Extreme ultraviolet multilayer for the FERMI@Elettra free electron laser beam transport system

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Abstract: In this work we present the design of a Pd/B_4C multilayer structure optimized for high reflectance at 6.67 nm. The structure has been deposited and also characterized along one year in order to investigate its temporal stability. This coating has been developed for the beam transport system of FERMI@Elettra Free Electron Laser: the use of an additional aperiodic capping layer on top of the structure combines the high reflectance with filter properties useful in rejecting the fundamental harmonic when the goal is to select the third FEL harmonic.

OCIS codes: (140.2600) Free-electron lasers (FELs); (230.4170) Multilayers; (310.4165) Multilayer design; (340.7480) X-rays, soft x-rays, extreme ultraviolet (EUV).

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1. Introduction

The free electron laser (FEL) FERMI@Elettra, at the Sincrotrone Trieste Laboratory in Italy, is based on a high gain harmonic generation (HGHG) seeding scheme [1], an approach that provides highly intense radiation pulses whose temporal structure, spectral distribution, and photon energy are stable from pulse to pulse, over time. These properties of the FEL beam must be preserved up to the end-user stations by the use of ad hoc optical systems: wavefront and pulse duration preservation, as well as improvement of monochromaticity and selection of spectral content, can be achieved in such FEL transport systems by the use of reflective nanometer-scale multilayer optical coatings [2,3]. From the experimental point of view, it is also mandatory to implement and realize pump-probe experiments using both the fundamental FEL radiation and both its higher harmonics content. Consequently, optical sections where the optical path is split and selection of the proper spectral content is achieved are foreseen in FERMI; again, nanometer-scale multilayer optics will represent fundamental elements of such schemes.

In Fig. 1 a delay-line arrangement under realization at FERMI is represented [4]. Such scheme is conceived for pump-probe experiments, in which it is necessary to pump the system with the fundamental wavelength and to probe it with a delayed harmonics one. The grazing mirror M1 represents a beam-splitter that divides the FEL fundamental-wavelength pulses in two parts. The two beams travelling along two different optical paths are then recombined by the M4 optical element. Two additional sets of 4 multilayer mirrors, with all the four mirrors working at 45° incidence, are used to adjust the optical path length difference, by moving together two of them in a compensated symmetric configuration. In the case of which it is interesting to pump the system with the fundamental wavelength and to probe it with the a delayed third harmonic, multilayer mirrors able to reflect the third harmonics while rejecting the fundamental must be used in one of the two arms of the system, to properly filter the beam, as shown in Fig. 1. A delay of a few nanoseconds can be achieved in this scheme by changing the distances between the multilayer mirrors, by moving simultaneously two of them for each arm. Such schemes will be adopted both on the Diffraction and Projection Imaging (DiProI) [5] and Low Density Matter (LDM) beamlines, which are now under development. For transient grating (TG)-based experiments, for example as requested by the Time Resolved (TIMER) beamline group [6, 7], it is important to extend the current standard TG technique to the Extreme Ultraviolet (EUV) spectral region, thereby permitting investigations of dynamics at the nanoscale. To accomplish this goal, the TIMER beamline, currently under development, will also employ the delay-line scheme

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presented in Fig. 1, in this case working at discrete wavelengths of 60, 40, and 20 nm for the fundamental-wavelength (20 nm is the shortest wavelength achievable with the FEL@Elettra). The correspondent third harmonics are 20, 13.5, and 6.67 nm. In this case multilayers optimized for third harmonics reflectance having high fundamental rejection capability are necessary. We quantify to the rejection capability of a multilayer by its Fundamental Rejection Ratio (FRR), a parameter we define as:

$$FRR = \frac{R(\lambda_{3rd})}{R(\lambda_{1st})} \tag{1}$$

where $R(\lambda_{fund})$ is the reflectance at the fundamental and $R(\lambda_{3rd})$ is the reflectance at the third harmonic ($\lambda_{fund} = 3 \cdot \lambda_{3rd}$).

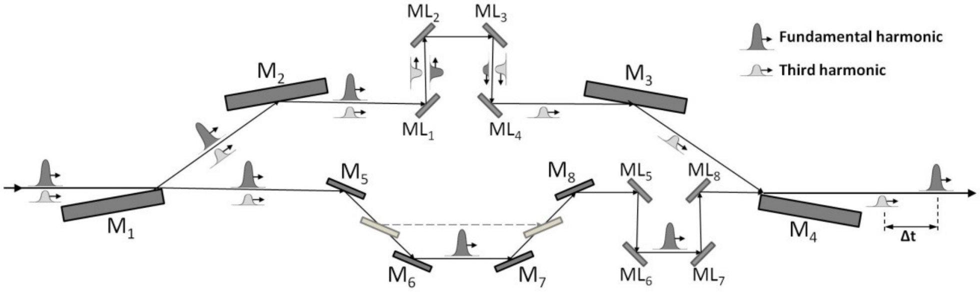

Fig. 1. Delay line system under realization at FERMI

In this work we have designed and simulated the performances of different multilayers. Some of them provide best reflectance, while other high FRR ratio. Among those, we have selected a multilayer which potentially combines both properties, representing a good compromise solution. Such multilayer coating uses the Pd/B_4C material couple, and it is optimized for high reflectance of S-polarized radiation at 45° of incidence, at a wavelength of 6.67 nm, which is just long-ward of the B K-edge at 6.6 nm. The use of an additional aperiodic capping layer based on the same material couple deposited on top of the structure is used to enhance the FRR ratio of the multilayer itself.

2. Materials and multilayer design technique

A prototype Pd/B_4C multilayer structure has been deposited and characterized, and then monitored over a period of one year, in order to establish the suitability of this coating for beam transport optics at FERMI@Elettra. The recently-developed Pd/B_4C multilayer system has been used only for shorter-wavelength X-ray applications up till now [8]. Its theoretical performance at 6.67 nm, simulated using the IMD program, is comparable to other B_4C-based multilayers that have already been investigated, including Mo/B_4C [9, 10], representing the state of art at this wavelength (experimental reflection ~25% in normal incidence), La/B_4C, recently developed for lithographic applications (experimental reflection ~40% in normal incidence) [10–12], and Ru/B_4C [13]. In Table 1 we have reported different possible multilayer designs based on the material couples just listed. Aperiodic structures have been optimized using a method described in the following to maximize the FRR ratio. Such aperiodic structures are based on a periodic stack on top of which an aperiodic capping layer is deposited. The structures have been all optimized for linearly polarized light at 45° incidence angle and simulated accordingly. The optical constants used in our simulations are those provided by the Center for X-ray Optics (CXRO), except for the case of molybdenum, whose optical constants are taken from reference [14]. The simulations were performed assuming perfectly smooth and sharp interfaces. Due to the linear polarization of the FEL

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beam, at least for the experiments of interest, hereafter we will consider the FRR calculated using only S-reflectance.

Optimization of the aperiodic capping layer structures was performed by considering the standing wave distribution [15] at the two different wavelengths of interest, the third harmonic (i.e. the wavelength for which the ML is optimized), and the fundamental. In this specific case, a layer by layer method has been applied. That is, starting from the optimized periodic structure, a top bi-layer designed to maximize the third harmonic peak reflectance is added and the FRR of the new structure is computed. If the FRR improves, another bi-layer is added and the optimization process continues, otherwise the last two layers must be removed and the structure is considered complete. The design method just described is summarized in the flowchart reported in Fig. 2.

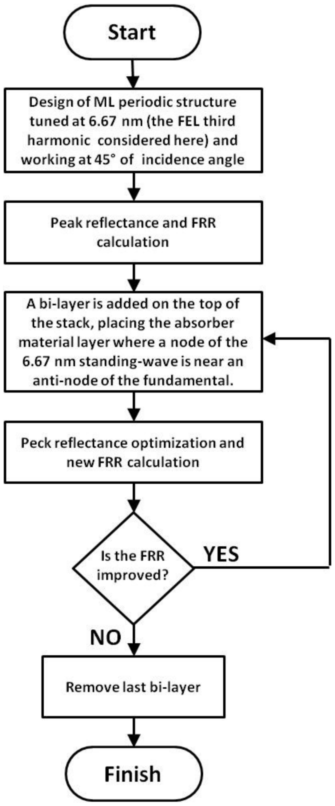

Fig. 2. The flowchart of the method adopted for designing the aperiodic structure reported in Table 1.

The standing-wave patterns generated at the two wavelengths in the Pd/B_4C multilayer case, both with and without the aperiodic capping layer, are shown in Fig. 3a and 3b, respectively. As it can be seen in these figures, in the periodic structure the anti-node of the fundamental always corresponds to a node at the third harmonic wavelength. When the periodic structure is overcoated by the aperiodic capping layer designed using the method described above, the absorber layer (the Pd layer in this case) is always placed near at a node of the third harmonic standing-wave and at an anti-node of the fundamental, with consequent suppression of the fundamental reflection.

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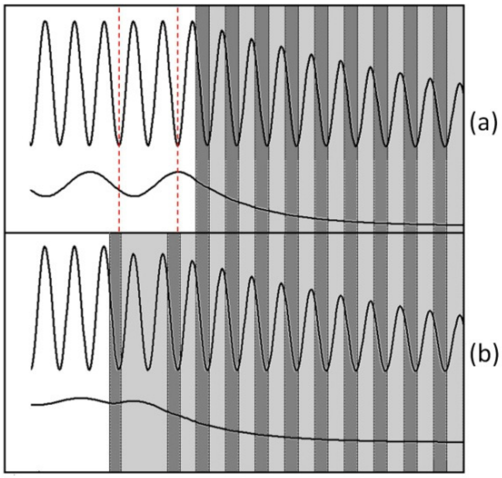

Fig. 3. Standing-wave distribution in a periodic Pd/B4C multilayer structure (a) and in a periodic Pd/B4C multilayer containing an aperiodic capping layer (b).

The performance of all four B_4C-based multilayers considered here is reported in Table 1. The simulations show that the periodic La/B_4C multilayer has the highest reflectivity, while the Mo/B4C multilayer containing an aperiodic capping layer has the best spectral rejection. The aperiodic-capped Pd/B_4C structure provides both high reflectance and good rejection of the fundamental, thereby representing a good compromise solution.

Table 1. B4C-Based Multilayers Performances Considered in this Work. For each material couple is reported the theoretical reflectance, the FRR and the FWHM with and without aperiodic capping-layer.

	Periodic structure	Capping-layer	R_S(peak)	FRR_S	FWHM (nm)
Mo/B_4C	d = 4.75nm $\Gamma = 0.45$ N = 100	B_4C (2.61 nm)	0.54	16.6	0.09
Mo/B_4C Aperiodic	d = 4.79nm $\Gamma = 0.54$ N = 100	B_4C (10.3 nm) Mo (2.4 nm) B_4C (2.61 nm)	0.52	1935	0.09
Ru/B_4C	d = 4.76nm $\Gamma = 0.45$ N = 100		0.58	16.8	0.12
Ru/B_4C Aperiodic	d = 4.76nm $\Gamma = 0.45$ N = 100	Ru(1.8 nm) B_4C(7.4 nm) Ru(2 nm) B_4C(2.5 nm)	0.58	330.7	0.12
Pd/B_4C	d = 4.76nm $\Gamma = 0.45$ N = 100		0.58	26.0	0.12
Pd/B_4C aperiodic	d = 4.76nm $\Gamma = 0.45$ N = 100	Pd(1.8 nm) B_4C(7.5 nm) Pd(2 nm) B_4C(2.5 nm)	0.58	1060	0.12
La/B_4C	d = 4.76nm $\Gamma = 0.45$ N = 100	B_4C (2.62 nm)	0.73	126.3	0.11
La/B_4C Aperiodic	d = 4.76nm $\Gamma = 0.45$ N = 100	B_4C (1.8 nm) La (6.5 nm) B_4C (2.62 nm)	0.70	335.7	0.11

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3. Results and discussion

Among all the possible structures designs reported in Table 1, at the beginning of this research we have decided to realize, for the first time, a Pd/B_4C multilayer for a soft-x ray application, which reflectance performances comply not only our application requirements, but in principle could have been comparable to the experimental ones of La/B_4C. Moreover, the use of Pd as a capping layer could be of interest to verify its stability with respect to the B_4C one, which has been reported to be not fully stable in air [9, 16]. A prototype of the capped Pd/B_4C multilayer described in Table 1 has been deposited at Reflective X-ray Optics LLC (New York, USA), by DC magnetron sputtering onto a polished Si(100) substrate measuring 16 mm x 16 mm. The EUV reflectance of this coating was measured three weeks after deposition at the Bending magnet for Emission Absorption and Reflectivity (BEAR) beamline at ELETTRA Synchrotron (Trieste, Italy). The reflectance was measured over the 5-25 nm spectral range at a 45° incidence angle in order to quantify both peak reflectance and FRR (Fig. 4a and 4b). In Fig. 4b is also reported the simulation of the periodic Pd/B_4C structure with period, Γ and interfaces roughness determined from the reflectance measurements fit as reported in Table 2. We find a peak reflectance at 6.67 nm of 42%. Such drop in reflectance with respect to theoretical expected value causes a diminishing of the FRR ratio, which has been experimentally determined to be ~332. Reflectance measurements in the range 5-8 nm that were repeated after a period of one year show no measurable variation relative to the initial measurements, suggesting good temporal stability (Fig. 5). During this time the sample was stored in a low vacuum atmosphere ($P \approx 10^{-3}$ mbar). A reflectance measurement at 10° incidence (near normal incidence) was also performed, driven by the potential utility of this coating to other applications; those results are shown in Fig. 6, where we measured a peak reflectance of 43% at a wavelength of 9.1 nm. The experimental reflectance curves obtained at both 10° and 45° incidence have been fitted with the same structural parameters reported in Table 2. The fits were carried out using IMD, assuming a polarization factor of 0.9, in order to approximate the polarization of the synchrotron beam used in the measurements.

Table 2. Multilayer Parameters Determined from Fits to Experimental Reflectance and FRR Measurements

Fit parameters	Capping-layer	R(6.67nm)	FRR
d = 4.78	Pd(1.82nm)	0.42	332.3
Γ = 0.46	B_4C(7.52nm)		
Roughness: 0.64nm	Pd(2.06nm)		
	B_4C(2.51nm)		

A comparison between the theoretical and experimental peak reflectance shows a relative difference of 26%, which can be explained by using an interface width of 0.64 nm in the simulation. Similar interface widths were obtained in the case of a Mo/B_4C multilayer, which was reported to show a 43% reduction in peak reflectance at normal incidence relative to the ideal theoretical case [9]. For La/B_4C multilayers, interface imperfections have been reported to cause a reduction in peak reflectance over than 40% [12] being in fact the peak theoretical value around 0.65 and the experimental one around 0.40 at 6.7 nm in normal incidence reflectance and un-polarized light; in order to reduce the interface imperfections to some extent, the use of a nitridation process at the interfaces during film deposition has been suggested [17]. In both these previously realized structures, the study and control of the status of the interface was addressed, and specifically in [9] the Mo/B_4C multilayer interface quality has been deeply studied. In case of Mo/B_4C multilayer, the best fit to the measured reflectance data was obtained with an interface roughness of about 0.5–0.6 nm per interface, therefore adopting similar values as those reported in Table 2 for the Pd/B_4C system. Nevertheless, the outcome data of specific analysis carried on to study the interface quality did not provide any supporting evidence that the interface roughness in Mo/B_4C is actually so

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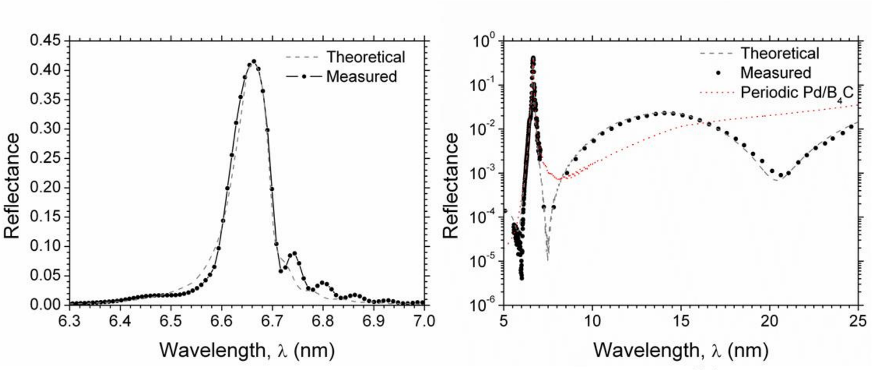
high. It is worth to mention that at this short wavelength (6.7 nm), the thickness of each layer is very small, and therefore inter-diffusion or compound formation occur over an extension comparable to the width of the layer itself; such effects are therefore dominant in the reflectance dropping. Grazing x-ray reflectance (XRR) measurement at Cu-Kα line ($\lambda = 0.154$ nm) has been also carried out on the aperiodic Pd/B_4C sample just after deposition. The experimental data and relative fit are reported in Fig. 7. The fitting was achieved with the same layer thicknesses distribution reported in Table 2, but it has been necessary to adopt a model in which the defined interfaces were replaced by a set of interlayers, characterized by an optical constant that smoothly varied from the Pd value to the B_4C one (i.e. graded interfaces); in the case of Pd over B_4C the width of such interface is 1 nm, while in the case of B_4C/Pd is of 0.3 nm. This demonstrate that the rms roughness value used in the fitting of the reflectance data is useful only to build a first simplify model, while the interfaces are in fact strongly inter-diffused with a diminishing of the optical contrast between spacer and absorber.



(a) (b)

Fig. 4. (a) Reflectance vs. wavelength of a Pd/B4C multilayer containing an aperiodic capping layer structure, measured at 45° incidence, compared with a simulation (dashed line) performed using the parameters reported in Table 2. (b) Reflectance of the same structure, measured over the range 5-25nm. Again, a simulation is shown (dashed line) performed using the structural parameters reported in Table 2. The simulation of the pure periodic structure, obtained using the period, Γ and interfaces roughness of Table 2, is also reported (dotted line) for comparison.

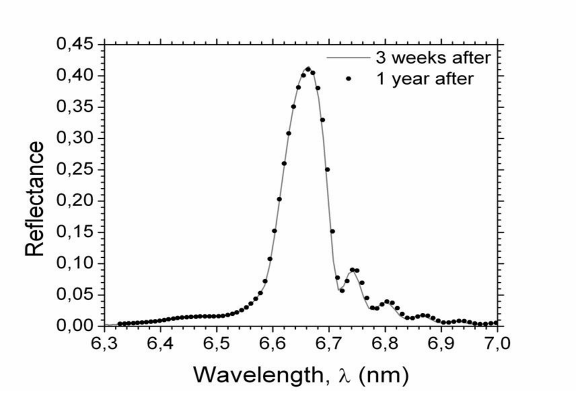


Fig. 5. Reflectance vs. wavelength of our prototype Pd/B_4C multilayer measured at 45° incidence angle just three weeks after deposition (continuous line) and one year after deposition (dot line) to verify its temporal stability.

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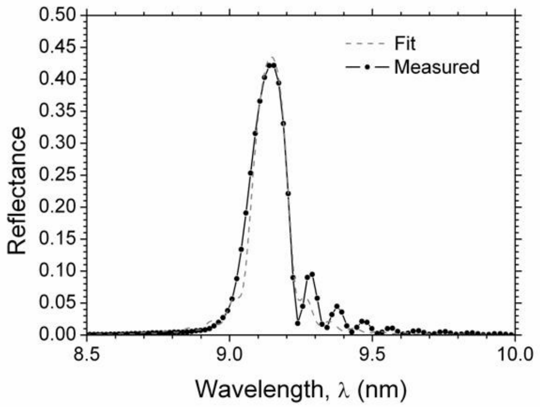

Fig. 6. Reflectance vs. wavelength of our prototype Pd/B_4C multilayer measured at 10° incidence; also shown is the simulation (dashed line) performed using the structural parameters reported in Table 2.

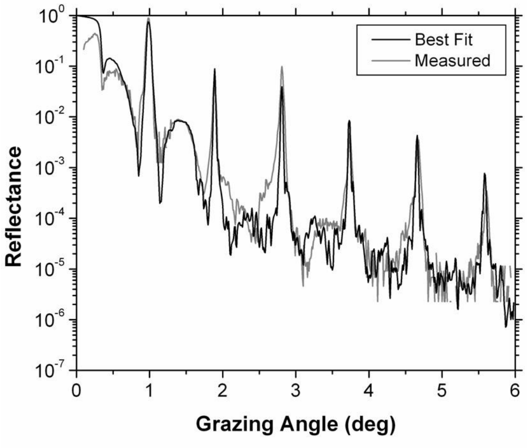

Fig. 7. XRR at λ = 0.154 nm (Cu-Kα line) of the Pd/B4C multilayer structure discussed in this work (black line). The figure shows also the best fit (grey line) obtained using a multilayer model with graded interfaces.

4. Conclusions

In summary, we have designed, fabricated and tested a Pd/B_4C multilayer coating comprising a conventional periodic stack and an aperiodic capping layer structure optimized for high reflectance at a wavelength of 6.67 nm. The theoretical reflectance peak for a linearly polarized light in S-configuration at 45° incidence angle is 58%, while experimental measurement provides a reflectance peak value of 42%; this drop must be attributed to a large intermixing at interfaces. Reflectance measurements repeated after a period of one year reveal no measurable performance changes, suggesting good temporal stability. Our experimental results indicate that this coating appears to be well-suited for a variety of emerging X-ray applications, including FEL beam transport optics, synchrotron and FEL beamline optics. In the specific case of FEL beam transport optics, our coating could be used with the FERMI@Elettra free electron laser to provide both high peak reflectance of the 3rd harmonic and good rejection of the fundamental in one configuration, or, alternatively, the coating could be used in a different configuration to provide high peak reflectance of just the fundamental. The fundamental harmonic rejection capability, expressed in term of FRR ratio for linear polarized light, has been experimentally determined to be 332; such a drop with

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respect to the theoretical value is related to the drop of experimental reflectance peak at 6.67 nm. To combine both the highest reflectivity of La/B_4C both the high rejection capability of the Pd/B_4C capping layer, it could be useful to make a further step toward a new solution, based on a combination of three material: a La/B_4C multilayer on top of which is deposited a Pd/B_4C capping layer. In this case a theoretical peak reflectance of 69% could be obtained with a calculated FRR of ~1500. The validation of any coating for FERMI@Elettra will required the analysis of thermal and irradiation stability, which has not experimentally addressed yet. Up to now, in fact, stability of multilayer under a FEL beam has been investigated mostly on Mo/Si based multilayer systems, using other facilities as FLASH in Hamburg [18]. Considering that FERMI@Elettra has different beam properties in term of pulse time duration and brilliance, it would be fundamental to carried out such type of experimental analysis on all different multilayer structures adopted in the beam transport system and experimental chambers. In the specific case of the Pd/B_4C, it is expected to be eventually used along a section in which the FERMI@Elettra beam is collimated, with an expected energy density of 4.5 mJ/cm^2; this energy density should be actually lower than the damage threshold determined for Mo/Si based multilayer, being such density ~45 mJ/cm^2 for a 10 fs single shot pulse at 13.5 nm. This experimental kind of tests will be object of a future research at FERMI@Elettra.

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