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Progress at the ESRF multilayer facility

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Abstract. The ESRF multilayer (ML) deposition facility is fully operational since 2009. By the end of 2011, almost 50 ML projects were completed using the new machine, bringing the total number to about 150 since 1998. Thanks to the new equipment and its improved performance the throughput could be significantly increased. The ESRF upgrade project caused strong demands for new ML optics, in particular dynamically bent KB focusing devices requiring very precise and steeply graded ML coatings. Thanks to this technology, the ESRF nano-imaging end-station ID22NI now provides the users with spot sizes of the order of 50x50 nm² at a photon flux of 10¹² ph/s. Among various in-house research and development activities the study of stress evolution during thin film and ML growth will be highlighted. Additional projects involving a PhD student and a PostDoc fellow cover the fields of wave optical simulations using curved MLs and the exposure of ML based monochromators to the white beam.

1. Multilayer deposition facility
The new ML deposition facility was installed in 2007 [1]. Fully operational since 2009, almost 50 coating projects were completed using the new equipment, bringing the total number to about 150 since 1998. Most of the MLs are deployed on KB focusing optics or as broad band monochromators on ESRF beamlines. Key assets of the sputter deposition machine are robust and reliable design and user-friendly operation and maintenance.

2. New instrumentation
More recently, the ESRF Multilayer Laboratory upgraded part of its instrumentation. The main goal was to facilitate existing ML characterization procedures and to extend them to new techniques.

2.1. In-situ curvature monitor
A curvature monitor was purchased from the company WinlightX (France) and installed on a free viewport of the deposition machine. Its operation is based on a two-beam deflection technique. The measured sample curvature change can be used to calculate the stress in the growing film or ML applying the Stoney equation [2]. The system allows for quasi in-situ stress studies since each film can be subdivided into thinner growth steps and measured without removing the sample from the vacuum chamber.

2.2. Micro-focus x-ray source
The laboratory x-ray reflectometer was upgraded with a Cu Kα micro-focus source and a Montel ML collimator optic purchased from the company Incoatec (Germany). Its small beam enables scans with high spatial resolution while maintaining the overall flux of a traditional x-ray tube.

2.3. Shack-Hartmann wave front analyser
A Shack-Hartmann wave front analyser was purchased from the company Imagine Optics (France). It has been used to perform in-situ surface figure studies during white beam irradiation experiments on MLs. It is a versatile instrument that can be deployed in rough environments where interferometers cannot operate.
3. Highlights

3.1. ID22NI nano-focusing KB

Recently, the KB focusing setup on the ESRF nano-focusing end-station ID22NI was upgraded. It was equipped with improved bending devices, shorter substrates, and steeper graded ML coatings customized to fit the reduced image distances of 180 mm (vertical) and 83 mm (horizontal), respectively [3]. Since 2011, the beamline can offer an ESRF record focus size of 43 nm (vertical) x 59 nm (horizontal) in user mode (Figure 1). Thanks to the combination of high total transmission ($\approx 50\%$) and broad bandwidth (7%) a photon flux of $10^{12}$ ph/s can be reached at a photon energy of 17 keV and a storage ring current of 200 mA.

![Figure 1. Knife edge scans through the focal spot on ID22NI in horizontal (left) and vertical (right) direction.](image)

3.2. Coherence preservation with multilayers

Flat field imaging experiments using various W/B$_4$C MLs have shown that the characteristic stripe contrast is essentially caused by the substrate figure errors that are printed through the ML stack [4]. Additional Talbot studies confirm that the degree of coherence preservation is independent of the ML coating.

4. Wave optical simulations

Wave optical calculations were developed for both curved mirrors and MLs [5]. The general framework relies on the Fresnel-Kirchhoff theory. In the case of curved graded MLs, a Takagi-Taupin-like approach was used to solve the Helmholtz equation inside the layered structure. The treatment includes the effect of refraction when the wave propagates into the ML stack. A computer code was written containing a web-based graphical user interface to define the fundamental simulation parameters. The program returns amplitude, phase, and reflectivity of the focusing element in and around the focal plane. An example of the simulated intensity distribution near the focal plane is given for a W/B$_4$C ML in Figure 2. A stochastic superposition of point sources can be applied to simulate x-ray sources of finite size and variable degree of spatial coherence. Experimental figure errors can be added to the ideally curved optical surface to estimate the performance of real mirrors.

![Figure 2. Example of simulated intensity distribution near the focal plane.](image)
The spot size simulations show a good agreement with the fundamental limit of diffraction given by

\[ D_{\text{diff}} = \frac{0.44 \cdot \lambda}{NA} \]

down to focal spots on the nano-meter scale. Here, NA is the numerical aperture and \( \lambda \) the wavelength. They also confirm that the frequently applied analytical expression

\[ \Lambda = \frac{\lambda}{2 \cdot \sqrt{n^2 - \cos^2 \theta}} \]

to calculate the d-spacing \( \Lambda \) of graded MLs as a function of the grazing angle \( \theta \) is a good approximation. Here, \( n \) is the average optical index of the ML stack.

5. White beam experiments

Various MLs were exposed to the full beam of the undulator source of the ESRF beamline ID06 [6]. Both in- and ex-situ reflectivity studies show no significant modification of the ML structure after irradiation doses of 60 kJ/mm\(^2\). In-situ surface figure measurements were performed with a Shack-Hartmann wave front analyser. The figure error caused by the intense x-ray beam can be simulated by Finite Element Analysis (FEA) calculations. So far, apart from minor surface alteration, no significant damage could be observed in the ML samples.

6. Stress investigations

The newly installed curvature monitor was used to study the stress evolution during the growth of various thin single films and MLs [7]. Most thin films build up considerable compressive stress from 0 to -3 GPa when deposited at low Ar pressures. Increasing the pressure generally leads to weaker compressive or even tensile stress. The situation is more complex for MLs since interface effects may have a strong impact on the total stress in the stack. Figure 3 shows the case of Ru/B\(_2\)C MLs grown at \( p(\text{Ar}) = 1 \mu \text{bar} \). In the left figure the curvature evolution with layer thickness is presented. From the local slopes the individual stress per 1nm thick sub-layer was derived as shown in the right figure.
Figure 3. Curvature evolution (left) and individual sub-layer stress (right) of Ru/B\textsubscript{4}C MLs. The points at t = 20 nm correspond to the single film limit.

It is evident that the Ru/B\textsubscript{4}C interface contributes a strong tensile component (positive slope) to the otherwise compressive stress (negative slope) in this ML system. It is important to know these features to avoid too high stress levels that may lead to damage or de-lamination of the coatings.

7. Summary and perspectives
Thanks to the new ESRF ML deposition facility the fabrication throughput could be improved significantly. New instrumentation facilitates the sample characterization and extends it to new fields such as stress investigations. The most important applications of ML mirrors at the ESRF remain KB focusing systems. The new nano-focusing end-station ID22NI offers a spot size below 60 nm to the user community. Within the ESRF Upgrade program this beamline will be extended and improved so that even smaller spots will be within reach in the near future. While coherence preservation by MLs seems sufficient for most imaging applications, the improvement of the flat field uniformity will require further efforts. New wave optical simulation tools provide deeper insight into both fundamental limits of nano-focusing by graded MLs and the performance of real optical elements. Studies of the white beam resistance of MLs appear promising for the design of future high flux monochromators.

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