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Laboratory GIXRF as a tool for fast screening of stratified samples with sub-nanometer thickness – the example of CrSc multilayer water window optics

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Abstract

Efficient multilayer optics for radiation in the water window range are difficult to manufacture due to extremely small layer thicknesses and severe intermixing of elements between the layers. Therefore, adequate analytics and short feedback loops are of utmost importance for manufacturers to improve performance and efficiency. We show the possibility for non-destructive elemental depth profiling with commercial laboratory equipment using four real-life CrSc multilayer samples. Comparative measurements at the laboratory of PTB at the synchrotron radiation facility BESSY II validate the results and prove the potential of laboratory equipment for the fast and reliable analysis of stratified materials with sub-nanometer layer thicknesses.  2019 Optical Society of America

1. Introduction

Periodic multilayers are important optical components designed to reflect electromagnetic radiation as optical systems often require redirection of photon beams. Depending on the material selection and structure design of the multilayers, these mirrors have high peak reflectivity at certain wavelengths. This leads to a wide range of applications. The CrSc based multilayer system is a promising candidate as high reflective mirror in the soft X-ray range and particularly in the water window (WW, between oxygen and carbon K absorption edge, from 2.3 to 4.4 nm) [1]. For radiation in this spectral range, the absorbance of biological material, of which carbon is the main component, is more than one order of magnitude larger than the usually surrounding water. This enables observing living specimen in aquatic environment and, thus, contributes to the development of scientific instruments

such as microscopes dedicated to biological samples [2-4]. A fine understanding of the substructure of a manufactured optic and the involved processes between the materials constituting the stratified structures is crucial for developing, optimizing and eventually improving the performance of the multilayer. Therefore, increased effort is dedicated to their characterizations, preferably in the laboratory.

CrSc is a suited candidate for the WW range, with a calculated reflectivity reaching 60 % [5]. In practice, the ML optics only reach 32 % due to well-known diffusion which directly reduces the optical contrast at the interfaces and consequently limits the optical performance of the mirror [6]. One solution for this problem is to introduce diffusion barriers which prevent interdiffusion. In this work CrSc ML samples with an additional B₄C layer are investigated. The

samples are deposited on sliced and polished Si (100) wafers using magnetron sputtering [7, 8].

Due to the sub-nanometer thin individual layers and the strong intermixing processes, characterization of the samples is challenging. As has been shown by Haase et al. [9], several complementary methods are necessary to find a consistent sample model with low uncertainties for all evaluated parameters. One possible nondestructive method which can be used to characterize the in-depth elemental composition of CrSc multilayers is grazing incidence X-ray fluorescence (GIXRF). This analytical technique employs X-ray radiation to generate X-ray fluorescence from the sample. This fluorescence is element specific, thus the reconstruction of the elemental distribution of the sample is possible. A depth gradient of the elemental composition within the sample is concluded from measurements under distinct incidence angles; each angle corresponding to a specific information depth. The result of a GIXRF measurement is an angular profile where the intensities for each relevant fluorescence line obtained from a deconvolution process are plotted over the angular position at which the corresponding spectrum was measured. When the incident X-ray radiation on the sample has high temporal and spatial coherence, i.e. high monochromaticity and low divergence, an X-ray standing wave (XSW) field is generated by interference of the incident beam with the reflected beam. Strong changes in the refractive index, i.e. sharp elemental boundaries, lead to increased reflection compared to smooth transitions, and thus a more pronounced XSW field. As a result a characteristic interference pattern can be observed in the angular profile. The shape of the interference pattern is predominantly influenced by the elemental depth distribution and the quality of the interfaces of the individual layers of the sample; a rough or diffuse interface lowers the contrast of the interference while the thickness of the layers shifts its angular position. To quantify the measurements the data are compared to simulated GIXRF profiles from sample models. The simulations are adapted to fit the measured data by varying initial sample parameters (e.g. layer thickness, density, roughness).

GIXRF is often used at synchrotron radiation facilities, due to high photon flux and because quantification of the measurements demands reliable knowledge about beam and geometry parameters. The latter follows from the quantification being carried out by fitting simulated data of a sample to the measured ones. Precise knowledge of experimental parameters thus lowers the uncertainty of the results.

As the access to beamtime at synchrotron radiation facilities is limited and direct laboratory access is desirable especially for materials development, laboratory setups for angular resolved XRF investigations are increasingly developed and tested [10-12]. As GIXRF is a nondestructive technique and no to minimal sample preparation is necessary for the measurements, a reliable laboratory setup facilitates the increased application of this technique e.g. in process control feedback loops.

We present measurements on four CrSc multilayer samples with laboratory equipment and validate the results with results obtained from experiments using synchrotron radiation.

2. Measurements

The laboratory measurements are performed with a Bruker S4 T-STAR™. This instrument was designed for total reflection XRF (TXRF) applications and upgraded with the option to perform angular scans and thus GIXRF measurements [13]. For the presented measurements, the Mo K characteristic radiation of the X-ray tube at 17.4 keV was monochromatized and focused on the sample by parabolic graded multilayer optics. The angular discrimination is achieved by tilting the sample relative to the beam in defined steps corresponding to angular positions and thus depths in the sample. While the hardware theoretically allows an angular step size of 0.0005° the achievable angular resolution is limited by the beam divergence to $\text{FWHM} = 0.014^\circ \pm 0.003^\circ$. The step size for the measurement was set to 0.001° . The real time of the measurement per angle was 15 seconds, which resulted in a measurement time of 75 minutes for a complete GIXRF scan. Fluorescence intensities were derived through the deconvolution software of the manufacturer (Spectra 7.8.2.0). As shown in a previous work [14], measurements of multilayers with bilayer thicknesses of several nanometers are feasible with the setup. However, the here investigated CrSc multilayers have bilayer thicknesses below 2 nm and suffer from strong intermixing of the individual layers.

To validate the capabilities of the commercial setup and gain reliable information about the in-depth composition of the samples, one CrSc multilayer (MP15004) is additionally measured with synchrotron radiation. The reference-free GIXRF experiments were carried out in the PTB laboratory at the electron storage ring BESSY II, employing the four-crystal monochromator beamline for bending magnet radiation [15]. PTB's in-house built instrumentation [16] for reference-free XRF experiments was used. The setup is installed in an ultra-high vacuum chamber equipped

with a 9-axis manipulator, allowing for a very precise sample alignment with respect to all relevant degrees of freedom. The emitted fluorescence radiation is detected by means of a calibrated silicon drift detector (SDD) mounted at 90° with respect to the incident beam. Additional calibrated photodiodes on a separate 2θ axis allow for X-ray reflectivity (XRR) measurements simultaneously with the reference-free GIXRF measurements as well as for the determination of the incident photon flux.

An incident photon energy of 6.5 keV was chosen which allows to excite both Cr-K and Sc-K shell fluorescence radiations. At each incident angle, the recorded fluorescence spectra are deconvolved using the known detector response functions [17] for the relevant fluorescence lines as well as physically modelled background contributions. Subsequently, the fluorescence intensities are normalized to detector efficiency, incident photon flux and life time and corrected for the solid angle of detection as described in [13].

3. Samples

Four multilayered samples were available with nominal layer thicknesses and compositions as listed in **Erreur ! Source du renvoi introuvable.** and shown in Figure 3. The substrate of the samples is 1 mm pure silicon with the multilayer structure magnetron sputtered on top, see [7] for details. In all samples the designed Cr layer is 0.6 nm thick, Sc 1.0 nm, the period is repeated 100 times with a 2.5 nm thick B₄C capping layer. The samples differ in their sequence of layers and the B₄C layer thickness.

Sample	Structure (from wafer)	Thickness Cr / nm	Thickness Sc / nm	Thickness B ₄ C / nm	Designed period / nm	XRR measured period / nm
MP15004	Cr/B ₄ C / Sc	0.6	1.0	0.3	1.9	1.645
MP15007	Cr/B ₄ C / Sc	0.6	1.0	0.6	2.2	1.720
MP15008	B ₄ C/Cr / Sc	0.6	1.0	0.6	2.2	1.718
MP15009	B ₄ C/Cr / Sc	0.6	1.0	0.9	2.5	1.833

Table 1: Design parameters of the investigated chromium scandium multilayer samples. The last column lists the period thickness obtained from X-ray reflectometry measurements [7].

All samples were analyzed with hard X-ray reflectivity [7] with the results displayed in the last column of

Erreur ! Source du renvoi introuvable. The deviation of the designed to the measured period thickness already suggests a severe intermixing of layers.

4. Evaluation

For the laboratory measurements, a Monte Carlo code was developed to calculate the solid angle of detection. To simulate the GIXRF profiles an in-house, C++ based code is employed. The code forward calculates from a sample model the angular dependent fluorescence intensities based on the solution of the Sherman equation for each relevant fluorescence line taking into account the beam divergence of the incident radiation. Additionally the algorithm is extended as introduced in de Boer [18] to account for refraction and reflection.

The goal of the evaluation is to find the sample model whose simulated angular fluorescence profile reproduces the measured one best. In an iterative fitting procedure, the parameters of the sample model are then changed to minimize the χ^2 value.

For the calculation the sample model must include the number and succession of layers. For each layer the composition, density as well as optical parameters are necessary. The analytical challenge is to establish initial parameters for this calculation and determine the minimal possible number of variable parameters. For example the number of thickness values can be reduced by one by introducing a fixed overall period thickness and assuming the same thickness for every period. Additionally, material parameters such as known stoichiometry or density can be introduced. If no convergence of simulated to measured values is achieved, the layered model must be changed successively.

In the presented case, the severe intermixing of layers was selected to be modelled with additional layers instead of roughness parameters. The best suited sequence and composition of the layers was found through sequential adding of intermixing layers starting from the designed sequence. For sample MP15004 this resulted in a sequence of CrSc/Sc/CrScB₄C/CrB₄C/Cr.

The densities of Sc and Cr are used as calculated by Haase et al. [9] to be 2.81 g/cm³ and 7.05 g/cm³ respectively due to similar layer thicknesses and production processes. For B₄C the tabulated value of 2.52 g/cm³ is assumed. The densities of the remaining layers are calculated from linear combinations according to the assumed composition, see Table 2. This also applies to the optical parameters for the layers which are extracted from the NIST database [19].

Layer	Density / g/cm ³	Composition / at%
CrSc	5.7	Sc60Cr40
Sc	2.81	Sc100
CrScB4C	4.8	Sc80Cr10(B4C)10
CrB4C	4.65	Cr50(B4C)50
Cr	7.05	Cr100

Table 2: Density and composition assumptions for the fitting routine.

Thus, the thicknesses of each individual layer are the only variable parameters in the algorithm with the minimum thickness of 0 nm. The maximum is restricted by the period thickness of the multilayer obtained by XRR measurements [7], see **Erreur ! Source du renvoi introuvable.**

5. Results

For the sample MP15004 two sets of data were collected, one with laboratory equipment and the one measured at BESSY II in order to compare performance and validate the laboratory data. The same initial parameters are used to fit the GIXRF data. The used algorithm differentiates solely in the deviating instrumental parameters between the two setups: the initial X-ray energy, the beam divergence and the solid angle of detection. As the laboratory setup is not known sufficiently well to calculate the absolute intensity values both calculations are normalized to an average intensity value for better comparison. Thus, the shape of the profiles and not the absolute intensities are fitted in the analysis.

The fitted layer thicknesses are shown in Figure 1 with estimated uncertainty values obtained from the square root of the diagonal elements of the covariance matrix of the fitting procedure. Therefore, the superior statistics of the laboratory experiment are included in the uncertainty values. In contrast, the assumed lower systematic uncertainty of the quantification due to the fully calibrated setup of the synchrotron measurements is not reflected in the uncertainty values. For all layers the fitted thicknesses are within the statistical uncertainties of the two data sets, indicating also low systematic errors for the laboratory setup. The values of the layer thicknesses from the laboratory fit are additionally listed in Table 3. The estimated uncertainty values might underestimate the actual confidence limits.

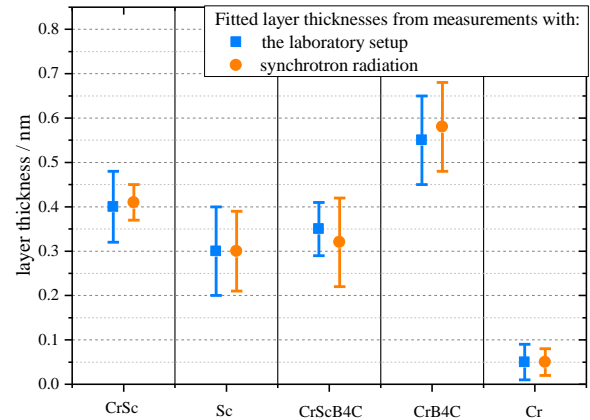


Figure 1: Results of the fitting procedure for data obtained from sample MP15004 with the laboratory setup and the setup at BESSY II.

A comparison of the relevant angular range of the measured GIXRF profiles from the laboratory and synchrotron radiation measurements with the simulated profile according to the fitted sample model is shown in Figure 2. The profiles show the interference features in the Cr and Sc K α profiles which are at different angular positions due to the different excitation energies. The laboratory data show superior counting statistics. The representation of the results demonstrates that the found sample model reproduces the measured GIXRF profiles by both experiments. Indeed, the difference in the two sample models is so small that when using the values derived from one measurement and simulating it for the other measurement, the shape of the GIXRF profile does not change significantly enough to be visibly discriminated.

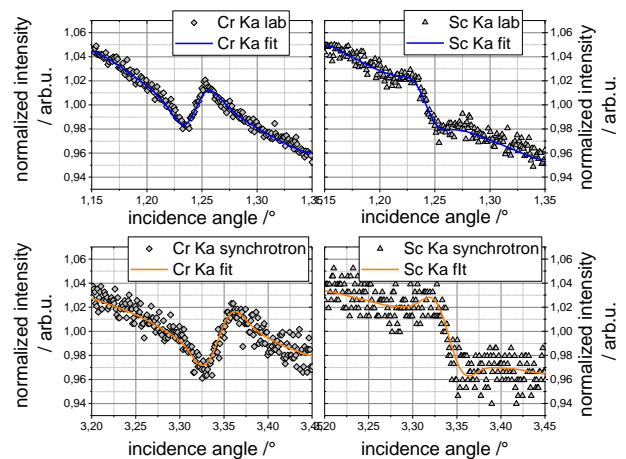


Figure 2: Measured (dots) and fitted (lines) GIXRF data obtained with the two setups for the Cr and Sc K α fluorescence line of sample MP15004.

The comparison of the fitting results of the sample MP15004 indicates that the laboratory GIXRF setup is characterized well enough concerning geometric parameters, divergence and sample adjustment that a modeling with sufficient sensitivity and accuracy is feasible. Thus, the remaining 3 samples, for which no synchrotron data are available, are modeled solely using the measured laboratory data. Other than the order of the individual layers the same routine for fitting is used for the samples as for MP15004 with initial parameters listed in Table 2 and 3. For all samples, the algorithm converged, and the calculated layer thicknesses including the estimated uncertainties are listed in Table 3. For none of the samples, a distinct chromium layer is found and the chromium is contained solely in the intermixing layers. This is visualized in Figure 3, where the designed and the corresponding fitted period is presented for all samples by stacked box plots.

In all samples, the measured period thickness is lower than the designed one indicating that the strong intermixing increases the density of the initial components. No clear trend for the influence of the sputtering order or the thickness of the B₄C layer can be observed but for safely drawing such conclusions, a larger number of samples should be measured. It is however observable that solely in the sample MP15004 with the thinnest B₄C layer, remnants of a chromium layer are found. The optimal solutions of all other samples resulted in samples without a pure chromium layer.

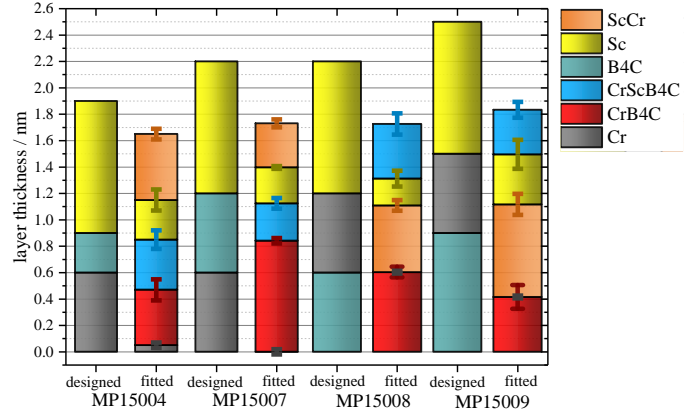


Figure 3: Designed and fitted layer sequence, composition and thickness for the 4 measured samples.

6. Conclusions

In this work a commercial laboratory GIXRF spectrometer is utilized for the analysis of multilayered samples with sub-nanometer single layer thicknesses and strong intermixing. The small thicknesses in combination with the intermixing pose challenges to the analytical interpretation of the data, rendering this investigation a showcase of the reachable certainty of measurements in the laboratory. A comparison to synchrotron data exemplifies that such measurements are feasible even with equal or superior counting statistics in the range of minutes to hours.

Sample-wise, the presented results strongly emphasize the difficulties of manufacturing and in the end employing CrSc multilayers as optics for water window radiation. The functionality of these multilayers crucially depends on the quality and stability of the interfaces between the individual layers within the sample which are disrupted by the intermixing processes. In the presented case of samples with sub-nanometer layer thicknesses, the addition of a B₄C layer does not inhibit intermixing substantially. The presented mathematical sample models might not be a full absolute representation of the samples, they however unequivocally demonstrate the intermixing and yield consistent and reproducible results.

Other investigations have shown that for the characterization of this type of samples a multitude of investigations is necessary [9, 20]. The possibility to perform quantification of multilayer samples with sub-nanometer layer thicknesses with strong intermixing in laboratories has great potential to support the investigation of novel multilayers towards the ultimate goal of high reflectivity. In fact, this work paves the way for short feedback loops and process control,

Layer	MP15004 thickness / nm		MP15007 thickness / nm		MP15008 thickness / nm		MP15009 thickness / nm	
	initial	fitted	initial	fitted	initial	fitted	initial	fitted
CrSc	0.12	0.40 ± 0.04	0.55	0.33 ± 0.03	0.3	0.51 ± 0.04	0.5	0.71 ± 0.08
Sc	0.5	0.30 ± 0.09	0.3	0.27 ± 0.01	0.4	0.21 ± 0.06	0.2	0.37 ± 0.11
CrScB ₄ C	0.22	0.35 ± 0.10	0.38	0.28 ± 0.04	0.26	0.41 ± 0.08	0.48	0.34 ± 0.06
CrB ₄ C	0.6	0.55 ± 0.10	0.44	0.84 ± 0.02	0.6	0.61 ± 0.04	0.6	0.42 ± 0.09
Cr	0.2	0.05 ± 0.03	0.05	0.00 ± 0.02	0.15	0.00 ± 0.01	0.05	0.0 ± 0.01

Table 3: Results of the thickness values for the individual layers of the four investigated samples with initial fitting values.

facilitating the development of novel structures and nanomaterials in an unprecedented way.

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