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Tungsten-rhenium alloy as a high-density absorber in ultra-short-period multilayer for hard X-ray mirrors

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Abstract. Ultra-short-period multilayer mirrors (period d below 5 nm) are key components for hard X-ray optics in the 20 keV–40 keV range, but their performance rapidly degrades when the period approaches a few nanometers due to density loss and/or interfacial defects (roughness or intermixing). In this work, we investigate a tungsten–rhenium alloy absorber (WRe, 50 at.% Re) in WRe/SiC multilayers and compare it with a pure W absorber. We also assess the impact of the magnetron sputtering mode, conventional direct-current magnetron sputtering (dcMS) versus high-power impulse magnetron sputtering (HiPIMS), on the optical contrast and reflectivity of WRe/SiC multilayers with $d \approx 3$ nm. Four periodic stacks [Abs/SiC]₄₀ (Abs = W or WRe) were deposited and characterized by multi-energy X-ray reflectometry from a laboratory diffractometer and synchrotron beamlines (BAMline-BESSY-II and BM05-ESRF). Simultaneous fitting of reflectivity curves at multiple photon energies yields estimates of period, densities, and interfacial roughness. This specific fitting process is based on a sequential approach, providing an accurate structure model. These results are complemented by high-resolution TEM analysis of the microstructure. Compared with W/SiC, WRe/SiC exhibits a higher absorber density and a lower spacer density (SiC). This significant increase in the density contrast between the absorber and the spacer results in higher peak reflectivity at comparable Bragg angles. Under these conditions, dense WRe layers are obtained by dcMS, and HiPIMS provides similar densification within experimental uncertainties, while preserving the interfacial quality. These results demonstrate that WRe/SiC multilayers are a promising alternative to conventional W/SiC systems for high-energy X-ray mirrors at nanometer-scale periods.

Keywords: Hard X-ray optics, Ultra-short-period multilayers, Tungsten–rhenium alloys, High-power impulse magnetron sputtering (HiPIMS), Interface roughness, Intermixing.

1 Introduction

High-efficiency X-ray mirrors in the 20 keV to 40 keV energy range require ultra-short-period multilayer (ML) structures with periods of 1 nm–5 nm, operating at grazing angles below 1°. Such mirrors are essential for numerous applications, including hard X-ray astronomy [1], advanced lithography [2, 3], synchrotron beamlines [4], medical imag-

ing [5], and plasma diagnostics for inertial confinement fusion experiments [6, 7].

At these high energies, ML reflectivity is primarily governed by two factors: the density difference $\Delta\rho = \rho_{\text{Absorber}} - \rho_{\text{Spacer}}$ between absorber and spacer layers, and the interfacial quality (roughness and intermixing). Conventional tungsten-based multilayers, such as W/SiC, W/B₄C, or W/C [8–10], can therefore suffer from reduced performance when the period is pushed to very small values and/or when the deposition conditions are not sufficiently optimized, leading to incomplete densification and increased interfacial broadening.

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In a previous simulation-based study [11], we demonstrated that integrated reflectivity of the first Bragg peak R_{int} exhibits strong sensitivity to $\Delta\rho$ at high energies. Figure 1 illustrates this trend for a $[\text{W}/\text{SiC}]_{40}$ multilayer in which the tungsten density is gradually varied. A modest change in the density contrast $\Delta\rho$ produces a pronounced variation in the integrated reflectivity R_{int} , whereas the peak reflectivity R_{max} is comparatively less sensitive. The integrated reflectivity is therefore the most relevant figure of merit at high photon energies: although R_{max} can be increased by raising the number of periods N (potentially up to several hundred, owing to the low absorption in this range), this enhancement is obtained at the expense of a reduced bandwidth and consequently a lower R_{int} .

This trade-off highlights the importance of maximizing $\Delta\rho$ for applications that require high performance across broad angular and spectral ranges.

Two complementary strategies can be pursued to enhance ML performance at ultra-short periods: (i) increasing the absorber density through material selection, and (ii) improving interfacial quality and film densification through advanced deposition techniques. In this work, we investigate both approaches by combining tungsten-rhenium (WRe) alloys and High-Power Impulse Magnetron Sputtering (HiPIMS) [12].

Tungsten-rhenium (WRe) alloys are a promising choice because the $\text{W}_{50}\text{Re}_{50}$ composition exhibits an amorphous phase over a wide temperature range. [13] This is beneficial for multilayer mirrors, since crystallization typically promotes strong roughness amplification and interface degradation. WRe alloys have been successfully employed as thin films in thermocouples [14], diffusion barriers [13], and corrosion protective coatings [15]. To the best of our knowledge, reports on WRe-based ultra-short-period multilayers remain sparse and are largely confined to early works from the 1980s [16–19]. Rhenium ($Z = 75$, bulk density $\rho_{\text{Re}} = 21.02 \text{ g cm}^{-3}$) [20] offers a density $\sim 9\%$ higher than tungsten ($\rho_{\text{W}} = 19.25 \text{ g cm}^{-3}$), while remaining more cost-effective than other high- Z refractory metals such as iridium, or platinum for magnetron sputtering applications requiring high amount of material.

Complementary to material optimization, HiPIMS deposition generates high-energy metallic ion fluxes that enhance adatom mobility compared to conventional direct current magnetron sputtering (dcMS) [21, 22]. This increased mobility promotes void filling and interface smoothing, resulting in denser films with reduced interfacial roughness [23, 24]. The combination of higher intrinsic density (WRe) and improved densification (HiPIMS) is expected to yield significant performance gains for ultra-short-period MLs.

In this article, we report a characterization of WRe/SiC MLs with a period of $d \approx 3 \text{ nm}$, comparing dcMS and HiPIMS deposition methods. Using multi-energy X-ray reflectometry (XRR), and high-resolution scanning transmission electron microscopy (HR-STEM), we provide quantitative insights into the influence of both material composition and deposition technique on interfacial quality, layer density, and reflective performance in the 20 keV to 40 keV range.

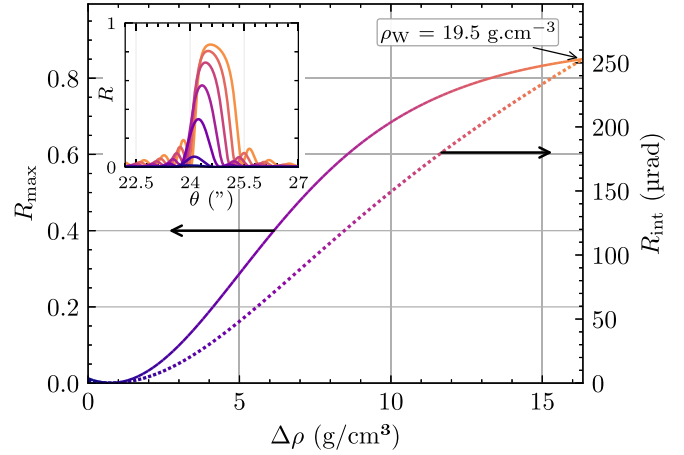


Figure 1. Simulated dependence of peak reflectivity R_{max} and integrated reflectivity R_{int} on density contrast $\Delta\rho$ for a $[\text{W}/\text{SiC}]_{40}$ ML with period $d = 3 \text{ nm}$, stacking ratio $\Gamma = 0.5$, and interfacial roughness $\sigma = 0.1 \text{ nm}$. The tungsten density is varied from bulk value (19.25 g cm^{-3}) down to that of SiC (3.16 g cm^{-3}). Inset: first Bragg peak profiles versus grazing angle θ for selected $\Delta\rho$ values.

2 Multilayer design and sample preparation

2.1 Optical principles

Hard X-ray multilayers consist of alternating layers of a high-density absorber and a low-density spacer. Each material is characterized by a complex refractive index $n(E) = 1 - \delta(E) + i\beta(E)$ and a mass density ρ , where E is the photon energy.

The real part of the refractive index, $1 - \delta(E)$, is directly related to the material mass density ρ through [25]:

$$\delta(E) = \rho \frac{r_e N_A}{2\pi} \left(\frac{hc}{E} \right)^2 \frac{\sum_i x_i f_{1,i}^0(E)}{\sum_i x_i \mu_i}, \quad (1)$$

where r_e is the classical electron radius, N_A the Avogadro constant, h the Planck constant, and c the speed of light. The term $f_{1,i}^0$ denotes the real part of the atomic scattering factor of element i , weighted by its atomic fraction x_i , and μ_i is the atomic weight of element i . This linear dependence of δ on mass density explains why increasing the absorber density directly enhances the optical contrast $\Delta\delta = \delta_{\text{absorber}} - \delta_{\text{spacer}}$, thereby increasing the Fresnel reflection coefficient at each interface and ultimately improving the multilayer reflectivity.

Constructive interference in a multilayer occurs when the Bragg condition is fulfilled:

$$2d \sin \theta \sqrt{1 - \frac{2\bar{\delta}}{\sin^2 \theta}} = m\lambda = m \frac{hc}{E} \quad (2)$$

where θ is the grazing incidence angle, d is the multilayer period, m is the Bragg order, and $\bar{\delta}$ is the period-averaged

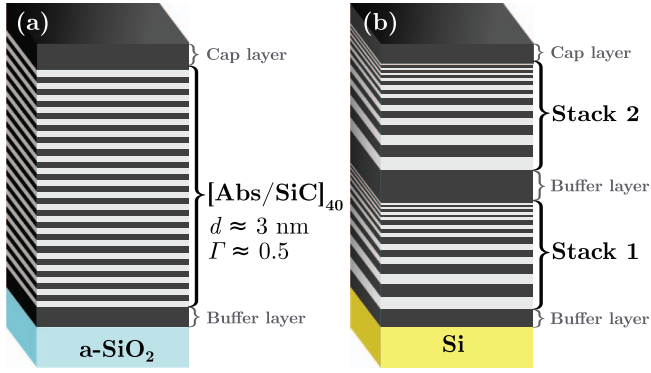


Figure 2. Schematic structures of samples prepared for (a) X-ray reflectometry and (b) transmission electron microscopy (TEM) measurements.

real part of refractive index. The square root term accounts for refraction within the multilayer structure.

For the first Bragg order ($m = 1$) at a photon energy of 30 keV, Bragg's law (Eq. 2) yields a period of $d \approx 3$ nm at a grazing angle of $\theta = 0.4^\circ$. The stacking ratio Γ , defined as the ratio of the absorber layer thickness to the total bilayer period d , is set to $\Gamma \approx 0.5$ for all samples, ensuring a balanced partition between absorber and spacer thicknesses. This balanced Γ value avoids excessively thin individual layers (the absorber or the spacer below ~ 1 nm), which would result in severe interface degradation and compromise multilayer performance [26].

Although B_4C and C exhibit comparable low densities and favorable optical properties for hard X-ray multilayer spacers [4], SiC was selected as the spacer material based on its low bulk density (3.16 g cm^{-3}), good chemical stability, and smooth interface formation in W-based multilayers, as well as on extensive previous in-house work [5–7, 23].

All samples include SiC buffer layers (5–10 nm thick) at the substrate of the ML and a cap layer to protect the multilayer from oxidation.

2.2 Periodic samples for X-ray reflectometry

Periodic multilayers were deposited on low-roughness silica substrates ($\sigma_{\text{rms}} < 0.3$ nm) to minimize substrate contributions to interface roughness and to have a good planarity. The structure consists of 40 bilayers of [Abs/SiC] where Abs = W or WRe, encapsulated between two SiC layers (~ 8 nm), as illustrated in Figure 2a. Four samples were fabricated to explore the effects of absorber material (W vs. WRe) and deposition mode (dcMS vs. HiPIMS):

- **W-DC-31** and **W-DC-25**: W/SiC multilayers deposited by dcMS, with different targeted periods to assess repeatability and period-dependent trends.
- **WRe-DC-46**: WRe/SiC multilayer deposited by dcMS, to quantify the effect of substituting W with WRe on density and interface quality.
- **WRe-HP-26**: WRe/SiC multilayer deposited by HiPIMS, combining the benefits of higher intrinsic density (WRe) and enhanced densification (HiPIMS).

Deposition parameters and structural characteristics extracted from multi-energy XRR fitting (see Sect. 3.1.1) are summarized in Table 1.

2.3 Aperiodic samples for transmission electron microscopy

Aperiodic multilayers for TEM were deposited on Si(100) wafers ($\sigma_{\text{rms}} < 0.25$ nm). Each sample consists of two distinct stacks deposited on the same substrate (Fig. 2b), enabling direct comparison between materials or deposition modes under identical TEM preparation and imaging conditions, thereby eliminating systematic uncertainties related to lamella preparation, sample alignment, or microscope settings. Each stack comprises seven subgroups of N bilayers with periods ranging from 1.5 nm to 6 nm, as detailed in Table 2. This aperiodic design allows simultaneous characterization of interface quality across a range of period thicknesses within a single cross-sectional TEM lamella, providing insights into the period-dependent evolution of interface morphology that cannot be obtained from periodic samples alone.

Two TEM samples were fabricated to isolate the effects of material composition and deposition mode:

- **TEM-73**: stack 1 is W/SiC (dcMS) and stack 2 is WRe/SiC (dcMS), to assess the influence of absorber composition on interface morphology while keeping the deposition process constant.
- **TEM-65**: stack 1 is WRe/SiC (dcMS) and stack 2 is WRe/SiC (HiPIMS), to assess the influence of deposition mode on interface quality while keeping the material composition constant.

2.4 Deposition system and conditions

All samples were deposited using a Plassys MP1000SL magnetron sputtering system at Laboratoire Charles Fabry. The chamber is equipped with four rectangular targets ($200 \text{ mm} \times 80 \text{ mm}$) distributed around the chamber on a circular geometry. The substrates are mounted on a rotating platter (substrate holder), so that during deposition, each sample passes sequentially above a target. Each magnetron has a confinement box that locally encloses the plasma above the target.

Tungsten (W) and tungsten-rhenium (WRe at 50–50 at.% Re) absorber layers were deposited using either dcMS or HiPIMS, while silicon carbide (SiC) spacer layers were deposited by radio-frequency magnetron sputtering (rfMS) in all cases. All targets have purity $> 99.9\%$. For the HiPIMS power supply, we use an HIPSTER-1 from Ionautics.

Depositions were performed at room temperature using argon as the sputtering gas. The base pressure was 5×10^{-8} Torr, and the working pressure was maintained at 2 mTorr with an Ar flow rate of 50 sccm. The target-to-substrate distance was 80 mm. Trapezoidal masks positioned between targets and substrate compensate for non-uniform sputtering flux distribution, ensuring lateral thickness uniformity across the deposition area.

Table 1. Deposition parameters and fitted structural properties of periodic multilayers for XRR. Uncertainties on fitted values are obtained from the sensitivity analysis of XRR fits at multiple energies.

Sample ID	W-DC-31	W-DC-25	WRe-DC-46	WRe-HP-26
<i>Multilayer structure</i>				
Absorber material	W	W	WRe	WRe
Absorber deposition mode	dcMS	dcMS	dcMS	HiPIMS
Spacer material	SiC	SiC	SiC	SiC
Spacer deposition mode	rfMS	rfMS	rfMS	rfMS
<i>Deposition parameters</i>				
Absorber: avg. power (W)	40	40	40	60
Absorber: avg. voltage (V)	262	262	322	710
Absorber: peak current (A)	—	—	—	14
Absorber: pulse frequency (Hz)	—	—	—	1000
Absorber: pulse duration (μ s)	—	—	—	30
Spacer: rf power (W)	200	200	200	200
<i>Fitted structural parameters</i>				
Period d (nm)	3.65 ± 0.01	3.28 ± 0.01	3.50 ± 0.01	3.34 ± 0.01
Stacking ratio Γ	0.47 ± 0.05	0.45 ± 0.05	0.41 ± 0.05	0.43 ± 0.05
ρ_{SiC} (g cm^{-3})	4.1 ± 0.5	4.5 ± 0.5	3.4 ± 0.5	3.3 ± 0.5
ρ_{abs} (g cm^{-3})	15.1 ± 0.5	15.3 ± 0.5	16.7 ± 0.5	17.1 ± 0.5
$\sigma_{\text{abs/SiC}}$ (nm)**	0.19 ± 0.05	0.33 ± 0.05	0.30 ± 0.05	0.35 ± 0.05
$\sigma_{\text{SiC/abs}}$ (nm)	0.22 ± 0.05	0.38 ± 0.05	0.23 ± 0.05	0.26 ± 0.05

**Roughness for absorber on SiC interface.

Table 2. Targeted aperiodic structure for TEM samples. Each subgroup consists of N bilayers with period d and stacking ratio $\Gamma \approx 0.5$. Subgroups are stacked vertically from substrate (subgroup 7, thinnest period) to surface (subgroup 1, largest period).

Subgroup	d (nm)	N	Subgroup thickness (nm)
7	1.5	2	3.0
6	2.0	2	4.0
5	2.5	2	5.0
4	3.0	2	6.0
3	4.0	2	8.0
2	5.0	1	5.0
1	6.0	1	6.0
Total stack thickness:			37.0

2.5 Simulation methods

Specular X-ray reflectivity curves are computed using the recursive Parratt formalism [27], implemented in a custom Python script. Optical constants (f_1^0, f_2^0) are obtained from the Henke database [28] for photon energies below 30 keV and from NIST-CHANTLER tables [29] at higher energies. Interfacial roughness is modeled using the Névot-Croce formalism [30], which accounts for a Gaussian distribution of interface height fluctuations with root-mean-square roughness σ .

3 Experimental results

3.1 X-ray reflectometry

3.1.1 Experimental setup

Specular X-ray reflectivity measurements were performed at multiple photon energies to enable robust structural characterization by simultaneously fitting multi-energy data sets.

In-house measurements were carried out using a Bruker D8 diffractometer equipped with a Cu tube source equipped

with a Göbel mirror to select $K\alpha$ lines ($E = 8.048$ keV, $\lambda = 0.154$ nm) in θ - 2θ geometry. These measurements provide high angular resolution over a wide angular range, enabling accurate determination of the multilayer period from Bragg peak positions, overall structural coherence from Kiessig fringes, and initial estimates of interfacial roughness. Measurements at 8.048 keV are presented in Figure 3 for all four samples with the final fitted curves (see Sect. 3.1.2).

High-energy reflectivity measurements were conducted at two synchrotron facilities to probe the first Bragg peak under conditions relevant for practical applications (20 keV to 40 keV):

- **BAMline at BESSY-II** [31]: Measurements at photon energy from 20 to 30 keV were performed using a photodiode detector with a 300 μm entrance slit ensuring adequate angular resolution while maintaining sufficient flux.
- **BM05 at ESRF** [32]: Measurements were performed in the 20 keV to 60 keV energy range, corresponding to the beamline's maximum flux region. The acquisition setup consists of an Andor Marana 11 sCMOS camera coupled to a 9×9 mm² Ce:YAG scintillator via a $2\times$ magnification objective. Each data point corresponds to an image acquired at multiple exposure times (from 0.01 s to 1 s), providing a high dynamic range while maintaining a relatively fast acquisition rate. This two-dimensional detector enables the selection of a region of interest after measurement.

The use of two independent beamlines with different detector technologies provides cross-validation of the measured reflectivity curves and eliminates potential systematic errors associated with a single experimental configuration.

3.1.2 Data analysis and fitting procedure

Reflectivity curves acquired at multiple energies (8 keV, 20 keV, 30 keV, and 40 keV) were fitted simultaneously using a single structural model to ensure self-consistency. In practice, we found that a direct approach (i.e., allowing all parameters to vary simultaneously from generic initial guesses) often fails to converge toward physically realistic solutions, due to strong parameter correlations at ultra-short periods (notably between Γ , layer densities, and interfacial roughness), and the presence of multiple local minima yielding comparably good agreement at a single energy. After testing several fitting strategies (including global refinements with randomized initial conditions), we identified a sequential procedure that provides more stable convergence and consistently yields physically meaningful parameters. While this approach is inherently more guided and, therefore, potentially more biased, it explicitly incorporates our prior knowledge of how each parameter affects distinct features of the reflectivity curves. The fits, illustrated in Figure 4, follow the sequential approach outlined below:

1. The multilayer period d is initially determined from the angular position of Bragg peaks in the 8 keV data.

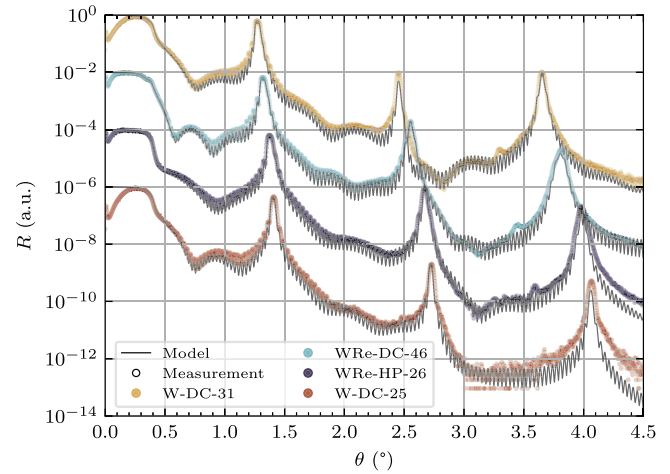


Figure 3. Reflectivity curve of all sample measures at 8.048 keV with fitted curve in solid lines.

2. The Γ ratio is adjusted to reproduce the overall reflectivity curve shape, relative peak intensities, and peak extinction (since $\Gamma \approx 0.5$, the even orders are strongly suppressed).
3. Interfacial roughness parameters ($\sigma_{\text{abs/SiC}}$ and $\sigma_{\text{SiC/abs}}$) are optimized using a genetic algorithm to match the decay of Bragg peak intensities with increasing order and the amplitude of Kiessig fringes.
4. Material densities (ρ_{abs} and ρ_{SiC}) are refined to simultaneously match: (i) the critical angle position, which depends on the period-averaged density $\bar{\rho}$, and (ii) the peak reflectivity magnitude, which depends on the density contrast $\Delta\rho = \rho_{\text{abs}} - \rho_{\text{SiC}}$.
5. The fitted model is tested against high-energy synchrotron data (20 keV to 40 keV). Steps 3 and 4 are iterated until convergence.

This multi-energy fitting approach strongly constrains the structural parameters, yielding more reliable estimates of layer densities and interface quality than single-energy analysis. Figure 4 demonstrates the good agreement between experimental reflectivity data and the fitted model across all photon energies (8 keV, 30 keV, and 40 keV) for all four samples, using the parameters listed in Table 1. The ability of a single structural model to reproduce the reflectivity curves at multiple energies validates the robustness and self-consistency of the estimated parameters.

We incorporated an angular-resolution parameter into the simulated curves to account for each instrument's angular resolution, as this can significantly influence the reflectivity curve [33]. We also introduced a relative d -spacing error to account for deposition-induced period variations during growth. This parameter broadens the high-order Bragg peaks and remains below 1 % for all samples.

Across the full dataset, the reflectivity curves acquired at different energies are well aligned, with the remaining small angular offsets staying below the alignment precision of the measurement setup. This further supports the benefit of the multi-energy approach, which averages out such

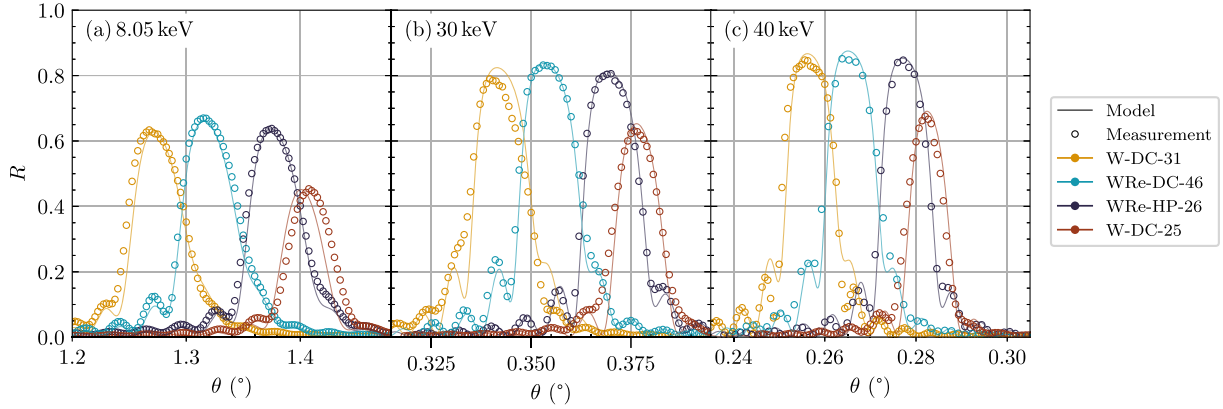


Figure 4. X-ray reflectivity measurements (symbols) and corresponding best-fit simulations (solid lines) for the four periodic multilayer samples at three photon energies: (a) 8 keV (Cu K α , Bruker D8), (b) 30 keV (W-DC-31 and W-DC-25 data from PTB-BAMline, others from ESRF-BM05), and (c) 40 keV (all from ESRF-BM05).

minor experimental shifts and yields the most representative, self-consistent reflectivity dataset.

3.2 Scanning transmission electron microscopy

3.2.1 Experimental setup

Cross-sectional lamellae for transmission electron microscopy were prepared by focused-ion-beam (FIB) milling in a scanning electron microscope (SEM) using a FEI ThermoFisher Helios Nanolab 660. High-resolution high-angle annular dark-field scanning transmission electron microscopy (HR-HAADF-STEM) imaging was performed using an FEI Titan G2 80–300 microscope operated at 300 kV at the Laboratoire de Mécanique Paris-Saclay.

The HAADF-STEM imaging mode exploits Z-contrast, where Z is the atomic number. This produces bright intensity from high- Z absorber layers (W: $Z = 74$, Re: $Z = 75$) and dark intensity from low- Z spacer layers (Si: $Z = 14$, C: $Z = 6$), providing excellent discrimination between materials with minimal diffraction contrast artifacts.

3.2.2 Quantitative analysis of TEM images

Individual layer thicknesses and interface profiles were quantified from intensity line profiles extracted perpendicular to the multilayer stack, as illustrated in Figure 5. We optimized image rotation to ensure that multilayer interfaces are aligned with the pixel grid, minimizing bias in the extracted profiles. To improve the signal-to-noise ratio, intensity profiles were averaged laterally over regions of ~ 10 nm width along the y -axis, corresponding to the vertical direction in the bottom panels of Figure 5b, c.

The intensity profile $I(z)$ was first normalized to the $[0, 1]$ range to enable consistent analysis across different imaging conditions. For each interface indexed by i , the precise interface position z_i and the transition width $\tilde{\sigma}_i$ were determined by fitting the normalized intensity profile in the region between consecutive local extrema (absorber and spacer peak positions) with an error function (Erf) (consistent with the error-function interface profile implied by the Névot-Croce roughness model [30]), as shown schematically in Figure 5a:

$$\text{Erf}_i(z) = \frac{1}{\tilde{\sigma}_i \sqrt{2\pi}} \int_{-\infty}^z \exp \left[-\frac{(z' - z_i)^2}{2\tilde{\sigma}_i^2} \right] dz' \quad (3)$$

where z_i is the center position of i th interface and $\tilde{\sigma}_i$ characterizes the interface transition width. A small value translates to a sharper interface. The fitted parameter $\tilde{\sigma}_i$ reflects the interfacial roughness, chemical intermixing, and instrumental broadening effects. Layer thicknesses e_i were calculated as the distance between successive interface centers: $e_i = z_{i+1} - z_i$ and the local period being $d_i = z_{i+1} - z_{i-1}$.

Despite using the same error function to describe the interface, it is important to note that $\tilde{\sigma}_i$ determined from TEM is not directly equivalent to the interfacial roughness parameter σ used in the Névot-Croce model for X-ray reflectometry, due to differences in the measurement process and sensitivity to different spatial frequency ranges. Therefore, TEM-derived interface widths provide complementary rather than directly comparable information to XRR-derived roughness values.

4 Discussion

4.1 Multi-energy reflectometry analysis and period-dependent performance

To assess the impact of period thickness, absorber composition, and deposition mode on multilayer performance, we analyze the X-ray reflectivity measurements across the 8 keV to 40 keV energy range. Figure 6 shows reflectivity curves measured at various photon energies for the four samples, in solid dotted line.

A direct comparison between samples with different periods requires careful consideration, as both the Bragg angle and the achievable peak reflectivity depend on the period thickness. We propose a meaningful comparison across energies and samples with a representation plotting the peak reflectivity $R_{\max}$ as a function of its corresponding Bragg angle $\theta_{\max}$ for the first-order peak. Those values are extrapolated from the multilayer model derived from all the measured data. This approach is illustrated in Figure 6 with thick solid lines. From the structural model of each

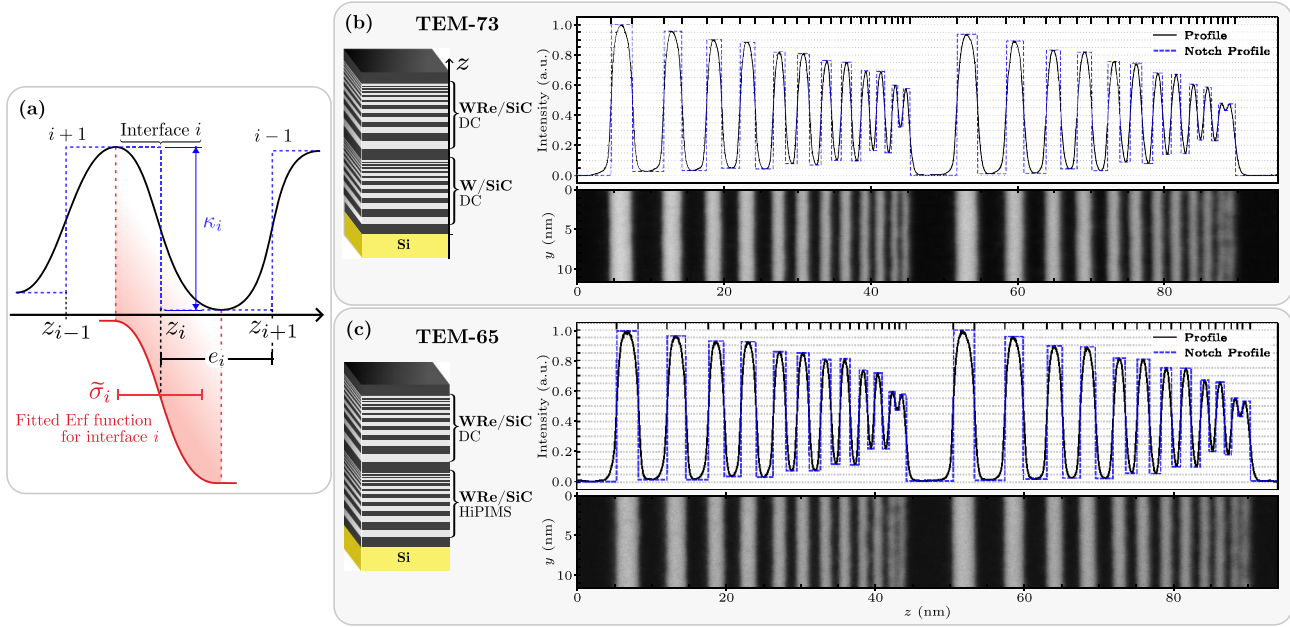


Figure 5. (a) Schematic illustration of the quantitative analysis procedure for HAADF-STEM images. Interface positions are determined by fitting the profile with the error function at each interface. (b) HAADF-STEM image and intensity profile for sample TEM-73, comparing WRe/SiC (top stack) and W/SiC (bottom stack), both deposited by dcMS. (c) HAADF-STEM image and intensity profile for sample TEM-65, comparing WRe/SiC deposited by dcMS (top) and HiPIMS (bottom). Brighter layers correspond to absorbers (W or WRe), darker layers to SiC spacers.

sample, we extract $(R_{\max}, \theta_{\max})$ by simulating angular reflectivity scans at varying photon energies, yielding a trajectory in the $R_{\max} - \theta$ space that characterizes each sample's performance independently of the specific measurement energy. We can observe features corresponding to the L -absorption edges of tungsten and rhenium around 1° on those computed curves. The thick solid lines in Figure 6 further validate the fitting procedure described in Section 3.1.2, demonstrating that a single structural model consistently reproduces the reflectivity at the different photon energies.

4.2 Effect of period thickness on W/SiC ML

The two W/SiC samples deposited by dcMS exhibit significantly different performance. Sample W-DC-31, with the larger period ($d = 3.65$ nm), achieves peak reflectivities approaching 85% at 40 keV. In contrast, sample W-DC-25, with a shorter period ($d = 3.28$ nm), shows significantly degraded performance with $R_{\max}$ of 64%.

This deterioration is primarily attributed to increased interfacial intermixing and roughness at reduced period thickness, as confirmed by the fitted roughness values in Table 1: $\sigma \approx 0.2$ nm for W-DC-31 versus $\sigma \approx 0.35$ nm for W-DC-25. At short periods, the transition zones become comparable to individual layer thicknesses [34], meaning that interfacial defects occupy an increasingly large fraction of the total structure volume. This phenomenon leads to a drastic reduction in optical contrast (electronic density) between the absorber and spacer, thereby limiting the maximum achievable reflectivity [35, 36]. This trend

is consistent with previous observations in W/B₄C systems [37].

4.3 Enhanced performance with tungsten-rhenium absorbers

Despite having lower period thicknesses ($d = 3.34$ nm for WRe-HP-26 and $d = 3.50$ nm for WRe-DC-46), both WRe/SiC samples outperform (or at least equal) the performance of the longer-period W/SiC sample (W-DC-31). This enhancement is clear in Figure 6, where the WRe/SiC samples exhibit higher peak reflectivities at comparable Bragg angles. The improved performance is primarily attributed to the increased density contrast $\Delta\rho$ between absorber and spacer layers. As shown in Table 1, the fitted absorber densities for WRe samples ($\rho_{\text{WRe}} \approx 16.7$ g cm⁻³ for dcMS, 17.1 g cm⁻³ for HiPIMS) exceed those of W samples ($\rho_{\text{W}} \approx 15.2$ g cm⁻³), resulting in a ~ 10 – 15% increase in $\Delta\rho$.

This density enhancement is further corroborated by calibration multilayer samples (with a reduced number of periods N) deposited prior to the main samples: XRR fits consistently show a shift in critical angle and higher fitted densities for WRe layers compared to pure W, confirming that the density contrast observed in the periodic multilayers is a robust and reproducible feature of the WRe absorber.

In addition, the fitted SiC spacer density (Table 1) is systematically lower for WRe/SiC samples ($\rho_{\text{SiC}} \approx 3.3$ g cm⁻³) compared to W/SiC samples ($\rho_{\text{SiC}} \approx 4.3$ g cm⁻³), despite identical rfMS deposition conditions for all spacer layers. This result enhances the density

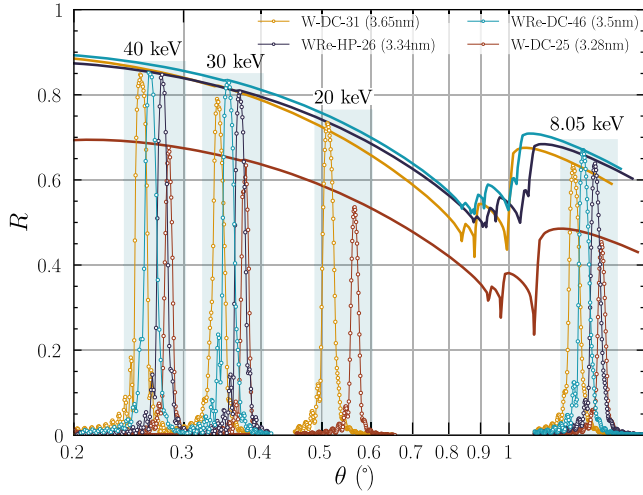


Figure 6. X-ray reflectivity curves measured (symbols), along with best-fit simulations (thin solid lines). The fitted structural parameters extracted from simultaneous fitting of multi-energy data are listed in Table 1. Bragg peaks and Kiessig fringes are clearly visible, indicating high structural quality. Thick solid lines are described in Section 4.1.

contrast $\Delta\rho$ in WRe/SiC multilayers, thereby improving their performance. The observed variation in apparent SiC density can have several origins. In W/SiC samples, the elevated fitted density ($\rho_{\text{SiC}} > 3.2 \text{ g cm}^{-3}$, the bulk value) likely indicates significant intermixing or interdiffusion of tungsten into the SiC layers, effectively increasing the average atomic mass within the spacer regions. Conversely, the lower fitted density in WRe/SiC samples suggests either reduced intermixing with the WRe absorber or different SiC growth dynamics on WRe versus W surfaces.

The reduced intermixing with WRe is likely thermodynamically driven. The interfacial energy balance between absorber, spacer, and their interface (γ_{abs} , γ_{SiC} , γ_i) determines both the growth mode and interdiffusion tendency. WRe may exhibit a higher interfacial energy or a lower chemical affinity for SiC than pure W, thereby reducing the thermodynamic driving force for intermixing despite its heavier atomic mass. Additionally, the WRe surface may provide different nucleation conditions for SiC growth, influencing the spacer microstructure (density, void content) and resulting in denser, less contaminated layers.

The fitted roughness values for WRe/SiC samples ($\sigma \approx 0.27 \text{ nm}$ for dcMS, 0.31 nm for HiPIMS) are slightly higher than those of the thicker W/SiC sample (W-DC-31: $\sigma \approx 0.21 \text{ nm}$), but comparable to or lower than those of the thinner W/SiC sample (W-DC-25: $\sigma \approx 0.36 \text{ nm}$) despite having intermediate periods. This indicates that the better performance of WRe/SiC ML is not primarily attributable to reduced interfacial roughness, but rather to the enhanced density contrast $\Delta\rho$, which increases by $\sim 10\text{--}15\%$ compared to W/SiC systems (Table 1). This highlights the main role of optical contrast at these ultra-short periods, consistent with the scaling predictions of Figure 1.

By analyzing the TEM images of the sample TEM-73 via the Figure 7a we can confirm that the interface widths $\tilde{\sigma}$ of WRe/SiC are less abrupt than W/SiC, which is consistent with the fitted roughness values from XRR.

In Figure 7, the extracted $\tilde{\sigma}$ values appear to decrease as the period d is reduced. This trend, however, is not consistent with the XRR fits reported in Table 1, which instead indicate a slight increase of the interfacial widths for the W/SiC samples (from $\sim 0.2 \text{ nm}$ up to $\sim 0.35 \text{ nm}$) as d decreases from 3.65 nm to 3.28 nm . Such interfacial broadening at shorter periods is also commonly reported in the literature. [26] This discrepancy arises from the data analysis procedure. In particular, the error-function fitting approach cannot return interface widths $\tilde{\sigma}$ larger than the local period d , which biases the extracted trend in the thinnest-period range. Nevertheless, the purpose of this analysis is primarily comparative, i.e., to assess relative differences between deposition conditions, rather than to derive absolute roughness values.

Regardless of the underlying mechanisms, the net effect is beneficial: the combination of higher absorber density and lower apparent spacer density in WRe/SiC systems maximizes $\Delta\rho$, directly enhancing reflectivity.

4.4 Impact of HiPIMS

The comparison between HiPIMS and dcMS deposition modes for WRe/SiC MLs reveals a moderate impact of HiPIMS, which differs from our previous observations on W/SiC systems [23]. Samples WRe-HP-26 (HiPIMS, $d = 3.34 \text{ nm}$) and WRe-DC-46 (dcMS, $d = 3.50 \text{ nm}$) exhibit comparable reflectivities across the 30 keV to 40 keV range (Fig. 6), with dcMS yielding marginally higher peak values, certainly due to a higher period.

The fitted structural parameters (Table 1) do not evidence any clear HiPIMS-induced densification within the density uncertainties: ($\rho_{\text{WRe}}^{\text{HiPIMS}} = 17.1 \text{ g cm}^{-3}$ vs. $\rho_{\text{WRe}}^{\text{dcMS}} = 16.7 \text{ g cm}^{-3}$, $\sim 2\%$ gain) compared to the $10\text{--}15\%$ improvements observed in pure W films.

TEM-derived interface widths $\tilde{\sigma}$ (Fig. 7b) show comparable values for WRe/SiC multilayers deposited by dcMS and HiPIMS, suggesting that the HiPIMS peak current of 14 A is not sufficient to significantly affect the microstructure.

However, the XRR-fitted roughness values for the WRe/SiC interface differ only slightly between HiPIMS ($\sigma_{\text{WRe/SiC}} = 0.35 \text{ nm}$) and dcMS ($\sigma_{\text{WRe/SiC}} = 0.30 \text{ nm}$). This $\Delta\sigma \approx 0.05 \text{ nm}$ remains on the order of our experimental repeatability and process-to-process variability, and it is further difficult to interpret unambiguously because the two samples have slightly different periods ($d_{\text{HiPIMS}} = 3.34 \text{ nm}$ vs. $d_{\text{dcMS}} = 3.50 \text{ nm}$). As a result, with our HiPIMS parameters, no statistically significant HiPIMS effect on interfacial roughness can be concluded from the present dataset.

These results point to a material-dependent response to HiPIMS processing. In particular, because pure Re is significantly less electrically conductive than W [20], the electrical conductivity of WRe alloys is also reduced, which is expected to modify the discharge characteristics in HiPIMS

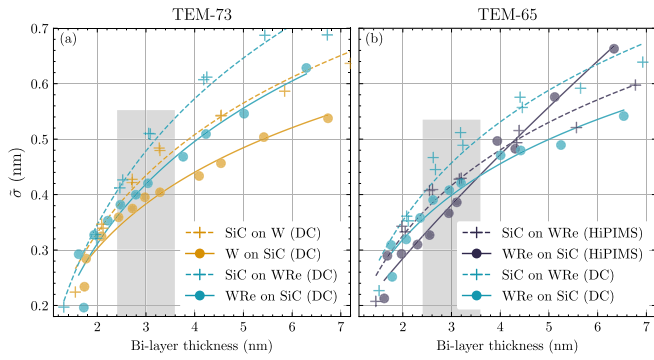


Figure 7. Two-by-two comparison of the MET sample groups: (a) W/SiC (dcMS) vs. WRe/SiC (dcMS) and (b) WRe/SiC (dcMS) vs. WRe/SiC (HiPIMS). The fitted interface widths $\tilde{\sigma}$ from TEM analysis are plotted as a function of the local period d_i for each interface. Tendency lines are added to guide the eye. Interest d-spacing zones are displayed in gray.

(plasma impedance, current waveform, and ion-to-neutral ratio) compared with pure W targets. Under our conditions, this altered discharge behavior may limit the potential densification benefits of HiPIMS and/or increase ion-induced interface degradation. Further optimization of HiPIMS parameters (pulse energy, duty cycle) specifically for WRe targets is therefore likely required.

Nevertheless, both WRe/SiC samples outperform conventional W/SiC ML at comparable periods, confirming that absorber density remains a primary determinant of reflectivity in the 20 keV to 40 keV range.

5 Conclusion

We investigated tungsten–rhenium alloy absorbers (WRe, 50 at.% Re) as a way to improve the performance of ultra-short-period multilayer mirrors for hard X-rays. Periodic $[\text{Abs}/\text{SiC}]_{40}$ stacks (Abs = W or WRe) were characterized by multi-energy X-ray reflectometry (8 keV to 40 keV) measurements, and interface morphology was further assessed by HR-HAADF-STEM on dedicated aperiodic samples.

Multi-energy simultaneous fitting provides self-consistent structural parameters and confirms the strong period-dependence of W/SiC performance: reducing the period from $d = 3.65$ nm to 3.28 nm leads to a significant reflectivity loss, consistent with the higher fitted values of interfacial intermixing/roughness. In contrast, WRe/SiC multilayers achieve peak reflectivity equal to or higher than that of W/SiC, primarily due to higher absorber density and therefore a higher density contrast. The fitted roughness values for WRe/SiC remain within a reasonable range and degrade performance (from ≈ 0.2 nm (W) to ≈ 0.3 nm (WRe)), indicating that optical contrast is a critical factor at these periods.

Finally, within the HiPIMS parameter set investigated here (peak current 14 A, 30 μ s pulses at 1 kHz), no clear densification of WRe beyond the dcMS case can be resolved within the experimental uncertainties, and any change in interface widths remains smaller than our measurement

and process repeatability. This suggests that WRe already reaches near the highest achievable densification in this range of period with dcMS in our configuration, and that further HiPIMS study with extended parameter values (peak current, pulse duration, bias, and ion-to-neutral ratio) will be necessary to achieve further optimization.

Although rhenium is scarcer than tungsten, its use in multilayer mirrors involves only sub-milligram quantities per device, making the raw material cost negligible at the device fabrication level. Moreover, rhenium remains significantly less expensive than other high-Z absorber candidates such as iridium or platinum [20], whose market prices exceed that of rhenium by one to two orders of magnitude [38]. Overall, WRe/SiC emerges as a robust, high-performing alternative to W/SiC for nanometer-period multilayer mirrors in the hard X-ray range, and appears to be a promising route to unlock reliable ultra-short-period coatings by alleviating the key performance limitations of conventional W-based stacks.

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Conflicts of interest

The authors declare no conflicts of interest.

Data availability statement

The data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

Author contribution statement

Conceptualization, A.L. and F.D.; *Methodology*, C.N.; *Software*, C.N.; *Validation*, A.L. and F.D.; *Formal Analysis*, C.N.; *Investigation*, C.N., A.L. and F.D.; *Resources*, E.M., TN.TC., C.G., and M.V.; *Data Curation*, F.D. and F.D.; *Writing – Original Draft Preparation*, C.N.; *Writing – Review & Editing*, F.D. and A.L.; *Supervision*, F.D. and A.L.; *Project Administration*, F.D. and A.L.; *Funding Acquisition*, F.D. and A.L.

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