricate multilayer coated neutron focusing optics. We present results from the first prototype multilayer coated neutron focusing optic which was recently tested at the National Institute of Standards and Technology.

The At-Wavelength Metrology facility for UV- and XUV reflection and diffraction optics at BESSY-II

F. Schäfers, F. Eggenstein, F. Senf, A. Sokolov

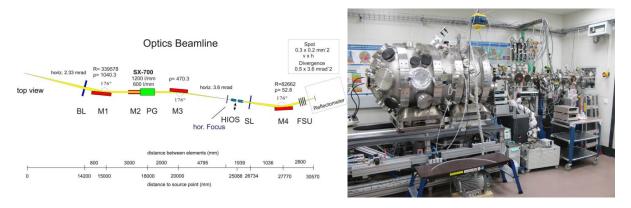
HZB-BESSY-II, Institute for Nanometre Optics and Technology, Albert-Einstein-Straße 15, 12489 Berlin

We have established a technology center for the production of high precision reflection gratings. Within this project a new optics beamline and a versatile reflectometer for atwavelength characterization of UV- and XUV reflection gratings and (nano-) optical elements has been set up in a clean-room surrounding at a bending magnet of the BESSY-II storage ring.

The Plane Grating Monochromator beamline operated in collimated light (c-PGM) is equipped with an old broad-range SX700 monochromator. The blazed gratings (600 and 1200 l/mm) have been exchanged by new ones of better performance produced in-house. Over the operating range from 10 to 2000 eV this beamline has very high spectral purity achieved by (1) a four-mirror arrangement of different coatings which can be inserted into the beam at different angles and (2) absorber filters for high-order suppression. Stray light and scattered radiation is suppressed efficiently by in-situ exchangeable apertures and slits. The beamline can be adjusted to either linear or elliptical polarization by use of off-plane synchrotron radiation.

The main feature of the novel 4-circle and 6-axes reflectometer is the possibility to incorporate real lived-sized gratings (up to >300mm in length). The samples are adjustable within six degrees of freedom by a specially developed UHV-tripod system compatible with a sample load up to 4 kg. Reflection properties can be measured between 0 and 89.9 degrees incidence angle for both s- and p-polarization geometry. A 360°-azimuthal rotation of the sample allows investigation of polarization properties.

This novel powerful metrology facility is in operation and is open for sophisticated reflection spectroscopy experiments. Results on optical performance and measurements on (multilayer-) gratings and interface structures will be presented at the workshop.



Financial support of the European Community is gratefully acknowledged (ERDF-contract no. 20072013 2/43)

REFERENCES

- [1] F. Schäfers et al., The At-Wavelength Metrology Facility for UV- and XUV-Reflection and Diffraction Optics at BESSY-II, Journal of Synchrotron Radiation, 23(1), 67-77 (2016)
- [2] A. A. Sokolov et al., *At Wavelength Metrology facility for XUV reflection gratings,* Rev. Sci. Instrum. **87**, 052005-1-7 (2016)

Characterization of chemical processes and interfacial diffusion in Pd/Y multilayers using HAXPES induced by standing waves

M.-Y. WU^{1,2}, P. Jonnard^{1,2}, J.-M. André^{1,2}, K. Le Guen^{1,2}

¹ Sorbonne Universités, UPMC Univ Paris 06, Laboratoire de Chimie Physique - Matière et Rayonnement, 11 rue Pierre et Marie Curie, F-75231 Paris cedex 05, France ² CNRS UMR 7614, Laboratoire de Chimie Physique - Matière et Rayonnement, 11 rue Pierre et Marie Curie, F-75231 Paris cedex 05, France. meiyi.wu@upmc.fr

We characterize Pd/Y multilayer and several derivative systems [1] designed to work in λ =7.5-11 nm range, using hard x-ray photoemission spectroscopy (HAXPES) combined with x-ray standing waves [2]. Experiments will be performed in September at the GALAXIES beamline of the SOLEIL synchrotron facility in France. Investigation of the real physical structure of the samples (interface roughness and diffusion, compound formation) and the relation to the experimental performance will help optimize the deposition process and improve its optical properties. The Pd L3 and M5, Y L3 and M5, B K, and C K HAXPES spectra of five samples with and without the B4C barrier layer of different thicknesses will be measured. The incident photon energy will be 10 keV in order to prbe a few periods of the stacks. The chemical processes taking place in the Pd/Y multilayers without diffusion barrier, or with B₄C layers of different thickness acting as diffusion barriers will be identified. Structural information of the multilayers will be revealed by comparing the experimental data with simulations. We expect to obtain a clear in-depth description of the stack from its chemical structure point of view. The effect of oxidation of the very first of the stack will be observed and the different chemical states of the various elements located, either at the interfaces or in the center of the layers. The positioning of x-ray standing waves enhancement for the emission is realized by rotating the grazing incident angle around Bragg angle as shown in figure 1 where the x-ray standing waves' anti-nodal plans are placed on Y-on-Pd interfaces as a result of constructive interferences, thus such the photoelectron emission on such interfaces will be enhanced while the emission at Pd-on-Y interfaces is minimized because of the destructive interferences.

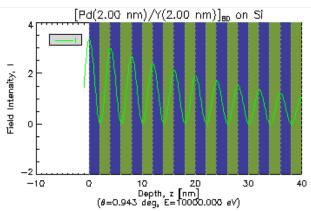


Figure 1: Simulation of field intensity in the Pd/Y multilayer stack. Grazing angle is set at the Bragg angle calculated with incident beam energy (10 keV) and multilayer period (4 nm).

References

- [1] Dechao Xu et al. Enhancement of soft X-ray reflectivity and interface stability in nitridated Pd/Y multilayer mirrors.
- [2] A. Giglia et al. Thermal effects on Co/Mo2C multilayer mirrors studied by soft x-ray standing wave enhanced photoemission spectroscopy, Proc. SPIE 8777, 87770I (2013).

Development of XUV multilayer coatings in IOF

Sergiy Yulin, Philipp Naujok, Robert Müller, Norbert Kaiser

Fraunhofer-Institut Angewandte Optik und Feinmechanik, Albert Einstein Straße 7, 07745 Jena,

Germany

Abstract

High-reflective XUV coatings are a key component for many applications including microscopy in the "water window" (2.3 - 4.4 nm), next generation lithography (λ = 13.5 nm), high-order harmonic generation (20 - 50 nm), astronomy and spectroscopy (5 - 80 nm) as well as plasma diagnostics and laser research.

At the Fraunhofer IOF Jena multilayer coating development covers the full spectral range from 1.0 nm to vacuum ultraviolet around 200 nm. This paper covers some theoretical design considerations, prospects of modern interface-engineered strategies (interface barriers and capping layers) and deposition techniques for controlled fabrication of XUV multilayer coatings. The paper summarizes our recent progress in preparation of high-reflective multilayer coatings with regard to minimum structure imperfections, enhanced stability and different possibilities in broadening of the angular reflective response.

Interface engineering method for ultra-thin Cr/Ti soft x-ray multilayer

Jingtao Zhu, ^{1,a)}Haochuan Li¹, Hongchang Wang², Mingqi Cui³, JinwenChen¹, Jiayi Zhang¹

MOE Key Laboratory of Advanced Micro-structured Materials, School of Physics Science and Engineering, Tongji University, Shanghai 200092, China

Abstract: Cr/Ti multilayers are one of the suitable candidates in the "water window" wavelength range (2.4-4.4 nm) because the theoretical near-normal incidence reflectivity is as high as 64% at wavelengths just above the Ti 2p edge (2.73 nm). However the reported reflectivity of real Cr/Ti multilayers at near-incidence angles is lower than 3%. [1] The large disparity between the theoretical and the measured reflectivity is caused by several physical limitations, among which the most important is the extreme sensitivity to interfacial imperfections for the ultra-short periods $(\Lambda \sim 1.4 \text{ nm})$ used. Recently, there has been a huge effort to develop techniques that can reduce the interface width of multilayers. One recent attempt involved the intentional incorporation of light-element impurities such as N into multilayers. It has been shown that the incorporation of N in Cr/Sc^[2] or B₄C/La^[3] multilayers can form nonstoichiometric nitride multilayers with reduced inter-diffusion or reaction between the constituent layers and leads to major improvements in the soft x-ray reflectivity. Concerning Cr/Ti multilayers, B or C is a better choice than N because of the stronger absorption of N in the applicable wavelength range of Cr/Ti multilayers, which is slightly lower than the N K edge (3.02 nm). In this work, we intentionally incorporated B and C into ultra-thin Cr/Ti soft x-ray multilayers by co-deposition of B₄C at the interfaces during DC magnetron sputtering deposition. The effect on the multilayer structure and composition has been investigated using x-ray reflectometry, x-ray photoelectron spectroscopy, and cross-section electron microscopy. The B and C is mainly bonded to Ti sites, forming a nonstoichiometric TiB_xC_v composition, which hinders the diffusion of Cr into the Ti layers and dramatically improves the interface quality of Cr/TiB_xC_y multilayers. As a result, the near-normal incidence soft x-ray reflectivity increases from 4.48% to 15.75% at wavelength of 2.73 nm.

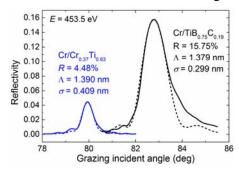


Fig. 1. Soft x-ray reflectivity from the Cr/Ti multilayers with and without B_4C .

Reference:

[1]N. Ghafoor, Per O. Å. Persson, J. Birch, F. Eriksson, and F. Schäfers, Appl. Opt. 45, 137 (2006).

_

²⁾ Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxfordshire OX11 0DE, United Kingdom

³⁾ Beijing Synchrotron Radiation Facility, Institute of High Energy Physics, Chinese Academy of Science, Beijing 100049, China

^[2] N. Ghafoor, F. Eriksson, E. Gullikson, L. Hultman, and J. Birch, Appl. Phys. Lett. 92, 091913 (2008).

^[3]T. Tsarfati, R. W. E. van de Kruijs, E. Zoethout, E. Louis, and F. Bijkerk, Thin Solid Films 518, 7249 (2010).

^{a)} Corresponding author's e-mail address: <u>itzhu@tongji.edu.cn</u>.

EBL2: high power EUV exposure facility

<u>Herman Bekman</u>, Edwin te Sligte, Norbert Koster, Freek Molkenboer, Peter van der Walle, Pim Muilwijk, Wouter Mulckhuyse, Bastiaan Oostdijck, Christiaan Hollemans, Bjorn Nijland, Peter Kerkhof, Michel van Putten, Andre Hoogstrate, Alex Deutz

TNO, Delft, The Netherlands

TNO is integrating EBL2, an open access facility to investigate the effects of EUV radiation on surfaces to enable future EUV HVM production.

A Sn-fueled Ushio LDP source is used to generate EUV. A two-stage grazing incidence collector system projects the EUV onto the mask location.

EBL2 will meet the intensity roadmap for all foreseen EUV litho tools and EUV sources, exposing at ~10 W total power. EBL2 will accept small test samples as well as EUV masks for both EUV exposure and XPS analysis. Masks with pellicles are also accepted.

MULTILAYER LAUE LENSES FOR HARD X-RAY MICROSCOPY

N. Bouet¹, H. Yan¹, J. Zhou¹, R. Conley², X. Huang¹, Y. S. Chu¹, A. Macrander², M. Vescovi¹, K. Lauer¹, E. Nazaretski¹, W. Xu¹ and P. Ilinski¹

¹National Synchrotron Light Source II, Brookhaven National Laboratory, Upton NY, USA ²Advanced Photon Source, Argonne National Laboratory, Lemont IL, USA

Recent progresses in thin film deposition and nanofabrication techniques have broadened the possibilities for the fabrication of new devices with larger design flexibility. Thus, Multilayer Laue lenses (MLLs), which consists of a depth-graded multilayer which is subsequently sectioned into a high—aspect ratio structure to become a usable optic have been receiving increased attention in the past few years [1]. Sectioning of multilayer films into high aspect ratios structures presents enormous challenges in order to produce high quality MLLs, especially as the aperture size continues to increase. Many grand challenges in growth, post-processing and characterization have been overcome in the past few years, allowing the production of a nearly-perfect MLL with focusing resolution approaching the diffraction limit [2]. Our developments led to the fabrication of MLL optics delivering a 15 x 15nm² focus at 12keV [3] to users for science experiments at the HXN beamline of the NSLS-II, and the fabrication of the largest MLL reported to date (102 microns) [4]. To reach higher spatial resolution and efficiency, wedged MLLs optics are needed. Wedged MLLs are based on the challenging deposition of profiled and depth-graded layers to form the multilayer. Our recent advances on wedged MLLs fabrication will also be presented.

References

- [1] H. Yan, R. Conley, N. Bouet, Y.S. Chu, J. of Physics D: Applied Physics, 47 (2014) 263001.
- [2] X. Huang, H. Yan, E. Nazaretski, R. Conley, N. Bouet, J. Zhou, K. Lauer, L. Li, D. Eom, D. Legnini, R. Harder, I.K. Robinson, Y.S. Chu, Sci. Rep., 3 (2013).
- [3] Y. S. Chu, H. Yan, E. Nazaretski, S. Kalbfleisch, X. Huang, K. Lauer, and N. Bouet, SPIE Newsroom, 14 August 2015, DOI: 10.1117/2.1201508.006068.
- [4] A.T. Macrander, A. Kubec, R. Conley, N. Bouet, J. Zhou, M. Wojcik, J. Maser, Appl. Phys. Letters, 107 (2015) 081904.

CeMOX, a Collaborative facility for Development of High Performance Multilayer Optics

<u>Blandine Capitanio</u>^a, David Dennetiere^a, Evgueni Meltchakov^b, Catherine Burcklen^b, Maël Dehlinger^b, Danielle Dowek^c, François Polack^a, Franck Delmotte^b

^aSynchrotron Soleil, L'orme des merisiers, Saint-Aubin, 91192 Gif-sur-Yvette, France. ^bLaboratoire Charles Fabry, Institut d'Optique, CNRS, Université Paris-Saclay, 91127 Palaiseau cedex, France.

^cInstitut des Sciences Moléculaires d'Orsay, Fédération Lumière Matière, Bat 350, Université Paris-Sud,91405 Orsay, France

Author Email: blandine.capitanio@synchrotron-soleil.fr

An X-ray optics community is organizing among Paris-Saclay University around the synchrotron and XUV ultrafast sources, in order to extend the user access to short wavelength experiments. This community is raising new demands of multilayer optics, with a particular stress to achieving small periods and precisely controlling period gradients.

In this context the CeMOX ("Couches Minces pour l'Optique X") managing collaboration has been renewed around Laboratoire Charles Fabry (CNRS UMR8501), SOLEIL Synchrotron and others laboratories form Université Paris-Saclay which are part of the Fédération Lumière Matière (CNRS FR2764). The CeMOX facility including multilayer magnetron sputtering and Cu K α grazing incidence x-ray reflectometry located at Institut d'Optique was moved in new and larger clean room.

Present work is mostly focused on three subjects.

- High efficiency gratings for synchrotron X-ray monochromators in the $1-4\ keV$ energy range.[1]
- Normal incidence multilayer coating for soft-X microscopy in the water window,[2]
- Mirrors for the ultra-fast X-UV sources within Paris-Saclay campus such as ATTOLAB [3], where a precise selection of the bandpass is required while maintaining a minimum pulse length.[4]

CeMOX has been mostly relying on its Plassys MP800 magnetron deposition machine which can host mirrors up to 260 mm long. It will soon receive a new MP1000 machine which will be able to host optics up to 350 x 100 x 100 mm³, and will be equipped with an auxiliary ion gun. This new equipment will help to achieve precise layer thicknesses and accurate period gradients, even on the large optics needed by synchrotron applications.

For the water-window microscopy project, the period gradient has to be exactly matched to the strong curvature of Schwarzschild mirrors. A new reflectometer under construction will allow to characterize these new optics on SOLEIL synchrotron beamlines.

Examples of main achievements and new developments within CeMOX facility will be presented and discussed.

- [1] F. Choueikani et al., Optics Letters 39, 2141 (2014)
- [2] C. Burcklen et al., Journal of Applied Physics 119, 125307 (2016)
- [3] http://attolab.fr
- [4] F. Delmotte et al., Proc. SPIE 9589, 958907 (2015)

Roger Coloma Ribera, Marko Sturm, Robbert van de Kruijs, Andrey Yakshin, Fred Bijkerk

Industrial Focus Group XUV Optics, MESA⁺ Institute for Nanotechnology, University of Twente, Enschede, The Netherlands

In-vacuo growth studies and thermal oxidation of ZrO₂ thin films

ZrO₂ thin films might be used as capping layers for protecting extreme ultraviolet (EUV) optics against oxidation and other chemical degradation processes. These coatings should be homogeneous and form a closed layer, not degrade the underneath barrier/mirror, and have a thickness in the nanometre range to keep good reflection properties of the mirror. The initial growth of ZrO₂ films by reactive magnetron sputtering on top of amorphous Si was monitored by in vacuo low-energy ion scattering (LEIS). With this technique, the atomic composition of the outermost atomic layer of a surface can be probed. By monitoring for which deposited amount of ZrO₂ the signal from the underlying Si vanishes, the ZrO₂ thickness required for forming a closed layer was determined. In this way, LEIS was employed to study how deposition parameters influence the sharpness of the interface between the cap layer and Si. Invacuo X-ray photoelectron spectroscopy (XPS) was used to find the optimal deposition conditions and the stoichiometry of the produced layers. Depending on deposition conditions, a fully closed layer can be formed with a deposited ZrO₂ thickness of 1.7 nm. Passivation of the Si underlayer by nitrogen or oxygen was found to have no influence on the sharpness of the interface with the ZrO₂ cap. Based on the smoothness of the as deposited layers, as probed by AFM analysis, it was concluded that intermixing, rather than island formation, is limiting the sharpness of the ZrO₂/Si interface. Finally, XPS was used ex-situ for probing oxygen diffusion through the capping layer after thermal oxidation in ambient. A 2 nm ZrO₂ cap on top of Si showed further intermixing up to 400 °C, without increase of the oxidation of the underlying Si. For higher temperatures, the cap does not block oxygen diffusion, resulting in further oxidation of the Si layer.

(Abstract PXNMRS 2016, University of Twente)

The new 1 – 5 keV high efficiency alternate multilayer grating for SOLEIL SIRIUS beamline.

<u>David Dennetiere</u>^a, Blandine Capitanio^a, Muriel Thomasset^a, Franck Delmotte^b, Evgueni Meltchakov^b, Catherine Burcklen^b, Regina Soufli^c, Philippe Fontaine^a, Gianluca Ciatto^a, François Polack^a

^aSynchrotron Soleil, L'orme des merisiers, Saint-Aubin, 91192 Gif-sur-Yvette, France. ^bLaboratoire Charles Fabry, Institut d'Optique, CNRS, Université Paris-Saclay, 91127 Palaiseau, France.

^cLawrence Livermore National Laboratory, 7000 East Avenue, Livermore, California 94550, USA Author Email: david.dennetiere@synchrotron-soleil.fr

Sirius is a beamline designed for studying soft interfaces and semiconductor or magnetic nanostructures. It has recently got a new multilayer grating monochromator covering the 1-4 keV band which complements its already existing two crystal monochromator covering the 3-11 keV energy range. Besides the grating, this kind of monochromator requires a mirror with a matched multilayer coating to reflect the beam back in it initial direction at any wavelength. The grating and the matched mirrors of Sirius monochromator have been fabricated at the CeMOX facility of Laboratoire Charles Fabry (LCFIO) and characterized both there and on SOLEIL Metrologie beamline. The monochromator is under commissioning and its first results look very promising. A first experimental EXAFS spectrum will be presented.

Cr and B4C have been selected as a suitable couple of material for the targeted range. The characterization of the achieved ML grating with 35 periods of Cr (2.5 nm) /B4C (4.1 nm), showed a 1st order of diffraction efficiency of 55% at 4.4 keV. A very special care has been given to the optimal match between the grating and the deflecting mirror. A very high sensitivity to period mismatch has been simulated and measurements have been done in order to reduce the impact of period inhomogeneity along the 250 mm length of the mirror. Design simulations, reflectivity measurements of actual grating and mirrors will be shown and compared to fits with different multilayer and grating models.

Design, Development and characterization of thin film filters for high brilliance sources in the UV-X-ray Spectral range.

Kety Jimenez

Padova University, information engineering department

High brilliance sources such as synchrotron and Free Electron Laser (FEL) are very important nowadays due to their multiples application in the development of science and technology. One strong requirement on the beam delivered by these sources besides brilliance, coherence and bandwidth is often related to the spectral purity; in fact, the beam can be the superposition of various harmonics. The rejection of high harmonics or diffuse light in order to improve the quality of the beam can be achieved by suitable optical systems acting as band pass filters [1-7].

Since materials present high absorption in the EUV spectral range, the selection of devices acting as filters in order to select suitable spectral bandwidth or reject harmonics is quite challenging. This project will be focused in the searching for potential materials, design, fabrication and characterization of self standing transmittance thin films filters between 4-20 nm and 20-100nm spectral range, which correspond to intervals into the Extreme Ultraviolet and soft- x ray spectra Region, to be inserted along Synchrotron and Free Electron Laser (FEL) transport optics.

Some properties like fragility, resistance against oxidation and carbonation of the films and resistance to the high power of these light sources have to be taken in consideration.

References:

- **1.** Hatayama et al., Japanese Journal of Applied Physics, 48, 2009.
- 2. M Fernandez Perea et al., Proc. SPIE 7101 (2008).
- **3.** Gann et al., RSI 83, 045110 (2012).
- **4.** Saitoh et al., RSI 71, 9 (2000).
- **5.** Mckinney et al.,RS1 66 2160 (1995)
- 6. Underwood et al., Journal of Electron Spectroscopy and related phenomena, 92, 265 (1998).
- **7.** F. Bencivenga et al., New Journal of Physics 15 (2013).

Nonequilibrium electron-phonon dynamics in ruthenium thin films exposed to ultra-short laser pulses.

I.Milov^{1*}, I.Makhotkin¹, S.Semin², C.Lee¹, E.Louis¹, A.Kimel² and F.Bijkerk¹.

¹Industrial Focus Group XUV Optics, MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, Netherlands

²Radboud University, Institute for Molecules and Materials, Heyendaalseweg 135, 6525 AJ Nijmegen, The Netherlands

*i.milov@utwente.nl

Abstract

The application of high repetition rate Free Electron Laser sources requires the development of robust optical coatings that will survive after being exposed to ultra-short, high peak intensity laser pulses. As a mirror for the case of grazing incidence reflectance, metal thin films are widely used. To study the lifetime of such optical coatings, the interaction of ultra-short laser pulses with metals must be understood. Since the pulse duration is comparable to or shorter than the thermalization time of the system, one needs to consider nonequilibrium electron-phonon dynamics that takes place after the laser pulse heats electrons at the surface.

The two-temperature model, with electron-phonon coupling, was used to calculate surface temperature changes of ruthenium thin films due to ultrafast laser heating. Time-domain thermoreflectance measurements were performed with 300 fs visible pump and probe pulses to monitor the surface reflectivity change.

Thermal stability and mechanical stress of B-based multilayers

P. Naujok^{1,2}, S. Yulin¹, K. Murray^{1,2}, N. Kaiser¹, and A. Tünnermann^{1,2}

¹ Fraunhofer Institute for Applied Optics and Precission Engeneering, Albert-Einstein-Straße 7, 07745 Jena, Germany

² Friedrich-Schiller-Universität Jena, Institute of Applied Physics, Abbe School of Photonics, Max-Wien-Platz 1, 07743 Jena, Germany

Abstract

In the last years, much effort has been put into the development of high reflective multilayer mirrors for a BEUV-lithography working at 6.x nm wavelength. It was previously shown that two main approaches lead to higher values of reflectivity: the introduction of ultrathin C-barriers in La/B₄C multilayers by Chkhalo et al. as well as the transition from La/B to LaN/B multilayers by Tsarfati et al. considerably enhanced the multilayers near normal reflectivity near the Boron K absorption edge $(\lambda = 6.6 \text{ nm})$.

In this contribution the authors will have a close look on the thermal stability of high reflective B-based multilayer mirrors, i.e. investigating their structure, reflective properties and chemical processes in the multilayer and at the interfaces for elevated temperatures up to $800\,^{\circ}$ C. La/B₄C and LaN/B₄C multilayers will be compared with various characterization methods like XRR, XRD, HRTEM, XPS and EUVR.

Furthermore, the coatings for various applications should have low residual stresses to avoid form deformation of precise substrates. The authors will present results on the residual stress of different B-based multilayer coatings. The physics of the temperature dependent development of the stress will be discussed, too.

- [1] Chkhalo, N. I. et al., Appl. Phys. Lett. 102(1), 2013.
- [2] Tsarfati, T. et al., Thin Solid Films 518(5),2009.

Fabrication, characterization and application of large aperture multilayer Laue lenses

S. Niese¹, S. Braun², R. Dietsch¹, J. Gluch³, T. Holz¹, A. Kubec²

¹ AXO DRESDEN GmbH, Gasanstaltstraße 8b, 01237 Dresden, Germany

Multilayer Laue lenses are an emerging type of X-ray lenses in hard X-ray microscopy at photon energies of 8 keV and beyond. Their application is related to beneficial properties in terms of efficiency and focus quality in comparison to Fresnel zone plates. The total thickness of the multilayer stack equals the size of the aperture of the resulting X-ray lens. Thus, the fabrication of multilayers with a total thickness of > 50 µm consisting of currently up to 12500 individual layers with a defined lateral and depth gradient is showed. Individual lenses are manufactured with micromachining methods and optionally bended to enhance the diffraction efficiency. The final assembly to a compact lens device allows a convenient integration in scanning and full-field X-ray microscopy instruments. Extensive characterization is performed during manufacturing in advance of the final measurement of the focusing properties using synchrotron radiation. A main application is point or line focusing of synchrotron radiation to dimensions well below 50 nm. Various methods such as nano X-ray diffraction, X-ray fluorescence and ptychography benefit from the small and intense X-ray focus. The status of the integration to a full-field X-ray microscope with a laboratory X-ray source is shown as well.

² Fraunhofer IWS Dresden, Winterbergstraße 28, 01277 Dresden, Germany

³ Fraunhofer IKTS, Maria-Reiche-Str. 2, 01109 Dresden, Germany

Numerical modelling of reflective multilayer based X-ray optics

P. Piault¹, C. Ferrero¹, F. Delmotte², S. Berujon¹, A. Rack¹, C. Morawe¹, J. P. Guigay¹, E. Ziegler¹

Artificially stratified films deposited on a mirror surface are envisaged as optical elements capable to either propagate a perfect wavefront from the source to the sample without degradation or to correct for wavefront imperfections.

Current multilayer-coated mirrors have shown to degrade the beam coherence and distort the reflected wavefront, although significant efforts were made to minimise them [1-3]. The propagation of phase distortions may result in the presence of undesired intensity variations in the recorded images as stripe-line modulations perpendicular to the diffraction plane. The most likely interpretation of these modulations is based on the explanation already given for silicon mirrors, i.e. imputing the wave distortion to the height deviation from a perfect surface [4-6]. However, this problem may get rapidly complicated if one tries to consider the role of every layer in the coating.

To address these issues the following project was initiated:

Firstly, images of W/B₄C multilayers were characterised at the ESRF instrumentation beamline BM05. The reflected monochromatic beam was recorded at various distances to detect a possible dependence of the stripes on the figure errors of the multilayer. Further experiments were performed to gain an insight into the way X-ray propagation in a multilayer in order to build an effective model for the reflection of a multilayer with imperfections. For instance, the speckle metrology technique [7] is used to recover the beam wavefront. The multilayer is also flipped from the vertical to the horizontal position to investigate the role of the diffracted beam coherence length on the image pattern.

Secondly, we started developing a numerical model capable of simulating the wave reflected by a multilayer. For a comprehensive simulation of the layered medium, topographies of the multilayer coating and/or substrate surfaces as recorded by Fizeau interferometry are needed. The model should provide height deviation maps as well as X-ray profiles propagating in the multilayer. Our first approach consists in modifying a model based on the Takagi-Taupin equations developed originally for a perfect flat or elliptical multilayer to simulate X-ray propagation in layered media perturbed by figure errors. In this case, these height deviations are implemented as suitable offsets of the susceptibility of the layered medium.

The ultimate goal of this work (in progress) is to explore and eventually propose ways to improve the quality of multilayer mirrors for forthcoming applications in present and future X-ray source facilities.

- 1. A. Rack, T. Weitkamp, M. Riotte, D. Grigoriev, T. Rack, L. Helfen, T. Baumbach, R. Dietsch, T. Holz, M. Krämer, F. Siewert, M. Meduna, P. Cloetens, E. Ziegler. J. Synchrotron Rad. 17, (2010), 496–510.
- 2. A. Rack, L. Assoufid, W.-K. Lee, B. Shi, C. Liu, Ch. Morawe, R. Kluender, R. Conley, N. Bouet. Radiat. Phys. Chem. 81 (2012) 1696-702
- 3. C. Morawe, R. Barrett, K. Friedrich, R. Klünder, A. Vivo. Spatial coherence studies on x-ray multilayers. In: C. Morawe, A.M. Khounsary, S. Goto. (Eds.) Advances in X-Ray/EUV Optics and Components VI, Proceedings of SPIE, (2011) vol. 8139, p. 813909.
- 4. P. Cloetens, R. Barrett, J. Baruchel, J.-P. Guigay. & M. Schlenker. J. Phys. D, 29, (1996). 133–146.
- 5. E. Ziegler, C. Morawe, O. Hignette, P. Cloetens. & R. Tucoulou. Ninth International Conference on Production Engineering, Osaka, Japan, (1999) pp. 285–291.
- 6. K. Yamauchi, K. Yamamura, H. Mimura, Y. Sano, A. Saito, K. Endo, A. Souvorov, M. Yabashi, K. Tamasaku, T. Ishikawa, Y. Mori. Appl. Opt. 44, (2005). 6927–6932.
- 7. S. Berujon, H. Wang, and K. Sawhney Physical Review A 86, (2012) 063813

¹ ESRF – The European Synchrotron, CS40220, 38043 Grenoble cedex 9, France

² Laboratoire Charles Fabry, Institut d'Optique Graduate School, CNRS, Université Paris-Saclay, 91127 Palaiseau Cedex, France

Thin film based Optical Elements for Analytical X-ray Applications

Andreas Stricker, Frank Hertlein, Uwe Heidorn, Jürgen Graf, Jörg Wiesmann

Incoatec GmbH, Max-Planck-Straße 2, 21502 Geesthacht, Germany

In this contribution we will be giving an overview on current developments of multilayer optics used for diffractometer instrumentation in the home-lab. We will be explaining the manufacturing process of the optics, summarizing the different types of optics and giving some examples of typical applications which benefit from the new possibilities, especially in combination with modern microfocus X-ray sources.

In the home-lab multilayer based Montel Optics are widely used as an essential component in modern X-ray diffractometers. These optics consist of bent substrates with shape tolerances below 100 nm, upon which multilayers are deposited with single layer thicknesses in the nanometer range and up to several hundreds of layer pairs. The multilayers are designed with lateral thickness gradients within ± 1% deviation of the ideal shape. Very low shape tolerances below 100 nm and figure errors below 5 arcsec are required for multilayer mirrors to ensure a superb flux density of up to several 10^{11} photons/s/mm² in combination with high-brightness microfocus X-ray sources, such as the air-cooled Incoatec Microfocus Source IµS or novel liquid metal jet X-ray sources. We use magnetron sputtering technology for deposition, optical profilometry in order to characterize the shape and X-ray reflectometry in order to characterize the multilayer thickness distribution both laterally and as indepth. For X-ray analytics the important beam parameters are monochromaticity, flux, brilliance and divergence. They demonstrate the quality of the combination of suitable X-ray sources with selected multilayer optics.

We will be showing some applications measured by diffractometers equipped with an I μ S. The I μ S is a low power air cooled X-ray source and is available with Cr, Co, Cu, Mo, and Ag anodes. The implemented multilayer optics form either a highly collimated beam with a low divergence (below 0.5 mrad) or a focusing beam with higher divergence (up to 10 mrad) and very small focal spots (diameter below 100 μ m).

Applications realized with an $I\mu S$ are small-angle X-ray scattering, texture, stress analysis, μ -diffraction, single crystal diffraction to name but a few.

In our presentation we will be presenting how multilayers can be characterized in-situ during their deposition by the method of grazing incidence small-angle X-ray scattering (GISAXS). This kind of experiments was done only at synchrotrons. With an IµS it becomes feasible in the home-lab.