# CHAPTER 1

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# Abbreviations

**AFM** atomic force microscopy.

**BESSY II** electron storage ring for synchrotron radiation.

**CCD** charge coupled device.

**DUV** deep ultraviolet.

**DWBA** distorted-wave Born approximation.

**EUV** extreme ultraviolet.

**EUVR** extreme ultraviolet beamline.

**FCM** four crystal monochromator.

**FEL** free-electron laser.

**FWHM** full width at half maximum.

**GISAXS** grazing-incidence small-angle X-ray scattering.

**GIXRF** grazing incidence X-ray fluorescence.

**HREELS** high-resolution electron energy loss spectroscopy.

**HZB** Helmholtz-Zentrum Berlin.

**LINAC** linear accelerator.

**MCMC** Markov-chain Monte Carlo.

**MLS** metrology light source.

**NMI** national metrology institute.

**PGM** plane grating monochromator.

**PSD** power spectral density.

## Abbreviations

**PSO** particle swarm optimization.

**PTB** Physikalisch-Technische Bundesanstalt.

**r.m.s.** root mean square.

**REUV** resonant extreme ultraviolet reflectivity.

**SASE** self-amplified spontaneous emission.

**SSD** silicon drift detector.

**SX700** soft x-ray beamline.

**TEM** transmission electron microscopy.

**TXRF** total reflection x-ray fluorescence.

**XRF** X-ray fluorescence.

**XRR** X-ray reflectivity.

**XSW** X-ray standing wave.

# Symbols

$\alpha_f$      scattering angle defined from the surface normal.

$\alpha_i$      angle of incidence defined from the surface normal.

$c$      speed of light in vacuum $c = 1/\sqrt{\epsilon_0\mu_0}$.

$e$      elementary charge.

$\epsilon(\omega)$      dielectric function $\epsilon(\omega) = \epsilon_1(\omega) + i\epsilon_2(\omega)$.

$\epsilon_0$      vacuum permittivity or electric constant.

$h$      Planck's constant, $h = 4.135667662(25) \times 10^{-15}$ eV s.

$\vec{k}$      wave vector; the wave number is $|\vec{k}| = k_0 = 2\pi/\lambda$.

$k_0$      modulus of the wave vector in vacuum (wave number) $k_0 = \omega/c = 2\pi/\lambda$.

$\lambda$      wavelength.

$m_0$      electron rest mass.

$\mu_0$      vacuum permeability or magnetic constant.

$n$      complex index of refraction, $n = \delta + i\beta$.

$\omega$      frequency.

$\vec{q}$      scattering vector / reciprocal space vector, $\vec{q} = \left(q_x, q_y, q_z\right)^T$.

$r_e$      classical electron radius $r_e = e^2/4\pi\epsilon_0 mc^2 = 2.82 \times 10^{-5}$.

$\rho_e(\vec{r})$      electron density at position $\vec{r}$.

$\frac{d\sigma}{d\Omega}$      differential scattering cross section.

$\theta_f$      azimutal scattering angle (out-of-plane scattering angle).

# 1

# Introduction

In 1959, Jack S. Kilby made an invention responsible for the technological revolution in the years to come. His development of the first integrated circuit was the realization of a logical element known as *flip-flop*, capable of storing a single bit, by implementing a layout that could host all required circuits on a single semiconductor wafer piece [28]. His achievement paved the way for the miniaturization of electronic circuits that enabled the technological advancements we experienced over the past 57 years and was awarded as part of the Nobel prize in physics in 2000 [45]. Only two years after the original invention, Robert N. Noyce submitted a patent on the fabrication of integrated circuits in monolithic single crystals using photo lithography to create the necessary artificial structure [43]. This technique of using light to transfer a pattern from a photomask onto a semiconductor wafer has prevailed over the course of the development and is still the primary method for the fabrication of computer chips today [38]. As the technology improved over time, the progress roughly followed *Moore's law* of doubling the transistor count on a unit area of the wafer every two years [41]. In consequence, the structured feature sizes on the wafers shrank to down to accommodate this large amount of circuits on a single chip. Today, structure sizes in the lower nanometer regime have been reached [25] but only through the implementation of additional methods appending the optical lithography. With the strong decrease in size, Moore's law is threatened to loose its validity [37, 48]. The technological requirements on the lithography systems used to fabricate those chips in mass production thus grew significantly.

A basic principle of optical resolution known as the *Rayleigh criterion* states, that the minimum structure size achievable with a purely optical system is proportional to the wavelength used [35]. Consequently, while the first lithography systems used in the semiconductor industry operated in the visible spectrum, wavelengths were reduced down to the deep ultraviolet (DUV) at 193 nm used nowadays to keep pace with Moore's law. However, with feature sizes of only a few tenths of nanometer now necessary, a significant further reduction of the wavelength can not be avoided as the lithography at optical wavelengths has reached its physical limits. The next-generation lithography uses wavelengths in the extreme ultraviolet (EUV) spectral range of 13.5 nm. This radiation is strongly absorbed by all materials, including air, challenging the design of

the optical lithography systems by effectively ruling out any optical design based on transmission lenses for focusing and imaging of the structures, as those are unavailable. With the semiconductor industry at the verge of a major technological change, the topic of reflective optical elements for EUV radiation has gained a large amount of attention and experienced extensive research efforts [7].

Back in 1972, Eberhart Spiller had proposed a new design for efficient mirror systems working at incidence angles near the surface normal for strongly absorbed radiation such as EUV. The idea was based on fabricating artificial layer systems reflecting portions of the incoming radiation at each interface that would interfere constructively at acceptable absorption levels overcoming the extremely low reflection otherwise seen from single surfaces [54]. The result are multilayer Bragg reflectors which fulfill the Bragg condition for constructive interference for specific pairs of wavelength and angle of incidence and thus require specific design. At angles close to the surface normal, layers with thicknesses in the order of half the wavelength are necessary, which requires fabrication methods capable of precisely depositing layers of only several nanometers in thickness. Since the original proposal, multilayer systems have been realized using evaporation and sputtering techniques and demonstrated to increase reflection [55, 59]. As the technology developed and more advanced sputtering techniques became available to fabricate at the necessary precision [57], the first important applications of focusing multilayer mirrors were space probes used for the observation of the sun in the EUV spectrum [15, 16, 56].

Theoretical models and calculations of candidate systems for large reflectivity close to normal incidence at a wavelength of 13.5 nm, show peak values of approximately 72 % by using multilayer systems based on molybdenum (Mo) and silicon (Si) [8, 9, 22]. State of the art systems fabricated today reach values closely above 70 % [5, 13, 14, 21, 39], which is still a few percentage points below the theoretical limit. This is of particular concern for the usage in EUV lithography systems, where 11 near-normal incidence reflections from the source to the wafer are required to image a structure [26, 60]. Even at the theoretical threshold, with 11 reflections only 3 % of the radiation reaches the wafer. Therefore, the small difference to the theoretical reflection limit additionally has a large impact on the total radiant power at the wafer level and is a very crucial point in the development of the next-generation lithography using EUV radiation.

While the semiconductor industry undoubtedly is a strong driving force in the development of EUV multilayer optics for 13.5 nm wavelength, mirrors for other spectral ranges suffer from the same issue. A system relevant to this work is a mirror designed to reflect radiation in the range of the so-called *water window* which is found between 2.3 nm and 4.4 nm. The water window is of special interest, because radiation in this spectral range shows low absorption in water, while it is absorbed by many elements, most importantly carbon and nitrogen, naturally occurring in organic molecules such as proteins [29]. This allows the study of biological systems in their native environment (water), where many proteins are biologically active. With the ability to produce radiation at those wavelengths at free-electron laser (FEL) sources [1, 49], more applications with strong and coherent pulses come in reach. High resolution imaging of protein samples, in addition to requiring the short wavelength, needs sufficient reflected radiation intensity and, more generally, optical elements capable of focusing and magnification. This can be achieved with high reflectance multilayer mirrors [24, 31]. A candidate system relevant in this range is made from chromium (Cr) and scandium (Sc) applying the very same principle as introduced above, however, having much thinner layer thicknesses due to the shorter wavelength. While the theoretical reflectance limit at 3.1 nm wavelength is

calculated to reach values above 50 % [47], state of the art mirrors only show reflectivities below 20 % [20, 62], less than half of the theoretically possible values.

The main reasons for radiation loss beyond unavoidable absorption inside the materials of both the Mo/Si and Cr/Sc multilayer systems are imperfections at the interfaces, such as compound formation, intermixing and roughness. As a result, the perfect multilayer system is distorted, since the interfaces are not chemically abrupt anymore. Thus, intermixing and compound formation lead to a diminished optical contrast and consequently to lower reflectance at the respective interface [42]. This is a known problem for multilayer optics, and measures to counteract this effect are the introduction of barrier layers hindering the formation of intermixing layers in some of the systems [13, 14]. In the case of roughness, the result of reduced optical contrast at the interfaces is the same on average for the impinging wavefield, however, in this case with the addition of diffuse scattering outside the specular beam direction [53], which is not present in the case of pure intermixing.

To minimize interface distortions and to ultimately increase the reflectivity of the respective systems, the research and industry groups concerned with fabricating multilayer mirrors require detailed information on the actual structural properties and the interface morphology of their samples. The characterization of those multilayer systems is thus a cornerstone in the effort for improvement and the fundamental understanding of the effects involved. There are several characterization techniques that exist and have been applied to assess and quantify the structure of the layer system, roughness and intermixing of materials at the interfaces of multilayer mirrors in the past. They can be roughly categorized as direct scanning methods and indirect ensemble methods.

Some widely used example of the first category is transmission electron microscopy (TEM), which establishes a microscopic approach to the problem of assessing the interface morphology with resolution at the nanoscale [6, 58]. By imaging the layer stack, interface imperfections can be made directly visible. In combination with high-resolution electron energy loss spectroscopy (HREELS), element specific interface profiles can be deducted giving insight into the intermixing behavior of two (or more) materials at the interfaces [17, 44]. A large downside of both methods, however, is the intrinsically local area of the image and thus the characterization of only very small local portions of the entire sample. Apart from that, the stack needs to be cut open to apply these techniques and thus leads to a destruction of the sample.

Another very popular method often used before and after deposition of a multilayer stack is atomic force microscopy (AFM) [10]. It is a scanning technique with nanometer resolution allowing to determine the morphology of a surface and thus investigate their roughness. However, it faces the same locality obstacle as TEM or HREELS and can only operate on exposed areas. Thus, the morphology of buried structures remains hidden for this method. Nevertheless, it is applied to determine the initial substrate roughness and the condition of the final top surface as an important prerequisite for high-quality multilayer mirror fabrication [6, 36].

Apart from the direct and local scanning techniques, indirect ensemble methods based on the elastic scattering of radiation are accurate and extensively used in multilayer characterization. Examples include X-ray reflectivity (XRR) and EUV reflectivity with resonant extreme ultraviolet reflectivity (REUV) as a variation of the latter. They are employed as standard approach in multilayer mirror fabrication and the subsequent characterization [6, 14, 33]. Other techniques sensitive to structural properties are spectroscopic ellipsometry and X-ray fluorescence (XRF). Ellipsometry delivers information

on the optical constants and layer thicknesses by measuring the altering of the polarization state of the impinging radiation after reflection from the sample [4, 34]. With XRF, fluorescence radiation of the materials inside the multilayer stack is exited using X-rays energetically closely above the respective absorption edges of these materials and detected to analyze the structure [27, 30]. The major advantage of all these techniques is, that they are destruction-free, contact less and deliver fast information on the buried structure as well as on the top surface condition. Furthermore, statistical information across a large area depending on the beam footprint of the impinging radiation is obtained in contrast to the aforementioned local methods. However, it is no longer possible to directly gain information on the multilayer stack as theoretical models are required in all above examples to calculate and compare the expected results from a certain model to the measurement outcome. This is known as the *inverse problem*. Reconstruction of the model parameters by fitting calculations to the experimental data raises the question of uniqueness and accuracy of the solution found. Even the applicability of the model and its limitations itself are of importance in these considerations. Several researches have shown, that the combination of EUV and XRR can lead to significant improvements in the accuracy compared to standalone measurements with each technique individually [61]. By XRF, similarly further complementary information can be added to assist in the solution of this problem [23].

While structural information on the layer stack can be obtained through reconstruction of a model by conducting these experiments, only limited information is gained on the roughness of the interfaces which can not be distinguished from intermixing. However, as only roughness causes diffuse scattering, the analysis of the off-specular intensity upon irradiation of a multilayer stack is a natural tool for characterization of the interface morphology. A lot of theoretical and experimental work has been conducted in relation to the study of diffuse scattering from multilayer samples in grazing incidence geometries using X-rays, e.g. by grazing-incidence small-angle X-ray scattering (GISAXS), at small incidence angles [11, 12, 32, 40, 46, 52, 53], but also in the optical and EUV regime [2, 3, 18, 19, 50, 51], to deduct the desired information on the interface roughness.

As the German national metrology institute (NMI), the Physikalisch-Technische Bundesanstalt (PTB) is dedicated to precise measurements related to all fields of physics and technology providing metrology as its core mission. In fact, the international metrology organization, the Bureau International des Poids et Mesures, defines* metrology as *"the science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology."*. In this spirit, this thesis seeks to provide metrology for the important field of multilayer optics. It is dedicated to the accurate and complete characterization of the structural properties and the interface morphology of multilayer mirrors.

For each method above, the uniqueness problem associated with any model based indirect characterization approach is largely unanswered and requires to be addressed. In this thesis, the data from experiments based on EUV reflectivity, XRR, REUV and XRF is analyzed with respect to answering this question. Experimental uncertainties inevitably associated with any measurement and model uncertainties are investigated with respect to the effect on the results obtained from each method employed. Based on theoretical optimization algorithms, this work extends the established characterization techniques to deliver confidence intervals for each reconstructed parameter of the underlying models, which allow to validate the results. As the layer thicknesses enter the sub-nanometer

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* Source: http://www.bipm.org/en/worldwide-metrology/

regime for the Cr/Sc multilayer mirrors designed for the water window regime, the model is extended to deliver a consistent result taking into account all of the aforementioned methods. As a result, those are assessed with respect to their applicability and necessity for the structural investigation of such samples.

Based on the methods of structural characterization, layer thicknesses and even the distortion of the interfaces become accessible. However, they lack in the ability to identify these distortions as either roughness or intermixing. This can only be achieved by combining the results of the structural characterization with a method sensitive to roughness and again validate the result with respect to accuracy. This issue is addressed here by measuring EUV diffuse scattering close to the wavelength of operation for the two mirror classes. This has some unique advantages as compared to GISAXS or other methods operating with grazing angles of incidence. Any measurement using small incidence angles is inherently limited to flat or convex surfaces. Focusing mirrors, however, usually are concavely curved and thus not suitable for this characterization technique. Instead, with EUV diffuse scattering with radiation impinging near normal incidence, it is possible to extract the roughness information for those samples as well. In addition, the wavelengths of operation for these mirrors are directly applicable to conduct this analysis. Based on the structural reconstruction, EUV diffuse scattering is established as a suitable method for completing the characterization of the multilayer mirrors with respect to their interface morphology.

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