Multilayer Reflectors for Improving the Primary Beam Intensity: Reflectivity Measurements on Coated Glasses

A. Knoll, L. Brügemann

► To cite this version:

A. Knoll, L. Brügemann. Multilayer Reflectors for Improving the Primary Beam Intensity: Reflectivity Measurements on Coated Glasses. Journal de Physique IV Proceedings, 1996, 06 (C4), pp.C4-385-C4-391. 10.1051/jp4:1996434 . jpa-00254317

HAL Id: jpa-00254317

<https://hal.science/jpa-00254317>

Submitted on 1 Jan 1996

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Multilayer Reflectors for Improving the Primary Beam Intensity: Reflectivity Measurements on Coated Glasses

A. Knoll and L. Brügemann*

Dorfbrunnenstrasse 2 B, 76744 Woerth am Rhein, Germany

** Siemens AG, AUT 37 E, 76181 Karlsruhe, Germany*

Abstract: Periodic multilayers are commonly used in various X-ray applications as primary beam conditioners. The newly designed so-called Goebel-mirror is a special type of multilayer which is used to convert a diverging incoming beam into e.g. parallel one.

The Goebel-mirror is bent parabolically or elliptically with the X-ray source lying in the focus of the parabola. The period of the multilayer is matched laterally to the incident angle so that the first order Bragg-condition is fulfilled for a specific wavelength band.

About 75% of the incoming $K\alpha$ radiation is reflected whereas $K\beta$ radiation is suppressed totally. In the case of the X-ray reflectometry technique the properties of a parabolically bent Goebel-mirror can be used to get an intensity gain up to about two orders of magnitude. This intensity gain can either be used to increase the measured angular range or to reduce the overall measurement time drastically. Investigations on thin film glass coatings will be presented which show the advantages which can be achieved by using the Goebel-mirror technique.

1. INTRODUCTION

Surface coating and different types of surface treatments are nowadays widely developed as techniques to modify the properties of surfaces. In this context, measurement techniques have been developed to get precise information of the physical parameters of the surface layers. X-ray reflectometry is one of these techniques which provides information about the properties of the surface layers. An overview is given by Zorn [1].

In 1931 H. Kiessig [2] described the X-ray reflectometry as a method to determine the thickness of thin films on substrates by making use of the total reflection of X-rays. The basic theory of the X-ray reflection can be treated just like the reflection of visible light, reflection and transmission being described by the Fresnel equations.

The measurement principle is based on the fact that the index of refraction n is slightly smaller than 1 for all materials for the regime of X-ray wavelengths. For X-rays the index of refraction is defined by:

$$n = 1 - \delta - i\beta$$

with δ describing the dispersion and β the absorption of a material at a certain wavelength. Both δ and β are of the order of 10^{-6} . As a consequence total external reflection occurs if X-rays hit a sample under a very small incidence angle.

The exact value for the critical angle of total external reflection α_c can be used to determine the density of the deposited films or the substrate according to the value of the critical angle

$$\alpha_c \cong \sqrt{2\delta} \quad \text{with} \quad \delta = \frac{N_A r_e}{2\pi} \frac{\rho}{A} (Z + f') \cdot \lambda^2$$

where N_A is the Avogadro's number, r_e the classical electron radius, ρ the mass density, A the atomic mass, Z the atomic number, f' the dispersion correction and λ the wavelength of the incident beam. The absorption is neglected in this formula.

The penetration depth of the X-ray wave is determined by the incident angle of the beam and the wavelength sensible value of the dispersion and the absorption correction. The penetration depth varies between 5 Å and up to several hundred nanometers. For large incident angles the penetration depth is then defined by photoabsorption. The minimal value of the penetration depth is defined by the X-ray density of the material. Therefore, X-ray reflectometry combines surface sensitivity and small penetration depth.

L. G. Parrat [3] extended Kiessig's theoretical approach by describing the X-ray reflectivity to more complicated systems containing more than one layer. The reflected intensity is determined by using a recursive formula calculating the Fresnel coefficients for all corresponding interfaces. The beams reflected from the different interfaces are added up in respect to their phase conditions. By changing the incidence angle, the phase condition is changed. Constructive interference results in intensity maxima called „Kiessig-fringes“ whose angular spacing is characteristic of the thickness of the layers. Although Parrat noticed the effect of surface and interface roughness on the reflectivity curves, a mathematically satisfying treatment was first published by L. Névet and P. Croce [4].

During the last decade, the grazing-angle X-Ray reflectometry technique was improved from a rarely used, exotic technique to a routine tool for surface and thin film analysis for research and quality control.

In the following the principles of the reflectometry measurement set-up and the principle of the graded multilayer design will be described. Comparative reflectivity measurements on coated glasses will be presented using both the technique with and without the graded multilayer as the primary beam conditioner.

2. THE EXPERIMENTAL SET-UP

2.1 The experimental set-up

All measurements were done on a SIEMENS D5000 $\theta/2\theta$ powder diffractometer equipped with the dedicated reflectometry sample stage. Figure 1 shows the principle of the reflectometry set-up.

Figure 1: Reflectometry set-up (without graded multilayer optics)

All experiments were executed using a copper long fine focus sealed tube. The generator power for all measurements was set to 40kV/40mA. A primary slit limits the beam height so that only the coated part of the sample is illuminated. The main components of the reflectometry sample stage are the vacuum sucker and the edge diaphragm opposite to the vacuum sucker. Both are mounted on translation stages whose positions are individually controlled by micrometric screws. The absolute position of the edge diaphragm is displayed by a dial indicator. The zero point of this dial indicator was calibrated by making reflectometry measurements on a silicon reference wafer. The edge diaphragm defines both the beam divergence and the illuminated area on the surface of the sample. Furthermore, it is used to align the surface orientation of the sample and the position of the surface in the centre of the goniometer. For all experiments the distance between edge diaphragm and sample surface was set to 10 μm .

During the measurements several orders of magnitude of intensity are covered. Therefore, an absorber is mounted into the diffracted beam path if measurements are executed in the regime of total external reflection.

The detector slit determines the angular resolution in 2θ . For all experiments a 0.03° detector slit in combination with a 2° secondary soller slit was used. In case of measurements executed without the Goebel-mirror a secondary graphite monochromator was used to avoid the influence of the bremsstrahlung and the $\text{CuK}\beta$ radiation.

2.2 The design of the graded multilayer

The parabolically bent graded multilayer used as primary beam conditioner for sealed X-ray tubes was first introduced by Schuster and Goebel [5] („Goebel mirrors“). These multilayers were applied as reflectors to convert a divergent X-ray beam from sealed X-ray tubes into a parallel beam. Parallel coupling (the scattering vector of the mirror reflection lies in the plane of the scattering vector of the sample) enables to exploit a wider solid angle range of the X-ray source and to obtain an intensity gain of about two orders of magnitude. Fig. 2 shows the principle of the graded multilayer as condensing reflector.

It is well known that a parabola reflects radiation emitted from its focus into a parallel bundle. The incidence angle of a ray emerging from the focus F on the parabola depends on the point of incidence.

Figure 2: The principle of the Goebel mirrors

For a parabola the incidence angle of the focal ray is given by

$$\alpha_i(f) = \arccot(\sqrt{2f/p - 1})$$

where f is the focal length (distance between the Focus and the incidence point). Because the multilayer Bragg reflector has to fulfill the Bragg equation at each point of the parabola, the period has to vary over the length of the parabola according to

$$d(f) = \frac{\lambda}{2 \sin(\arccot(\sqrt{2f/p - 1}))}$$

where λ is the wavelength of the radiation. For Cu K α radiation these periods correspond to Bragg angles between 3° and 0.6° . The integral reflectivity of the multilayer is mainly defined by the electron density of the reflector material. A high integral intensity is obtained for a combination of two materials with a large contrast in the electron density. Simultaneously, the absorption coefficient must be low. Furthermore, the number of double layers influences the reflectivity and the angular width of the reflection. The angular width defines the angular divergence of the graded multilayer. The ratio of the individual layer thickness is defined to suppress the second order Bragg reflection.

For our experiments a W/Si graded multilayer with 50 layer pairs was used. The thickness of the layer pairs varied from 3.0 nm to 4.0 nm. The multilayer was fabricated by N. Grupido (Osmics) by sequential sputtering on a (100) silicon wafer substrate. The wafer was bent and laminated onto a parabolically formed steel support with appropriate parameters of the parabola. The reflectivity of the first order Bragg reflection was about 75% with an angular width of about 0.05° . Cu K β was totally suppressed by this type of graded multilayer.

3. MEASUREMENTS AND RESULTS

Two coated glasses were investigated using the X-ray reflectometry technique. Sample A consisted of a In₂O₃/Ag/In₂O₃ coating and sample B was coated with a 57 nm thick TiO₂ layer. First, a reflectometry measurement was executed with graphite secondary monochromator to remove the bremsstrahlung and the Cu K β radiation. The measurement was then repeated with a Goebel-mirror in the primary beam path. In this case, the secondary monochromator was removed because the graded multilayer separates pure Cu K α radiation. Both measurements were composed of five angular ranges with different measurement parameters up to an incidence angle of 3° . For both measurements the measuring parameters were identical, except for the step time which was selected to obtain similar counting statistics within each range. The step size was 0.005° in α_i .

Figure 3a: Reflectometry measurements of sample A with (solid line) and without (dotted line) Goebel mirror.

Fig. 3a shows the result for sample A. The reflected intensity is plotted on a logarithmic scale as a function of the incidence angle. The solid line represents the measurement with the Goebel mirror while the dotted line represents the measurement without the Goebel mirror. The overall measuring time was about 5 hours for the second type of measurement whereas only 38 minutes were necessary to collect data with the same counting statistics in the former type of Fig. 3b measurement.

Figure 3b: Reflectometry measurements of sample A with Goebel Optics (solid line) and without (dotted line). The intensity scale factors are shown.

Fig. 3b shows both measurements on a linear intensity scale. Each reflectivity curve is split into five parts. To make all interference fringes visible, the intensity of each part is scaled by the number given on the figure. Both measurements show nearly the same absolute intensity. To conclude, using in this example the graded multilayer as primary beam conditioner enabled to collect data with the same quality eight times faster than usual.

Fig. 4 shows the reflectometry measurements for sample B. Measuring parameters were equal to the measurement of sample A. Both intensities were scaled in respect to the the maximum value of intensity to be unity. With the Goebel mirror (solid line) it was possible to enlarge the dynamic range of the measurement by one order of magnitude compared to the measurement without Goebel mirror (dotted line). Additionally, measuring time was decreased by nearly one order of magnitude.

Figure 4: Reflectometry measurements of sample B scaled to unity.

4. CONCLUSION

It was shown that the use of parabolically bent graded multilayer in the X-ray reflectometry technique with sealed X-ray tubes enabled to collect data with the same statistics in a drastically reduced time compared to standard reflectometry measurement. Furthermore, it was shown that the intensity gain can be used to extend the dynamical intensity range covered by the reflectivity measurements.

References

- [1] G. Zorn, Analytical Application Note No. 337, Siemens Corporate Research, Munich, 1994
- [2] H. Kiessig, *Ann. d. Phys.*, **10** (1935) 769-788
- [3] L.G. Parrat, *Phys. Rev.*, **5** (1954) 359-369
- [4] L. Nevot, P. Croce, *Rev. Phys. Appl*, **15** (1980) 761-779
- [5] H. Göbel, M. Schuster, *J. Phys. D: Appl. Phys.*, submitted