#### NANOMETER SIZED LAYERED STRUCTURES

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### 1. Introduction

Nanometer-size layered structures with characteristic dimensions in the nm range have found wide interest both in research and applications. Important nanometer sized layered structures are multilayer (ML) mirrors applied as optical elements in the soft X-ray spectral region. The basic demands on the MLs preparation techniques are tight control of deposition process, sharp and smooth boundaries between layers. When the layers in a ML stack are very thin, the interfaces dominate the ML properties. In general low mutual solubility and/or immiscibility of material combinations are preferred to avoid the interface mixing. The interface (geometrical) roughness is the result of the growth process itself. It is affected also by the substrate (underlying layer) roughness, which is replicated to some extent. Low interface roughness and replication are needed to maximize the specular reflectivity and imaging contrast [1].

In this work the ML interface analysis by grazing incidence small angle X-ray scattering GISAXS and X-ray reflectivity techniques XRR are used to analyze the Mo/Si multilayers prepared by various deposition methods.

#### 2. Experimental details

The XRR measurements were done on an X-ray diffractometer (Bruker D8 Discover) equipped with Cu X-ray rotating anode. The mean roughness and thickness of the Mo and Si layers were determined by fitting the experimental XRR data by a modified genetic algorithm.

The GISAXS measurements were done with synchrotron radiation and a microfocus X-ray source. The synchrotron measurements were performed at BW4 beamline at the HASYLAB, Hamburg [3] at the angle of incidence 0.7°. Supplementary GISAXS measurements were done on a table-top GISAXS system Nanostar (Bruker AXS) equipped

with a microfocus X-ray source (wavelength 0.154 nm). The details of the theory of GISAXS are described in Ref. 2. From the GISAXS experimental data the information on multilayer period, gamma value, interface roughness, lateral correlation length, Hurst parameter and vertical correlation length can be obtained [4].

The Mo/Si multilayer mirrors were prepared by e-beam evaporation, e-beam evaporation on heated substrate, e-beam evaporation with ion beam polishing, ion beam sputtering and rf sputtering [1].



Fig. 1: The Mo/Si multilayer deposited by e-beam evaporation. (a) X-ray reflectivity (b) GISAXS pattern (c) FWHM of the 2nd Bragg sheet (d) The intensity decay of the 2nd Bragg sheet.

#### 3. Results and discussion

The evaluation procedure of the XRR and GISAXS experimental data is presented in for Mo/Si multilayer prepared by e-beam evaporation at room temperature (Fig. 1).

The multilayer period  $\Lambda = 8.37$  nm, together with the thickness and roughness of Mo and Si layers and their standard deviations were determined by simulation of the XRR (Fig. 1a). The synchrotron GISAXS pattern is shown in Fig. 1b. Here, three Bragg sheets point at the correlated roughness of the ML interfaces. The plot in Fig. 1c shows the FWHM of the 2<sup>nd</sup> Bragg sheet ( $q_z = 1.75$  nm<sup>-1</sup>) and the effective number of correlated periods  $N_{eff}$ . The spatial frequencies of roughness lower than 1 nm<sup>-1</sup> are completely replicated throughout the multilayer stack ( $N_{eff} = 9$ ). The intensity of the 2<sup>nd</sup> Bragg sheet was integrated within the interval  $\Delta q_z = \pm 0.05$  nm<sup>-1</sup> and plotted as a function of  $q_y$  in Fig. 1d. The simulation of the 2<sup>nd</sup> Bragg sheet intensity as a function of  $q_y$  gives the mean lateral correlation length  $\xi = 1.7$  nm and mean Hurst parameter H=1.

A similar evaluation procedure was performed for all Mo/Si MLs. The parameters of the multilayer interfaces are summarized in Table 1. From the data it follows, that the ion beam and rf sputtering as well as the e-beam evaporation combined with ion beam polishing produce smooth interfaces.



Fig. 2: The intensity profiles of the Bragg sheets extracted from the reciprocal space maps of the Mo/Si mirrors deposited by different techniques: 1) e-beam evaporation, 2) e-beam evaporation on heated substrate, 3) e-beam evaporation combined with ion beam polishing,
4) ion beam sputtering and 5) rf sputtering.

Table 1: Parameters of the ML interfaces for Mo/Si MLs determined from XRR and GISAXS data.  $\Lambda$  - multilayer period,, N – number of periods,  $\sigma_{Si}$  - Si interface roughness,  $\sigma_{Mo}$  -Mo interface roughness,  $\xi$  - lateral correlation length, Neff - number of correlated periods (vertical roughness correlation), H - Hurst parameter. Sample number corresponds to the method of preparation 1) e-beam evaporation, 2) e-beam evaporation on heated substrate, 3) e-beam evaporation combined with ion beam polishing, 4) ion beam sputtering and 5) rf sputtering

| Sample | Λ    | Ν  | $\sigma_{\scriptscriptstyle Si}$ | $\sigma_{\scriptscriptstyle Mo}$ | ξ [nm] | N <sub>eff</sub> | Н   |
|--------|------|----|----------------------------------|----------------------------------|--------|------------------|-----|
| 1      | 8.37 | 10 | 0.67                             | 0.76                             | 1.7    | 9                | 1   |
| 2      | 6.78 | 30 | 0.38                             | 0.79                             | 42     | 25               |     |
| 3      | 6.63 | 50 | 0.28                             | 0.45                             | 1.4    | 20               | 0.3 |
| 4      | 6.61 | 40 | 0.21                             | 0.52                             | 7      | 18               | 0.5 |
| 5      | 10.7 | 30 | 0.22                             | 0.51                             | 1.9    | > 12             | 0.3 |

In Fig 2 the intensity profiles (cuts at extracted from the GISAXS reciprocal space maps through the 2<sup>nd</sup> Bragg sheet) of Mo/Si MLs are summarized. It is evident that directly from the GISAXS data a qualitative comparison for the interface morphology can be performed. Based on the integral intensity under the curves one can easily figure out the best deposition technique that produces the lowest scattering in the reciprocal space.

## 4. Conclusion

The interface morphology of Mo/Si MLs prepared by various deposition techniques was investigated by XRR and GISAXS techniques. From the GISAXS pattern the unambiguous information on autocorrelation functions describing the interface roughness can be obtained. The analyzed samples were deposited by e-beam evaporation at room temperature and/or on heated substrate, e-beam evaporation combined with ion beam polishing, ion beam sputtering and rf magnetron sputtering. The latter three deposition techniques produce interfaces with a low intrinsic roughness required for highly reflecting soft X-ray mirrors. However, the ion beam polishing of the deposited layers was not found to be effective for different metal layers.

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