



Optical constants of Sc, V, Cr, Co, Ni, Nb, Mo, Rh, Pd, Hf, W, Re, Os, Ir, Pt, Au, and ZrO₂ in the extreme ultraviolet range

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Abstract: Transition metal coatings are of fundamental value for extreme ultraviolet (EUV) optics. Often, the reported optical constants for transition metals in the EUV range exhibit inconsistencies across different publications. Therefore, additional studies to determine optical constants in the EUV range for transition metals are highly relevant to further develop optical components. In our work, the optical constants of scandium, vanadium, chromium, cobalt, nickel, niobium, molybdenum, rhodium, palladium, hafnium, tungsten, rhenium, osmium, iridium, platinum, gold, and zirconium dioxide are determined in parts of the EUV spectral range. The optical constants were determined from angle-dependent reflectance measurements collected using monochromatized synchrotron radiation. We also test capping solutions for thin coatings of scandium, vanadium, cobalt, niobium, molybdenum, hafnium, tungsten, and rhenium. All of our results are presented in comparison with literature data. For many cases, our results present either significant scale deviation or previously unresolved fine-structure in the widely used optical data compilations.

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1. Introduction

At a wavelength of 13.5 nm, 8 nm resolution can now be achieved in the resist image using extreme ultraviolet lithography (EUVL) scanners [1]. That is feasible for scanners with a numerical

aperture (NA) of 0.55 [1], yet, imaging optics for EUVL scanners with a NA of 0.75 aiming at higher resolution (~ 6 nm) are currently under study [2]. The high-volume manufacturing (HVM) capabilities of EUVL systems are rapidly expanding to cope with market demand, and they are receiving substantial developments. However, EUVL requires components such as pellicles that meet stringent criteria including high extreme ultraviolet (EUV) transmission and high thermal stability [3]. EUVL systems are also energy intensive [4], and complex elements such as aperiodic multilayer reticles might be needed for later developments [5]. The current importance of EUVL and its demanded developments give research on the interaction of EUV radiation with solids a priority. In line with that, research on optics and photoresists relevant to the projected beyond EUV lithography (BEUVL) technology at 6.7 nm is advancing [6,7]. We hereafter refer to the radiation within the spectral range 1.0 nm to 50.0 nm as EUV. The applications of EUV radiation surpass lithography. Spaceborne solar observation [8], ultrafast magnetodynamics studies [9], magneto-optical spectroscopy [10], coherent diffractive imaging (CDI) [11], hydrogen desorption [12] are a few of many.

EUV applications and their projected developments rely on dedicated optical components for radiation collection, monochromatization, polarization selection, etc., multilayer mirrors (MLMs), zone plates, and transmissive films are examples of such components. Here, transition metals are particularly interesting as constituents of EUV optical components, they have been discussed in this scope before [13]. Most transition metals have high melting points, a property that is crucial for the effective operation of EUV optics, especially when exposed to considerable thermal loads. Also, transition metals are compatible with coating techniques like magnetron sputtering, enabling coating large substrates with dense thin films of low surface roughness. That is particularly important for free-electron laser (FEL) optics, where the deposition length can exceed 1 m [14]. Magnetron sputtering of metals can yield thin films with near-bulk mass density, see ref. [15] as an example for Sc. Transition metal coatings are also alloyable (*e.g.*: ref. [16]), which allows for tuning optical responses. Some transition metal alloys were shown to form metallic glasses, with ultra-smooth surfaces [17]. Additionally, it is typical to use very thin layers of transition metals as auxiliaries, rather than primary coatings, such as interdiffusion barriers, capping (see refs. [18–20]), binders and seed layers. Those applications stem from the advantage that metallic films can form isotropic and stable layers even when coated at very thin thicknesses. With ion-beam sputtering, there are indications that some transition metals – such as Ni, Nb, Rh, W, and Pd – form optically isotropic thin films at thicknesses slightly below 5 nm [21]. Furthermore, their coatings can be treated using techniques like nitridation (ref. [22]) and carbonization for enhancing stability. Rehman *et al.* discussed hydrogen radical interactions with some coatings of transition metal carbides and nitrides [23,24]. That is relevant since EUV optics are especially vulnerable to water vapor and reactive oxygen species (ROS) in certain environments [25,26]. These features besides others distinguish transition metals for EUV applications and optics.

Simulation is an essential step in the design of optical components, for which the fundamental optical parameters of the relevant materials are needed in order to correctly predict their optical properties. The optical constants are both parts of the complex index of refraction $\tilde{n}$, which is a function of the wavelength (λ) given as: $\tilde{n}(\lambda) = 1 - \delta(\lambda) + i\beta(\lambda)$. The real part ($1 - \delta$) – known as the index of refraction – and the imaginary part (β) – known as the extinction coefficient – are reported by many researchers [27–36], but inconsistencies are often observed. Such inconsistencies sometimes lead to significant discrepancies when trying to simulate the optical response of an element, see ref. [37] as an example. In the same direction, synchrotron beamlines' optics can cost hundreds of thousands of Euros, and their production can take more than a year [38]. It is essential to ascertain the behavior of such optical components *in silico* before fabrication. That is only possible if the optical constants of the coated materials are accurately known.

Determining optical constants in the EUV has a few inherent challenges. Materials in the EUV are highly absorbing, and environmental contamination is difficult to avoid. Minimal contamination on optical surfaces affects the measured optical responses. That in turn biases the determined optical constants, especially at the long-wavelength band of the EUV. Corresponding to that, measurement setups require vacuum conditions. Also, EUV lab-based sources have high intensity only at discrete bands. Their stability and spectral tunability are limited. The majority of research conducted on the determination of optical constants within the EUV and soft X-ray ranges is done using synchrotron radiation. Synchrotrons provide broad and continuous spectral coverage, but they have a high operation cost.

Inconsistencies in optical data of transition metals are often observed. Perhaps, in addition to the inherent challenges addressed before, another contributing factor is that most transition metals exhibit multiple oxidation states. For example, Cr and Os have eleven known oxidation states [39,40]. Meta-stable states are also found in nano-layers. Furthermore, inconsistencies in data were found even for exceptionally stable metals such as Ta and platinum-group metals like Pt and Ir [41–43]. Also, some transition metal coatings are prone to aging effects and silicidation. The latter is unfavorable here because Si and quartz are the two most widely used substrates. Such phenomena make resolving the optical parameters of elemental transition metals more challenging. Estimating the impact of oxidation and interdiffusion is often complex when simulating the optical response of a sample. Moreover, for platinum-group metals and Hf in particular, obtaining highly pure and elemental thin films is more involved. For Hf, it is found together with Zr and their separation is very difficult [44]. For Rh, Re, Os, and Ir, they are among the least abundant elements in Earth's crust [45]. As an example, the separation and refinement of Rh require extensive processes [46], one reason is its complicated aqueous chemistry and limited access to relevant research [47]. Impurities in thin films of platinum-group metals and others such as Hf might be an additional factor leading to inconsistencies in tabulated optical data.

However, in addition to the development of optics, dispersion and absorption profiles contain fundamental details about materials' nature. Transition metals have absorption edges in the EUV range, resolving such features is of principle importance to better understand their electronic structure [48,49]. X-ray absorption spectroscopy (XAS) techniques like near edge X-ray absorption fine structure (NEXAFS) among many others also benefit from updated optical data. Also, the available theoretical estimations of optical constants close to absorption edges for many transition metals are not accurate. Therefore, optical constants determination in the EUV – not only for transition metals – remains an active and important field, as examples, the optical constants of Be and Al in the EUV were reexamined in recent years [50,51].

Here, we present the determination of optical constants in the EUV spectral range from sixteen transition metals, ascendingly with respect to the atomic number; Sc, V, Cr, Co, Ni, Nb, Mo, Rh, Pd, Hf, W, Re, Os, Ir, Pt, and Au in addition to one oxide; ZrO_2 . The optical constants were determined from reflectivity measurements collected using monochromatized synchrotron radiation. The samples here are coatings on Si substrates. The investigated materials are very relevant for EUV applications, with numerous studies referring to them. For brevity, we include one representative reference where each of the studied materials was or has been of interest [52–68].

We select reflectometry for determining optical constants as it allows determining both parts of the complex index of refraction ($\bar{n}$), simultaneously, from a single coating. Although ellipsometry and transmission-mode measurements can be used for the purpose [50,51,69], they have additional requirements that are unnecessary for reflectometry. Ellipsometry requires additional optical components such as analysers and retarders. High quality retarders dedicated for the EUV range are not readily available. For transmission measurements, samples with differing thicknesses are needed to achieve reliable accuracy, especially when studying atmospherically reactive elements.

Overviews of our results were presented in conferences before [70–73]. Also, the formalism used here for investigating reflectometry data is established and was reported in publications [74]. For every material, our results are shown in comparison with literature.

2. Experimental methods

2.1. Samples fabrication

The investigated samples are categorized based on the production laboratory and are described in this sub-section. The following list presents seventeen samples from different sources:

2.1.1. Sc and V samples fabrication

In MESA + Institute for Nanotechnology, the Sc and V layers were sputtered with DC magnetron sputtering onto Si substrates using a modified box coater from Leybold GmbH (Cologne, Germany). The substrates' orientation is (100). The 4-inch sputter targets had target-to-substrate distance of 26 cm. The base pressure of the deposition system was 1×10^{-8} mbar, with a working pressure of 1.4×10^{-3} mbar during sputter deposition. Ru interdiffusion barriers were deposited on the substrates prior to Sc and V deposition. C capping layers were deposited directly after deposition of Sc and V layers. All layers were deposited in the sputter system without breaking vacuum. Prior to each layer deposition, the targets were sputter cleaned behind a closed substrate shutter. The deposition rates were controlled by adjusting the sputter power, which is shown in Table 1 alongside the nominal thickness of the two systems.

Table 1. Target sputter power and nominal layers' thicknesses of the Sc and V samples

Sample	Target sputter power/watt				Layer thickness/nm			
	Ru	Sc	V	C	Ru	Sc	V	C
Sc sample	332	525	-	708	3	40	-	5
V sample	300	-	350	750	3	-	40	4

The resulting film thicknesses were verified using X-ray reflectometry (XRR). XRR fits indicated Ru-Si formation for the case of the Sc system. Finally, a separate C coating with a nominal thickness of 40 nm was deposited onto a Si (100) substrate, using the aforementioned sputtering settings of the V capping layer.

2.1.2. Co sample fabrication

The Co sample was described and characterized in a previous work [75]. The sample was coated on a super-polished 300 mm Si wafer using DC magnetron sputtering, by optiX fab GmbH. The sputtering chamber was evacuated to a base pressure lower than 1×10^{-7} mbar before the coating process. High-purity Ar gas (6N) was purged as the sputtering gas at a working pressure of 1×10^{-3} mbar. The nominal structure consists of the following layer thicknesses: C capping (5 nm)/Co layer (40 nm)/Ru underlayer (2 nm) all deposited on Si substrate. The native oxide layer of the substrate was not etched before deposition. After coating, a square coupon with a side length of 25 mm was diced from the wafer.

2.1.3. Mo sample fabrication

A Mo thin film with thickness of 39.9 nm was deposited by magnetron sputtering onto a 1 mm thick and square n-doped Si (100) substrate with a side length of 30 mm. A C capping layer with a thickness of 5 nm was coated on top. The fabrication took place in Laboratoire Charles Fabry. The purity of the targets is 99.95%. The Mo deposition is made in DC-mode (current of 0.06 mA) while the C layer is deposited in RF-mode (power of 150 watts). The distance between

the substrate and the targets is 10 cm. The substrate rotation rate is 50 Revolutions Per Minute (RPM). The base pressure in the deposition chamber was 2.7×10^{-8} mbar and the working gas (Ar of a 99.9999% purity) pressure was 2.67×10^{-3} mbar at a flow rate of 50 standard cubic centimeters per minute (SCCM). The effective deposition rate is within 0.15–0.3 nm/s.

2.1.4. Rh, Re, Os, and Ir samples fabrication

Regarding the fabrication of the Rh, Re, Os, and Ir samples, they were prepared by magnetron sputtering, using Helmholtz-Zentrum Hereon in-house designed sputtering facility. The Rh, Os, and Ir samples were single layers on Si substrates but the Re sample had additionally a C capping. The power settings, deposition rates, and the nominal thicknesses are given in Table 2.

Table 2. Key parameters relevant to deposition of the Rh, Re, Os, and Ir samples

Parameter	Material				
	Ir	Os	Re	Rh	C
Target sputter power/watt	100	80	80	80	300
Deposition rate/nm s ⁻¹	0.12	0.08	0.08	0.18	0.003
Nominal thickness/nm	43	50	40	50	5

The chamber was evacuated to a base pressure below 1×10^{-7} mbar before the start of the deposition process. All sputtering targets had a purity of at least 99.9%, and were 3 inches in diameter. The metallic films were deposited on Si substrates (20 mm × 60 mm × 0.7 mm) that have a native oxide layer and a (100) orientation parallel to the substrate normal. The substrates were mounted on a custom-made substrate holder and were rotated at a rate of 1 Hz during deposition to obtain homogenous film thickness. The distance between the target and the substrate was 14 cm. Ar gas of purity 99.99999% at a flow rate of 28 SCCM was used as sputtering gas and the sputtering pressure was maintained at 2×10^{-3} mbar. The thin films were grown from the sputtering targets using DC modes.

2.1.5. Pd and ZrO₂ samples fabrication

For the fabrication of Pd and ZrO₂ samples, Pd and ZrO₂ layers were deposited onto Si wafers using sputtering techniques. The substrates are with a diameter of 25.4 mm and a thickness of 6.35 mm. The surface flatness of the substrates is characterized at $\lambda/10$ at a wavelength of 632.8 nm, and the surface roughness is measured to be less than 0.2 nm root mean square (RMS). For the deposition of the Pd coating, a Ti adhesion layer was first applied on the substrate. This process created an interfacial layer of titanium oxide between the substrate and the Pd layer. In contrast, for the ZrO₂ layer, a native oxide layer of Si is anticipated at the interface between the sputtered materials and the Si substrate. Both coatings were characterized using atomic force microscopy (AFM). Table 3 gives additional information on the Pd and ZrO₂ samples.

Table 3. Selected parameters relevant to the Pd and ZrO₂ samples

Parameter	Sample	
	Pd	ZrO ₂
Estimated layer thickness/nm	48.6	42.5
Surface roughness/nm (RMS) (1μm × 1μm)	0.60	0.47
Ti adhesion layer thickness/nm	4.4	-

2.1.6. Cr, Ni, and Pt samples fabrication

For the investigated Cr, Ni, and Pt coatings, they were fabricated by imec. Considering the Cr and Ni coatings, prior to the physical vapor deposition (PVD) of the metallic films, the 300 mm

Si wafers received an Ar-preclean to remove the native oxide, while no pre-treatment was done of the substrate before Ni deposition. The two films were deposited with a nominal 30 nm thickness by means of DC magnetron Ar ion sputtering in a 300 mm cluster system with base pressure below 1×10^{-7} mbar and operating at room temperature. The within-wafer deposited thickness uniformity is expected to be less than 3% (1 standard deviation).

As to the Pt sample, a Pt thin film with a nominal thickness of 30 nm was coated on a Si substrate using DC magnetron sputtering. The sputtering system base pressure was estimated to be 10^{-7} mbar. Ar was used as process sputtering gas with working pressures in the range 1×10^{-4} mbar to 5×10^{-3} mbar. The film is expected to be polycrystalline and the RMS surface roughness value is < 0.1 nm.

2.1.7. Nb sample fabrication

In respect of the Nb sample, it was coated at the Physikalisch-Technische Bundesanstalt (PTB) using a UNIVEX cluster sputter system on a standard 3-inch Si-Wafer. The wafer was subjected to pre-cleaning for 2 minutes using Ar plasma at a power of 50 watts. The nominal system of the sample is the following: HfTi (3 nm)/Nb (40 nm)/HfTi (4 nm)/Si substrate. Prior to magnetron sputtering, the Nb target was conditioned for 2 minutes with the shutter closed with Ar gas flow rate of 30 SCCM and power of 500 watts. The HfTi target was also conditioned for a duration of 3 minutes with the shutter closed with 20 SCCM Ar flow rate under a power of 50 watts. The HfTi target composition (weight) ratio is 50% – 50%. HfTi was sputtered with the same parameters as those of conditioning, yielding an approximate deposition rate of 6 nm/min. Nb was sputtered with Ar flow rate of 30 SCCM and power of 500 watts, yielding an approximate deposition rate of 43 nm/min. For all of the mentioned processes here, the working pressure is 1×10^{-2} mbar.

2.1.8. Hf and W samples fabrication

The Hf and W samples were deposited at the Center for X-Ray Optics (CXRO). The deposition system uses 3 magnetron sputter guns with 4-inch diameter targets. The base pressure was approximately 10^{-7} mbar and the substrates were standard 100 mm Si wafers. Considering the Hf sample, it has the following nominal structure: C capping (5 nm)/Hf (40 nm)/Ru underlayer (3 nm)/Si substrate. The Hf and C layers were grown using DC sputtering with a power of 100 watts. The Ru underlayer was deposited at 100 watts with RF mode. The Ar sputtering gas pressure was 2.7×10^{-3} mbar.

For the W sample, its nominal structure consists of the following: C capping (5 nm)/W (40 nm)/Ru underlayer (3 nm)/Si substrate. The W layer was deposited using RF sputtering at 90 watts. The Ru base layer was deposited at 100 watts and the C capping layer was deposited at 70 watts, both materials using DC mode. The Ar sputtering gas pressure was 4×10^{-3} mbar.

2.1.9. Au sample fabrication

For the Au sample, it was coated by DC magnetron sputtering at Helmholtz-Zentrum Berlin für Materialien und Energie (HZB), using a deposition system made by Bestec GmbH (Berlin, Germany). Using an 8-inch Au-target, a 35 nm coating of Au was sputtered on a standard 100 mm Si wafer. The Ar sputtering gas pressure was 3×10^{-3} mbar. The coating thickness $35 \text{ nm} \pm 1 \text{ nm}$ was measured using a Bruker Dektak stylus profiler.

2.2. Reflectometry using synchrotron radiation

Reflectivity data were measured using reflectometers of three different beamlines in two storage ring facilities. The soft X-ray radiometry beamline of the PTB (SX700 beamline) and Optics Beamline of HZB in the storage ring facility BESSY II, in addition to the EUVR beamline in

metrology light source (MLS). Table 4 specifies the beamline(s) used for measuring each sample, with references detailing the technical characteristics of each beamline.

Table 4. The studied samples beamline allocation. Details about the beamlines are available in the references therein.

Sample	Beamline	Beamline Technical Details
Sc, V, Ni, Nb, Mo, Pd, Hf, W, Re, Os, Ir and Au	Optics Beamline	Refs. [76–78]
Cr, Co, Rh, Pt and ZrO ₂	SX700	Refs. [74,79,80]
Rh	EUVR	Refs. [74,79–81]

All measurements were carried out in linear S-polarized radiation geometry and the reflectometers have GaAsP photodiode detectors. In each experimental setup, one detector was used to measure both incident and reflected radiation. This minimizes systematic errors by ensuring an identical response when measuring normalized reflectivity. Photodiode readings were dark-current corrected and normalized to a simultaneous monitor diode to account for storage ring current variations. Finally, the relative reflectance is obtained from the ratio of the normalized reflected to normalized incident signals.

3. Modelling formalism and determined optical constants

Reflectometry is a robust method for determining optical constants and is compatible with substrates, thin films, and MLMs. The main difficulties for reflectometry when determining optical constants are the requirements for samples to have minimal morphological and interfacial defects, and the need to solve an inverse-problem. The two difficulties are intertwined, but the latter is the focus now. In the relevant inverse-problem, the optimization's objective is to simulate the measured reflectivity data. For simulations, we assume a plausible model that describes the sample, based on which Parratt's formalism is used to calculate dynamical reflectance [82]. The effects of interfacial imperfections are estimated with Névot–Croce damping coefficients [83], which hold that the vertical heights of surface roughness are normally distributed [84]. Constructing the best-fit solution from the assumed expectation ranges of the problem is assisted with Markov chain Monte Carlo (MCMC)-based Bayesian inferences [85,86]. The stopping criterion here is the prolonged lack of major improvements in the fits with additional iterations. At that point, we assume convergence if the majority of the MCMC chains exhibit similar behaviour and explore the same region of parameter space. The formalism used here for determining optical constants was presented and discussed in detail before [74].

The optical constants among other target parameters of the relevant inverse-problems here are referred to in our study as sampling parameters. The adopted MCMC-based Bayesian inferences (ideally) explore the entire parameter space given the assumed expectation ranges. That returns a distribution correlated with a modal value for each target parameter, instead of estimated single best-fits as in classical optimization methods. The calculated distributions serve for projecting estimated confidence intervals [85]. The advantages of MCMC-based Bayesian inferences are discussed elsewhere [87].

It is to be noted that our aim in essence is the determination of optical constants, not the high-resolution modelling of coatings' structures. Our models are simplified depictions of the samples, that allow for a plausible description of the measured reflectivity. In our models, ideal lateral and vertical homogeneities are assumed for the layers. That can hardly be the case of real structures, where defects such as impurities and transitional non-stoichiometric profiles are often found. Interfaces often have particularly complex composition and density gradients due to interdiffusion. However, adapting more realistic models requires electromagnetic field solvers, employing finite element methods (FEMs). FEMs are needed to enable spatial discretization of the complex geometry. An alternative approach can be realized with assuming additional

(transient) layers (*e.g.*: ref. [88]). However, that could make the inverse-problems here intractable, or it might increase the risk of over-fitting. To mitigate the formalism's simplifications, expanded error parameters are used [74,89]. Also, parts of the experimental data were not attempted to be analyzed, rather omitted and are not shown in our work. For example, but not limited to, in the vicinity of the total external reflection region below a grazing angle of 3° to 6° , the beam's footprint is enlarged due to geometrical projection. Arguably, if such data are included, the made assumptions might lead to computational artifacts given the expected nonuniformity in surface layers and the (unaccounted for) complexity of interfacial imperfections.

The targeted optical constants are floating parameters here, and initialized around the CXRO's tabulations [27,28,90]. For modelling the samples, unless otherwise stated, the optical constants of the Si substrates and the introduced carbonaceous contamination layers are fixed from CXRO database [27,90], similarly for the optical constants of Ru and Ti (TiO_2) regarding the samples with Ru or Ti seed layers.

3.1. Sc sample

Sc thin films form oxidation layers at room temperature [91,92]. Larruquert *et al.* used C for protecting their Sc samples dedicated for EUV transmission measurements [93]. A 5 nm of C capping is sufficient to preserve the metallic state of a coating, even for highly reactive Y [94]. To avoid oxide formation, we also use C capping in addition to Ru as an interdiffusion barrier with the substrate. An interdiffusion barrier with the substrate's native oxide is needed to avoid the

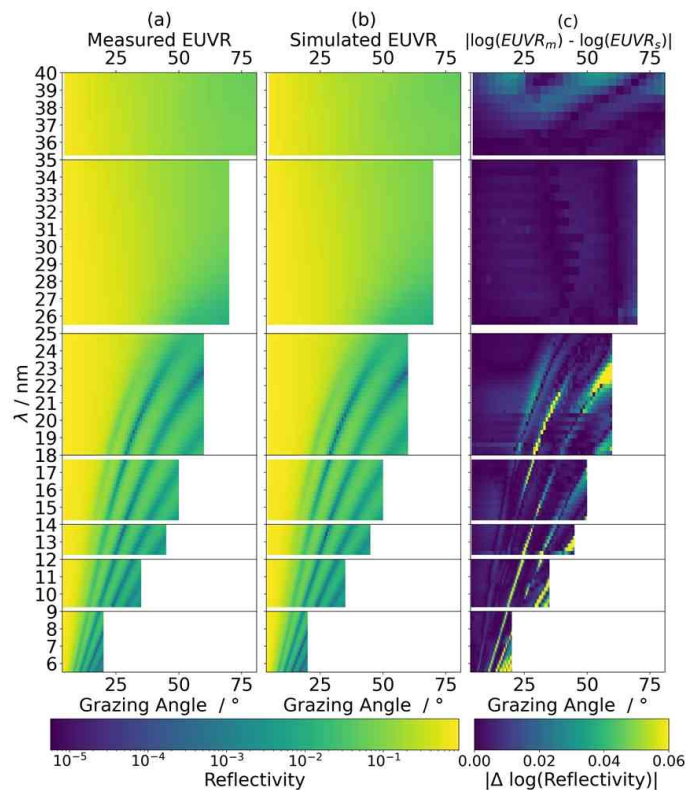


Fig. 1. Processed interpolated mapping of measured and simulated reflectivity data of the Sc sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations. Plotted using Ref. [97].

growth of a buried oxidation layer. Ru is a well-known interdiffusion barrier and is also used as a capping material [95,96]. The nominal system of the sample is as follows: C capping (5 nm)/Sc coating (40 nm)/Ru underlayer (3 nm)/Si substrate.

In Fig. 1, the measured reflectivity from the sample in the spectral range 5.5 nm – 40 nm is shown. For the purpose of determining optical constants, the model-based simulations rely on the nominal fabrication settings. A trilayer (on a substrate) system is assumed with the thickness of the capping fixed as 5.0 nm. The data were divided into two parts for a better adjustment of the sampling process. The 1st part consists of the data measured between 5.5 nm and 25.0 nm. The 2nd part consists of the rest. This partition is effective in reducing the computational effort. Also, we intend to stabilize the simulation of the data measured at the longer wavelengths ($\lambda > 25.0$ nm) with the calculated model from shorter-wavelength data. The sensitivity of shorter-wavelength data to the geometry of the sample's system is higher, especially for the buried layers.

For the 1st part, floating variables included the optical constants of C and Sc, the thicknesses of the Sc and Ru layers, and the interfaces roughness coefficients. After arriving at a stable solution in the simulations, the modal values for the Sc and Ru layers' thicknesses are 40.2 nm and 3.6 nm, respectively. The simulated reflectivity is in very good agreement with the measurements as indicated by Fig. 1. Regarding the residual plots of Fig. 1(c), we define residuals in our work as the absolute difference between the logarithms of the measured and simulated values. All residual maps here have their scales clipped below the real maxima. This allows better discernibility of simulations' deficiencies apart from the typical residual peak regions, such as troughs of fringes and the tails of reflectivity profiles. In such regions the reflectance is often minimal.

For determining Sc optical constants in the range 25.25 nm – 40.0 nm, the optical constants of the capping layer were fixed from the data shown in Fig. 2. That data is determined from another C coating with a nominal thickness of 40 nm. This coating was fabricated using the same sputtering setup of the 5 nm capping layer, and from the same sputtering target. These measures are necessary to further mitigate the large dimensionality of the inverse-problem. Which is concurrent with reduced sensitivity for the optical constants, given the smaller penetration depths for the longer wavelengths. Figure 3 shows the relative uncertainties of the capping layer's optical constants between 14.0 nm and 25.0 nm. In Fig. 3, the relative uncertainties increase

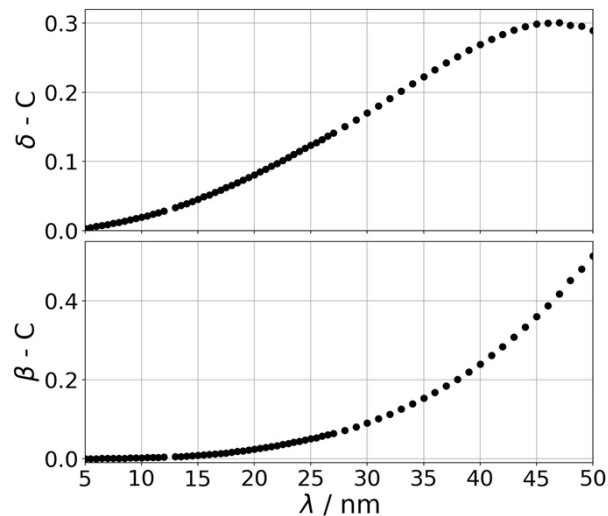


Fig. 2. Determined optical constants for the 40 nm-thick C layer in the spectral range 5.0 nm – 50.0 nm. Top: profiles of the refractive component. Bottom: profiles of the extinction coefficients.

towards ~25 nm. This behaviour is particularly problematic given coinciding disappearance of the fringes observed in the reflectivity profiles (Fig. 1(a)) with increasing wavelengths. That leads to non-uniqueness in the solution of the inverse problem. Consequently, when the inverse-problem is left unadjusted, strong cross-correlations with other parameters further increase the uncertainties and render the inverse problem unstable, or intractable.

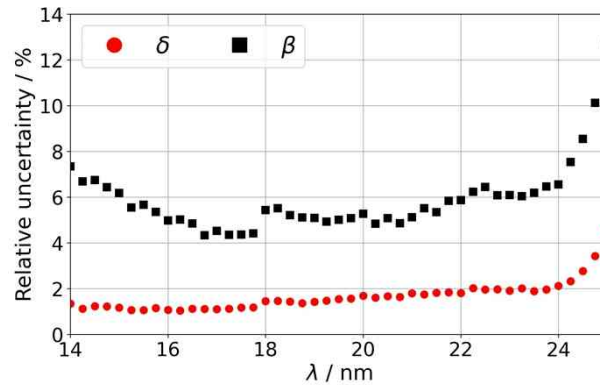


Fig. 3. The relative uncertainty for the calculated optical constants of the capping layer in the spectral range 14 nm – 25 nm.

In such cases, user intervention in the form of data partitioning and parameter constraints can improve stability. For this, and to ensure a smooth transition at the boundary wavelength (25.0 nm), given the data from Fig. 2, the dispersive components and the extinction coefficients were scaled with factors of 1.268 and 1.426, respectively. These factors were determined from the ratios between the sampled capping's optical constants and those shown in Fig. 2 at (the boundary) wavelength of 25.0 nm. Equation (1) illustrates for the refractive components:

$$\text{Scaling factor}_\delta = \frac{\delta_{\text{capping}}(\lambda = 25 \text{ nm})}{\delta_{40 \text{ nm coating}}(\lambda = 25 \text{ nm})} \quad (1)$$

The model was fixed as calculated from the shorter-wavelength data, except for the uppermost roughness parameter. Although we took optical constants of a C thin film coated using the same setup as those of the Sc capping layer, the carbonaceous layers are not expected to be exactly the same. C is known for its pronounced allotropism. The optical constants of 40 nm C thin film are not necessarily identical to those determined from a 5 nm capping layer. Moreover, contamination and other surface effects are assumed to significantly influence the optical constants of the capping layer. For these reasons, the uppermost roughness parameter was left as floating variable. The deficiencies in the assumed model could be partially mitigated given the additional degree of freedom.

The simulations considering the 2nd part data are also close to the measurements, as shown in Fig. 1, but discrepancies are observed at and above 38.0 nm. That is noticeable when examining the residual mapping shown in Fig. 1(c). Our calculated optical constants of Sc are plotted in comparison with literature data in Fig. 4 [27,28,98–100]. The determined optical constants of the capping layer are shown in Fig. 5. The absorption peak observed in the extinction coefficients is expected given the known $M_{2,3}$ edge [100]. The grey shaded region shows the Confidence Interval (CI) with $\pm 3\sigma$ (three standard deviations). Throughout our report, uncertainties are plotted as $\pm 3\sigma$.

For the dispersive parts, our results for Sc are in excellent agreement with the shown literature [27,28,98–100]. For the extinction coefficients, we have excellent agreement with the updated CXRO data and Fernández-Perea *et al.* results till ~28 nm, and above ~34 nm. In comparison

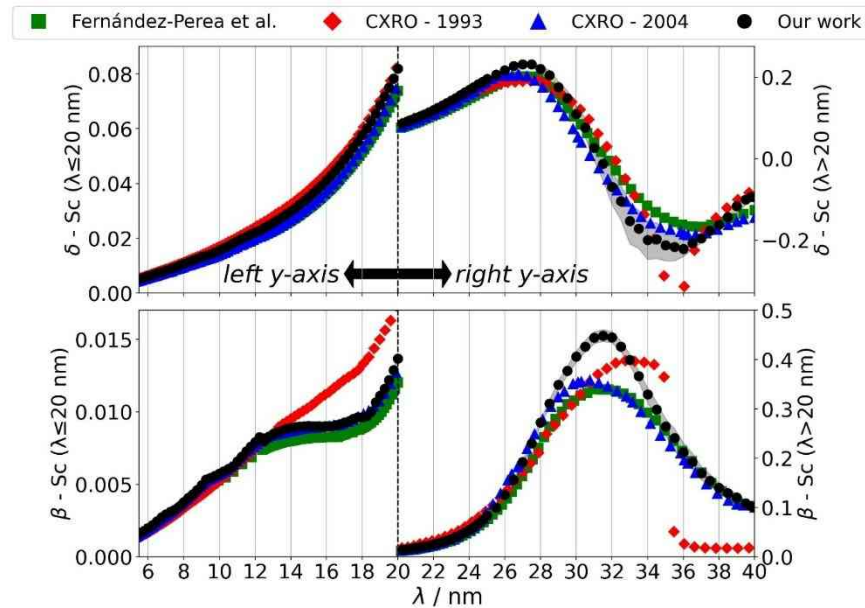


Fig. 4. Determined optical constants for Sc in the spectral range 5.5 nm – 40.0 nm. Optical constants from literature are shown for comparison [27,28,98–100]. Left y-axes: $\lambda \leq 20.0$ nm; right y-axes: $\lambda > 20.0$ nm.

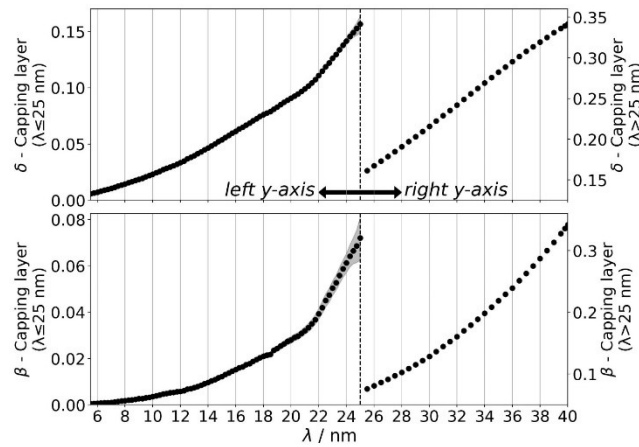


Fig. 5. Optical constants of the Sc carbonaceous capping layer determined in the 5.5 nm – 25.0 nm spectral range. Data for $\lambda > 25.0$ nm were obtained by scaling values from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 25.0$ nm; right y-axes: $\lambda > 25.0$ nm.

with these two datasets [27,28,98–100], the absorption peak's scale at ~ 31.8 nm in our data is clearly different. The calculated optical constants for the capping layer depict smooth increasing functions as expected from C in this range [74].

3.2. V sample

The fabricated V sample is nominally a trilayer system, consisting of the following layers, descendingly: C capping (4 nm)/V coating (40 nm)/Ru underlayer (3 nm)/Si substrate. Capping

is particularly important here since V oxidizes at room temperature [101,102]. We measured reflectivity from the sample in the spectral range 5.0 nm – 40.0 nm, as shown in Fig. 6(a).

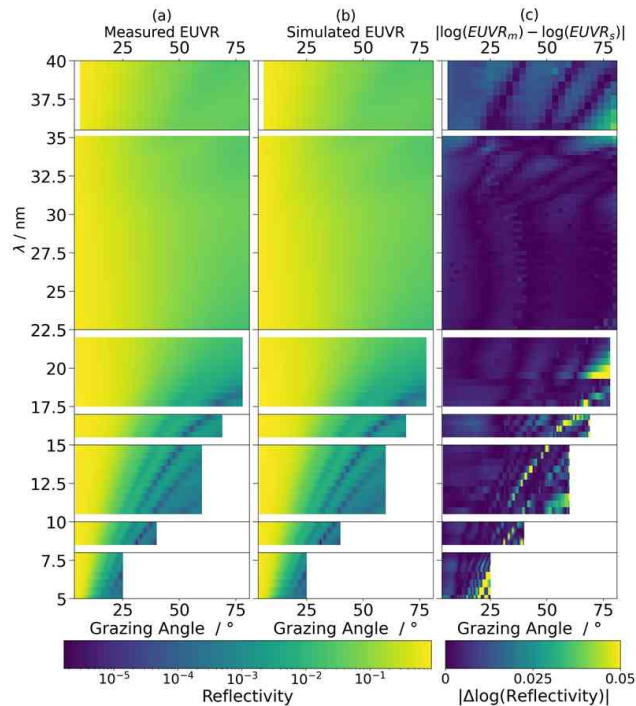


Fig. 6. Processed interpolated mapping of measured and simulated reflectivity data of the V sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

The needed sample's model for determining optical constants is based on the nominal fabrication settings of the trilayer system. Similar to the analysis scheme of the Sc sample, the analysis approach divides the experimental data into two parts. For the first part, the measurements in the range 5.0 nm – 19.0 nm, the optical constants of both of the carbonaceous capping layer and those of V were floating variables, in addition to the structural parameters. Upon arriving at a stable solution, the calculated thicknesses of the layers are the following: C capping (4.3 nm)/V coating (38.6 nm)/Ru underlayer (3.5 nm).

For the 2nd part, the data measured in the range 19.5 nm – 40.0 nm, the structural model was fixed as calculated from the data collected at the lower wavelengths, except for the capping layer's thickness and the two uppermost roughness parameters. Also, the optical constants of the capping layer were scaled from the data shown in Fig. 2. The dispersive components and the extinction coefficients were scaled with factors of 0.962 and 1.139, respectively. The two factors were determined from the ratios between the sampled capping's optical constants and those shown in Fig. 2 at wavelength of 19.0 nm. The calculated thickness of the capping layer from the data measured in the range 19.5 nm – 40.0 nm is 4.6 nm. The slight mismatch between the calculated thicknesses of the capping layer given the different wavelength ranges is rather expected. The discussion in sub-sec 3.1 regarding the variability of C optical data among different coatings is also applicable here. Basically, the simulations are quite close to the measured data, as manifested by Fig. 6, with the residuals plots shown in Fig. 6(c). Our calculated optical constants from the V coating and its capping are shown in Fig. 7 and Fig. 8, respectively.

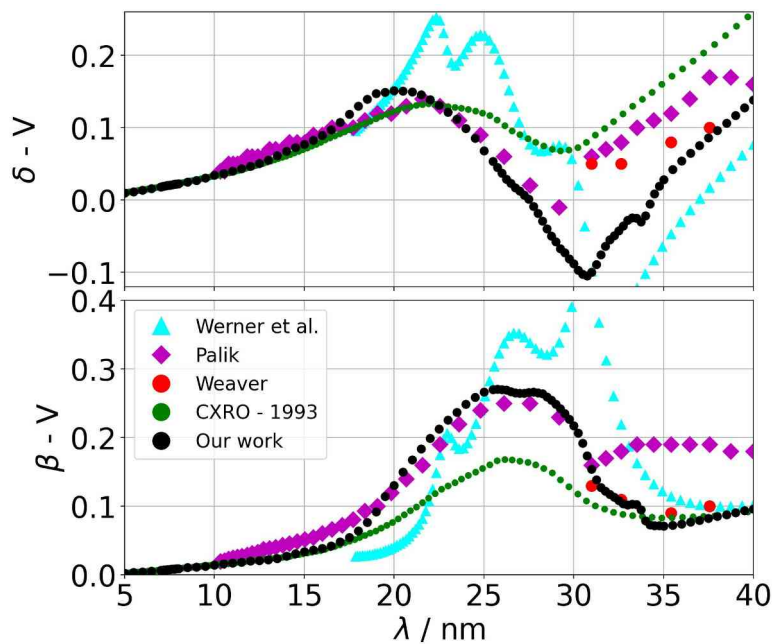


Fig. 7. Determined optical constants for V in the spectral range 5.0 nm – 40.0 nm. Optical constants from literature are shown for comparison [27–30,33,103].

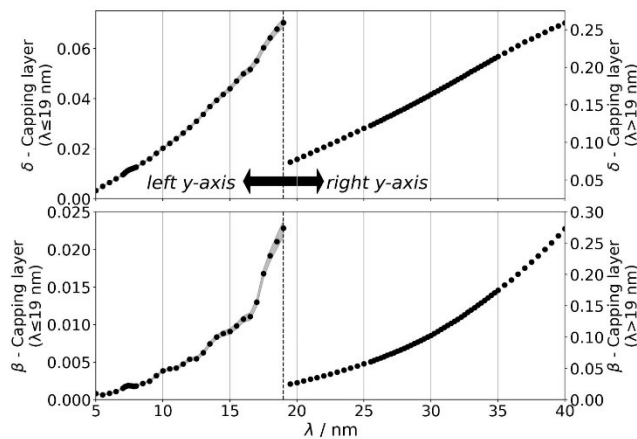


Fig. 8. Optical constants of the V carbonaceous capping layer determined in the 5.0 nm – 19.0 nm spectral range. Data for $\lambda > 19.0 \text{ nm}$ were obtained from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 19.0 \text{ nm}$; right y-axes: $\lambda > 19.0 \text{ nm}$.

For our results shown in Fig. 7 with comparison with external literature [27–30,103], the data of Palik and Weaver were imported via IMD database [29]. Our optical constants are in general agreement with those of CXRO between 5 nm and ~16 nm, also for the extinction coefficients in the investigated range above ~34 nm [27]. In comparison with Palik, our results here are generally close considering the spectral range ~10 nm to ~28 nm [29,30]. The data of Weaver are also close to our results for the extinction coefficients in the available range [29]. However, Werner *et al.* data (ref. [103], imported via ref. [33]) scale differently and reveal fine-structure that is not observed in any literature. For our data, the uncertainties are smaller than the plotted

dots in Fig. 7 and do not influence the comparison with literature values. For the remainder of the report, uncertainties of our optical data are shown only when they noticeably exceed the marker (black dot) size.

For the optical constants of the carbonaceous capping layer, generally, both profiles of the refractive components and extinction coefficients trace increasing functions given the wavelength as shown in Fig. 8.

3.3. Cr sample

This sample consists of a Cr layer with a nominal thickness of 30 nm on a Si substrate. Reflectivity data were collected in the spectral range 5.0 nm – 20.0 nm from the sample, as shown in Fig. 9(a).

Cr thin films are known to develop oxide layers when exposed to air, our sample was exposed to air for more than two years. Delmotte *et al.* introduced an oxide layer with the stoichiometry Cr_2O_3 for modelling their Cr thin films and membranes [104]. Wang *et al.* reported the existence of Cr_2O_3 among other chemical species on sputtered Cr thin films [105]. For modelling our sample, we assume the presence of an oxide layer with the stoichiometry Cr_2O_3 on top of the Cr coatings. Additionally, a thin C contamination layer is also assumed to compensate for the contaminants on the optical surface. For solving the inverse-problem, a trilayer system on Si substrate – as shown in Fig. 10 – is adopted.

Ideally, in a problem targeting the optical constants from a material with an oxide on top, the optical constants of both the targeted material and its oxide are to be determined independently.

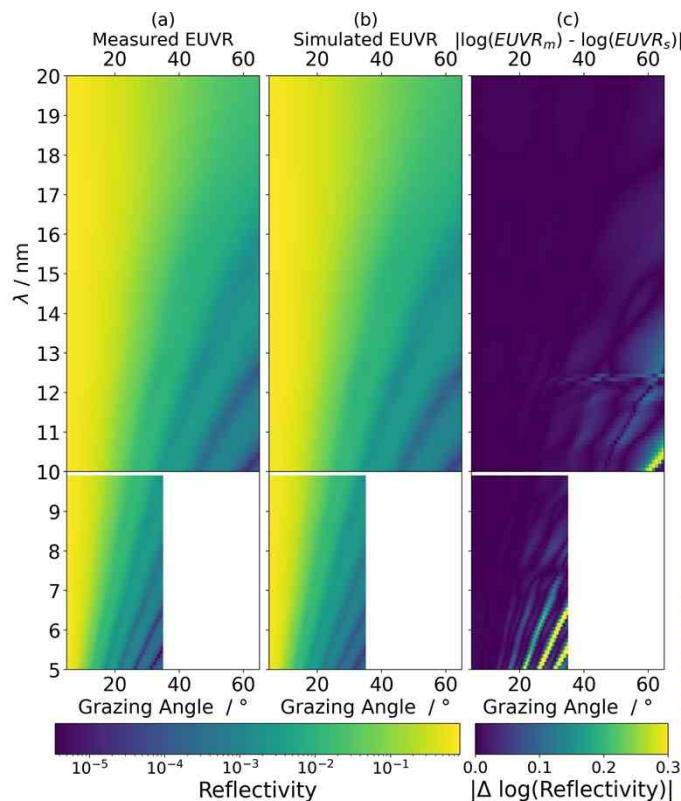


Fig. 9. Processed interpolated mapping of measured and simulated reflectivity data of the Cr sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

However, such an ideal approach doubles the number of free parameters in the problem. Even if the ideal approach is followed, the optical constants of the oxide layer are expected to be determined with relatively large uncertainties given the ultrathin nature of that surface layer. It is well-known that the optical contrast of a thin film is linked to its thickness. The optical constants of Cr and its oxide can be linked using the atomic scattering factor (ASF) through the independent atom approximation (IAA) (ref. [27]), where $\bar{n}$ as a function of the wavelength is given as:

$$\bar{n}(\lambda) = 1 - \delta(\lambda) + i\beta(\lambda) = 1 - \frac{r_e}{2\pi} \lambda^2 \sum_{j=1}^N k_j f(\lambda)_j \quad (2)$$

In Eq. (2), r_e is the classical electron radius, k_j denotes the number of atoms of type j per unit volume where $f(\lambda)_j$ stands for the complex ASF for an atom with type j , and is given as:

$$f(\lambda) = f_1(\lambda) - if_2(\lambda) \quad (3)$$

Where f_1 and f_2 in Eq. (3) relate to the dispersion and absorption of a propagating electromagnetic wave in a defined medium, respectively. Figure 10 explains how the ASFs of Cr were coupled between the Cr coating and its oxide layer. The ASFs of Cr were sampling parameters and those of O were fixed from CXRO [27]. The calculated thicknesses in the trilayer stratification were as follows: C (1.6 nm)/Cr₂O₃ (1.9 nm)/Cr (27.8 nm). The calculated density of the oxide layer was 4.48 gm/cm³, where the theoretical density of Cr₂O₃ is reported to be 5.22 gm/cm³ [106]. The oxidation layer modelling seems to be plausible then.

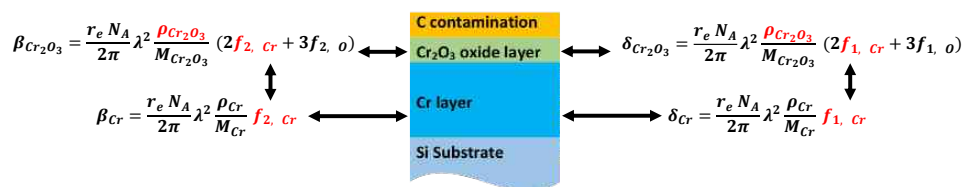


Fig. 10. Drawing of the assumed trilayer structure on Si substrate for the Cr sample. The symbols N_A , ρ , and M denote Avogadro constant, the mass density and the molar mass, respectively. Figure adapted from ref. [107].

This approach is an efficient solution to estimate the optical constants of the oxide layer in the inverse-problem. We used it for the case of Ta thin films previously [107], and the results were convincing. However, the validity of this approach is limited at and in the vicinity of absorption edges. The IAA approximation does not take into account chemical shifts and other effects that can be pronounced near atomic resonance energies. A discussion about the validity of the IAA in estimating optical constants is given in section 6.5 of ref. [74].

The simulated reflectivity is generally close to the measured as shown in Fig. 9. However, the residual peaks are noticed for troughs of the fringes in the spectral range 5.0 nm to ~7.5 nm. Also, for the highest oblique angles in the reflectivity data collected between 10.0 nm to ~11.0 nm, and for the region around 12.4 nm. Presumably, the latter is due to non-stoichiometric layers forming at the Si-Cr interface. Our calculated optical constants are shown in Fig. 11.

As shown in Fig. 11, our calculated optical constants converged close to the updated CXRO data (of 2018, refs. [27,104]). The resolved fine-structure's features between ~12 nm and 20 nm are in general agreement considering our data and those updated by CXRO [28]. This also supports the credibility of our modelling formalism given that we set the expectation ranges around those ASFs of the original tabulations of Henke [27], not the updated ones. Around and below ~11 nm, for the investigated spectral range our optical constants and those two datasets of

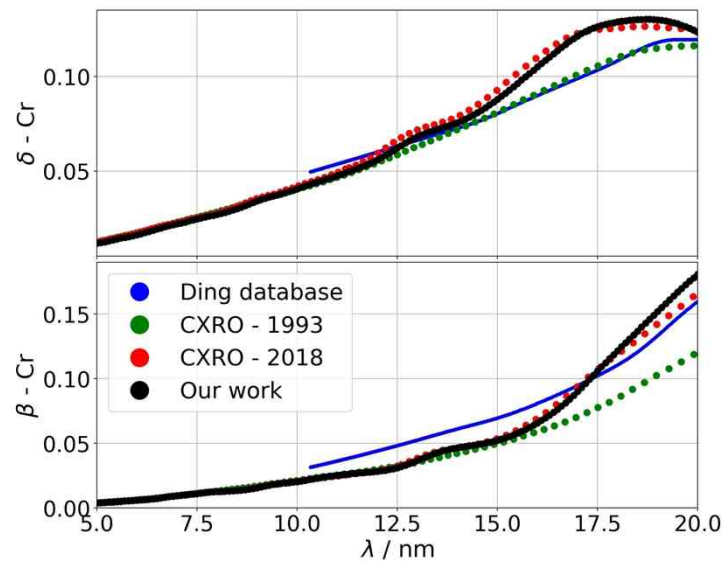


Fig. 11. Our determined optical constants for Cr in the spectral range 5.0 nm – 20.0 nm. Each profile of our data consists of 151 discrete data points. Optical constants from literature are shown for comparison [27,28,31]. Top: profiles of the refractive component. Bottom: profiles of the extinction coefficients.

CXRO are very close [27,28]. However, our results are inconsistent with those of Ding database [31]. That data was determined via reflection electron energy-loss spectroscopy (REELS) [108].

Throughout our work, data originating from publications posted on the Database of Ding's Microsolid Lab are cited in the text as Ding database [31], while the corresponding individual publications are listed separately in the reference list [108].

3.4. Co sample

The sample is a trilayer (on substrate) system with the following nominal thicknesses: C capping (5 nm)/Co layer (40 nm)/Ru underlayer (2 nm)/Si substrate. The Co sample was measured in the spectral range 5.0 nm – 9.0 nm. An interpolated representation of the measured data is shown in Fig. 12(a).

The region marked by the red rectangle in Fig. 12 is for omitted data from the fitting, due to low signal to noise ratios. Some omitted data points have values below 10^{-5} . For determining optical constants, in addition to the nominal structure of the sample, a thin SiO₂ layer is assumed between the substrate and the Ru underlayer. In the simulation, the optical constants of the SiO₂ layer were fixed from the PTB database [109]. Both optical constants of the C capping layer and the Co coating were sampling parameters.

Upon reaching a stable solution, the simulated reflectivity data was found to be generally close to the measurements as shown in Fig. 12(b). The calculated thicknesses of the tetralayer system were as follows: C (5.5 nm)/Co (40.4 nm)/Ru (1.9 nm)/SiO₂ (1.6 nm). The optical constants of Co and the carbonaceous capping layer are shown in Fig. 13 and Fig. 14, respectively.

Our calculated optical constants of Co are in general agreement with those of CXRO and Palik as shown in Fig. 13 [27,30]. The optical constants from our previous work shown in green were first shown in ref. [75]. However, our extinction coefficients show a distinct departure from linearity for the values between 6.5 nm and 8 nm, those of CXRO and Palik do not [27,30].

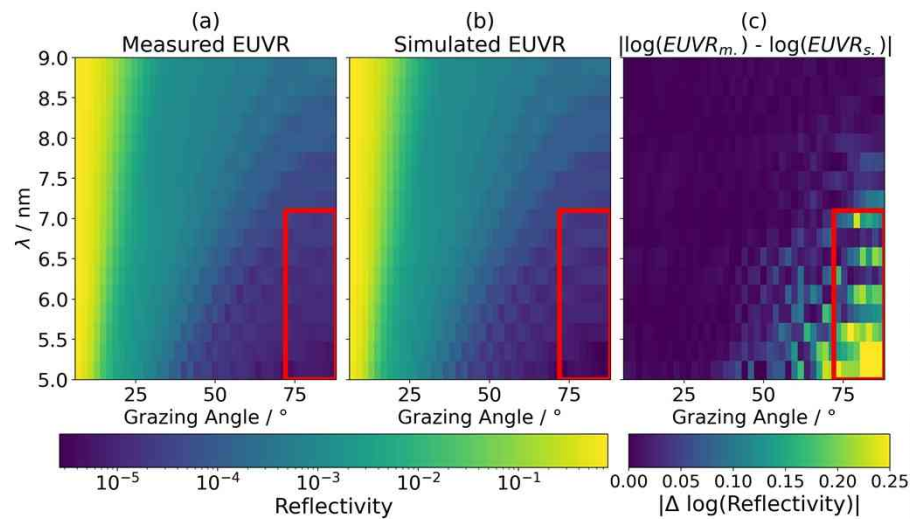


Fig. 12. Processed interpolated mapping of measured and simulated reflectivity data of the Co sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

The optical constants of Palik shown here were imported from IMD database [29]. In the overlapping range (8.0 nm – 9.0 nm) of our current and previous studies on Co, the results are in very good agreement. Figure 14 shows the calculated optical constants of the carbonaceous capping layer with their corresponding CIs. When the CIs of the extinction coefficients are considered, it could be argued that the slight increase noticed in the overlapping range (8.0 nm – 9.0 nm) of our current and previous studies is due to larger densification or deposition of

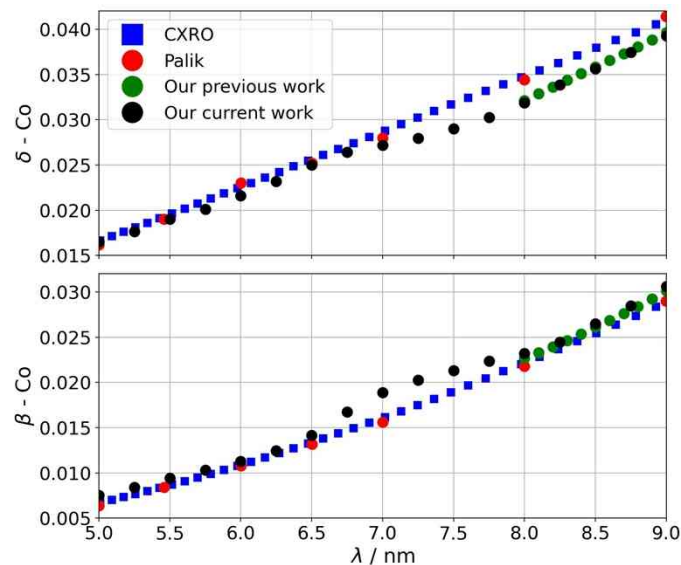


Fig. 13. Determined optical constants for Co in the spectral range 5.0 nm – 9.0 nm. Optical constants from our previous work and external literature are shown for comparison [27–30,75].

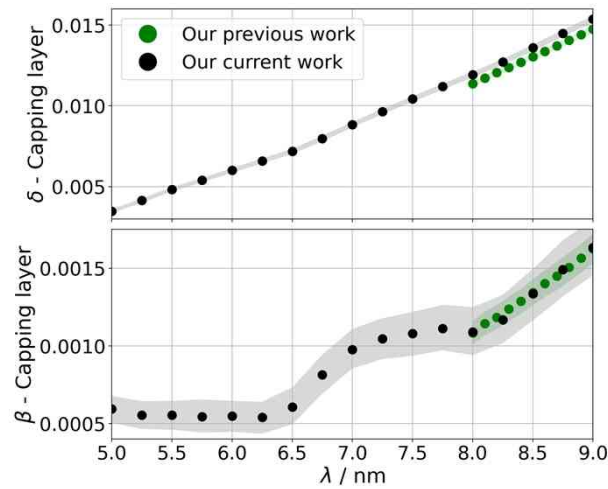


Fig. 14. Optical constants of the Co carbonaceous capping layer determined in the 5.0 nm – 9.0 nm spectral range. Optical constants from our previous work are shown for comparison [75].

contaminants. However, the optical constants of the capping layer are not presented as those of pure C. The nature of the carbonaceous capping layer was not investigated. This work focuses on determining the optical constants of elemental Co and the capping layer serves only to prevent oxidation.

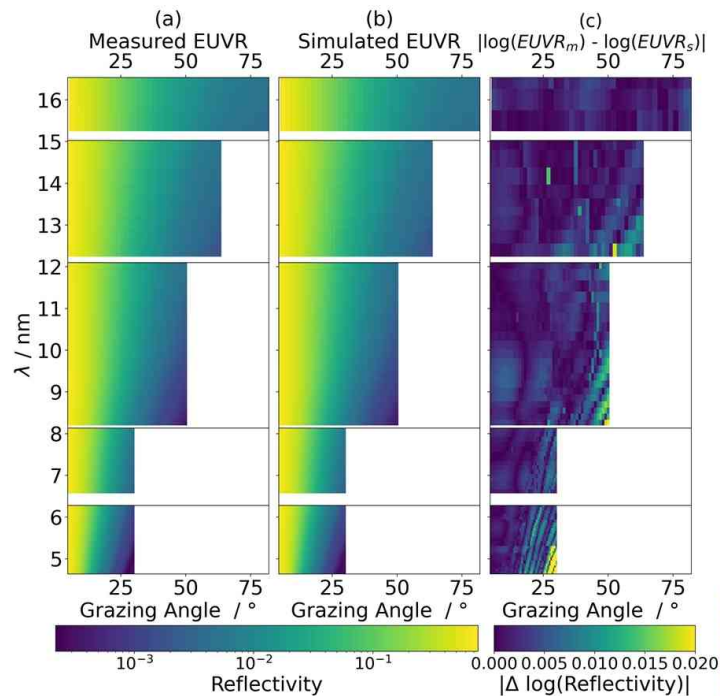


Fig. 15. Processed interpolated mapping of measured and simulated reflectivity data of the Ni sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

3.5. Ni sample

The fabrication settings of this sample targeted a 30 nm Ni layer on a Si substrate. The Ni sample was measured in the range ~4.6 nm to ~16.5 nm, as shown in Fig. 15(a). The gap in the vicinity of 6.45 nm is due to failed scans. Ni thin films are reported to develop thin oxide layers in ambient conditions [110,111]. Therefore, we assume the presence of an oxide layer on top of the film. The assumed model of the sample consists of four layers: carbon contamination, oxide layer, Ni coating on top of the SiO₂ layer. The latter's optical constants were fixed from PTB database [112].

The same IAA-based formalism used for the Cr sample is also applied here. The ASFs of Ni are coupled with the assumed oxidation layer with the stoichiometry of NiO₂. Upon arriving at a stable solution in the sampling process, the simulated reflectance of the sample is in good agreement with the measurements, as shown in Fig. 15. The calculated thicknesses of the four layers are as follows: C (1.6 nm)/NiO₂ (1.4 nm)/Ni (27.2 nm)/SiO₂ (1.2 nm). The calculated density of the assumed oxide layer is 6.1 g/cm³. Our calculated optical constants of Ni are shown in Fig. 16 in comparison with literature data [27,31,113–116].

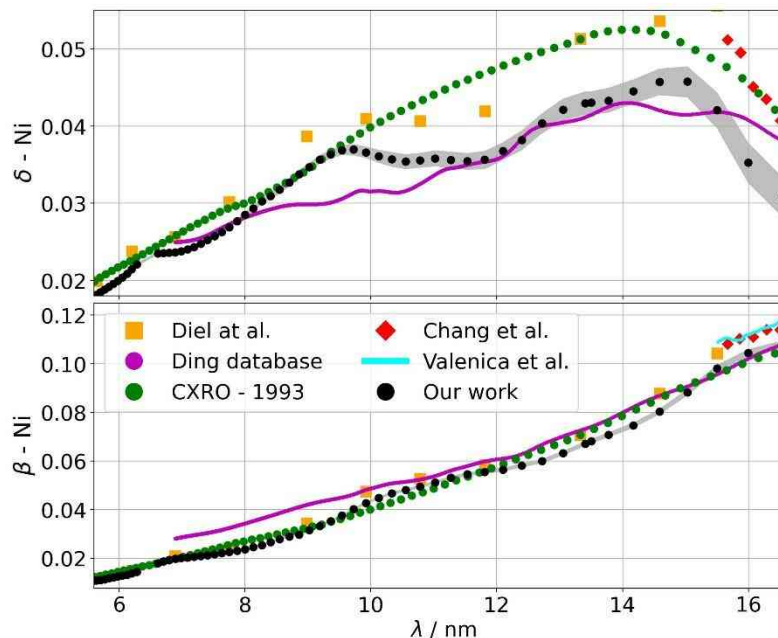


Fig. 16. Determined optical constants for Ni in the spectral range ~4.6 nm to ~16.5 nm. Optical constants from literature are shown for comparison [27,31,113–116].

For the extinction coefficients, our data seems closer to the cited literature than the case of refractive components [27,31,113–116]. For the refractive components, the inconsistencies between our results and the cited literature are noticed given dissimilar resolved features [27,31,113–116], especially after ~8 nm. For Fig. 16, the plotted data of Valencia *et al.* and Chang *et al.* were obtained via digitization of figures in refs. [113,114]. This procedure may affect the extracted values.

3.6. Nb sample

Nb thin films develop oxidation layers upon exposure to air [117]. Coating Nb directly on a passivated Si substrate in our case might lead to issues. The formation of Nb-silicide, which

is also intermixed with oxidation below the Nb film might form. Because of that, we opted for coating Nb between two ultrathin HfTi layers to prevent oxidation and potential silicide formation. HfTi layers are known to be compatible with Nb from their coupled use in Josephson junctions [118]. Based on the aforementioned, we coated the following trilayer system: HfTi (3 nm)/Nb (40 nm)/HfTi (4 nm) on a Si substrate. We present reflectivity data measured from the Nb sample in the spectral range 6.0 nm – 20.4 nm, as shown in Fig. 17(a).

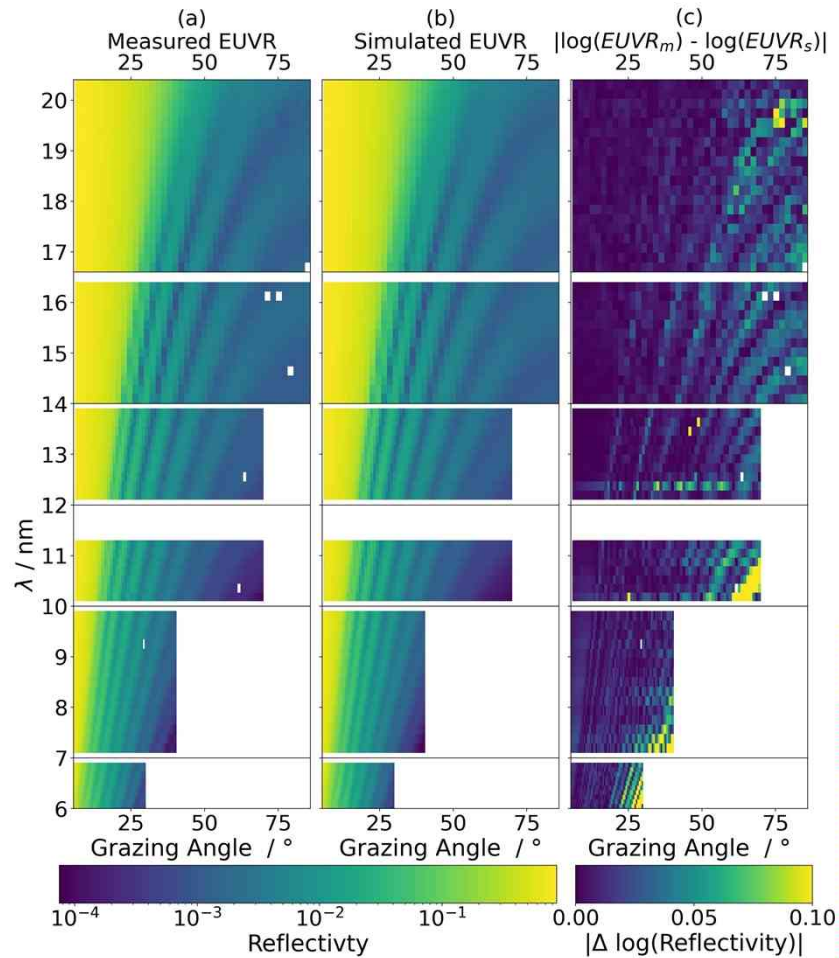


Fig. 17. Processed interpolated mapping of measured and simulated reflectivity data of the Nb sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

The absence of data in the vicinity of 11.7 nm results from acquisition failures during measurements. The clear modulations of Kiessig fringes (ref. [119]) in Fig. 17(a) do not indicate severe oxidation or strong interdiffusion, which is a good sign for the quality of the sample. For the simulations, we used a trilayer (on a substrate) system based on the nominal fabrication settings. The optical constants of both of Nb and HfTi were sampled, in addition to the geometrical parameters. The calculated thicknesses of the trilayer model are as follows: HfTi (5.2 nm)/Nb (39.6 nm)/HfTi (4.2 nm)/Si substrate. The simulated reflectivity data are in general agreement with the measurements as shown in Fig. 17(b). However, discrepancies appear in the tails of the reflectivity profiles for wavelengths $\leq \sim 11$ nm. This may be due to the simplification of assuming identical optical constants for both HfTi layers. The upper HfTi layer is exposed

to ambient conditions, whereas the buried layer may react with Si. The determined optical constants of Nb and HfTi are shown in comparison with external literature in Fig. 18 and Fig. 19, respectively.

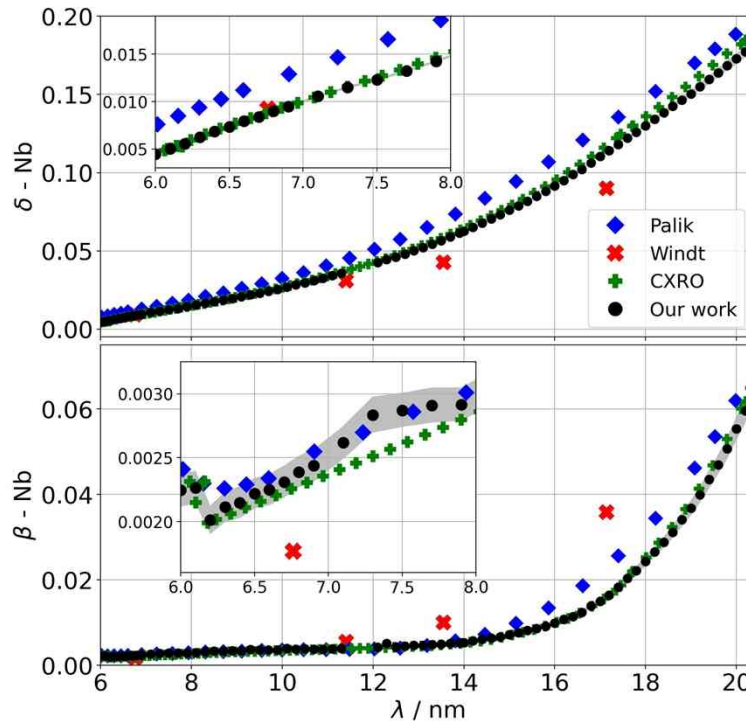


Fig. 18. Determined optical constants for Nb in the spectral range 6.0 nm – 20.4 nm. The plotted insets better show the data between 6.0 nm and 7.5 nm. Optical constants from literature are shown for comparison [27–30,120].

Our Nb optical constants are in very good to excellent agreement with the constants taken from CXRO and Palik [28,30]. Generally, the data of Windt *et al.* (ref. [120]) also indicate increasing trends, but exhibit differences with our data. Moreover, the noticed fine-structure in the subplots of Fig. 18 considering the extinction coefficients is expected, because of the reported Nb $-M_4$, and $-M_5$ levels around 6 nm [121].

For the determined optical constants of HfTi in Fig. 19, an accurate comparison with literature cannot be made due to the unknown mass density of the deposited HfTi layers. However, when assuming a mass density of 9.0 g/cm^3 , our dispersive components have a similar trend as those predicted by the IAA based on ASFs taken from CXRO data [28]. Though, they are slightly smaller. For the extinction coefficients, in comparison with CXRO estimated values (ref. [28]), inconsistencies are noticed given that our values are smaller in the spectral ranges 6.0 nm to ~ 13 nm and ~ 9 nm to 20.4 nm, but slightly larger between ~ 14 nm and ~ 17 nm.

3.7. Mo sample

Mo thin films are known to develop oxide layers at room temperature [122], because of that, capping is needed. A C capping layer with a thickness of 5 nm was coated on a Mo thin film with a thickness of 40 nm, all on a Si substrate. As shown in sub-Fig. 20(a), we measured the reflectivity from the sample in the spectral range 6.0 nm – 40.0 nm.

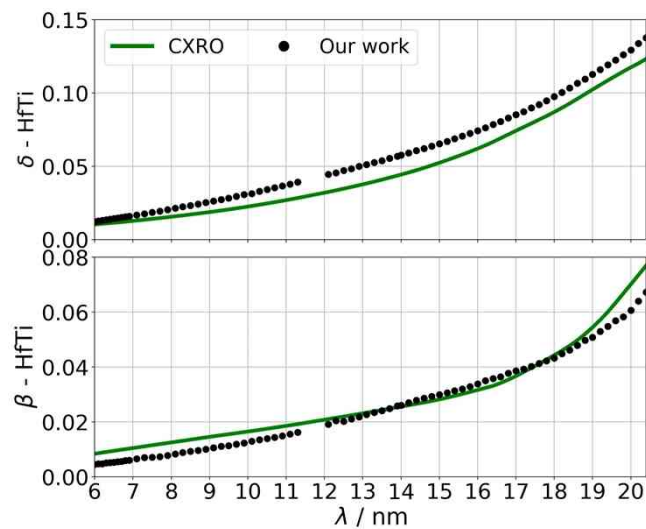


Fig. 19. Determined optical constants for HfTi in the spectral range 5.0 nm – 20.4 nm. Approximate optical constants from CXRO are taken via ref. [90].

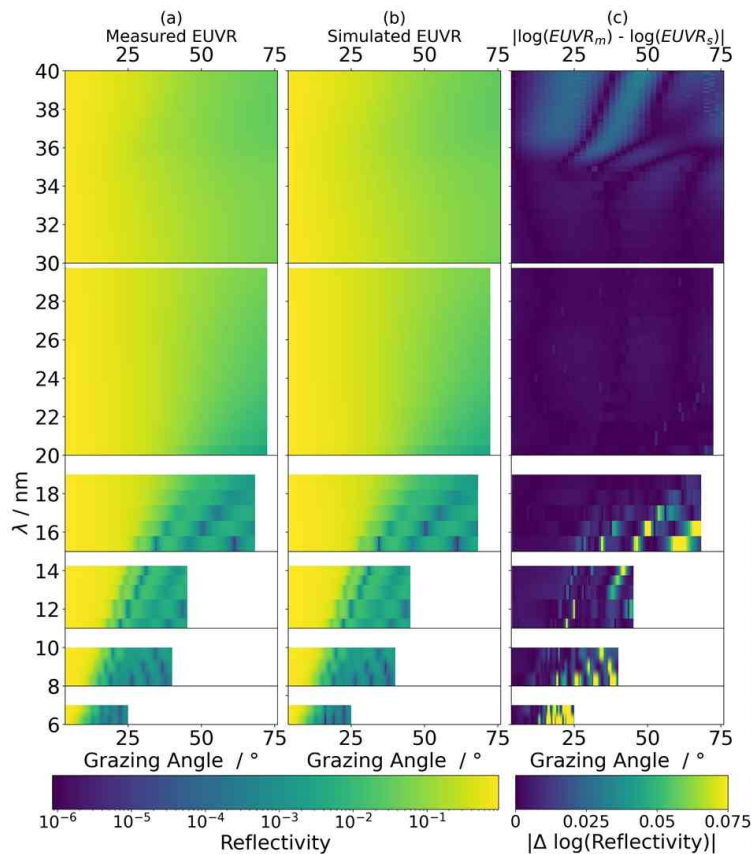


Fig. 20. Processed interpolated mapping of measured and simulated reflectivity data of the Mo sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

To determine the optical constants, for the range 6.0 nm – 21.5 nm, the optical constants of the capping and those of the Mo layer were sampled, besides the structural parameters. The calculated thicknesses of the sampled layers are as follows: C capping (6.04 nm)/Mo coating (40.06 nm). For the data measured above 21.5 nm, the calculated geometrical parameters were fixed and the optical constants of Mo were sampled. For the optical constants of the capping, they were scaled from the C data shown in Fig. 2. The refractive components and the extinction coefficients were scaled with factors of 0.792 and 0.915, respectively. These factors were calculated with dividing the sampled capping's optical constants and those shown in Fig. 2 at wavelength of 21.5 nm.

From sub-Fig. 20(c), it can be observed, the model does not fully simulate the experimental data accurately at certain wavelengths. Miss-fits are pronounced for the wavelengths measured at 6.0 nm. Also, for the data measured above ~34 nm. Yet, we present our results in the latter range given the reported issues in the available Mo data above ~25 nm [123]. The simulated reflectivity data are in excellent agreement with the measurements for the range 7.0 nm – 33.0 nm.

The determined optical constants of Mo are shown in Fig. 21 in comparison with our previous work and external literature [27–30,124–127]. The determined optical constants of the capping layer are plotted in Fig. 22. In our previous work, we determined the optical constants of Mo in the spectral range 10.0 nm – 19.8 nm [126]. As shown in Fig. 21, the optical constants from our previous work are overlapping with our current results.

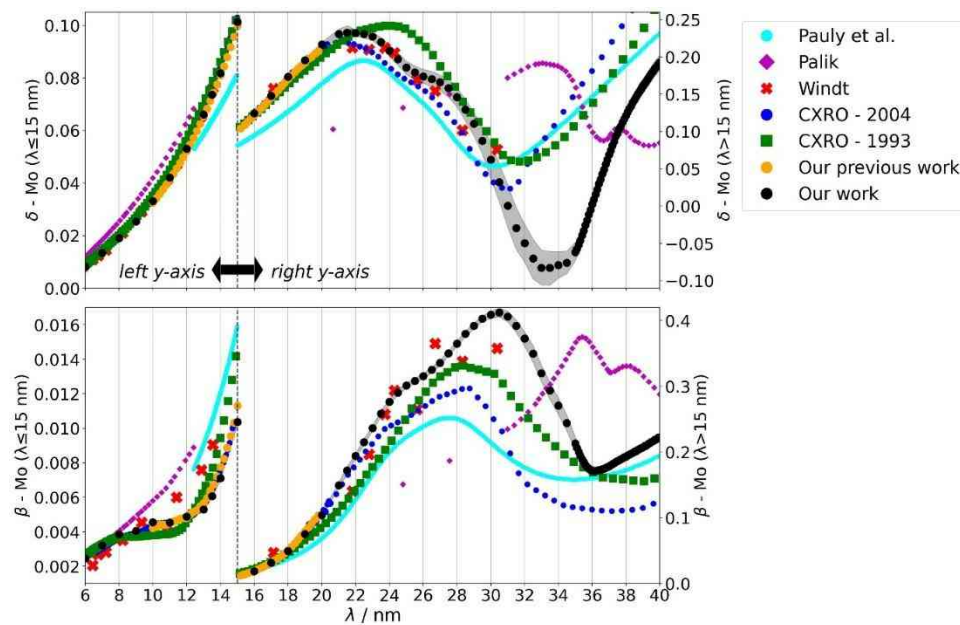


Fig. 21. Determined optical constants for Mo in the spectral range 6.0 nm – 40.0 nm. Optical constants from literature are shown for comparison [27–30,124–127]. Left y-axes: $\lambda \leq 15.0$ nm; right y-axes: $\lambda > 15.0$ nm.

Up to ~20 nm, both of our two datasets are in excellent agreement with CXRO updated data [28,124,125]. In the range between ~20 nm and ~30 nm, the dispersive components we determined diverge slightly from CXRO's data, but above ~30 nm the inconsistencies are significant [28,124,125]. For the extinction coefficients, our data peaks at ~31 nm and is generally inconsistent with CXRO data [28,124,125].

Pauly *et al.* data (ref. [127]) were determined using REELS, and they follow similar trends as those of CXRO [28,124,125]. However, they also show clear inconsistencies with our results above ~25 nm and ~30 nm for the for the extinction coefficients and the dispersive components,

respectively. For Windt's data (ref. [29]), they are in general agreement with our results up to ~ 20 nm, but they develop in a slightly different manner above ~ 20 nm. Regarding the quality of our simulations, even though good agreement is not achieved at all wavelengths, a 2nd reason for showing our results is to bring out possible issues with Palik's data [30]. The modulating fine-structure in Palik data is not observed in our data, or in the addressed literature [27–30,124–127]. Moreover, we highlight that our unprocessed reflection spectra at grazing angles do not show such modulations. That is noteworthy because reflection spectra measured at grazing angles resemble dispersion features.

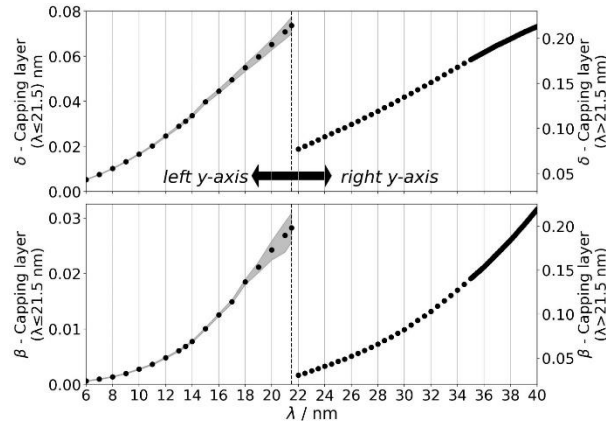


Fig. 22. Optical constants of the Mo carbonaceous capping layer determined in the 6.0 nm – 21.5 nm spectral range. Data for $\lambda > 21.5$ nm were obtained by scaling points from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 21.5$ nm; right y-axes: $\lambda > 21.5$ nm.

The determined optical constants of the capping layer are shown in Fig. 22. Similar to the optical constants of V capping layer, they also trace increasing functions given the wavelengths after 6 nm.

3.8. Rh sample

The nominal thickness of the Rh coating is 50 nm. The presented reflectivity data of Rh are for the spectral range 4.6 nm – 30.0 nm, shown in Fig. 23(a). For the determination of optical constants, a trilayer model of the sample is assumed. C contamination/Rh coating/SiO₂/Si substrate. The optical constants of the SiO₂ layer were fixed from the PTB database [109,112]. The density of the carbonaceous layer as a factor was a floating parameter additionally.

The measurements here were obtained using two beamlines at different time periods. The data for the spectral range 10.1 nm – 30.0 nm were measured first and then the rest. Because of that, two different sampling trials are used for the same sample. Upon sampling the presented data and arriving at quasi-stable solutions, the simulations are in excellent agreement with the measurements as shown in Fig. 23. The calculated thicknesses from the data collected in the spectral range above 10.1 nm are the following: C (1.2 nm)/Rh (49.7 nm)/SiO₂ (1.1 nm)/Si substrate, while for remaining data the following model is calculated: C (1.09 nm)/Rh (49.8 nm)/SiO₂ (1.89 nm)/Si substrate.

The determined optical constants of Rh are shown in Fig. 24. Maehara *et al.* published data determined using reflectometry and photoelectric yield measurements [128]. In Fig. 24, the legends Maehara *et al.* – EY and Maehara *et al.* – R denote the constants determined using photoelectric yield and reflectivity measurements, respectively.

Below ~ 15 nm, our profiles (Fig. 24) are in general agreement with CXRO and Palik data [27,30]. Above ~ 15 nm, our profiles diverge from those of Palik [30], and those of CXRO are the

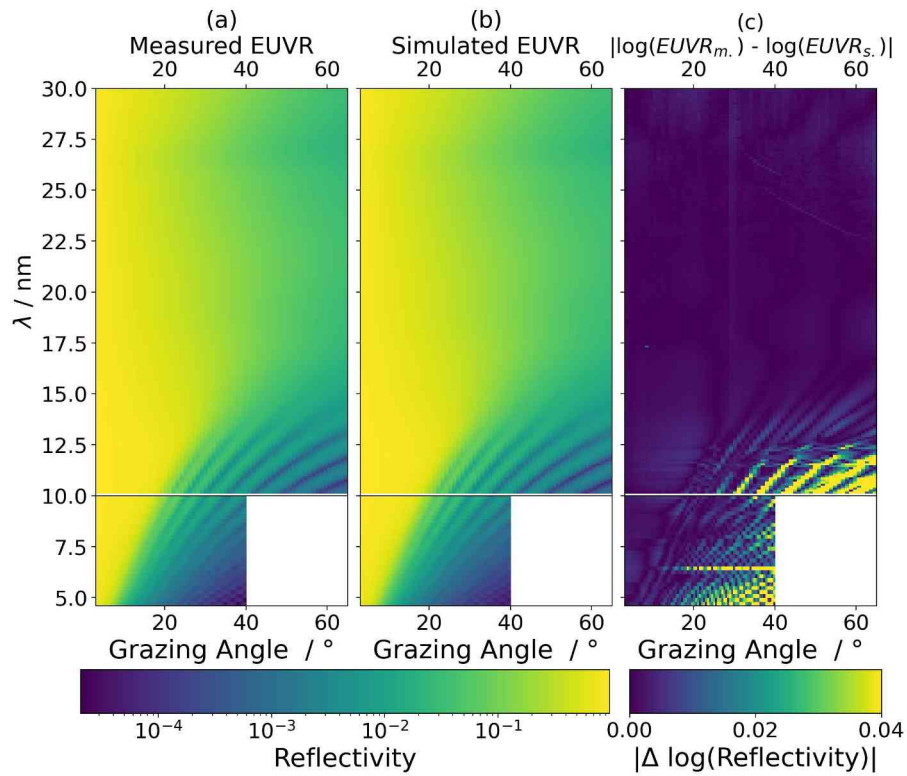


Fig. 23. Processed interpolated mapping of measured and simulated reflectivity data of the Rh sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

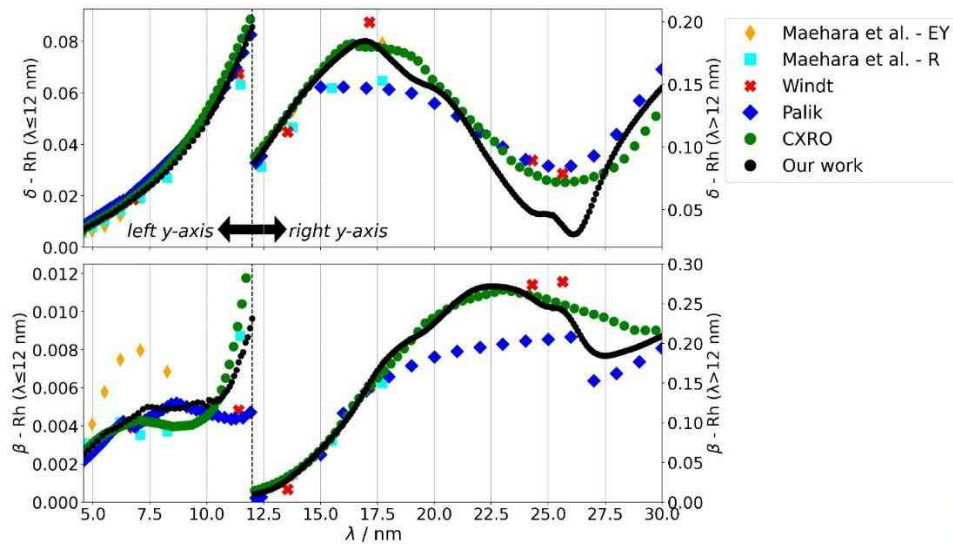


Fig. 24. Determined optical constants for Rh in the spectral range 4.6 nm – 30.0 nm. Our data shown in black dots consists of 228 points for each profile. Optical constants from literature are shown for comparison [27,29,30,120,128]. Left y-axes: $\lambda \leq 12.0$ nm; right y-axes: $\lambda > 12.0$ nm.

closest among the presented literature [27]. For the spectral range above ~ 16 nm, our profiles reveal fine-structure unseen in the literature data shown here. The abrupt change in the profiles is noticeable around 27.0 nm. That is here because of the change in absorption given Rh $-N_{2,3}$ levels [129]. In the spectral range we investigated, Windt *et al.* show 6 points [120], hence, no conclusive comparison could be carried out. However, it is interesting to point out that the data of Maehara *et al.* (ref. [128]) determined using reflectivity are closer to ours, than their data determined using photoelectric yield measurements.

3.9. Pd sample

This sample is a 48.6 nm Pd coating on a 4.4 nm Ti seed layer on top of a Si substrate. Ti seed layers were found beneficial for enhancing the quality of Al coatings dedicated for the EUV and the ultraviolet (UV) range [130,131]. Lee *et al.* also reported that Ti seed layers can help in obtaining smoother sputtered Mo thin films [132]. We assume that (oxidized) Ti seed layers have the same effect for our coating here. We present the reflectivity data measured from the sample shown in Fig. 25(a).

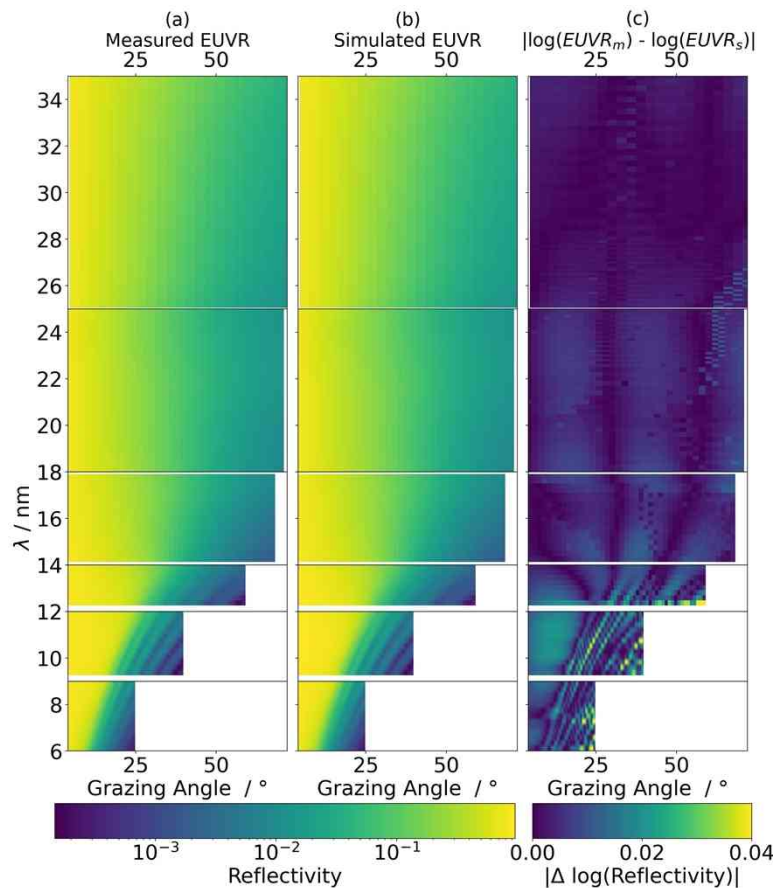


Fig. 25. Processed interpolated mapping of measured and simulated reflectivity data of the Pd sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

For the simulations, we introduce an additional carbonaceous contamination layer on the surface. No oxidation layer is assumed given the indicated oxidation resistance of Pd coatings

[133]. For the spectral range 6.0 nm – 20.0 nm shown here, the following thicknesses were calculated upon assuming convergence: C (1.8 nm)/Pd (49.8 nm)/TiO₂ (1.7 nm)/Si substrate. For the remaining data at the longer wavelengths, the calculated model was fixed. Overall, as shown in the subplots of Fig. 25(b) and (c), the simulations and residuals indicate good agreement with the measurements. Misfits are noticed for the grazing angular region below 14 nm. The determined optical constants are shown in Fig. 26 in comparison with external literature data [27,30,31,134,135].

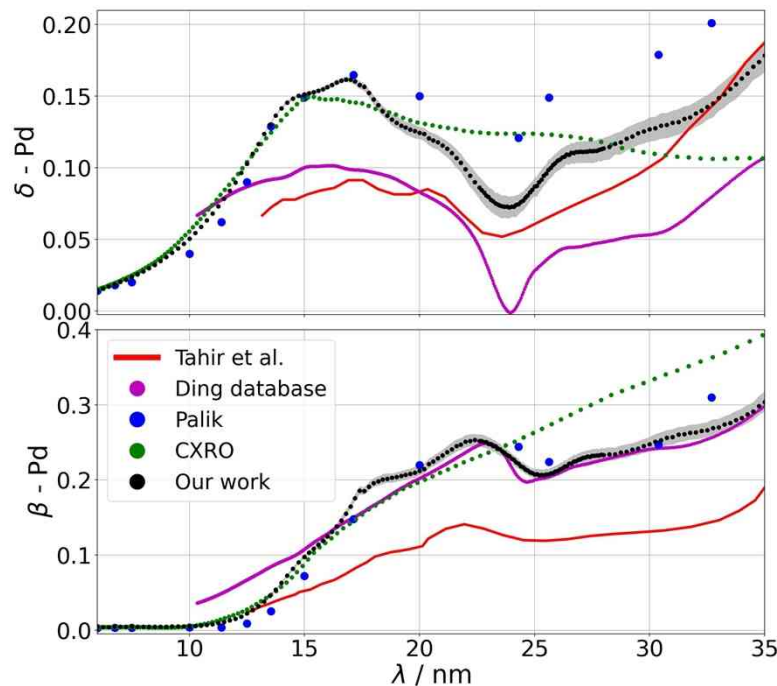


Fig. 26. Determined optical constants for Pd in the spectral range 6.0 nm – 35.0 nm. Optical constants from literature are shown for comparison [27,30,31,134,135].

The data of Tahir *et al.* were digitized from the plots shown in ref. [134]. Artefacts are therefore expected. For the spectral range 5.0 nm to ~15 nm, our results are in good agreement with CXRO (ref. [27]) and Palik (ref. [30]). Above ~15 nm, our results do not agree with CXRO tabulations (ref. [27]) as what can be observed from the very different trending behaviors of the curves. Palik (ref. [30]) data above ~15 nm is in agreement with our data for the extinction coefficients, but not for the refractive components.

The dip observed at wavelength 24 nm in our refractive components is also pronounced in the data from Ding database (refs. [31,135]), but not in CXRO (ref. [27]) and not clearly for Palik (ref. [30]). However, the scale of the refractive components of Ding database (refs. [31,135]) is smaller than ours. Tahir *et al.* data were determined using REELS [134].

Generally, both refractive components and extinction coefficients profiles have smaller values than ours. For the extinction coefficients we determined below ~10 nm, there are indications of noise. That might be due to the deficiencies of the assumed layer structure.

3.10. Hf sample

Hf is known to oxidize at room temperature [136]. Aiming for studying metallic and oxygen-free Hf, the following trilayer system was coated: C capping (5 nm)/Hf layer (40 nm)/Ru underlayer

(3 nm)/Si substrate. After that, we measured the system's reflectivity in the spectral range 5.0 nm – 50.0 nm, as shown in Fig. 27(a).

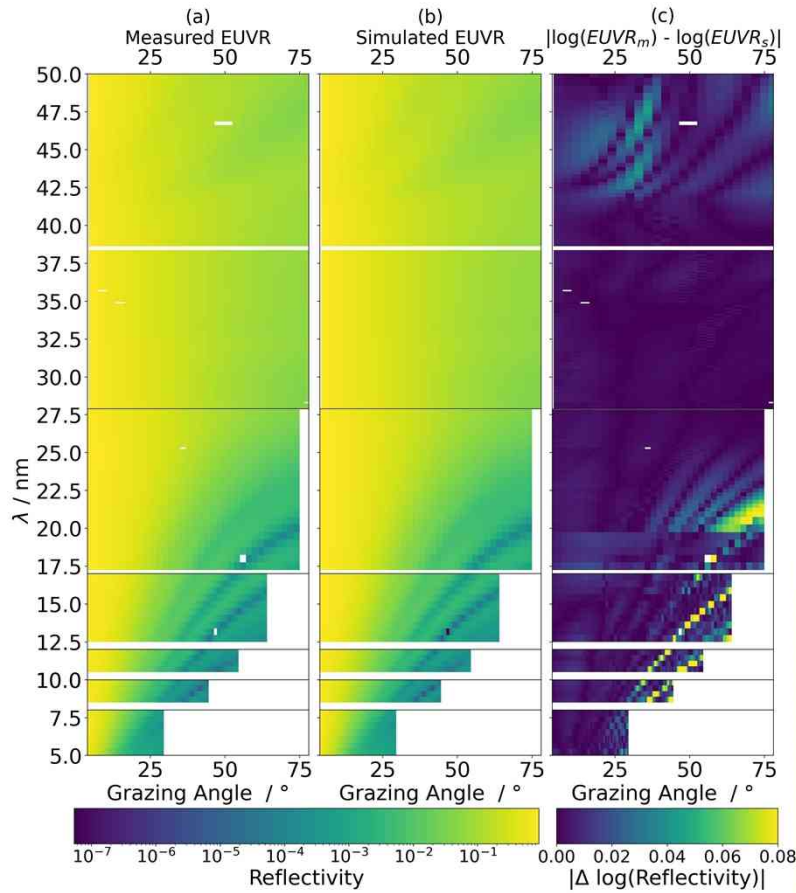


Fig. 27. Processed interpolated mapping of measured and simulated reflectivity data of the Hf sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

In comparison with the other samples, this one was measured in a relatively broader spectral range given the stretched fine-structure observed in the reflection spectra at grazing angles. For the reflectivity data measured in the spectral range 5.0 nm – 19.5 nm, the optical constants of the capping layer and Hf were floating parameters, in addition to the geometrical parameters of the coated system. The calculated thicknesses of the layer system are as follows: C capping (5.8 nm)/Hf layer (39.2 nm)/Ru underlayer (3.2 nm). The calculated thicknesses are quite close to the nominal fabrication settings. For the simulations considering the data measured above 19.5 nm, the calculated geometrical model at the shorter wavelengths was used, except for the uppermost roughness coefficient, it was left as a sampling parameter. Also, the optical constants of the capping layer were scaled from the data shown in Fig. 2. The dispersive components and the extinction coefficients were scaled with factors of 1.046 and 1.056, respectively. The factors were calculated from the ratio of the capping layer optical constants to those in Fig. 2 at 19.5 nm. Additionally, for wavelengths above 31.0 nm, the optical constants of Si were taken from refs. [33,137], since the CXRO data (refs. [27,28]) do not fully cover the investigated spectral range. The resultant simulations are in very good agreement with the experimental reflectivity data

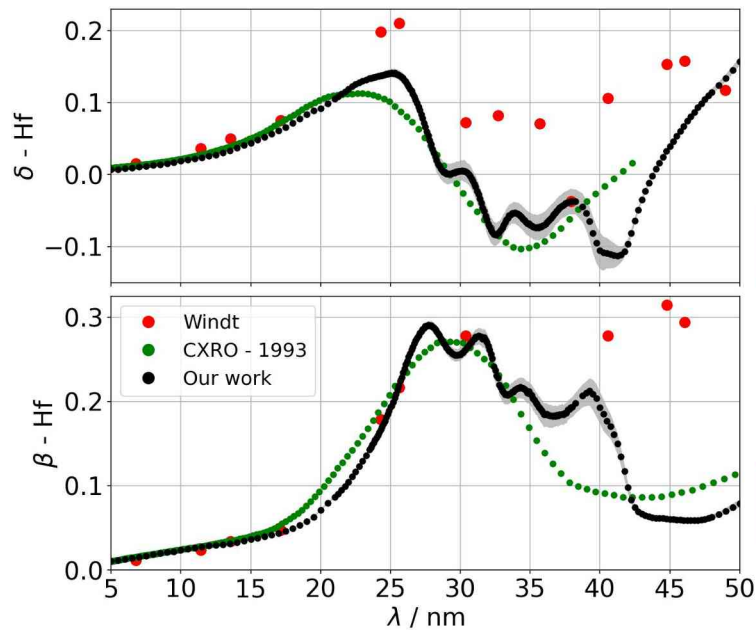


Fig. 28. Determined optical constants for Hf in the spectral range 5.0 nm – 50.0 nm. Optical constants from literature are shown for comparison [27,29].

as shown in Fig. 27. The determined optical constants of Hf and its carbonaceous capping are shown in Fig. 28 and Fig. 29, respectively.

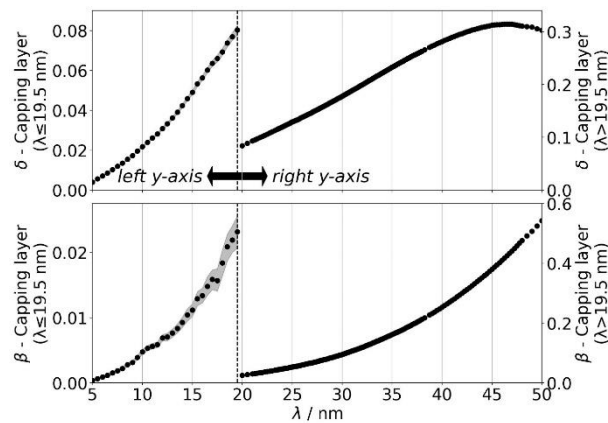


Fig. 29. Optical constants of the Hf carbonaceous capping layer determined in the 5.0 nm – 19.5 nm spectral range. Data for $\lambda > 19.5$ nm were obtained by scaling points from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 19.5$ nm; right y-axes: $\lambda > 19.5$ nm.

In comparison with CXRO and Windt data [27,29], our results are in general agreement for the range below ~ 18.0 nm. Above ~ 25 nm, our determined optical constants reveal fine-structure unidentified in the literature we compare our results with [27,29]. However, the presence of fine-structure is to be anticipated given the known Hf $-N_4$, $-N_5$, $-O_1$, $-O_2$ and $-O_3$ levels in the investigated spectral range [121]. Also, older optical absorption data for Hf in the EUV range clearly indicates modulating extinction coefficients [48]. Moreover, recent optical data for HfO_2

obtained using REELS corroborates the existence of modulating fine-structure for Hf in the EUV spectral range [138]. Irrespective of that, optical data for metallic Hf in the EUV range are very rare.

Pertaining to the determined optical constants of Hf carbonaceous capping layer, they resemble increasing functions of the wavelength, similar to what is observed for the data shown in Fig. 2, Fig. 5, and Fig. 8.

3.11. W sample

The investigated W sample is nominally a trilayer system, made up of the following layers, descendingly: C capping (5 nm)/W coating (40 nm)/Ru underlayer (3 nm)/Si substrate. Reflectivity measurements in the spectral range 5.0 nm – 34.0 nm from the sample are shown in Fig. 30(a).

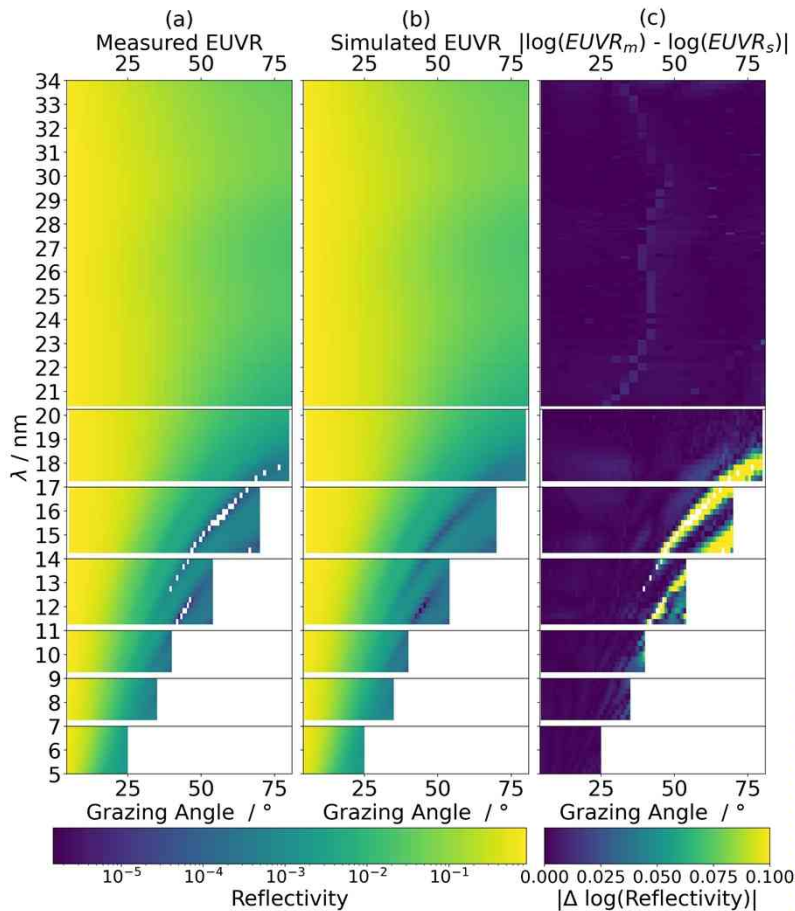


Fig. 30. Processed interpolated mapping of measured and simulated reflectivity data of the W sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

For the analysis, we introduce an additional SiO_2 layer on top of the substrate with a thickness of 2.5 nm. The substrate roughness RMS value was also fixed as 0.5 nm. Initially, the data measured in the range 5.0 nm – 20.25 nm were sampled for the optical constants of both of the C capping and the W layer, with the remaining geometrical parameters. The calculated thicknesses of the sampled layers are as follows: C capping (5 nm)/W coating (39 nm)/Ru underlayer (2.7 nm). For analyzing the measurements at and above 20.5 nm, the calculated model was fixed as obtained

for the data at the shorter wavelengths, except for the uppermost roughness parameter. Also, the optical constants of the capping layer were scaled from the data shown in Fig. 2. The refractive components and the extinction coefficients were scaled with factors of 1.06 and 1.324, respectively. The latter two were determined by taking the ratio of the capping layers' optical constants to those plotted in Fig. 2 at a wavelength of 20.0 nm. The boundary wavelength for this sample is 20.25 nm. Since no data of the 40 nm-thick C layer (Fig. 2) are available at this wavelength, the optical constants at 20.0 nm were used instead. Nevertheless, the simulated reflectivity is generally close to the measured, as observed in Fig. 30. The determined optical constants for W and the capping layer are shown in Fig. 31 and Fig. 32, respectively.

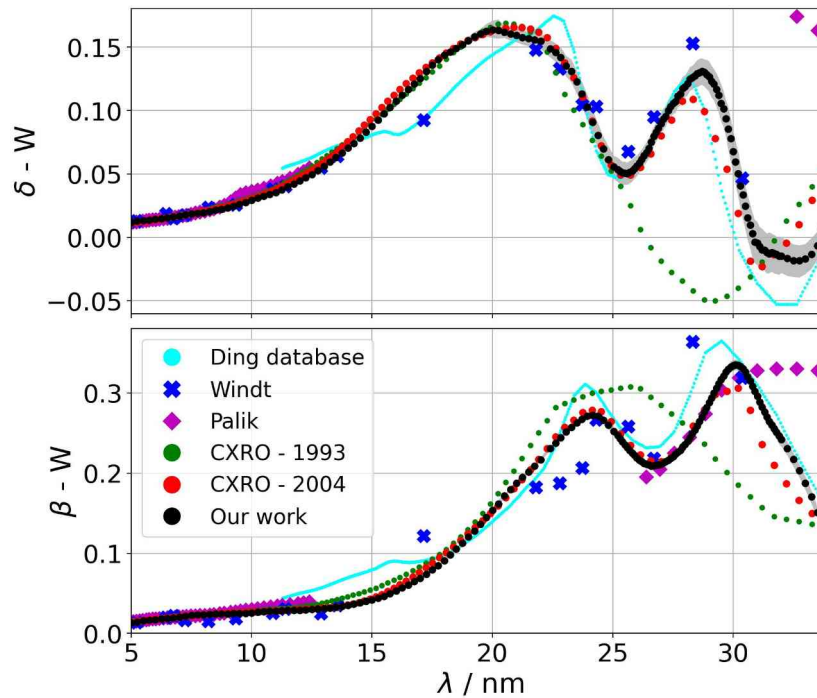


Fig. 31. Determined optical constants for W in the spectral range 5.0 nm – 34.0 nm. Optical constants from literature are shown for comparison [27–31,139].

When comparing our results with external literature [27–31,139], very good agreement is noticed between our data and those updated from the CXRO, except for the dispersive components above ~30 nm [28,124]. Palik data here is imported from IMD database [29]. Below ~15 nm, our data have similar trends to Windt, Palik and CXRO [27–30,124].

The most recent literature we compare our results with is that of Ding database [31,139], the semi-flattening noticed in the dispersive components above ~31 nm is also visible in their data.

As discussed for the optical constants of the carbonaceous layers for Sc, V and Co samples, we do not present the optical constants shown in Fig. 32 to be those of pure C. The optical constants in this case are particularly susceptible to the presence of environmental contamination and other effects.

3.12. Re sample

There is evidence that Re thin films coated with atomic layer deposition (ALD) develop oxidation [140]. There are also indications for the formation of ReO₃ on Re thin films obtained by pulsed laser evaporation (PLE) [141]. Additionally, Cox *et al.* noted evaporated Re films losing very

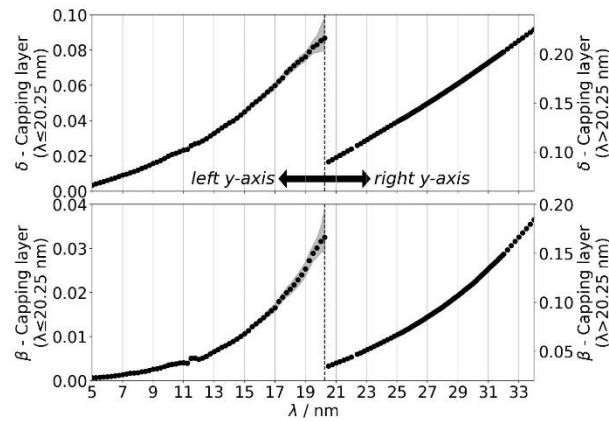


Fig. 32. Optical constants of the W carbonaceous capping layer determined in the 5.0 nm – 20.25 nm spectral range. Data for $\lambda > 20.25$ nm were obtained by scaling points from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 20.25$ nm; right y-axes: $\lambda > 20.25$ nm.

small reflectance upon air contact, and that they form a surface layer when exposed to plastic fumes [142]. To avoid oxidation and other surface effects, carbon capping is desired. The studied Re sample here is nominally a bilayer (on a substrate) system, consisting of the following: C capping (5 nm)/Re coating (40 nm)/Si substrate. We measured the reflectivity from this sample in the spectral range 5.0 nm – 36.0 nm, as shown in Fig. 33.

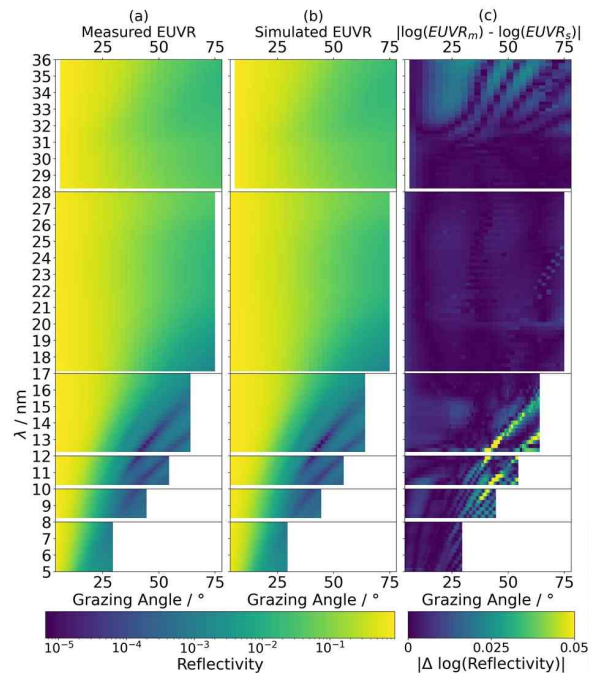


Fig. 33. Processed interpolated mapping of measured and simulated reflectivity data of the Re sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

For determining optical constants, the geometrical parameters and the optical constants of both layers were sampled for the data measured between 5.0 nm and 20.0 nm. The calculated thicknesses for the layer system were as follows: C capping (5.4 nm)/Re coating (41.2 nm). The calculated geometrical parameters at the shorter wavelengths – except for the uppermost interface roughness coefficient – were fixed for the simulations of the data measured in the spectral range 20.25 nm – 36.0 nm. Additionally, for that data, the optical constants of the carbonaceous layer were scaled from the data shown in Fig. 2. The refractive components and the extinction coefficients were scaled with factors of 0.943 and 1.353, respectively. The factors were obtained by dividing the optical constants of the capping layers by those shown in Fig. 2 at a wavelength of 20.0 nm. However, the simulations are in noticeable agreement with the experimental data as shown in Fig. 33, and our determined optical constants of Re and its capping layer are plotted in Fig. 34 and Fig. 35, respectively.

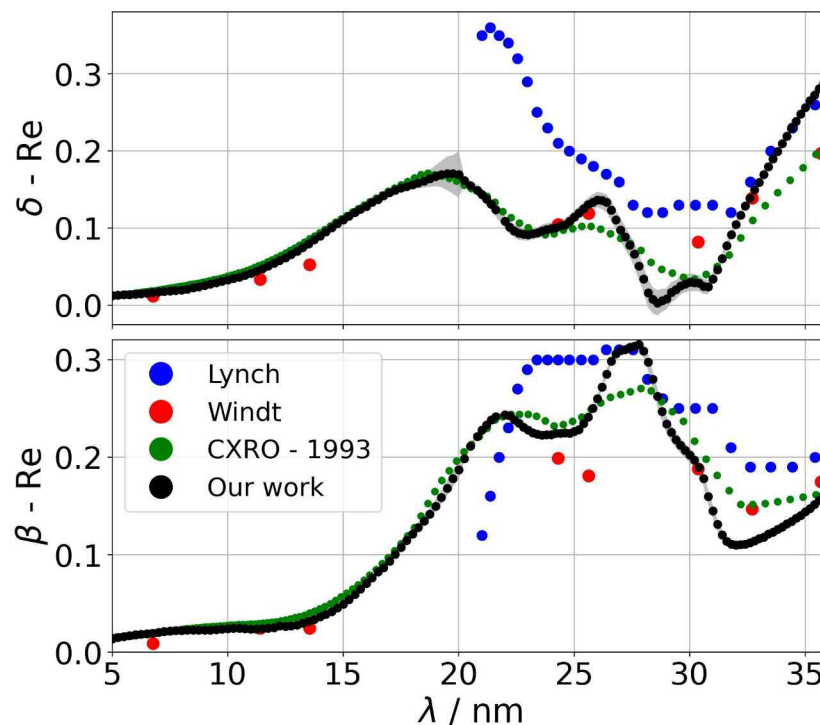


Fig. 34. Determined optical constants for Re in the spectral range 5.0 nm – 36.0 nm. The apparent black line consists of 139 discrete points. Optical constants from literature are shown for comparison [27,29].

For Re, in comparison with CXRO data [27], we have very good agreement for the spectral range 5.0 nm to ~ 21 nm as shown Fig. 34. However, the number of data points considering Windt (ref. [29]) is too sparse for a conclusive comparison. For the data of Lynch shown in Fig. 34, it was imported from IMD database [29], they show considerable differences compared to our results.

The resolved fine-structure in our Re profiles is rather expected given the reported Re $-N_6$, $-N_7$, $-O_1$, $-O_2$ and $-O_3$ levels in the wavelength range ~15 nm to ~35 nm [121]. Also, Haensel *et al.* reported fine-structure in the absorption spectra of Re at the range we investigate here [49].

The calculated optical constants of the carbonaceous capping layer in the spectral range 5.0 nm – 20.0 nm are plotted with their corresponding CIs in Fig. 35. The discussions relevant to the

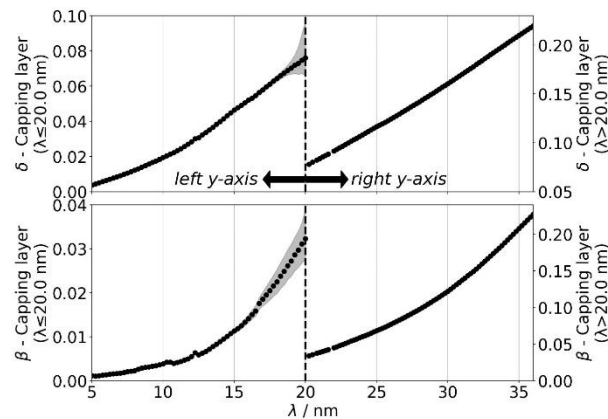


Fig. 35. Optical constants of the Re carbonaceous capping layer determined in the 5.0 nm – 20.0 nm spectral range. Data for $\lambda > 20.0$ nm were obtained by scaling points from the 40 nm-thick C layer. Left y-axes: $\lambda \leq 20.0$ nm; right y-axes: $\lambda > 20.0$ nm.

optical constants of the capping layers used for Sc, V, Co, and W samples also apply here. The necessity of adjusting the optical constants of this ultrathin layer beyond a wavelength of 20 nm becomes evident by examining how the CIs enlarge proportionally with the wavelength.

An additional check to validate our simulated model was carried out. The differentiation-assisted fast Fourier transform (FFT) approach of Poust *et al.* (ref. [143]) was used to estimate the thickness of the Re layer and its capping. Based on a reflectivity profile measured at a wavelength of 1.0 nm, as shown in Fig. 36.

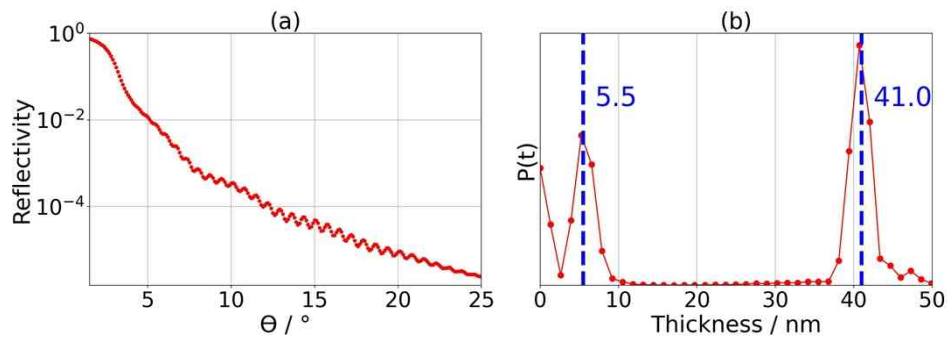


Fig. 36. Re sample thickness estimation using the differentiation-assisted FFT approach. (a) Measured soft XRR profile from the sample. (b) Interpolated calculated FFT spectrum. The vertical blue dashed lines are assumed to indicate layers' thicknesses found in the system [143,144].

In Fig. 36, the FFT spectrum peaks at ~5.5 nm and at ~41.0 nm. These values are very close to the calculated thicknesses from the other EUV data. This cross-check is presented given the significance of the resolved-fine structure.

3.13. Os sample

The studied data here was measured from the Os coating in the spectral range ~4.54 nm to ~34.4 nm. The coating's nominal thickness is 50 nm. The measurements are shown in Fig. 37(a). In order to determine the optical constants, we considered a trilayer model consisting of C

contamination/Os coating/SiO₂ layer all on top Si substrate. Cox *et al.* reported that evaporated Os films develop very thin oxide layers at room temperature, and that only 1% reflectivity loss was noticed after one month of exposure to air [145]. Therefore, we only consider C contamination on the surface. The optical constants of the SiO₂ layer were fixed from the PTB database [109]. Upon arriving at an assumed state on convergence, the following layer thicknesses were calculated for trilayer system: C (1.8 nm)/Os (50.2 nm)/SiO₂ (1.1 nm). The simulated reflectivity is shown in Fig. 37(b). The determined optical constants of Os are shown in comparison to external literature in Fig. 38 [27,29,30,146].

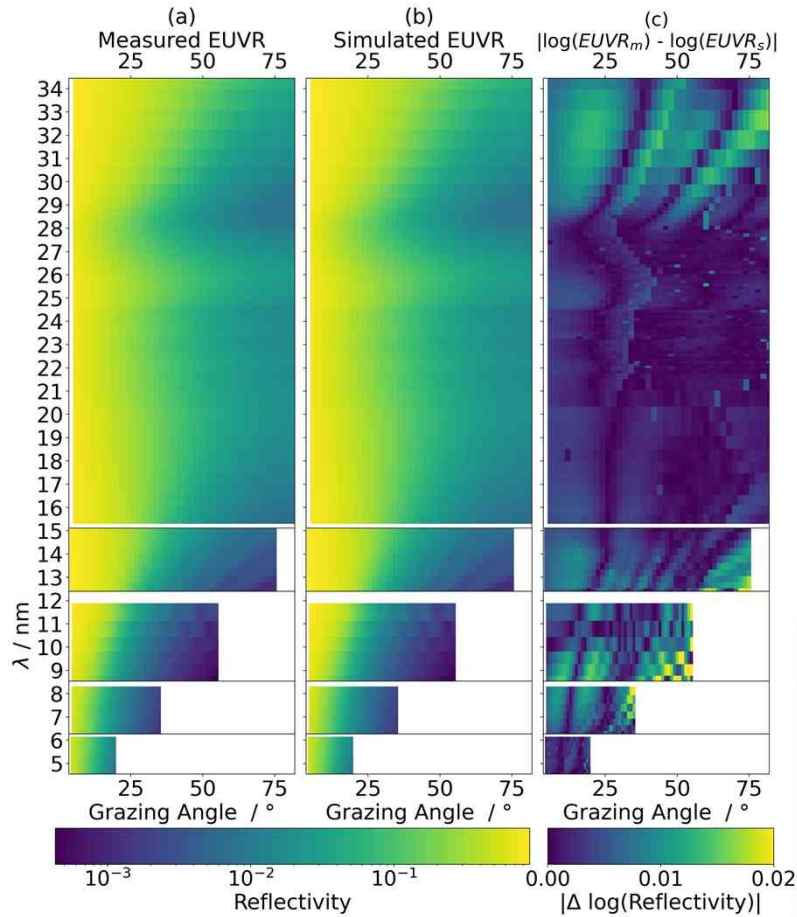


Fig. 37. Processed interpolated mapping for the measured and simulated reflectivity data of the Os sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

Fine-structure is observed in our results between 21 nm and 30 nm. Indications for the existence of fine-structure can be inferred from the observed anomalous reflection spectra in the corresponding spectral range. The data from Lynch *et al.* (ref. [146]) and Palik (ref. [30]) shown here were imported from the database of IMD software [29]. Generally, aside from the fine-structure region, the optical constants of the presented literature here (except for Lynch *et al.*) are in relative agreement with our results' trends below ~17 nm and above ~29 nm.

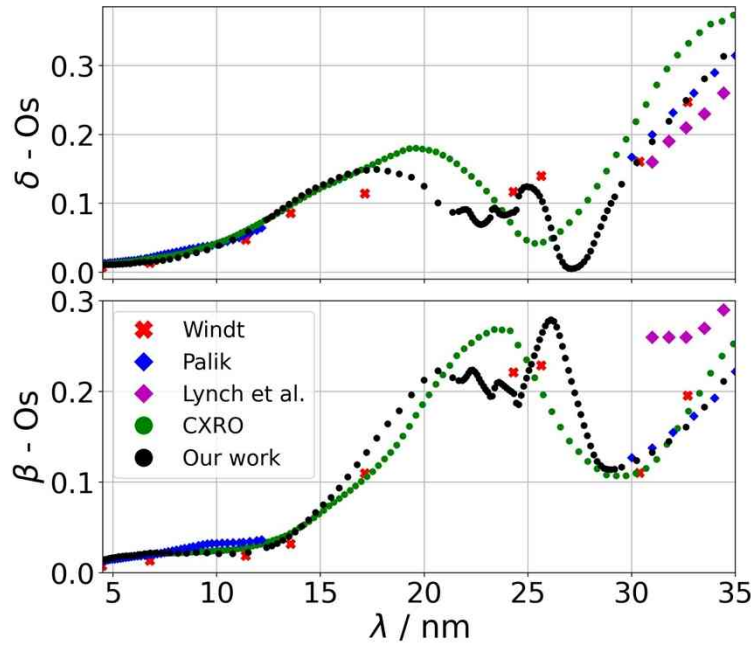


Fig. 38. Determined optical constants for Os in the spectral range ~ 4.5 nm to ~ 34.4 nm. Optical constants from literature are shown for comparison [27,29,30,146].

However, the resolved fine-structures in our profiles are not achieved in the cited literature [27,29,30,146]. According to ref. [121], Os $-N_6$, $-N_7$, $-O_1$, $-O_2$ and $-O_3$ levels are between ~ 14.7 nm and ~ 26 nm. That explains the existence of the resolved features in our results.

Because of the importance of the resolved fine-structure, in Fig. 39 the differentiation-assisted FFT approach is again used for a cross-check to validate the simulated model [143]. Given reflectivity data measured at a wavelength of 1.04 nm, the FFT spectrum peaks at ~ 50.5 nm as shown in Fig. 39. That is in agreement with the calculated thickness obtained using the EUV data from which the optical constants were determined.

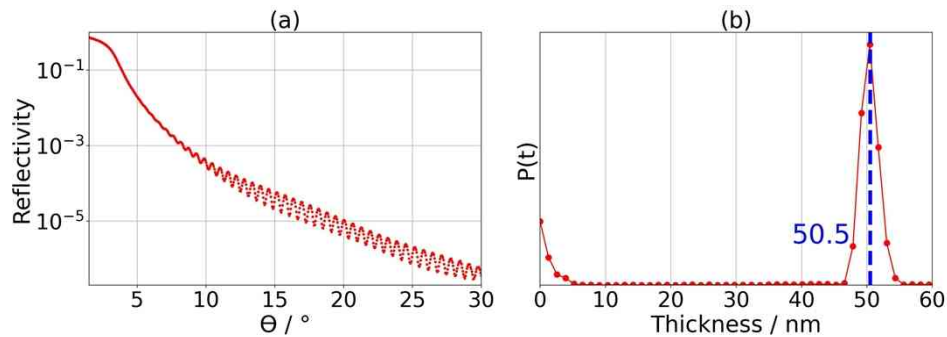


Fig. 39. Os coating thickness estimation using the differentiation-assisted FFT approach. (a) Measured soft XRR profile from the sample. (b) Interpolated calculated FFT spectrum. The vertical blue dashed line is assumed to indicate the thickness of Os coating [143,144].

3.14. Ir sample

The studied data for the Ir sample was measured in the spectral range 5.0 nm – 35.0 nm. The measurements are shown in Fig. 40(a). The Ir layer nominal thickness is 43 nm.

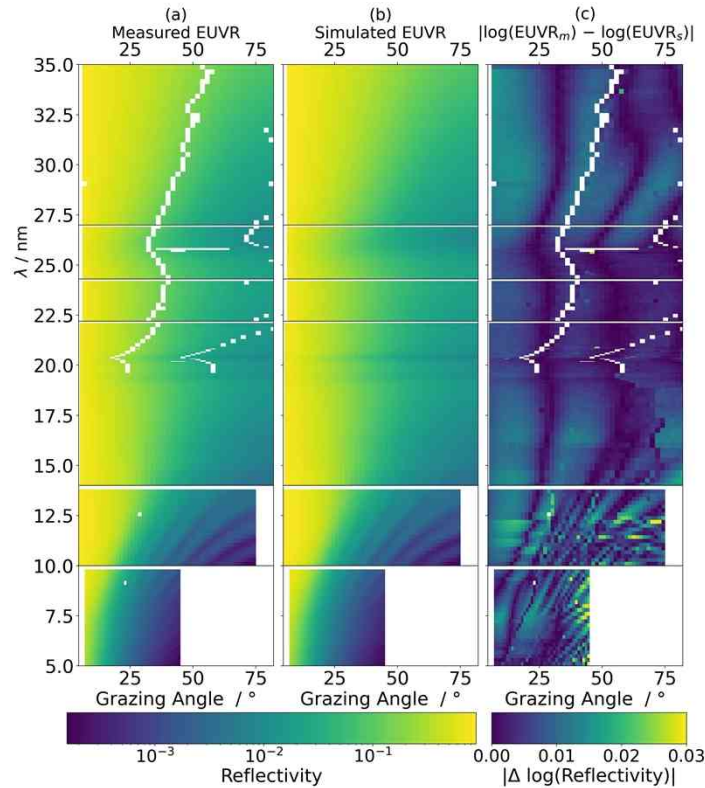


Fig. 40. Processed interpolated mapping for the measured and simulated reflectivity data for the Ir sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations. The white pixels appearing in the measured data are masked values.

For the simulations, we assume a trilayer model consisting of C contamination/Ir coating/SiO₂ all on top Si substrate. To adjust the structural model of the sample, the data between 5.0 nm to ~17 nm were taken, and the calculated thicknesses of the model are as follows: C (1.8 nm)/Ir (42.8 nm)/SiO₂ (1.0 nm). This calculated model was fixed for determining optical constants from the remaining data. The simulations are generally in agreement with the measurements as shown in Fig. 40(b) and (c). Our determined optical constants are shown in Fig. 41 in comparison with external literature values.

Ding database (refs. [31,147]) values for the extinction coefficients above ~15 nm are not very far from ours, while the fine-structure we resolved between ~19 nm to ~21 nm is not present there. However, for Ding database (refs. [31,147]), the refractive components are relatively smaller than our data.

For the investigated range here, Windt data consists of 8 points in each profile [29], because of that, no certain comparison could be carried out. Palik data (ref. [30]) also do not fully cover the range we investigated. CXRO data are in general agreement with our results, except for the lack of resolved fine-structure in their data [27]. However, the data digitized from Graessle *et al.* (ref.

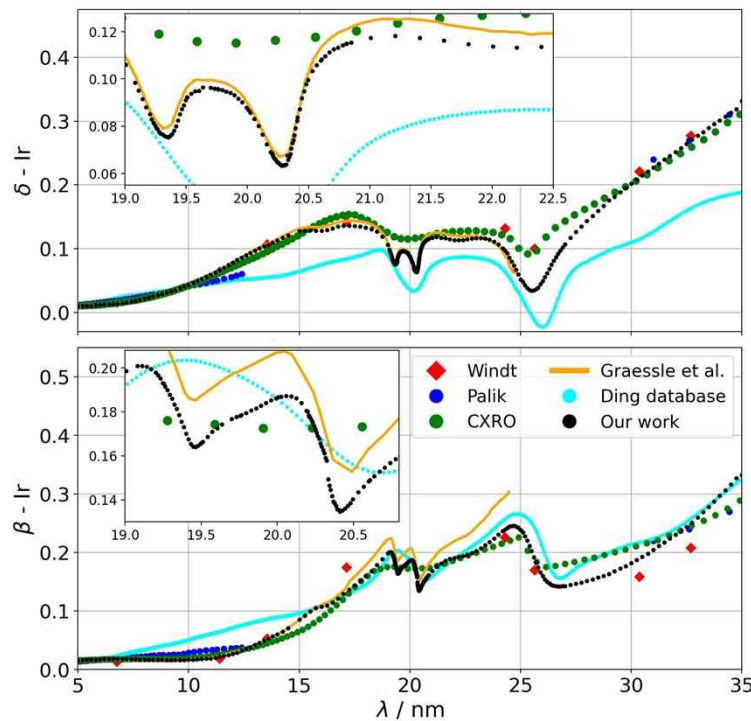


Fig. 41. Determined optical constants for Ir in the spectral range 5.0 nm – 35.0 nm. The plotted insets better show the resolved fine-structure. Optical constants from literature are shown for comparison [27,29–31,43,147].

[43]) are in very good agreement with our results, especially for the refractive components. The shown data of Graessle *et al.* (ref. [43]) may be affected by inaccuracies arising from digitization.

From ref. [121], Ir $-N_6$, $-N_7$, $-O_1$, $-O_2$ and $-O_3$ levels coincide with the spectral range spanning ~12.9 to ~24.3 nm. Therefore, the presence of fine-structure is expected. The discussion in ref. [148] is relevant for more details on the observed fine-structure.

3.15. Pt sample

The studied sample is a nominal 30 nm Pt coating on a Si substrate. Previously, we presented optical constants from this sample in the spectral range 10.0 nm – 18.0 nm [126]. Here, we investigate reflectivity data measured in the spectral range 5.0 nm – 27.8 nm, as shown in Fig. 42(a).

The model used to determine the optical constants includes a carbonaceous contamination layer on the surface and the (presumed) native substrate oxidation (amorphous SiO₂ layer). After assuming the state of convergence in the simulations, the following layer thicknesses were calculated: C (1.6 nm)/Pt (28.8 nm)/SiO₂ (1.7 nm). The simulated reflectivity profiles and the residual mapping are shown in Fig. 42. The simulations mostly agree well with the measurements, with discrepancies observed at the high oblique angles for the shorter wavelengths. That could be due to a substrate related effect. The determined optical constants of Pt are shown in Fig. 43 and Fig. 44 in comparison with our previous work and external datasets [27,29,42,126,149].

The profiles of Palik and Windt are retrieved from the database of IMD software [29]. The data shown are all in agreement below ~14 nm. Above ~14 nm, CXRO data of 1993, those of Palik, Windt and Sharma *et al.* demonstrate divergence with our current and previous works,

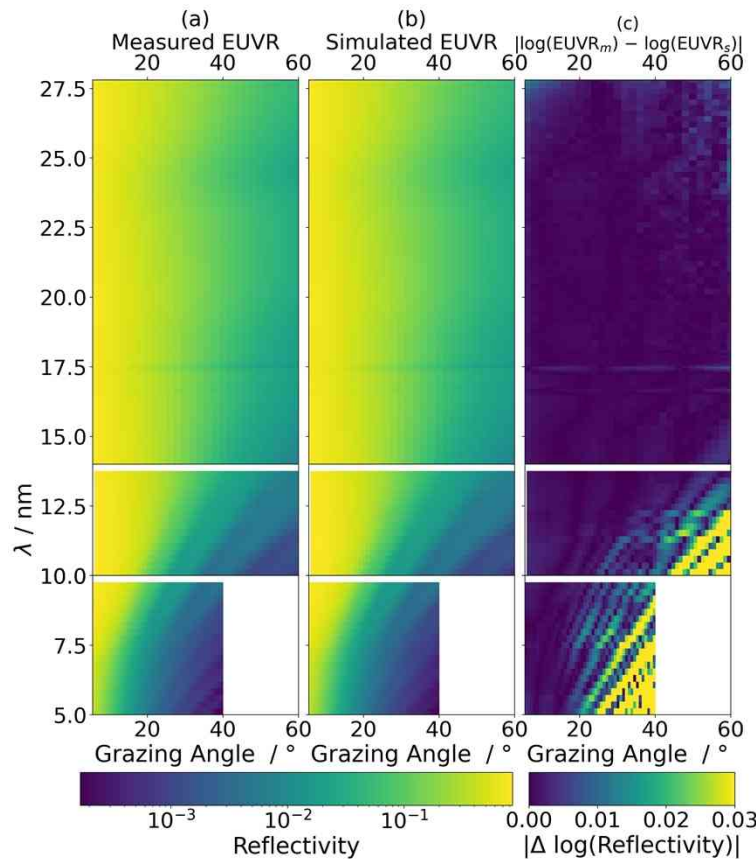


Fig. 42. Processed interpolated mapping for the measured and simulated reflectivity data of the Pt sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

besides that of Soufli *et al.* [27,29,42,126,149]. Pt $-N_6$, $-N_7$, $-O_1$, $-O_2$ and $-O_3$ levels fall within the range ~ 12.1 to ~ 24.3 nm [121]. The observed fine-structure has been resolved and reported before by Soufli *et al.* [42]. A clearer comparison between our results and literature considering the observed fine-structure is possible by studying Fig. 44.

The extinction coefficients we determine here and the results of our previous work on Pt are in remarkable agreement with that of Soufli *et al.* [42,126]. The refractive components determined here are clearly smaller than our previous work and those of Soufli *et al.* [42,126]. Possibly, that could be ascribed to surface contamination or possible aging behavior, since the coating is slightly more than five-year-old.

3.16. Au sample

This sample is nominally a 35 nm Au coating on a Si substrate. The presented data shown in Fig. 45(a) is measured from the sample in the spectral range 5.0 nm – 33.0 nm. For the modelling formalism, we introduce the substrate's native oxide as an additional separate layer. The calculated thicknesses in the model are as follows: Au (32.6 nm)/SiO₂ (2.4 nm). The resultant simulations are in general agreement with the measurements as indicated by Fig. 45(b) and (c). Misfits are noticed for the simulations regarding the measurements at the highest oblique angles for data collected in the spectral range 5.0 to ~ 7 nm.

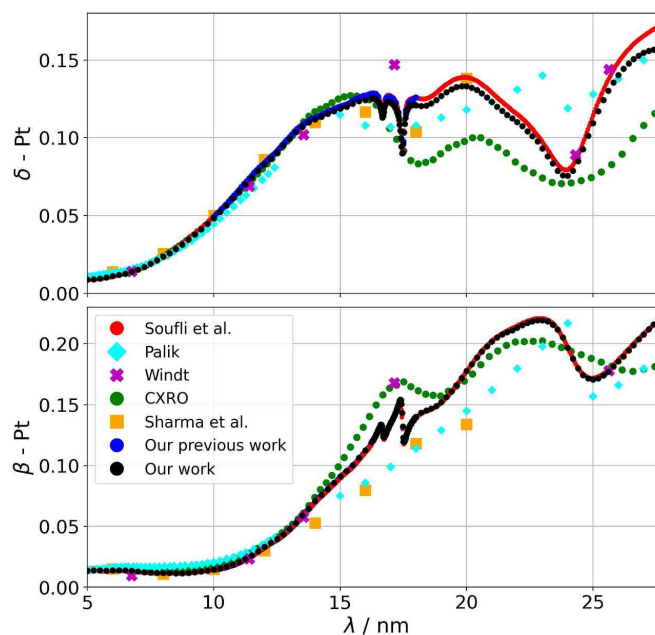


Fig. 43. Determined optical constants for Pt in the spectral range 5.0 nm – 27.8 nm. Optical constants from previous work of ours and external literature are shown for comparison [27,29,42,126,149].

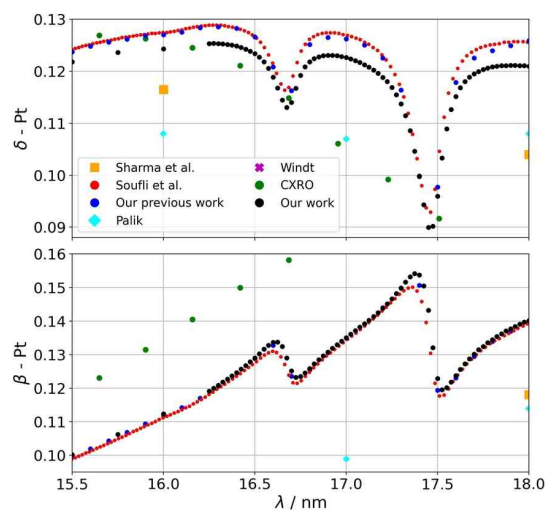


Fig. 44. Determined optical constants for Pt in the spectral range 15.5 nm – 18.0 nm. Optical constants from previous work of ours and external literature are shown for comparison [27,29,42,126,149].

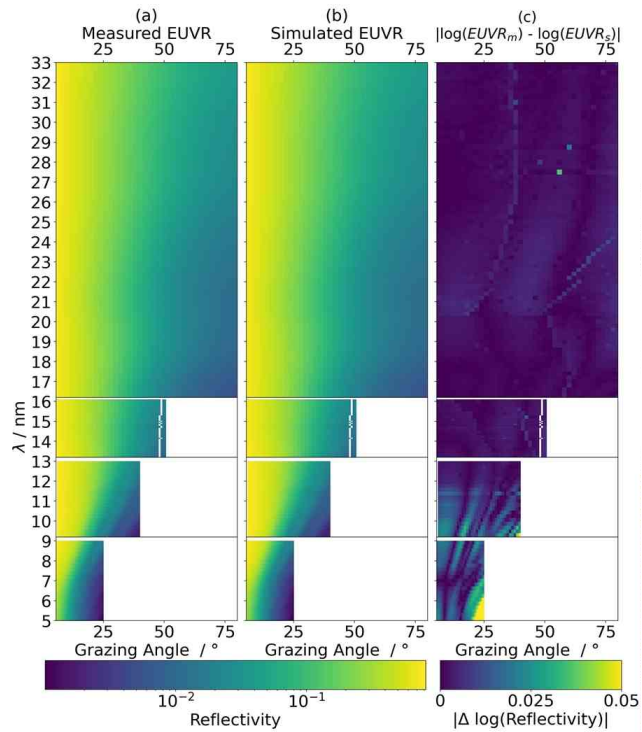


Fig. 45. Processed interpolated mapping for the measured and simulated reflectivity data for the Au sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

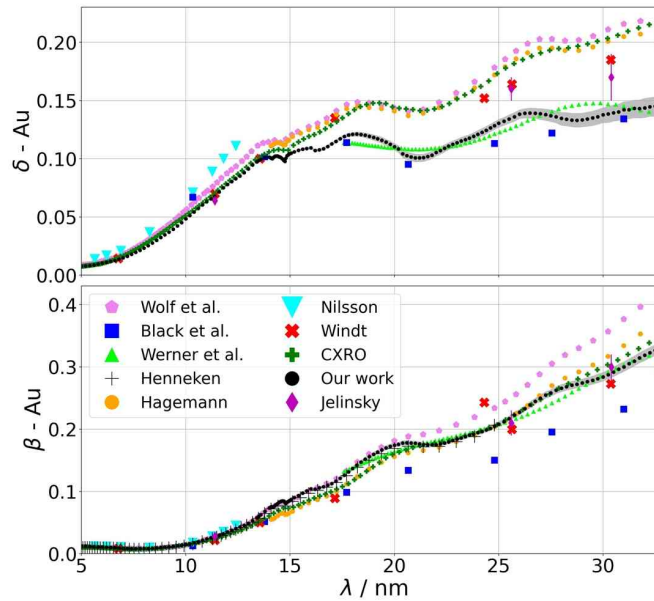


Fig. 46. Determined optical constants for Au in the spectral range 5.0 nm – 33.0 nm. Optical constants from literature are shown for comparison [27,29,67,150–152].

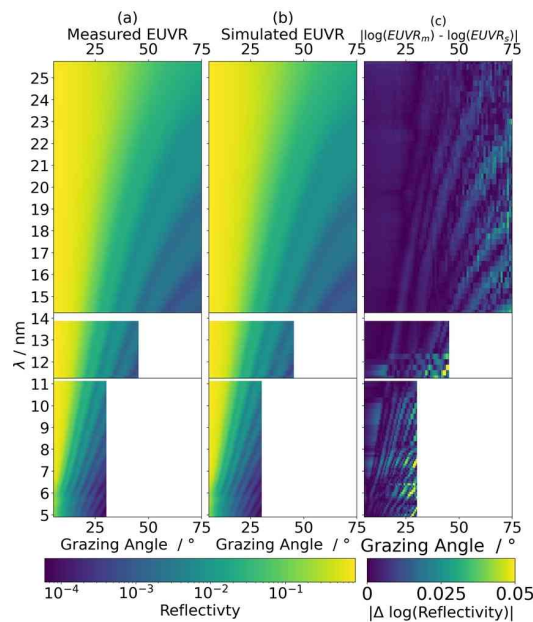


Fig. 47. Processed interpolated mapping for the measured and simulated reflectivity data for the ZrO_2 sample. (a) Measured reflectivity data, (b) simulated data, and (c) mapping of the residual between measurements and simulations.

The determined optical constants are plotted in Fig. 46 in comparison with external literature data [27,29,67,150,151]. The data presented for Nilsson and Hagemann was obtained from the IMD database [29]. The data for Werner *et al.* were imported via ref. [33,103]. The extinction coefficients of Henneken are PTB data determined with measuring total electron yield [152]. For the extinction coefficients, generally, with the exception of Windt (ref. [29]) and Black *et al.* (ref. [67]) data points, the presented literature data are in good agreement with our results. However, our results demonstrate clear scaling inconsistencies for the spectral range above ~ 15 nm for the refractive components with CXRO, Windt, Hagemann and Wolf *et al.* data [27,29,151]. Our refractive components have the same scale as those of Werner *et al.* [103]. The modulating profiles are rather expected. According to ref. [121], Au $-N_6$, $-N_7$, $-O_1$, $-O_2$ and $-O_3$ levels fall in the range ~ 11.5 nm to ~ 22.9 nm.

In the bulk form, with respect to some criteria, Au is regarded as the most noble element [153,154]. Au bulk surface is also exceptionally stable [154]. As well, Au is widely used for coating gratings and as a capping material. Oxidation or effects that lead to surface degradation are assumed to be ruled out. It is evident that Au optical constants were extensively investigated from the data shown in Fig. 46. Hence, we did not expect the addressed large deviations in the spectral range above ~ 15 nm for the refractive components.

Wolf *et al.* investigated Au optical constants in the range between ~ 0.92 nm to 31 nm, and wrote that the observed inconsistencies between the sources they examined are “unusually large” [151]. Predehl *et al.* showed differences between the optical constants they determined and one literature dataset in the spectral range 5 nm – 20 nm [155]. Outside the range we investigated, but close, Kandaka *et al.* determined optical constants of an evaporated Au film (in the range ~ 44 nm to 82.6 nm) and showed differences with one literature data [156].

Apparently, although Au is highly inert and widely used, its optical constants in the EUV range were not accurately known. However, it is to be noted that our sample was transported to the

reflectometer's vacuum the next day after fabrication, and our measurements began four days after that. Therefore, contamination and aging effects are (presumably) excluded here.

3.17. ZrO_2 sample

The nominal fabrication settings of this sample are to coat a 42.5 nm ZrO_2 film on a Si substrate. We measured the reflectance in the spectral range 4.9 nm – 25.75 nm from the sample, as shown in Fig. 47(a). For determining the optical constants, we introduced a carbonaceous contamination layer on the optical surface, and the substrate's native oxide is also included. The latter's optical constants were fixed from PTB database [112]. Upon arriving at a stable solution of the inverse-problem, the following layers' parameters were calculated: C (1.6 nm)/ ZrO_2 (43.2 nm)/ SiO_2 (3.1 nm). The simulations are consistent with the measurements as shown in Fig. 47(b) and (c). Our determined ZrO_2 optical constants are shown in Fig. 48 together with Singh *et al.* results and CXRO predictions based on the IAA [27,157]. In Fig. 48, CXRO predictions are calculated assuming a mass density of 5 g/cm³ [27].

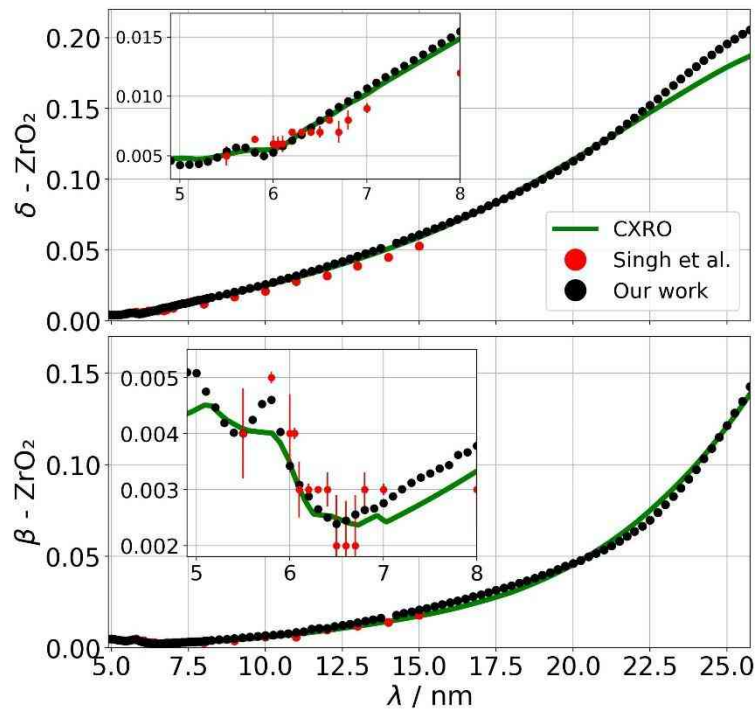


Fig. 48. Determined optical constants for ZrO_2 in the spectral range 4.9 nm – 25.75 nm. Optical constants from literature are shown for comparison [27,157]. Approximate optical constants from CXRO are taken via ref. [90].

We were able to describe the measured reflectivity of the coating using a simplified trilayer (on a substrate) model. That is demonstrated with the relatively low residuals in Fig. 47(c). This strongly indicates the homogeneity of the ZrO_2 layer, since our simulations assume ideal uniformity through each layer's thickness. The clear Kiessig fringes (ref. [119]) seen in the spectral range 4.9 nm to ~22 nm are another indication for the quality of the coating. Therefore, we assume the formation of a homogenous thin film.

Generally, our results are in agreement with the cited literature above ~7 nm [27,157]. Above ~7 nm, for both the dispersive components and the extinction coefficients, the values trace a

smooth profile in the presented spectral range. Below 7 nm, fine-structure is rather expected given the reported Zr $-M_4$ and $-M_5$ levels in the vicinity of ~ 6.7 nm [121].

The reported mass density of ZrO₂ coatings varies between 4.5 g/cm³ and 6.5 g/cm³ [68,157,158]. The discussion given in ref. [68] is highly relevant. For the comparison here, Singh *et al.* estimate their e-beam deposited coatings' density around 4.5 g/cm³ [157]. We estimate that our coatings' density is well-within that range and likely close to 5 g/cm³, based on comparison with CXRO (ref. [27]) data. The proximity of our results to literature values suggests the presence of a (near-) stoichiometric thin film [27,157].

4. Summary and outlook

Transition metals are highly relevant for the development of EUV optics, not only as main coatings in optical components, but also as capping layers, binders, interdiffusion barriers, and seed layers. Transition metals were also used for substrates [159], and a few metal-based photoresists were found to possess enhanced properties [160]. Some transition metals, especially refractory metals and those of the platinum-group, are important for optical applications where high thermal loads are met. That is critical for applications like FELs, synchrotron radiation and space-borne astronomy.

However, it is often the case to see inconsistencies when comparing optical constants regarding different sources for the same metal. That probably relates to impurities, contamination, aging or oxidation. Most transition metals have multiple stable oxidation states. Silicide formation is another phenomenon often observed upon coating transition metals on Si and quartz substrates, which are the most widely used. All that makes the determination of optical constants from thin film coatings challenging. Not only for reflectometry, but also when using other methods.

Here, we present the determination of optical constants from Sc, V, Cr, Co, Ni, Nb, Mo, Rh, Pd, Hf, W, Re, Os, Ir, Pt, Au, and ZrO₂ in the parts of the EUV spectral range. The optical constants were determined from angle-dependent reflectivity measurements collected with monochromatized synchrotron radiation. The nominal thicknesses of the investigated coatings are between ~ 30 nm to 50 nm. In the investigated spectral ranges, we report very good agreement between our results and literature for the cases of Cr [104], Co [27,30], Nb [27,28,30], Ir [43], Pt [42] and ZrO₂ [157].

Considering Sc, in comparison with CXRO, our results are in general agreement for the dispersive parts, but between ~ 28 nm and ~ 34 nm our determined extinction coefficients deviate [27,28,98–100]. For Mo, we report very good agreement between our data and that of CXRO (refs. [27,28,124]) in the spectral range 6.0 nm to ~ 25 nm. However, in the wavelength range 25.0 nm – 40.0 nm, our Mo data are in disagreement with the presented literature [27–30,124–127], especially for the dispersive components. Regarding W, our data and that of CXRO are in noticeable agreement below ~ 30 nm [28,124], and similarities are clear with Ding database above ~ 25 nm [31,139]. For Rh, we present optical data with higher resolution than the discussed literature [27,30,120,128]. The validity of using the IAA for the case of Cr was substantiated by the proximity of our results to those Delmotte *et al.* from 2018 [104]. But for the case of Ni, arriving at a conclusion is not straight forward since the discussed literature (refs. [27,31,113–115]) demonstrates inconsistencies itself, especially for the refractive components.

The most significant inconsistencies addressed here are for V, Pd, Hf, Re, Os and Au. For V, Pd, Hf, Re and Os, we present fine-structure not resolved in the main optical constants databases [27–30]. For Au, our extinction coefficients are in very good agreement with many sources. However, our determined dispersive components scale at lower values than the main optical constants databases [27–30]. Overall, our findings motivate further research to revisit optical data for V, Ni, Mo, Pd, Hf, Re, Os, Au, and for many other materials, too.

In broad terms, considering the spectral ranges we investigated and up to the wavelengths between ~ 10 nm to ~ 15 nm, we observe general agreement between our work and the tabulations

of the main optical constants databases [27–30]. But the most serious inconsistencies are more noticeable at wavelengths longer than ~ 15 nm. That can be explained with two arguments. First, a considerable part of external literature data we compare our results with were obtained using transmission measurements. In that formalism, the extinction coefficients are often calculated with neglecting the dynamical nature of radiation. Depending on the material, this simplification has an effect on the accuracy of the obtained extinction coefficients, especially at the longer wavelengths [161]. Second, a few monolayers of contamination or oxidation bias the optical response in the EUV range. Naturally, this bias magnitude is inversely-proportional to the penetration depth of radiation. Given the reduced penetration depths of photons with increasing wavelength, optical data collected at longer wavelengths are more vulnerable to surface effects. That in turn yields optical constants with lower accuracy when approaching the longer wavelength band of the EUV range.

For determining optical constants, reflectometry is an indirect characterization method. The measurements are investigated by assuming a model that describes the sample. But models are hardly accurate representations. Complex surface effects, morphological defects and inhomogeneities are likely. In most cases here, model deviations exceeded measurement uncertainties. This indicates that imperfections in the model are the dominant source of uncertainty in the determined optical constants, not the measurements errors. However, it is not a straightforward solution to assume additional parameters to better describe the samples. Here, the tackled inverse-problems have large dimensionalities, with strong cross-correlations of the sampled parameters. For example, the simulations for determining optical constants from the Sc sample in the spectral range 5.5 nm – 22.0 nm have 276 variables. The case of Os had 231 variables. Convergence becomes intractable if large numbers of additional variable parameters were added. Additional degrees of freedom can also cause over-fitting. R. Bellman referred to the associated issues accompanying highly dimensional inverse-problems with the term “curse of dimensionality” [162]. Consequently, we see the exponential increase in complexity and computational cost as unjustifiable when it yields only minor refinements. This is noteworthy to mention since the optical constants with their associated uncertainties depend on the assumed model, which is always imperfect. Such peculiarities are sometimes indicated as main disadvantages of reflectometry for investigating optical constants.

In this context, the high dimensionality of the simulations results from sampling both parts of $\bar{n}$ over a large number of wavelength scans. The structural parameters of the samples are restricted to layer thicknesses and interfacial roughness coefficients. Seemingly, the addressed measures to mitigate modeling approximations and computational constraints do not severely affect the accuracy of the determined data. The remarkable agreements between our results and recent external literature for the cases of Cr and Nb [28,104] are reassuring. For Re and Os, the near-match between the calculated thicknesses from EUV data simulations and soft XRR FFT inversion is convincing. For Hf and Ir, the existence of fine-structure is expected, given the observed features in older photoabsorption measurements [48,148].

Overall, the models give simulations in very good agreement with most of the measurements. This shows that the critical physical aspects are well captured. The agreement also supports the high quality of the samples. Figure 49 shows reflectivity profiles measured from ten samples at a wavelength of 13.5 nm with their corresponding simulations. The pronounced Kiessig fringes observed in Fig. 49 indicate a high degree of coating uniformity and well-defined interfaces. In contrast, these fringes disappear with increasing surface roughness or strong interfacial intermixing. In such cases, accurate modelling of the layer geometry becomes very challenging and is unlikely to be achieved using the simplified approach we employed. More details on the nature of the observed fringes are found in ref. [163].

For the impact of our work, it is very useful to compare data considering different sources as cross-checking and validation become manageable. That is particularly important when

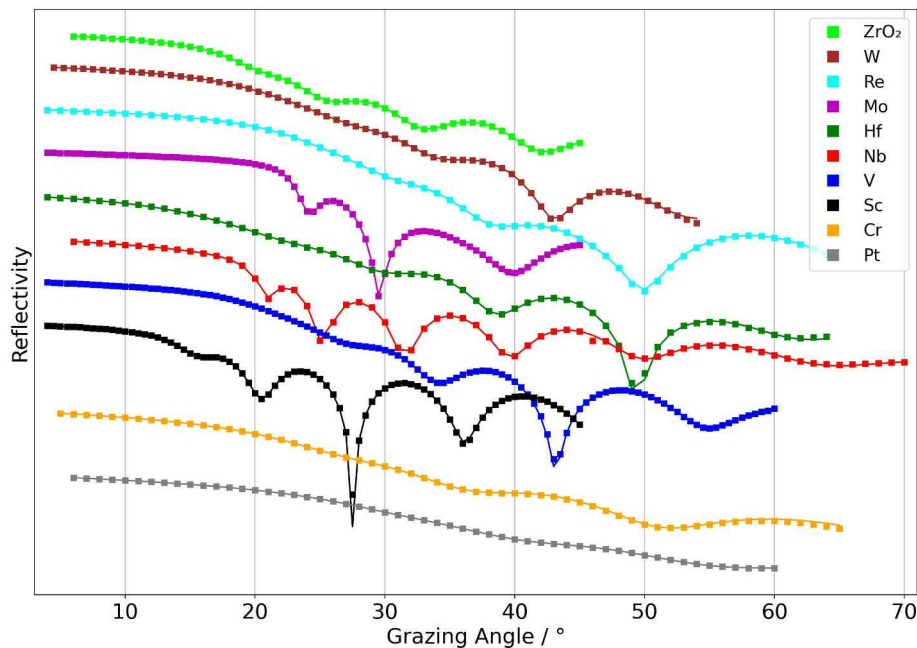


Fig. 49. Measured and simulated reflectivity profiles of the Pt, Cr, Sc, V, Nb, Mo, Hf, W, Re, and ZrO₂ samples at wavelength of 13.5 nm. Squares denote experimental data, and lines denote simulations. The legend entries are arranged in a single column following the sequence of the reflectivity profiles.

discussing determining optical constants with non-photometric methods, such as Electron-Energy-Loss Spectroscopy (EELS). Or when attempting to understand the deficiencies of theoretical calculations of optical data. Updated data facilitate MLM design and help in understanding optical components' responses. For example, it was explicitly stated that more accurate data for Pd can aid in the developments of relevant MLMs [164]. Optical Simulation software (*e.g.*: refs. [165,166]) can greatly benefit from our results, especially for the cases demonstrating significant inconsistencies with other literature. Moreover, the determined optical data can be relevant for materials selection processes considering the projected BEUVL technology, anticipated at ~ 6.7 nm [166].

Besides, updated optical data of one material can also help in examining the accuracy of another. For instance, thin W barrier layers were used for fabricating Dy samples dedicated for measurements for optical constants determination [167]. Within a similar scope, Buckway *et al.* determined the extinction coefficients of Fe in part of the EUV using a sample with Au capping for oxidation protection [168].

Additionally, EUV dispersion profiles of transition metals contain fine-structures. Resolving such features is relevant for calibrating optical components [169]. The performance and utilization of EUV-dedicated synchrotron beamlines can be also improved as a result of resolving such fine-structure.

For determining optical constants, we further address capping layers for mitigating surface oxidation and contamination. It can be argued that *in situ* deposition and characterization of coatings all in vacuum is a superior approach. However, surface contamination and EUV-induced oxidation are still observed in vacuum conditions [170,171]. Also, vacuum setups are prone to leaks and failures. The associated air-free techniques required for handling air sensitive samples are demanding. The use of thin C buffer layers was also found to reduce the interface roughness

[172]. Therefore, capping layers are more robust and sustainable as a solution for preserving the coated metals. HfTi alloy thin layer was used as capping for Nb. Carbonaceous capping layers were used here for coatings of Sc, V, Co, Mo, Hf, W, and Re. The optical constants relevant to the latter seven are shown together in Fig. 50 for comparison. Despite the advantages of such capping layers, their use necessitates approximations when determining optical constants of the capped material.

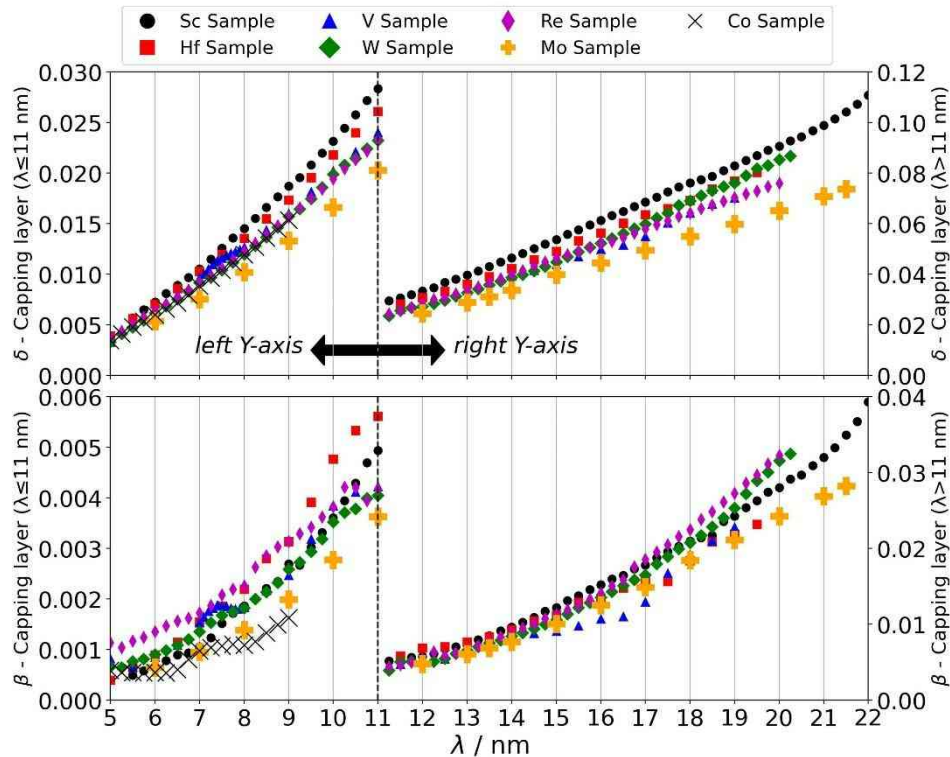


Fig. 50. The determined optical constants of the capping layers used for the Sc, V, Co, Mo, Hf, W, and Re samples. The values for the Sc sample are shown up to 22.0 nm. Left y-axes: $\lambda \leq 11.0$ nm; right y-axes: $\lambda > 11.0$ nm.

We reviewed the optical constants of C from seven different sources in the EUV, and clear inconsistencies were identified [74]. It is well-known that C has allotropes with significantly different optical properties. The layers' internal structures could be a factor [173]. As a results, it is not expected for the optical constants of the capping layers to be equal, but rather close, and that is clearly emphasized by Fig. 50. Hence, we see the assumptions behind the use of scaled optical data for C capping layers justified. A summary of the calculated scaling factors for the capping layers of the Sc, V, Mo, Hf, W, and Re samples is given in Table 5. The factors were calculated from the ratios between the sampled constants of the capping layers and the corresponding constants of the 40 nm-thick C layer (Fig. 2). The ratios were evaluated at the respective boundary wavelengths. An exception was made for the W sample, because the reflectivity of the 40 nm-thick C layer was not measured at exactly 20.25 nm (the boundary wavelength in this case). Accordingly, the optical constants at 20.0 nm were used instead.

Correspondingly, the optical constants of the carbonaceous capping layers are not to be taken as representative of pristine C. The maximum nominal capping thickness is 5 nm. Which is close to the thickness of surface contamination layers due to ambient conditions. Trace formation of

Table 5. Summary of the optical-constant scaling procedure for the capping layers of the Sc, V, Mo, Hf, W, and Re samples

Sample	Boundary wavelength/nm	Scaling factor	
		δ	β
Sc	25.0	1.268	1.426
V	19.0	0.962	1.139
Mo	21.5	0.792	0.915
Hf	19.5	1.046	1.056
W	20.25	1.06	1.324
Re	20.0	0.943	1.353

carbides is also possible. Therefore, the nature of those ultrathin capping layers is easily affected by environmental factors.

This work has clear advantages for spectroscopy and solid-state physics. Most XAS studies focus on *K*- and *L*-edges; by contrast, *M*-edges of *3d* transition metals are far less commonly investigated [174]. Historically, absorption spectra at *N*- and *O*- edges for heavy metals were not easy to investigate [175], due to instrumentation limits mainly. A considerable part of the existing literature on *O*- edges is for actinides, *e.g.*: ref. [176]. For transition metals, as solids, literature covering *M*-, *N*-, and *O*- series is substantially rarer than that for *K*- or *L*- series, but such studies can provide complementary information. However, the spectra of the outer shells tend to be broader and overlapping, making it difficult for interpretation and measurements.

Our results provide insight into the electronic properties of solids, similar to the way photoionization cross-section studies inform about the electronic structure of atoms. Photoionization cross-section measurements are essential for atomic physics, plasma physics and astrophysics. Extensive work was done for determining photoionization cross-sections of atomic transition metals – and their ions – in the EUV range [177–181]. Although those experiments relate to evaporated metals, there is resemblance between the absorption spectra of solid and gas phases in the long wavelength band of the EUV [182]. In particular, for V, Mo, Rh, Pd, Hf, W, Re, Os, Ir, Pt, and Au, the determined data here are quite useful when trying to understand the spectroscopic characteristics in the soft X-ray and EUV spectral ranges.

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Disclosures. The authors declare no conflicts of interest.

Data availability. 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. The determined optical constants for the metals and ZrO₂ are available on the PTB’s online Optical Constants Database (OCDB); [183].

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