State-of-the-art Thin Film X-ray Optics For Conventional Synchrotrons And FEL Sources

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Abstract. Selected aspects of simulation, preparation and characterization of total reflection and multilayer X-ray optics will be discussed. The best multilayer is found by calculating the optical properties of the coating. Sophisticated improvements in deposition technology allow the precise realisation of the specified parameters when manufacturing the X-ray optics. The quality of the shape of the substrate for the optics is measured with the aid of profilometry. X-ray reflectometry measures both film thickness as well as their lateral gradient. Last but not least we will be showing results of the development of carbon coatings as total reflection mirrors for FEL (free electron laser) sources. Over the past years we have developed optimized optics for the XUV range up to 200 eV. First FEL irradiation tests have shown that carbon coatings offer high reflectivity >95%, high radiation stability, good uniformity in thickness and roughness. An optimized coating of two stripes for different beam energies was produced especially for a tomography beamline, where a Ru/C multilayer was chosen for energies between 10 and 22 keV and a W/Si multilayer for energies between 22 and 45 keV.

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INTRODUCTION

Nowadays, a large variety of thin film based reflective optics exists for home-lab X-ray instruments [1,2] and synchrotrons [3,4]. The most widespread optics at synchrotrons are total reflection optics, where a single layer reflects the incident beam under a very flat angle – typically below 2°. On the other hand, bragg diffraction on single crystals was used for directing synchrotron beams. More and more multilayer optics are now also used for shaping and reflecting the synchrotron beam in a defined manner. Additionally the energy spectrum of the reflected beam can strongly be influenced. The reflection profile of the multilayer can be designed exactly to the user's requirements.

Reflective X-ray optics can basically be characterized by two features: their shape and their surface, which in most cases is modified by deposition of single or multilayer films. Parabolically shaped optics cause a collimated beam, elliptical shapes cause a focused beam [1]. By modifying the surface with a multilayer, a reflection of up to > 90% can be achieved. Lateral gradients of the multilayer thickness are needed to fullfil Bragg's Law at every point along the substrate. The thickness of single layers is typically in the nanometer region, thus requiring perfection of a few tenth parts of a nanometer. The lateral gradients need to be within a few percent of the ideal gradient. Perfect substrate surfaces are needed to enable perfect multilayer growth, consequently leading to high reflectivity. A range of 0.3 nm or even lower is required for the RMS (root mean square) roughness.

DESIGNING AND MANUFACTURING REFLECTIVE X-RAY OPTICS

We design, manufacture and characterize optics with the aim to achieve the best quality for the specific application [2]. The optical properties are simulated to find the best multilayer materials. In addition, the optimum

layer thickness profile and substrate shapes are selected. If necessary complete beam paths can also be calculated. For many lab optics the manufacturing process includes the preparation of shaped substrates by bending silicon wafers. The most important part of the process is the deposition of thin layers for total reflection and multilayer optics. We use the very reliable and reproducible method of magnetron sputtering. Last but not least the optics are characterized by profilometry to examine the shape of the optic and by X-ray reflectometry to check the quality of film growth.

Designing Multilayer Optics

Simulating the optical properties enables us to develop optimized optics. When chosing multilayer materials, their optical constants dispersion δ and absorption β are determined according to the application. In addition, the optimum layer thickness profile and substrate shapes are selected. Furthermore, the complete beam path can be calculated by so-called ray tracing methods. Optical constants are accessible from the Internet (http://www-cxro.lbl.gov/). With suitable freely available simulation programs, e.g. (http://cletus.phys.columbia.edu/~windt/idl/) the characteristics of multilayers such as reflectivity, monochromatization or bandwidth can be calculated for all desired wavelengths. In fact, not all types of multilayers which are in theory the best possible, can actually be manufactured. In reality, their properties are often degraded by other specific material parameters such as interdiffusion and surface roughness. The actual material density is changed when two materials inter diffuse. Unfavorable surface energies reduce the wetting of one material on the other, thus leading to high roughness at the interfaces. Parameters such as actual density (in thin films the material densities are often reduced) and roughness are taken by the simulation programs into account.

Besides helping to choose the right multilayer, ray tracing enables us to calculate the optimum substrate form and length. Calculating with ray tracing also takes the multilayer properties, the emission profile of the X-ray source, the complete wavelength spectrum as well as the geometrical dimensions of the source into consideration. The divergence and the beam flux can therefore be calculated at every place of the beam course.

Making the Substrates

Typical substrates for X-ray optics require extremely high shape precision. For example, the parabolic shape of parallel beam optics for lab X-ray diffractometry has a sag of 49 µm, and should show a slope error smaller than 12“ and for high resolution applications smaller than 5“. This means that local variations of the ideal profile must be well below 0.5 µm. These shapes are either produced by etching, lapping, burnishing etc. from a block – slope errors down to 0.1“ are possible - or by bending thin silicon wafers which are then glued to a backing plate. The latter is the more economic technology which, however, causes a significantly larger (10“ - 20“) slope error.

For the synchrotron applications shown in section Applications ultra-precise silicon substrates were used. In order to preserve the beam quality, for FEL applications high accurate Si mirrors are needed, these mirrors were been provided by Carl Zeiss Laser Optics. Slope error values in the order of 0.05“ (rms) could be achieved by means of ion beam figuring (IBF) for relatively large flat mirrors (510 mm) and spherical gratings (seeding monochromator). The surface figure errors were been measured by Interferometry (Carl Zeiss D100) for flats and spheres, other geometries were been tested by high precision coordinate measurement technique (Carl Zeiss M400). Achieved microroughness values are in the order of 0.1 nm, qualified by microinterferometry (Micromap Promap 512).

The plane Si substrates for a multilayer spectrometer for the Swiss Light Source were also provided by Carl Zeiss. The <111> crystal orientation on the optical surface could be adjusted better than 0.01°. Also by means of IBF slope errors of 0.04“ (rms) could be achieved, the required microroughness within the high spatial frequency range, responsible for the multilayer performance, was around 0.1 nm, measured by AFM.

Deposition Technology

High precision is necessary when producing the multilayers for X-ray optics. The single layers should grow with very smooth surfaces. For this physical vapor deposition (PVD) processes are used. They work at low pressure and provide a high surface mobility of the particles during growth. Smooth interfaces without larger pores can be derived. Fig. 1 shows a typical magnetron sputtering system (left) and the plasma inside the chamber (right). In most applications, the single layers must not show measurable variations of the layer thickness in depth, as otherwise peak reflectivity is reduced with the reflected beam showing higher divergence. Optics for collimation as well as for

focusing purposes require a lateral d-value gradient with a maximum allowable error of few percent from the ideal d-value dependence.

Incoatec uses sputtering technologies to fulfill the above requirements. By magnetron sputtering X-ray optics on 6” wafers can be produced within layer homogeneity to ± 0.1%. Furthermore, rectangular substrates can be coated with lengths up to 50cm. Optics up to 150 cm in length will be available in the near future. During the coating procedure the substrates are not heated, as the growth of amorphous single layers is preferred. Crystalline layers would not allow a film growth with smooth d-value gradients. Substrate cooling is not essential either, because during the coating process the temperature does not rise above 100°C.

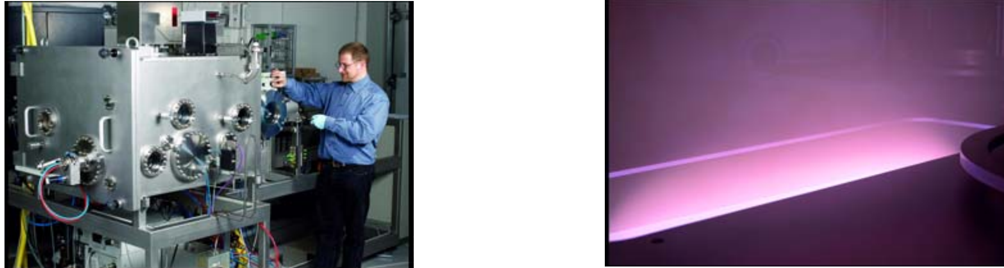

FIGURE 1: left: PVD coating machine for X-ray optics; right: Plasma of a magnetron source inside the chamber

Due to the large number of varying applications, many different coating materials are used for multilayer X-ray optics. We have experience with heavy elements and compounds, such as W, WSi_2, Mo, La, Ni, Cr, Fe, TiN, and TiO_2 as reflectors. As a spacer we use light materials and their alloys, for example C, Si, B, CN, BN or B_4C. Conductive materials can be sputtered with DC-sputtering, while for the semi-conducting or isolating materials RF- and MF-technologies are required. To achieve high precision layer profiles, the substrates are moved in the coating chamber on complex paths. Often, the manufacturer’s special knowledge is based in these details.

Characterizing X-ray Optics

The multilayer properties can be characterized by X-ray reflectometry (XRR). Using XRR, reflectivity, roughness and layer thickness can be determined. The lateral layer thickness profile can be determined by measurements at various positions along the mirror length. Fig. 2a shows a typical multilayer reflectometry plot with intensity maxima as a function of Bragg’s angle.

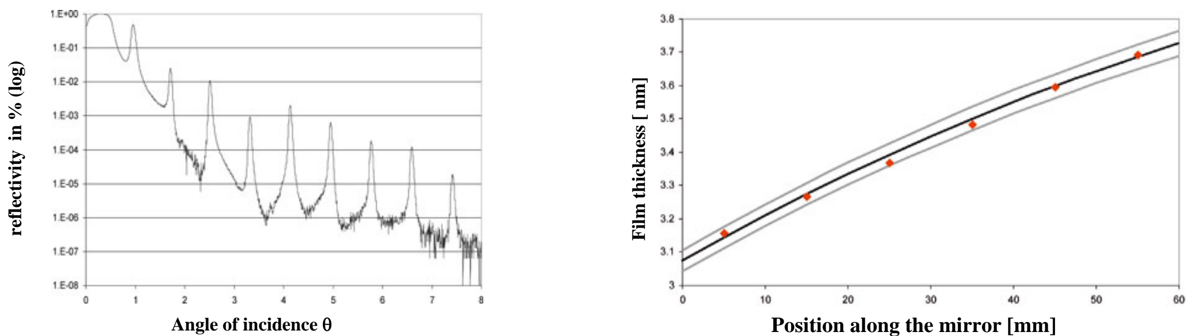

Figure 2: a) X-ray reflectometry measurement of a multilayer; b) Multilayer thickness profile along the mirror position

Sharp Bragg reflections with high angles show that the multilayer has grown very homogeneously in depth and that it has smooth interfaces. For d-value graded structures, measurements like those shown in Fig. 2a are performed at various positions along the mirror. The plot in Fig. 2b shows the resulting first-order Bragg angles, together with the theoretical values (solid line) given by the mirror design. Fig. 2b shows the high conformity between theoretical and experimental layer thicknesses (points), as a result of our advanced, highly controlled deposition process. The variations are clearly smaller than 1% (grey lines indicate a ± 1% range). During the development stage, selected multilayer systems are characterized by transmission electron microscopy (TEM). With this method the multilayer growth can be examined in detail. Fig. 3a shows a typical TEM micrograph of a synchrotron multilayer Ru/C with single layer thicknesses of 2 nm and 100 pairs. Fig. 3b shows the d-values and local deviations. The film shows a very homogeneous distribution of the thickness.

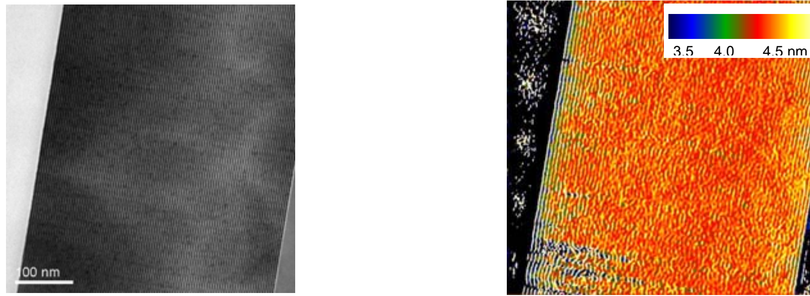

FIGURE 3: a) TEM image of a Ru/C multilayer, b) False colour display of the local change of the bilayer thickness with respect to the medium thickness (TEM Measurements of D. Häußler and W. Jäger, University Kiel)

APPLICATIONS

The following shows two applications with our X-ray optics. In close collaboration with GKSS we developed special carbon coatings as total reflection optics with a very high density for the FEL test facility at Desy, Hasylab. Fig.4a shows, that in the area of interest in the energy range of 50 to 200 eV the carbon film exhibits a very high reflectivity above 96%. Simultaneously the roughness is quite low. In comparison, the red curve shows an ideal diamond with a density of 3.5g/cm^3 and zero roughness. The film properties are quite close to the optimum below 200eV. First experiments with the optics in the FEL beam show a good resistance to radiation damages [5,6]. Fig 4b shows a multistrip optic for the tomography beamline at the Swiss Light Source. We produced a Ru/C multilayer with 100 layer pairs and a single layer thickness of 2nm for the reflection of energies in the range of 10 to 22 keV. A second multilayer underneath consists of W/Si with single layer thicknesses of 1.5nm for the energy range of 22 to 45 keV. In between these two multilayers the Si 111 substrate also acts as a reflecting element. The optical element has a length of 30cm. The multilayers were deposited with a precision of single layer thickness of below ± 1%.

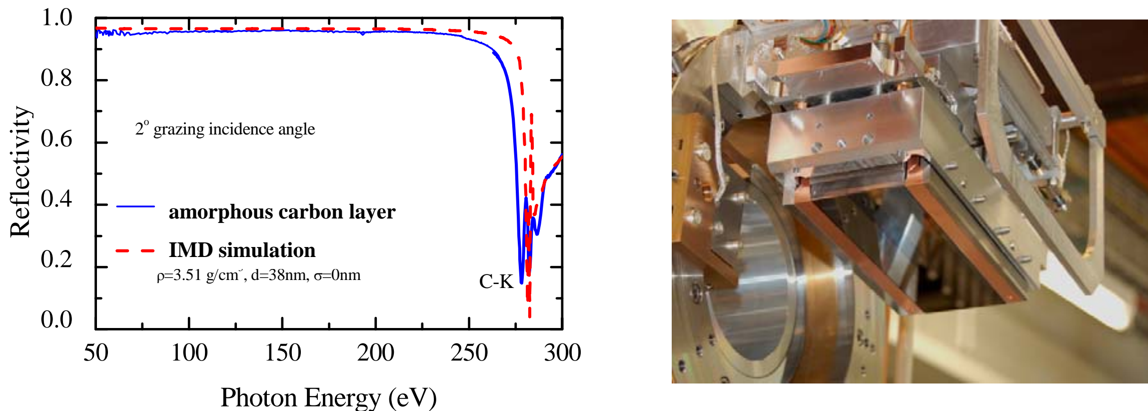

FIGURE 4: Two applications of X-ray optics: a) Carbon layers as total reflection optics with a thickness of 38 nm, b) multi-strip multilayer for reflecting of a wide range of energies at a tomography beamline (Foto courtesy of M. Stampanoni, PSI-SLS)

REFERENCES

1. M. Schuster, H. Göbel, L. Brügemann, D. Bahr, F. Burgäzy, C. Michaelsen, M. Störmer, P. Ricardo, R. Dietsch, T. Holz, and H. Mai, "Laterally graded multilayer optics for x-ray analysis" in *Proc. SPIE 3767*, pp. 183 (1999)
2. C. Michaelsen, J. Wiesmann, C. Hoffmann, K. Wulf, L. Brügemann, and A. Storm, "Recent developments of multilayer mirror optics for laboratorary x-ray instrumentation" in *Proc. SPIE 4782*, pp. 143-151 (2002)
3. M. Störmer, A. Liard-Cloup, F. Felten, S. Jacobi, B. Steeg, J. Feldhaus, R. Bormann, "Investigations of large x-ray optics for free electron lasers" in *Proc. SPIE 5533*, pp. 58-65 (2004)
4. B. Steeg, L. Juha, J. Feldhaus, S. Jacobi, R. Sobierajski, C. Michaelsen, A. Andrejczuk, and J. Krzywinski, "Total reflection amorphous carbon mirrors for vacuum ultraviolet free electron lasers", *Appl. Phys. Lett.* **5, 84**, pp. 657-659 (2004)
5. S. Jacobi, B. Steeg, J. Wiesmann, M. Störmer, J. Feldhaus, R. Bormann, C. Michaelsen, "Characterization of amorphous carbon films as total-reflection mirrors for XUV free-electron lasers" in *Proc. SPIE 4782*, pp. 113-121 (2002)
6. L. Juha, M. Bittner, M. De Grazia, J. Feldhaus, S. Guizard, S. Jacobi, M. Kozlová, J. Krása, J. Krzywinski, H. Merdji, C. Michaelsen, T. Mocek, et al., "Radiation damage to amorphous-carbon optical coatings" in *Proc. SPIE 5917* (2005)