Development of aperiodic multilayer mirrors operated at W-Lbeta line for plasma diagnostics application

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Abstract

Multilayer interference mirrors play a pivotal role in spectroscopic diagnostic systems, which probe electron temperature and density during inertial confinement fusion processes. In this study, aperiodic Mo/B4C multilayer mirrors of varied thicknesses were investigated for X-ray plasma diagnostics at the 9.67-keV W-Lbeta line. The thickness distribution of the aperiodic multilayers was designed using the 1st Bragg diffraction condition and then optimized through a simplex algorithm to realize a narrow bandwidth and consistent spectral response. To enhance spectral accuracy, further refinements were undertaken by matching the grazing incidence X-ray reflectivity data with actual structural parameters. X-ray reflectivity measurements from the SSRF synchrotron radiation facility on the optimized sample showed a reflectivity of 29.7% $±$2.6%, flat-band range of 1.3 keV, and bandwidth of 1.7 keV, making it suitable for high-temperature plasma diagnostics. The study explored the potential of predicting the 9.67 keV reflectivity spectrum using the fitting data from the Grazing incidence X-ray reflectivity (GIXRR) curves at 8.05 keV. Additionally, the short-term thermal stability of an aperiodic multilayer was assessed using temperature-dependent in situ X-ray measurements. Shifts in the reflectivity spectrum during annealing were attributed to interdiffusion and interfacial relaxation. The research team recommends the aperiodic Mo/B4C multilayer mirror for operations below 300 ℃.

Full Text

Preamble

Development of Aperiodic Multilayer Mirrors Operated at W-Lβ Line for Plasma Diagnostics Application

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Keywords: X-ray multilayer mirror; Aperiodic multilayer; Flat response; Sputtering deposition; Synchrotron radiation

Abstract

Multilayer interference mirrors play a pivotal role in spectroscopic diagnostic systems that probe electron temperature and density during inertial confinement fusion processes. In this study, we investigated aperiodic Mo/B4C multilayer mirrors with tailored thickness distributions for X-ray plasma diagnostics at the 9.67-keV W-Lβ line. The thickness distribution was initially designed using the first-order Bragg diffraction condition and subsequently optimized through a simplex algorithm to achieve a narrow bandwidth and consistent spectral response. To enhance spectral accuracy, further refinements were undertaken by matching grazing incidence X-ray reflectivity data with actual structural parameters.

X-ray reflectivity measurements from the SSRF synchrotron radiation facility on the optimized sample demonstrated a reflectivity of 29.7% $\pm$ 2.6%, a flat-band range of 1.3 keV, and a bandwidth of 1.7 keV, making it suitable for high-temperature plasma diagnostics. The study explored the feasibility of predicting the 9.67-keV reflectivity spectrum using fitting data from grazing incidence X-ray reflectivity (GIXRR) curves at 8.05 keV. Additionally, the short-term thermal stability of the aperiodic multilayer was assessed through temperature-dependent in situ X-ray measurements. Shifts in the reflectivity spectrum during annealing were attributed to interdiffusion and interfacial relaxation. Based on these results, we recommend operating the aperiodic Mo/B4C multilayer mirror

below 300 °C.

1. Introduction

Over recent decades, high-temperature plasma diagnostics has gained significant attention due to its critical importance in laser inertial confinement fusion (ICF). X-ray optical diagnostics analyzes the spectrum and angular distribution of characteristic emission lines from metals such as Fe, Cu, and W at elevated temperatures, enabling examination of hotspot electron temperature and density [?]. High-resolution X-ray spectroscopy tools are vital for distinguishing signals amidst intense background radiation in extreme heat environments. However, practical applications demand that X-ray devices possess several key optical properties, including sufficient bandwidth to accommodate variable spectra and changes in emission line breadth and position under extreme conditions [?]. A consistent spectral response is crucial for accurately capturing and imaging desired emission lines, and the device's spectral performance should remain stable under short-term and localized high temperatures, particularly during intense X-ray exposures. We selected the W-Lβ emission line as the focal energy point for this analysis because higher photon energy X-rays from the hotspot diminish interference from the self-emission of ablating materials.

Multilayer interference mirrors, constructed from alternating layers of materials with high and low refractive indices, are fundamental components in X-ray and EUV spectral regions [?]. Periodic multilayers maintain a constant bilayer thickness that adheres to Bragg diffraction conditions, offering high reflectivity in a narrow spectral range through constructive interference between bilayers [?]. Conversely, aperiodic structures exhibit variable layer thicknesses that enable phase modulation across a broader spectrum. Such designs can achieve flat reflectance by continuously optimizing the thickness distribution [?]. However, when pursuing this flat spectral response, the reflectivity of Kiessig oscillations—or side lobes—might increase due to random changes in each bilayer's thickness ratio [?, ?]. Suppressing these side lobes during optimization is essential for maintaining a high signal-to-noise ratio (SNR) in plasma diagnostics. A design and preparation approach for aperiodic multilayers that achieve a flat spectral response around the 8.05-keV Cu-Kα line has been presented previously [?], and this structure was deemed suitable for extracting the 9.67-keV W-Lβ line. Yet, the energy-dependent reflectivity prediction and optimization method in Ref. [?] warrants validation, especially since standard thin-film laboratories lack a direct X-ray source for the 9.67-keV W-Lβ line.

Predictions made using 8.05-keV data might introduce inaccuracies due to variations in interfacial structures, bilayer thicknesses, and densities between different X-ray energies (8.05 keV and higher) [?, ?]. Errors from 8.05-keV characterizations could also result in significant deviations in predictions. This paper explores the viability of employing fitted structural parameters from 8.05-keV X-ray measurements to predict optical performance at 9.67 keV.

We engineered aperiodic multilayers to realize a reflectivity plateau with confined spectral bandwidth at a grazing angle of 0.8°. We selected the Mo/B4C material combination over commonly used W-based multilayers to circumvent pronounced absorption at the W L-edge. The thickness distribution for the aperiodic structure was formulated using the first-order Bragg diffraction condition and refined with the simplex algorithm. Grazing incidence X-ray reflectivity (GIXRR) measurements ascertained the precise thickness distributions and interfacial widths of fabricated samples. By harnessing structural data from GIXRR curve fits, we projected the reflectivity spectrum while accounting for specified roughness and density of the aperiodic multilayers. We subsequently assessed the energy-dependent X-ray reflectivity of the refined multilayer mirror to validate the proposed prediction approach and analyzed the aperiodic multilayer's optical performance and thermal stability through in situ X-ray evaluations at varying annealing temperatures.

2. Methods and Experiments

Designing aperiodic multilayers requires determining the optimal layer thickness distribution for the stack, which is achieved by starting with a suitable initial array of layer thicknesses and refining it through numerical optimization. Various methods can inform the initial array selection, including periodic sequences [?], block-periodic methods [?], depth-graded sequences [?], or specialized algorithms [?, ?]. Numerical optimization techniques encompass genetic algorithms, degenerate algorithms, and simplex algorithms. Given the stringent requirements for the full width at half maximum (FWHM) of reflectivity curves and constraints on the total number of bilayers, this study employed an initial array based on the periodic sequence that satisfies the first-order Bragg diffraction condition, which was further optimized using the simplex algorithm for the aperiodic multilayer design.

Optimization hinges on minimizing a merit function that represents the disparity between desired reflectivity R_0 and calculated reflectivity R [?]. In this research, the merit function MF, which encompasses both range flattening and side lobe suppression, is articulated in equation (1). The first term focuses on achieving a uniform reflectivity plateau, where E_i denotes a discrete photon energy point within the response domain and m signifies the total number of discrete energy points assessed within that domain. The subsequent term suppresses side lobes beyond the flat-band domain, where E_j represents a discrete photon energy point outside the response domain and n denotes the aggregate of discrete energy points beyond the said domain. Within this energy spectrum, the desired reflectivity R_0 is set at zero. The coefficient a balances the importance between central flattening and side lobe suppression.

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The aperiodic multilayers comprised a total of 50 layers, representing a balance between attaining high theoretical reflectivity and managing cumulative effects of interfacial width. During optimization, all layer thicknesses—defined in an array—served as variables to be optimized within a range of 1.7–4.5 nm. Initially, each interface width was considered zero and material density was based on bulk values. For further refinement, specific roughness and density values were informed by experimental data. Fig. 1 [Figure 1: see original paper] illustrates the computed reflectivity curves for both periodic and aperiodic Mo/B4C multilayers, each having 50 layers, at a grazing angle of 0.8°. The spectral center for each multilayer mirror aligned at 9.67 keV, consistent with the targeted energy point, and the reflectivity curve of the aperiodic multilayer showcased the sought-after reflectivity plateau with an FWHM no greater than triple that of the periodic multilayer. Computational outcomes revealed a designed reflectivity of $(36.9\% \pm 0.1\%)$ within a flat-band span of 1.2 keV, with a target FWHM established at 1.5 keV to encompass the variable spectral line and provide a suitable field of view.

We fabricated multilayer samples on 20×20 mm^2 ultra-smooth silicon substrates using direct-current (DC) magnetron sputtering. These substrates exhibited a surface roughness of approximately 0.3 nm. The system was maintained at a base pressure below 2×10^{-4} Pa, while the working pressure with high-purity Ar sputtering gas was kept at 1.5×10^{-1} Pa. The sputtering powers were set at 40 W for the B4C target and 20 W for the Mo target. The thickness distribution of the sputtered aperiodic multilayers mirrored that of the computational models. To calibrate the deposition rate for each target, we deposited periodic Mo/B4C multilayers with varying bilayer thicknesses, each containing a consistent count of 10 bilayers. These were categorized into two sets: samples 1–4 exhibited varying B4C thicknesses with Mo layers held at approximately 3.8 nm, and samples A–D exhibited variable Mo thicknesses with a consistent B4C layer thickness near 3.0 nm. We determined the thicknesses and interfacial widths of these periodic Mo/B4C multilayers using GIXRR measurements. With a stable magnetron sputtering apparatus, the deposition rate of a single target remains effectively consistent, making layer thickness linearly correlated with deposition duration. We meticulously monitored the deposition process to ensure sub-angstrom precision.

GIXRR measurements, utilizing the $\omega - 2\theta$ specular geometry, were conducted using a Panalytical Empyrean XRD diffractometer equipped with a high-quality Cu-Kα line X-ray source at 8.05 keV. We performed individual fittings five times for each GIXRR curve to yield average fitting parameters. Energy-dependent reflectivity evaluations were carried out at the BL09B beamline of the SSRF, spanning a photon energy range of 8.5 keV to 11.5 keV. The BL09B beamline employs a sequence of optical devices, including a double crystal monochromator (DCM), compound refractive lens (CRL), and toroidal mirror (TM), delivering high-resolution X-rays that cover the 5–40 keV range with a minimal unfocused spot measuring less than 300 μm (V) $\times$ 500 μm (H) [**?**, **?**]. During experiments, we gauged reflectivity intensity using a four-circle diffractometer paired with a slit and PIN diode throughout the DCM energy scan, with reflectivity determined by the ratio of the reflective beam's measured current to the direct beam's current. We facilitated in situ X-ray reflectivity assessments at varying temperatures using a computer-controlled hot stage located outside the experimental hutch. This hot stage was situated inside a sealed chamber equipped with two gas conduits for generating a gas flow environment, with pure helium serving as the shielding gas during thermal procedures. In the course of the experiment, the multilayer sample atop the heat stage underwent continuous annealing at intervals of 100 °C, 200 °C, 300 °C, and 400 °C, each for 10 min. The temperature ramp rates for both heating and cooling were set at 100 °C/min, and we captured energy-dependent X-ray reflectivity curves at each designated temperature.

3. Experimental Results and Discussion

Fig. 2 Figure 2: see original paper displays the GIXRR curves for samples 1–4, while Fig. 2(b) depicts those for samples A–D. The specular reflectivity curves feature numerous distinct Bragg peaks, suggesting the formation of coherent and continuous interfaces by the periodic Mo/B4C multilayers. We determined structural parameters of the periodic Mo/B4C samples—including layer thickness, density, and interfacial width—using Parratt's standard formula paired with the Névot–Croce factor [**?**, **?**], facilitated by a genetic algorithm. The derived data are compiled in Table 1 . The fitted thicknesses for the Mo and B4C layers correlate linearly with deposition time, exhibiting a coefficient of determination surpassing 0.999. Nonetheless, the fitted density does not maintain a linear relationship as Mo or B4C thickness increases, likely due to Mo crystal growth and subsequent interdiffusion between neighboring layers [**?**]. For samples A–D, the fitted interfacial width of the B4C-on-Mo interface remains nearly consistent with increasing Mo layer thickness, probably due to minimal bombardment from settling B4C clusters. In contrast, samples 1–4 reveal unpredictable variations in the Mo-on-B4C interface, attributed to the elevated kinetic and surface energies of settling Mo atoms. For spectral analysis of aperiodic multilayers, averages of the fitted parameters are pivotal, notably interfacial width means of $\sigma_{\mathrm{Mo}} = 0.30$ nm and $\sigma_{\mathrm{B4C}} = 0.45$ nm, as well as density averages of $\rho_{\mathrm{Mo}} = 0.93$ and $\rho_{\mathrm{B4C}} = 1$.

We analyzed the GIXRR curves of the aperiodic Mo/B4C multilayer to discern the actual thickness distribution. Fig. 3(a) depicts the GIXRR curve as blue dots, while the predicted reflectivity curve is illustrated by a black dotted line. Instead of using direct light intensity, we normalized the GIXRR curve to the peak of total external reflection to eliminate horizontal size effects stemming from a line spot. The designed aperiodic multilayer, intended for a flat response around 9.67 keV, displayed an intrinsic figure error in the flat-band region at 8.05 keV in the calculated curve. The GIXRR measurement reveals a response range from 0.87° to 1.02°, with an observed average reflectivity of 28.0%, slightly lower than the predicted 32.4%. Although the angular positions of the response center and side lobe peaks in Fig. 3(a) align with theoretical expectations—suggesting a consistent thickness distribution—a noticeable dip in the reflectivity plateau, with a peak deviation of 8.6%, indicates potential errors in effective thickness and interfacial impacts. To probe the origins of this spectral dip, we fitted the GIXRR curve to determine the thickness distribution. In this fitting process, we held the interfacial width constant to sidestep further discrepancies in the fitted thickness distribution, basing this constant value on the average from the fitted data of periodic Mo/B4C multilayers.

In Fig. 3 Figure 3: see original paper, the fitting curve closely aligns with the GIXRR measurements. Meanwhile, Fig. 3(b) displays the average of five repeated fitting data points, represented by red square dots, with the desired thickness distribution illustrated by blue round dots. To offer clearer insight into fitting variance, we included an error bar for the thickness sequence across more than 50 variables. Calculations indicate that the maximum deviation in layer thicknesses does not exceed 0.25 nm, with an average random deviation of 0.08 nm—approximately a 3.0% relative deviation. However, for other Mo/B4C aperiodic samples prepared using consistent parameters, GIXRR measurements consistently showed the same spectral characteristics, suggesting that the spectral depression cannot be solely attributed to random deposition errors. We also assessed the effect of interfacial width, estimating it between 0.3–1.0 nm to account for potential peak roughness accumulation. Although this contributes to pronounced attenuation of reflectivity, it does not appear to correlate directly with a noticeable error in the reflectivity profile.

The observed depression in the GIXRR curve may result from interdiffusion between adjacent Mo and B4C layers, leading to a phase error in constructive interference. This phase mismatch is evident in the fitting density: in the periodic sample data, Mo layer density rises from 90% to 96% as Mo thickness increases from 1.78 nm to 3.11 nm, while B4C density shows inconsistent variation, ranging from 90.1% to 110% with increasing B4C thickness. In this context, density deviation can be associated with noticeable figure error in the reflectivity profile, mirroring the effects of random thickness deviation.

Given that the stable compound at the Mo/B4C multilayer interfaces has not been uniquely identified among the contenders—MoB, MoB2, and Mo2C—there is no standard value for the optical constants of the interdiffusion zones to reference. Consequently, we utilized the fitting densities of the Mo and B4C layers to depict the additional phase effects of these interdiffusion layers. We finetuned the thickness distribution of the aperiodic Mo/B4C multilayer based on specified interfacial width and density to enhance spectral performance. Incorporating these fixed parameters as optimization constraints inevitably complicates achieving a stable solution. To strike a balance, we tweaked parameter a in the merit function MF to emphasize response flattening, thereby fostering higher low-angle side lobes in the reflectivity curves. The fixed parameters were drawn from the fitting data of periodic Mo/B4C samples: average interfacial widths of $\sigma_{\mathrm{Mo}} = 0.30$ nm and $\sigma_{\mathrm{B4C}} = 0.45$ nm, along with average densities of $\rho_{\mathrm{Mo}} = 0.93$ and $\rho_{\mathrm{B4C}} = 1$. We produced the revised design under identical preparation conditions as the original sample. Fig. 4 [Figure 4: see original paper] displays the GIXRR measurements of the enhanced sample, denoted by the red dotted line. Notably, the original depression was markedly mitigated, yielding a more consistent response within the angle range of 0.88–0.98°. However, the spectral response presented undulating variances and a noticeable gradient near its diminishing edge. This discrepancy in the GIXRR curve primarily stems from the fluctuating diffusion zone with expanding bilayer thickness, diverging from the static state assumed in the design. The low-angle side lobes span the angle range of 0.6–0.75°, resulting in diminished SNR for imaging diagnostic experiments. Fortunately, this spectral segment can be effortlessly isolated using basic optical system structures. With these optical specifications met for X-ray optical diagnostics, we subsequently analyzed the energy-dependent reflectivity curves for this sample series.

Fig. 5 [Figure 5: see original paper] presents the energy-dependent spectrum of the optimized sample, recorded at SSRF BL09B. The measured reflectivity profile shows an evident shift toward higher energy by approximately 300 eV compared with the design, largely due to incident angle error during measurement—a 300 eV shift equivalent to a minor angle error of 0.03° induced by the uneven sample stage surface. The full width spans from 9.3 keV to 10.6 keV, totaling 1.3 keV, while the FWHM of the reflectivity peak measures 1.7 keV. The average reflectivity on the plateau reads 29.7%, below the projected 36.9%, with an uncertainty of roughly 0.2%. Reflectivity fluctuation within the flat response was $±$2.6%. The curve derived from GIXRR measurement fitting data is depicted as a solid line in Fig. 5. We fixed structural and interfacial parameters in the design: average interfacial widths of $\sigma_{\mathrm{Mo}} = 0.30$ nm and $\sigma_{\mathrm{B4C}} = 0.45$ nm, and average densities of $\rho_{\mathrm{Mo}} = 0.93$ and $\rho_{\mathrm{B4C}} = 1$. The grazing angle was set at the actual angle of 0.77°. This calculated curve aligns well with experimental data, signifying that predicting energy-dependent X-ray reflectivity with GIXRR fitting data is feasible. Yet, the calculated reflectivity profile is not entirely accurate, especially for photon energies above 10 keV, possibly due to heightened sensitivity of high-energy X-rays to thin-layer structures. The relative discrepancies between predictive and measured curves are 4.4% for average reflectivity, 7.1% for flat-band width, 5.1% for reflectivity fluctuation, and 1.4% for FWHM.

Fig. 6 [Figure 6: see original paper] depicts the energy-dependent reflectivity curves of the Mo/B4C multilayer during in situ thermal processing, represented by hollow dots. The samples tested were created with identical thickness distribution and preparation parameters as the optimized design. The reflectivity curve's response range at room temperature (25 °C) aligns with calculated data, signifying that the grazing incident angle was precisely maintained at 0.8°. As annealing temperature increased, the bilayer thickness of the aperiodic multilayer exhibited overall growth, causing the flat-band center of the reflectivity curves to shift consistently toward the low-energy side. The annealing process also influenced average reflectivity within the response range: there was a notable increase when temperature rose from 25 °C to 200 °C, followed by a decrease at temperatures above 200 °C.

Several studies on periodic Mo/B4C multilayers have documented significant interdiffusion in layer thickness and interface evolution [?]. These studies highlighted that B4C thickness dictates dual paths of thickness evolution due to interdiffusion depth: period expansion for periods with thick B4C layers and period contraction for those with thin B4C layers [?]. The pivotal thickness for these evolution directions fluctuates between 1.5 nm and 2 nm, contingent upon annealing temperature and duration. Given this, the aperiodic multilayer's thickness distribution, which spans 1.7–4.5 nm, could exhibit dual evolution directions under identical temperature conditions as periodic counterparts. With brief annealing at lower temperatures, the diffusion length of surplus Mo atoms does not suffice to entirely replace the neighboring B4C layer, leading to formation of low-density compounds in interfacial regions and contributing to overall thickness increase. This period expansion also dominates thickness evolution at the elevated temperature of 400 °C. Nevertheless, in some bilayers, interdiffusion spanned almost the full extent of the B4C layer with thickness below 2 nm, inducing densification and period contraction. This contradictory evolution in these bilayers, set against an overall expanding thickness sequence, could introduce further phase match errors across the intended flat-band area. A discernible figure error became evident in the reflectivity curve when annealed at 400 °C. To gauge interface evolution, we illustrate curves derived from GIXRR measurement fitting data as red lines in Fig. 6. The simulated data reveals a decline of several angstroms in interfacial width at temperatures below 200 °C, a consequence of roughness relaxation during the thermal process. However, as temperatures continued increasing, interfacial width steadily expanded due to significant interlayer growth. Given that the central energy of the reflectivity plateau must remain around 9.67 keV, we advise that aperiodic Mo/B4C multilayers should function at temperatures not exceeding 300 °C.

Conclusion

This study introduced an aperiodic Mo/B4C multilayer mirror designed for operation at 9.67 keV (W-Lβ line), featuring a reflectivity plateau with narrow bandwidth. Initially, the thickness distribution was conceived via Bragg diffraction and later refined via a merit function paired with the simplex algorithm. The projected reflectivity curve reveals a design reflectivity of 36.9% alongside a flat-band width of 1.2 keV. Adhering to a calibrated deposition rate, we fabricated aperiodic Mo/B4C multilayers on ultra-smooth silicon substrates via DC magnetron sputtering. Fitting data suggests the mean deviation in thickness distribution was approximately 3.0%. We further optimized the thickness distribution considering specific roughness and density values to enhance spectral performance. The GIXRR curve for the enhanced sample demonstrated notable improvement in response consistency. The energy-dependent X-ray profile of this improved sample, captured at the SSRF, revealed an average reflectivity of 29.7% with a fluctuation margin of \$±\$2.6%. The flat-band width spans 1.3 keV from 9.3 keV to 10.6 keV, and the FWHM measures 1.7 keV—showing potential for high-temperature plasma diagnostic applications. The prediction curve derived from GIXRR fitting data aligns closely with experimental findings, barring a minor discrepancy in the region above 10 keV. Disparities between computed results and actual measurements remained under 10% for primary structural parameters, underscoring the viability of this method for X-ray spectrum prediction. Energy-dependent X-ray reflectivity assessments during annealing recorded a general shift of the flat-band response area toward the low-energy side, linked with prevalent period expansion. The observed transformations in thickness and interface can be attributed to interdiffusion and interface relaxation dynamics. This evaluation of thermal stability suggests an optimal operational temperature of 300 °C for aperiodic Mo/B4C multilayers.

Author Contributions

All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Zehua Yang, Jingtao Zhu, Shangyu Si, Yunping Zhu, and Zhongliang Li. Experimental resources and funding were provided by Jingtao Zhu, Hongxin Luo, Li Zhao, Kuan Ren, Feng Wang, and Rongqing Yi. The first draft of the manuscript was written by Zehua Yang, and all authors commented on previous versions. All authors read and approved the final manuscript.

Data Availability Statement

The data that support the findings of this study are openly available in Science Data Bank at https://www.doi.org/10.57760/sciencedb.10936 and https://cstr.cn/31253.11.sciencedb.10936.

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Note: Figure translations are in progress. See original paper for figures.