Experimental Multilayer Survey in the VUV

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ABSTRACT

We present an experimental survey of the performance of various multilayer systems to be used in the soft x-ray range with special emphasis on the water window. The multilayers have been designed as high reflectance normal incidence mirrors and, for polarimetry purposes, as detectors for circularly polarized synchrotron radiation, respectively. Seven different multilayer systems with spacer materials of C or the transition metals Sc, Ti, V, Cr in combination with the absorber materials Fe, W and Ni were investigated. At the 1s- and 2p absorption edges, respectively, they show a strong resonant enhancement of the reflectance due to anomalous dispersion. By tailoring the layer thickness and the thickness ratio for use at and below the resonance energy in normal incidence ($\theta=90^\circ$) and at $\theta=45^\circ$, respectively, an excellent performance with respect to reflectance, transmission and polarizance, respectively, in the water window was achieved for multilayers with period thicknesses down to 1.4 nm.

Keywords: multilayers, reflection, transmission, synchrotron radiation, soft x-ray radiation, water window, circularly polarized light, polarimeter, reflectometer

1. INTRODUCTION

Multilayers with periods in the atomic range of 1-2 nm (nanolayers) are currently of great interest. This is related to their widespread use as e.g. radiation-stable dispersive optical elements /1-3/ and polarizers for synchrotron radiation /4-8/, as dispersive elements for x-ray diagnostics of high-temperature plasmas /9/, as normal incidence reflectors for (soft-) x-ray microscopy in the water window (284 eV- 530 eV) /10/ and for applications in x-ray astronomy /11/.

All these applications require a high radiation- and thermal stability, a high spectral selectivity and, in particular, a high reflectance. A normal incidence reflectance of 5 % is considered to be sufficient for many applications, e.g. for double mirror systems in telescopes and microscopes. For photon energies up to the C 1s-edge (284 eV) mirrors, including spherical ones, on the basis of Fe/C and Cr/C with reflectancies between 6 and 13 % are available /12,13/. However, for photon energies above 284 eV the reflectance has not until recently exceeded 3% /14,15/.

It has recently been shown that specially optimized multilayer systems working both in reflection and transmission (Mo/Si, 95 eV /4/ and Cr/C, 265 eV /5-7/) can be used effectively for a complete polarization analysis of soft x-ray radiation distinguishing between linear, circular and unpolarized components, i.e. all four Stokes parameters. At 95 eV this system is also routinely used for a complete conversion of linearly into circularly polarized radiation, since the Mo/Si transmission multilayer operates as a perfect quarter-wave plate /8/. For higher photon energies above the C 1s edge phase-retarding transmission multilayers have been proposed /6/, but so far not been realized.

The layer thickness scales with the wavelength. Thus, with increasing energy the performance of a multilayer is increasingly influenced by the interface roughness σ , according to the Debye-Waller type term in the reflectance: $R=R_0 \exp(-(2\pi m \sigma/d)^2)$, with R_0 being the reflectance of an ideal structure with $\sigma = 0$ nm, and m being the reflection order. Thus, with decreasing period d the reflectance tends to be dominated by the Debye-Waller factor and the ratio σ/d rather than by the optimum choice of material pairs in terms of their x-ray optical constants.

Consequently, the optimum material pairs will be those that yield structures with minimum interfacial roughness. For example, for pairs like W/Sb, W/Sc /13,16/ and W/B₄C /17/ no increase of roughness with decreasing period has been

reported. For such a widely used pair as W/Si, however, no high-reflectance structures with $d < 1.6$ nm have so far been reported.

The interest in multilayers containing a 3d-transition element Sc, Ti, V or Cr is called forth by the high reflection in the range of the anomalous dispersion at their 2p-absorption edges. Ni/V and Ni/Ti have been successfully used for x-ray- and neutron optics /18-21/. For W/Sc, Cr/Sc and Fe/Sc a high reflectance was realized /14,22,23/, but efforts to produce high reflectance structures of Cr/Sc and Fe/Sc with $d < 2$ nm have failed so far.

This paper presents a systematic experimental investigation with VUV-synchrotron radiation on the following multilayer systems: W/C, Fe/C, Cr/C, Cr/Sc, Ni/Ti, W/Ti and Ni/V. The period and the thickness ratio of the multilayers was tailored to their specific application with special emphasis on their use right below the absorption edges of the spacer materials C (1s edge at 284 eV), and the 2p-edges of the 3d-transition elements Sc (399 eV), Ti (454 eV), V (512 eV) and Cr (574 eV), respectively. They were optimized for the following synchrotron radiation applications:

- 1) High reflectance normal incidence mirrors for the the water-window.
- 2) Polarimetry in the soft x-ray range (detectors for polarized light)
 - a) Reflection multilayers to analyze linearly polarized light (45° , Rabinovitch detectors)
 - b) Transmission multilayers to convert circularly into linearly polarized light (phase retarders; $\lambda/4$ -plates)

Application 2 is part of a project of extending the working range of multilayer polarimeters to higher photon energies above the C 1s edge /6,24/. Other results including the performance of the transmission multilayers are published in /25/ for Cr/Sc and in /26/ for the other multilayer systems investigated.

2. THEORY

With increasing photon energy the refractive index of absorber and spacer materials of a multilayer approaches 1 and thus the reflectances at the interfaces decrease. Nevertheless, the reflectance is drastically enhanced right below the absorption edges of the involved materials due to the low imaginary part and the high real part of the Fresnel reflection coefficient in these regions.

Fig. 1 Survey of theoretical multilayer performance in the water window region. The maximum normal-incidence reflectance for ideal multilayers, assuming no roughness, is plotted. The d-spacing is varied with the photon wavelength according to $\lambda = 2d$. In the calculation 100 periods and a thickness ratio of 0.3 were assumed for all multilayers. Best performance is obtained at and below the absorption edges of the respective spacer materials.

Fig. 2 Plot of imaginary versus real part of the normal-incidence reflection coefficients for a variety of materials at a photon energy of 573 eV, right below the Cr 2p edge, according to /29/.

In Fig. 1 the maximum achievable normal incidence reflectance was calculated on the basis of the Henke-tables /27/ for all investigated multilayer systems. For the calculations using the BESSY-REFLEC-program /28/ 100 periods, a thickness ratio of absorber and spacer materials of $\gamma = 0.3$ and ideal structures with no roughness were assumed. The d-spacing of the multilayer was varied with photon wavelength λ according to $d = \lambda/2$. The C-, Sc-, Ti-, V- and Cr-resonances show up with ideal reflectances of up to 45 %. At the C-edge Fe as absorber is superior to W and Cr. The Cr/C and the Cr/Sc system can be used on both absorption edges.

Due to the enhancement of the multilayer performance near absorption edges, we concentrated the investigation to the near edge-region. According to the procedure described in /29/ the choice of the optimum material combination for a certain photon energy is done on the basis of a complex plot of the reflection coefficients. Such plots are available for the C-1s edge in /30/, for Sc 2p in Ref. /25/, for Ti and V 2p in /26/. For the Cr 2p edge (574 eV) this is shown in Fig. 2, where the imaginary part of the normal-incidence reflection coefficient is plotted against its real part. As already seen in Fig. 1, the transition metal- (Fe, Ni, Co) Sc combination is superior to a noble metal- (W, Ir, Os, Pt) Sc combination due to the higher absorption of the latter. According to the selection criterion of Fig. 2, Cr/Sc seems not appropriate due to the poor contrast. However, this criterion does not take into account roughness, which, according to the previous experimental data /22,23/ favours this material combination.

3. EXPERIMENTAL

All multilayers have been produced in various laboratories using magnetron sputtering devices: The Cr/C samples are from Sincrotrone Trieste, Italy, the W/C, W/Ti, Ni/Ti and Ni/V samples were made at the PSI, Switzerland, and the Fe/C and Cr/Sc multilayers have come from Nizhny Novgorod, Russia. They were first characterized with monochromatic unpolarized Cu-K α radiation or, additionally for the Cr/Sc-systems, using soft-ray radiation emitted from an x-ray tube source at wavelengths of 4.47 nm (C K α), 3.14 nm (Ti L β), 2.74 nm (Ti L α), 1.76 nm (Fe L α), 1.6 nm (Co L α), 1.46 nm (Ni L α), 1.33 nm (Cu L α), 0.99 nm (Mg K α) /23/.

Fig. 3 Schematic drawing of the experimental setup at BESSY. The triple-axis UHV-reflectometer / diffractometer is coupled to the exit slit of the bending magnet beamline PM 4 /31-33/

The synchrotron radiation measurements described in this report have been done using the UHV-reflectometer chamber at the BESSY-beamline PM 4 /31,32/ in 3 beamtime shifts in 1996/97. The experimental arrangement is shown schematically in Fig. 3. The plane mirror / plane grating (1200 l/mm) -combination of the SX700-type Petersen-Monochromator (PM 4) /33/ delivers the dispersed synchrotron radiation onto the ellipsoidal mirror which focusses the light vertically onto the fixed exit slit with strong demagnification of the source. For reduction of higher orders the monochromator can be operated in the higher order suppression mode by changing the incidence angles onto the mirror/grating combination (c-factor, standard mode: $c=2.25$). For the transmission measurements c was set to 1.65. In the standard mode the higher order contribution was measured to be between 10 % and 25 % in the range from 200 to 450 eV, at $c=1.65$ it is less than 15 % /34/. This, in combination with the increasing detector efficiency for higher energies leads to an underestimate of the reflectance data between 1.1 and 1.6. The results shown in this article have, however, not been corrected with respect to higher orders. The linear polarisation is estimated to be better than 90 % in the energy range of interest.

The BESSY-triple axis reflectometer /31/ (Fig. 3) is operated with all 3 motor-driven goniometers in vacuum and is used as multipurpose instrument for optical characterization at any UV- or soft x-ray beamline. A differential pumping stage separates the 10^{-7} mbar pressure of the reflectometer from the 10^{-9} mbar pressure of the beamline. The reflectometer is equipped with entrance pinholes of various sizes between 0.14 mm and 2.5 mm, positioned behind the exit slit close to the position of the horizontal focus of the ellipsoidal mirror. The incident intensity is monitored during the measurements with Au- or Cu-meshes. For the intensity reflected from the sample and for the direct incident beam the same GaAsP-diode /35/ with a noise level of 30 fA is used.

For an efficient data collection a new sample holder accepting up to 3 samples of any dimension up to $150 \times 50 \times 50$ mm³ was constructed. A 3-dimensional scan option (Bragg-scan) was implemented into the computer control which enables to scan simultaneously the incoming photon energy while scanning the multilayer around the Bragg-peak ($h\nu$ - θ - 2θ scan). This feature is especially useful for an investigation of near-edge absorption structures. As an example, Fig. 4 shows the Bragg curves (θ - 2θ scans) in the region of the Cr $2p_{3/2}$ and $p_{1/2}$ absorption edges, demonstrating the drastic changes in reflectance across these resonances.

Fig. 4 Bragg-scan ($h\nu$ - θ - 2θ) of a Cr/Sc multilayer around the Cr $2p_{3/2}$ and $p_{1/2}$ absorption edges. For each photon energy a θ - 2θ scan around the Bragg-peak was performed. The projection shows the peak reflectance data.

4. RESULTS

Fig. 5 Experimental survey of multilayer for the water window. For each of the seven investigated multilayer systems one sample is shown.

For all investigated multilayers the reflectance was measured in the total working range from normal incidence ($\theta=87^\circ$) to grazing incidence ($\theta=10^\circ$) either as function of the photon energy at fixed incidence angle or as function of the incidence angle at fixed energy (θ - 2θ scan). In addition, the near edge reflectance was recorded using the Bragg-scan feature (see Fig. 4). The energy resolution is given as the FWHM-value of the energy dependence of the reflectance curve or, it has been extracted from their angular width by $E/\Delta E = \tan\theta/\Delta\theta$.

4.1. Survey

The figure 5 shows the peak reflectance behaviour for one example out of each of the seven investigated multilayer systems in the region of the absorption edges of the spacer materials C, Sc, Ti, V and Cr. The resonances clearly show as points of maximum reflection. Note, that the incidence angle changes with energy, the lowest energy for each sample corresponds to the normal incidence value. The performance of these multilayer systems in the near-edge energy range of the respective spacer materials C, Ti and Sc will be shown in detail in the following.

Typical near-normal incidence reflection curves are given in Fig. 6 for six of the investigated multilayer systems. The

Fig. 6 Normal incidence peak reflectance data for selected examples of the multilayers systems investigated. The incidence angle was set to 87° . Full curves: experimental data, dotted curves: calculation with fitted roughness values as given in the figure.

experimental data (full curves) are compared to a calculation with the roughness σ as fit parameter. All samples exhibit well pronounced Kiessig fringes indicating the homogeneity of the individual layers. Note that some of these multilayers have the highest

Fig. 7 Reflectance curve of a Cr/C multilayer (θ -2 θ scan) at a photon energy of 195 eV in near normal incidence. The peak reflectance is 10.7 %. Symbols: experimental data, dotted curve: calculation with a fitted roughness of 0.5 nm.

Fig. 8 Peak reflectance data for multilayers with C as spacer material, plotted as function of the photon energy, in the region of the C 1s-edge. The data were extracted from Bragg-scans like shown in Fig. 4. The incidence angle changes with energy as indicated, the lowest energy point corresponds to normal incidence ($\theta=87^\circ$).

reflectances measured so far /36/: The W/C sample ($d=2.62$ nm) reaches 7.9 % at 236 eV. The least roughness is obtained with Cr/Sc (0.3 nm), followed by W/C (0.42 nm) and Ni/V (0.45 nm).

4.2 Cr/C, Fe/C, W/C

The reflectance curve of the Cr/C multilayer is given in Fig. 7. The fit delivers a roughness of 0.5 nm. In normal incidence, corresponding to a photon energy of 195 eV, a reflectance of 10.8 % was reached.

Fig. 8 shows a comparison of the 3 multilayer systems with C as spacer. These peak reflectance data were extracted from Bragg-scans like shown in Fig. 4. The angle of incidence changes with energy, the lowest energy corresponds to the normal incidence value. The cut-off at the C 1s-edge is clearly seen in all samples. Obviously, Cr/C and W/C have the best performance, while Fe/C, despite of the larger number of periods, suffers from a severe roughness of 0.65 nm, as seen already in Fig. 6.

4.3 Ni/Ti, W/Ti

The energy dependence of the peak reflectance of the multilayers with Ti as spacer material is given in Fig. 9 for selected samples with Ni and W as absorbers. As expected, the reflectance increases strongly up to a maximum right below the Ti $2p_{3/2}$ -absorption edge at 454 eV, which is followed by a sharp cut-off. The spin-orbit split $2p_{3/2}$ component is also clearly resolved by the W/Ti samples. Note, that one of the W/Ti samples was matched for normal incidence at this resonance. A peak value of 2.2 % at 441 eV, which is presently the best value, was found.

Fig. 9 Peak reflectance data for multilayers with Ti as spacer material, plotted as function of the photon energy, in the region of the Ti 2p-edge. The data were extracted from Bragg-scans like shown in Fig. 4. The incidence angle changes with energy, as indicated. Note, that the normal-incidence reflectance of the W/Ti sample is 2.2 % at 441 eV!

Fig. 10 Peak reflectance data for multilayers with Cr as spacer material, plotted as function of the photon energy, in the region of the Cr 2p-edge. The data were extracted from Bragg-scans like shown in Fig. 4. The incidence angle changes with energy, as indicated.

4.4 Cr/C, Cr/Sc

The reflectance curves for selected multilayers containing Cr (as spacer or absorber, respectively) are given in Fig. 10. Again, the resonance behaviour of the performance is clearly seen for the region of the spin-orbit split Cr $2p_{3/2}$ and $2p_{1/2}$ levels. With decreasing period the reflectance goes down due to the increasing angle of incidence. Note, that the smallest period Cr/Sc ($d=1.66$ nm) is matched for 45°-application close to the absorption edge and has a remarkable reflectance of 5 %.

Another Cr/Sc-sample with an even smaller period of $d=1.57$ nm has been matched to normal incidence close to the Sc-2p edge at 399 eV. The experimental result is given in Fig. 11. Note, that an extraordinary reflectance of 7 % has been observed which corresponds to the highest normal incidence reflectance measured so far at this energy of 397 eV. The fit yields a roughness of 0.39 nm which corresponds to the roughness of the wafer-substrate.

4.5. Energy resolution

Corresponding to the resonance behaviour of the peak reflectance, also the energy resolution of the multilayers changes throughout a resonance due to the changing penetration depth of the light falling onto the multilayer. This is shown in Fig. 12 for selected multilayer samples. The resolution is mainly determined by the total thickness of the multilayer stack contributing to the reflection, i.e. by the number of periods. This number determines also the maximum possible resolution. This maximum is nearly reached by all samples below the absorption edge. As seen in Fig. 12, the resolution is seriously reduced at the absorption edge due to increased absorption. As expected, the resolution values obtained from energy scans (not shown) are identical to those obtained from θ - 2θ scans. It is interesting to note, that in the vicinity of absorption edges this may be different and that the energy resolution can even exceed the theoretical limit. For a Ni/V sample with 75 periods, for instance, a maximum resolving power of $E/\Delta E=165$ was measured (not shown). This can be easily explained by the behaviour of the Bragg-peak when it moves across the absorption edge. When crossing the edge the Bragg-curve gets smaller and asymmetric due to absorption on the high energy side of the peak. This effect can, of course, not be seen in angular scans at fixed energy.

Fig. 11 Normal incidence peak reflectance for a Cr/Sc multilayer. The period of the multilayer was matched to a normal incidence energy right below the Cr 2p edge. The peak reflectance is 7.0 %. Symbols: experimental data, dotted curve: calculation with a fitted roughness of 0.39 nm.

Fig. 12 Energy resolution for selected multilayer systems as function of the photon energy. The absorption edges of the spacer materials are indicated.

5. CONCLUSION

We have presented a systematic investigation of sputter-deposited ultrathin multilayers of different composition using tuneable synchrotron radiation in the soft x-ray range. The period spacing varied in the range between 1.5 and 5.4 nm, which determines the working range to be in the soft x-ray range with special emphasis on the water window. An excellent performance was measured close to the 1s or 2p-absorption edges of the spacer materials C, Sc, Ni, V and Cr (284 eV-574 eV) where the reflectance and energy resolution is resonantly enhanced. A value of 7 % for the normal incidence reflectance at 395 eV was measured for a Cr/Sc-multilayer with a period of $d=1.57$ nm, matched right below the resonance energy. This is the highest value measured so far at this energy.

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 36. for further information on the multilayer survey see: <http://www-cxro.lbl.gov/multilayer/survey.html>