La/B_4C multilayer mirrors with an additional wavelength suppression

Philipp Naujok,[1,2,*] Sergiy Yulin,[2] Anna Bianco,[3] Nicola Mahne,[3] Norbert Kaiser,[2] and Andreas Tünnermann[2]

[1] *Friedrich-Schiller-Universität Jena, Institute of Applied Physics, Max-Wien-Platz 1, 07743 Jena, Germany*
[2] *Fraunhofer IOF, Albert-Einstein-Straße 7, 07745 Jena, Germany*
[3] *Sincrotrone Trieste S.c.p.A., Strada Statale 14 - km 163,5 in Area Science Park 34149, Trieste, Italy*
**philipp.naujok@iof.fraunhofer.de*

Abstract: In this paper, the authors report on La/B_4C multilayer mirrors designed for an incidence angle of 45° with both maximum reflectivity at a wavelength of 6.7 nm and reflectivity suppression at a wavelength of 20.1 nm. These mirrors were deposited for the EIS-TIMER at the FERMI@Elettra Free Electron Laser. The multilayer structure and optical properties were characterized using grazing incidence X-ray reflectometry with Cu-K_α radiation and EUV reflectometry in the spectral region of 6.5 - 21.0 nm. An anti-reflective coating designed at the wavelength of 20.1 nm had to be deposited on top of the high reflective La/B_4C multilayer mirror optimized at a wavelength of 6.7 nm. Measured reflectivities of 53.4% at the wavelength of 6.72 nm and 0.15% at the wavelength of 20.1 nm were simultaneously achieved. It is shown that the reflectivity loss at the wavelength of 6.7 nm due to the utilization of antireflective coating designed at the wavelength of 20.1 nm can be minimized up to 1.0%.

OCIS codes: (230.4170) Multilayers; (040.7480) X-rays, soft x-rays, extreme ultraviolet (EUV).

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1. Introduction

High reflective La/B multilayer mirrors for 6.x nm spectral region are of great general interest for next generation lithography systems [1–3], as well as for a specific application related to the development of the EIS-TIMER beamline [4] at the FERMI free electron laser (FEL) [5]. The TIMER project [4] aims to develop a time resolved instrument capable of extending transient grating (TG) methods in the EUV/soft x-ray spectral range. As compared with optical TG, the larger wave vector of VUV/soft x-ray photons will allow to investigate e.g. collective atomic dynamics in the 0.1-1 nm-1 wave vector range. Such a range, presently inaccessible by conventional methods, is of the highest relevance for the study of dynamics in disordered systems and nanostructures [4].

In a basic TG experiment two pulses (pump) interfere on the sample producing a spatial periodic variation of the material properties. A second beam (probe), possibly of different wavelength, impinges on the induced grating at the Bragg angle, thus yielding to a diffracted beam. The intensity of the diffracted radiation is thus recorded as a function of the time delay between the pump and probe pulses, and carries out information on the investigated material. An essential prerequisite to carry out such a kind of experiments, that are based on third-order coherent non-linear processes termed four-wave-mixing, is the high degree of longitudinal (i.e. temporal) coherence of the radiation [6]. The seeding scheme adopted at FERMI, based on the principle of high gain harmonic generation with fresh bunch injection technique [7], allows to exploit the EUV/soft x-ray spectral region maintaining the coherence properties of seed laser radiation, which are adequate for the proposed experiments [6]. The basic idea of TIMER experiments is to use the first and third harmonics of the FEL radiation as the pump and probe pulses, respectively. The FEL beam will be split into two parts and the third harmonic (probe) will be isolated and delayed using a set of 4 suitable multilayer mirrors working at an angle of incidence (AOI) of 45°. To achieve maximum performances, the optical system must have enhanced reflectivity of the third harmonic while suppressing the fundamental wavelength as much as possible.

In this work we describe the performances obtained with a new La/B_4C multilayer coating at $\lambda = 6.7$ nm, one of the wavelengths of interest for the TIMER project. Maximum calculated reflectivity of 78% at wavelengths higher than 6.5 nm can be achieved by La/B

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multilayer mirrors mainly due to low absorption of boron near its K absorption edge ($\lambda \approx 6.5$ nm). The first successful attempt to deposit coatings satisfying the requirements of the TIMER project at 6.7nm was made in 2012 using Pd/B_4C multilayer mirrors [8]. It was shown that reflectivities of 42% and 0.13% at the corresponding wavelengths of 6.7 nm and 20 nm can be experimentally achieved by a combination of high-reflective Pd/B_4C multilayer mirror designed at $\lambda = 6.7$ nm and an antireflective (AR) coating designed at $\lambda = 20$ nm. The new La/B_4C multilayer coating, presented in this paper, shows better performances compared to the previously used Pd/B_4C.

2. Experiment

The La/B_4C multilayer mirrors were deposited using a commercial DC magnetron sputtering system "Nessy-3" designed by Leybold Optics GmbH. Further information can be found in [9]. For research purposes, all La/B_4C multilayers were grown onto super polished Si (100) substrates (RMS ~0.15 nm), even though quartz-substrates with the same surface roughness have been used for mirrors applied at the FEL. According to the Bragg equation [10], the bilayer thickness (H) of the La/B_4C multilayer coating designed at AOI = 45° should be approximately 4.8 nm for the resonant reflection of s-polarized light with $\lambda = 6.7$ nm. Assuming an optimal thickness ratio ($\Gamma = d_{La}/H$) of about 0.4, the single layer thicknesses of lanthanum (d_{La}) and boron carbide (d_{B4C}) in the La/B_4C multilayer stack should be 1.9 nm and 2.9 nm, respectively. To reach theoretical reflectivity of 70% at $\lambda = 6.7$ nm, 120 La/B_4C bilayers were deposited for all studied multilayers. A B_4C-capping layer with fixed thickness of 3.0 nm was used to prevent oxidation of the La/B_4C multilayer stack.

Grazing incidence X-ray reflectometry (GIXR) with Cu-K_α radiation ($\lambda \approx 0.154$ nm) was used for multilayer characterization. Crystalline structure of La- and B_4C-layers was investigated by conventional X-ray diffraction (XRD). The final test of optical performance was carried out at PTB, Berlin [11] in two different EUV spectral regions of 6.5 … 6.9 nm and 18 … 22 nm at a fixed incidence angle of 45 degrees.

3. Results

To achieve an anti-reflective effect at wavelength $3\lambda \approx 20.1$ nm ($R_{20.1} < 0.3\%$), an AR-interference coating has to be added on top of the high-reflective La/B_4C multilayer mirror designed at the wavelength of 6.7 nm. Using *Film Wizard* by *SCI* [12], the designs of two La/B_4C multilayer mirrors with and without AR-interference coating were optimized, where Henke's optical constants [13] and La and B_4C bulk material densities were used. Special attention was paid on the AR-coating, because many different designs might be possible. Besides its AR-effect at the fundamental wavelength of 20.1 nm, the AR-coating reduces the reflection at $\lambda = 6.7$ nm mainly due to additional absorption in these layers. Hence it appears that the AR-design optimization should simultaneously meet two requirements: the highest possible reflection at the wavelength of 6.7 nm and minimizing the reflection below 0.3% at $\lambda = 20.1$ nm. La and B_4C are well suited to form the AR-coating due to their low absorption at $\lambda = 6.7$ nm. Even though other materials could provide better AR-effects, our calculations showed that this would lead to higher losses at $\lambda = 6.7$ nm. Besides this, problems of material comparability of the AR-coating and the periodic multilayer stack are avoided, if only La and B_4C are used in the multilayer design.

Figure 1 shows the calculated optical performances of different optimized La/B_4C AR-coatings, where the number of layers in the AR-coating is varied. It should be noted, that all designs have to end up with a 3 nm thick B_4C layer to prevent oxidation of the multilayer stack. As it can be seen, at least three layers are required to achieve a sufficient AR-effect of $R_{20.1} < 0.3\%$. According to our calculations, the application of more layers in the AR-coating would not enhance the AR-effect noticeable, but reflection losses at $\lambda = 6.7$ nm would grow further.

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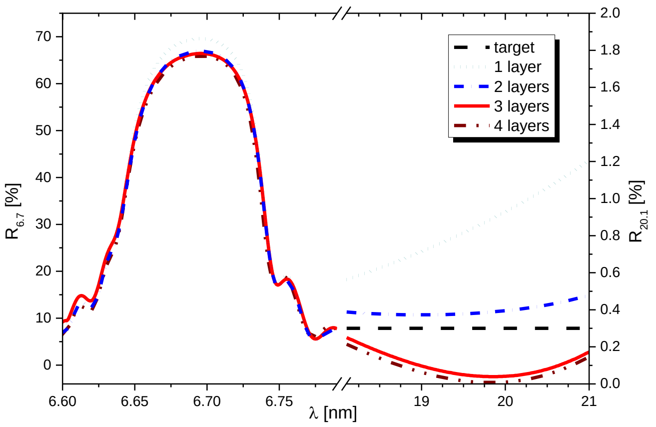

Fig. 1. Evolution of the reflective properties of La/B_4C multilayers with different numbers of layers in the AR-coating. To achieve a reflection of less than 0.3% at the wavelength of 20.1 nm (marked as a continuous line), at least three layers have to be used in an optimized AR-coating.

Table 1 summarizes the optimized theoretical designs and optical performances of both studied mirrors. In addition, the most important previous results from Corso et al. [8] are shown. Besides the calculated reflectivities at 6.7 nm ($R_{6.7}$) and 20.1 nm ($R_{20.1}$), the suppression ratio S = $R_{6.7}/R_{20.1}$ is also included in Table 1 for further discussion.

Table 1. Calculated designs and optical properties of multilayer mirrors with and without AR-coatings

Mirror	**H [nm]**	**t_{cap1} (B_4C) [nm]**	**t_{cap2} (La) [nm]**	**t_{cap3} (B_4C) [nm]**	**$R_{6.7}$ [%]**	**$R_{20.1}$ [%]**	**S = $R_{6.7}/R_{20.1}$**
La/B_4C	4.80	3.0	-	-	69.7	0.90	77
La/B_4C + AR	4.80	11.5	3.7	3.0	67.0	0.04	1675
Pd/B_4C + AR [8]	4.76	see ref [8].			58	0.05	1060

Compared to the Pd/B_4C mirrors presented by Corso et al. in [8], the calculated peak reflectivities of the La/B_4C multilayer mirrors at λ = 6.7 nm are about 10% higher. Additionally, one can see that the reflectivity suppression ratio S can be enhanced up to 1675 due to the application of La/B_4C multilayer combination.

The measured GIXR-spectra of both studied mirrors together with corresponding best fits are shown in Fig. 2. The layer thicknesses extracted from best fittings are shown in Table 2. As can be seen in Table 2, all film thicknesses are close to targeted values (Table 1). The coinciding Bragg peaks in both GIXR measurements indicate that the high reflective La/B_4C

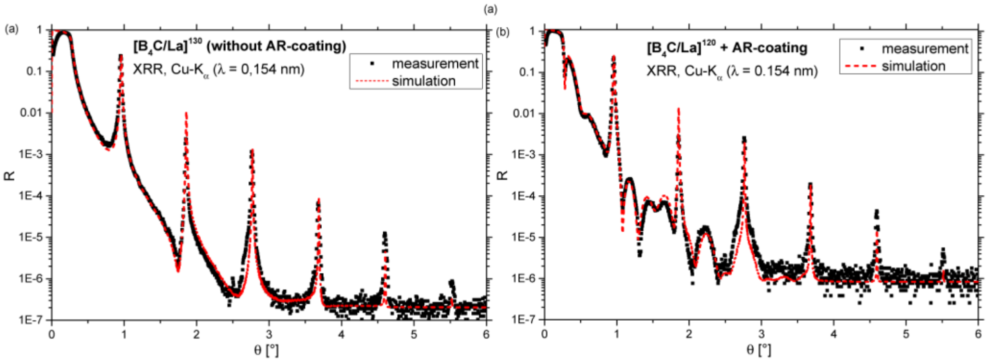

Fig. 2. Measured (dots) and fitted (dashed line) GIXR curves from La/B_4C multilayers without (a) and with AR-coating (b). The conspicuous oscillations between the Bragg peaks in (b) are caused by the AR-coating.

Table 2. Comparison of layer thicknesses extracted from GIXR simulations and targeted values

Mirror		H [nm]	t_{cap1} (B_4C) [nm]	t_{cap2} (La) [nm]	t_{cap3} (B_4C) [nm]
La/B_4C	cal.	4.80	3.0	-	-
	exp.	4.81	3.6	-	-
La/B_4C + AR	cal.	4.80	11.5	3.7	3.0
	exp.	4.81	11.7	3.7	3.3

multilayer stacks designed at λ = 6.7 nm of both studied mirrors are similar, i.e. having the same period, thickness ratio, and interface roughnesses (σ). In addition to film thicknesses, which can be measured quite accurately, interface roughnesses σ on both interfaces (La-on-B_4C and B_4C-on-La) were extracted from GIXR simulations. Even though the extracted values σ may not fully describe the real interface structure (interface roughness and diffusion intermixing), a first hint about the interface quality can still be obtained. According to our best GIXR simulations, the B_4C on La interfaces seem rougher ($\sigma_{B4C\text{-on-La}}$ = 0.9 nm) than the La on B_4C interfaces ($\sigma_{La\text{-on-B4C}}$ = 0.4 nm), as it has also been reported by others [1,14,15].

This asymmetry in interface roughnesses between La on B_4C and B_4C on La interfaces can also be found within the AR-capping, where layer thicknesses and interface roughnesses can be simulated more precisely. Figure 2(b) shows the obvious additional maxima between the Bragg-peaks on the GIXR curve of the La/B_4C multilayer mirror with AR-coating. These maxima are caused by the three top-layers forming the AR-coating. The simulation results (of the three top-layers) indicate that an increase in the thickness of La layers from 1.9 nm in the periodic La/B_4C multilayer stack to 3.7 nm in the AR-coating results in a development of interface roughness of B_4C on La interfaces from 0.9 nm to 1.1 nm. In contrast, $\sigma_{La\text{-on-B4C}}$ = 0.4 nm was found for the 11.7 nm thick B_4C film in the AR-capping and in the multilayer stack. Hence, the interface roughness of La on B_4C interfaces seems to be independent from the thickness of B_4C-layers, whereas the interface roughness of B_4C on La interfaces is developing by increasing La film thickness. A similar phenomenon was found in the conventional Mo/Si multilayer system [16] and could be explained by various levels of crystallinity of layers with different thicknesses. XRD of the La/B_4C mirror without AR-capping revealed a broad peak at 2θ ≈27° (Δ2θ ≈10°), matching with La (002) in a hexagonal crystalline system [17]. A medium size of the La-crystallites of about 1.5 nm in La layers with a thickness of 2.0 nm was calculated by Scherrer's formula [18]. Probably even larger crystallites have formed in a 3.7 nm thick La layer in the AR-capping, causing an even higher surface roughness there. Tsafari et al. [1] also investigated the crystalline structure of La/B_4C

multilayers with different periods and they reported quite similar results. So, the interface roughness of B_4C on La interfaces is critical for optical performance of La/B_4C-multilayer mirrors and strongly depends on the layer thickness and corresponding crystalline state of La layers.

The measured EUV reflectivities (EUVR) with corresponding data for both studied La/B_4C multilayer mirrors are shown in Fig. 3 and Table 3. Experimental peak reflectivities of more than 50% at the wavelength of 6.72 nm were achieved on both multilayer mirrors. Compared to Pd/B_4C multilayer mirrors having $R_{6.7}$ = 42% [8], measured reflectivities of La/B_4C multilayer mirrors at a wavelength of ~6.72 nm are more than 10% higher. Assuming that four consecutive mirrors are used at the FEL, an increase of the intensity of the probe beam of about 2.5 times can be achieved. Note, that the comparatively thick three-layer AR-coating with a total thickness of nearly 20 nm results in a reflectivity loss by only 1% at a wavelength of 6.72 nm. On the other hand, a considerable AR effect at 20.1 nm can be observed. For the proposed application, it is necessary to reduce reflectivity at 20.1 nm below 0.3% and to have a suppression ratio better than 100. In our case, $R_{20.1}$ = 0.15% and thereby S = 356 was achieved with an AR-coating consisting of only three layers, whereas Corso et al. [8] used four layers and obtained $R_{20.1}$ = 0.13% and S = 332. It should be mentioned, that the calculated suppression of the AR-coating almost five times exceeds our experimental value. This can be explained by surface oxidation of the top B_4C layer. Even though B_4C is not a highly oxidative material, a thin oxide layer may be formed at the surface. This causes both an additional radiation absorption and layer thickness changes, which increase the reflection at λ = 20.1 nm and lower the reflection suppression. According to our simulation a transformation of the B_4C top layer to a mixture of B_4C and B_2O_3 can explained this phenomenon.

Without an additional AR-coating, a suppression ratio of S = 124 was measured, which is already a value higher than the theoretical one. This disagreement between calculated and experimental data can also be explained by surface oxidation of the B_4C capping layer with a corresponding reflectivity drop to 0.44% at the wavelength of 20.1 nm (instead of calculated $R_{20.1}$ = 0.9%). The measured EUVR in Fig. 3 were modeled by a simple replacement of the top B_4C layer with an initial thickness of 3.6 nm by a combination of a B_4C and B_2O_3 layer with thicknesses of 1.0 nm and 2.9 nm, respectively.

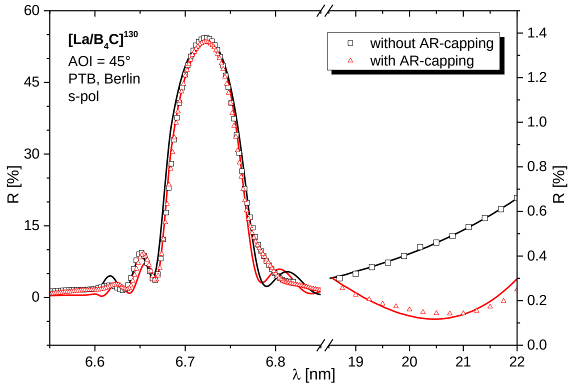

Fig. 3. Measured EUV reflectivities (points) and best fits (lines) of La/B_4C multilayer mirrors with (triangles) and without (squares) AR-coating designed at the wavelength of 20.1 nm.

Table 3. Comparison of calculated and measured EUVR data

Sample		$R_{6.7}$ [%]	$R_{20.1}$ [%]	$S = R_{6.7}/R_{20.1}$
La/B_4C	cal.	69.7	0.90	77
	exp.	54.4	0.44	124
La/B_4C + AR	cal.	67.0	0.04	1675
	exp.	53.4	0.15	356

EUVR simulations were also performed to model interdiffusion processes at the interfaces. As described by Tsarfati et al. [1,15], the interdiffusion processes during multilayer deposition result in a formation of amorphous LaB_6 compounds at the interfaces. Due to this reason, a four layer model of La/LaB_6/B_4C/LaB_6 with perfect smooth interfaces was used in our EUVR simulations. According to the results of our best fittings, the LaB_6 thicknesses on the different interfaces (La on B_4C and B_4C on La) are not the same. LaB_6 interlayers with thicknesses of 0.7 nm and 1.0 nm were found at the La on B_4C and B_4C on La interfaces, respectively. The thicknesses of pure B_4C and La layers were 2.5 nm and 0.6 nm. These results are in good agreement with the GIXR fitting results. Again, the B_4C on La interfaces are broader than the La on B_4C ones, exactly as it was shown in GIXR simulations.

It should be finally noted that the total number of layers contributing to the constructive interference of reflection can be found through additional Kiessing oscillations [19] besides the main Bragg peak. The best EUVR simulations for both La/B_4C multilayer mirrors were achieved with 90 bilayers, although 120 bilayers have been deposited. This marked disagreement was previously described in [14] and can be explained by both a slight aperiodicity in multilayer stack and enhanced radiation absorption inside of real La/B_4C multilayer mirrors. Both of these multilayer imperfections can be minimized due to optimization of multilayer design and deposition process.

4. Summary

Two La/B_4C multilayer mirrors designed at the wavelength of 6.7 nm with and without additional AR-coating optimized at the wavelength of 20.1 nm were compared in terms of optical properties and interface performances. The highest peak reflectivity of 54.4% at the wavelength of 6.72 nm was measured on a conventional periodic La/B_4C multilayer mirror without AR-coating. At the same time, it was shown that a combination of this multilayer mirror with an additional AR-coating provides both a high suppression ratio of 356 and peak reflectivity of 53.4% at the wavelength of 6.7 nm. It was experimentally proven that transition from Pd/B_4C to La/B_4C multilayer mirrors results in a considerable peak reflectivity improvement up to 12% at the wavelength of 6.7 nm. Due to optimization of multilayer design, AR-capping design and deposition process of La/B_4C multilayer with AR-coating, the reflectivity loss at the wavelength of 6.7 nm was minimized up to 1.0%.

The thickness asymmetry of different interfaces (La on B_4C and B_4C on La) in real La/B_4C multilayer mirrors was simultaneously proven by both GIXR and EUVR fittings. The asymmetry of the different interfaces can be explained by specific growth features of thin La and B_4C films.

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