Nanometer multilayers as monochromators for the X-ray analysis

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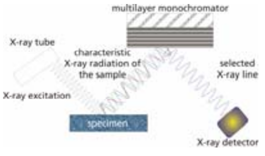
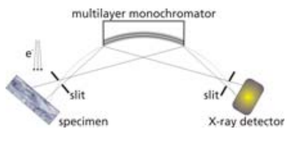
Introduction

The principle of X-ray analytical methods like wavelength-dispersive X-ray fluorescence (WD-XRF) analysis and electron probe micro analysis (EPMA) is based on the excitation of characteristic radiation of chemical elements contained in the specimen under investigation. In order to separate the emission lines of different elements, monochromators like crystals or multilayers are used. This poster focuses on the development and improvement of multilayer monochromators for the detection of light elements of the periodic table (Be – S).



Scheme of the WD-XRF working principle



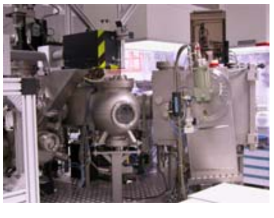
Scheme of the EPMA working principle

The photon wavelengths of the characteristic radiation of light elements are in the range of 0.7-11.4 nm. Correspondingly, the period thickness of the multilayers has to be in the same order. However, with smaller period thicknesses the requirements for the interfaces between adjacent layers within the multilayer significantly increase. It has to be ensured that both interface roughness and interface diffusion has to be minimized at the same time. Additionally the stability of the deposition process must be extremely high since the multilayers consist of up to 1200 single layers.

Multilayer fabrication

Nanometer multilayers with single layer thicknesses in the range between 0.5 nm and 20 nm are synthesized using UHV thin film deposition techniques like sputtering or pulse laser deposition (PLD). For XRF and EPMA multilayers, the magnetron sputter deposition (MSD) is applied to produce multilayers with outstanding specifications:

- layer thickness uniformity: ≥ 99.9 %
- run-to-run reproducibility: 99.8 % - 99.9 %
- layer microroughness (rms): 0.15 nm – 0.25 nm



Photograph of the thin film deposition machine “UHV cluster tool” combining two deposition chambers, the handling system, one sample magazine and the load-lock. In both deposition chambers, MSD and PLD, up to four materials can be used in the multilayer period. The typical substrate size is ∅ = 150 mm, the maximum size can be up to ∅ = 250 mm.

Multilayer requirements

Key parameters for X-ray analytical purposes

- high reflectance => low detection limits
- high resolving power => high selectivity
- low background scattering => high signal-to-noise ratio
- suppression of higher order reflections
- long-term stability

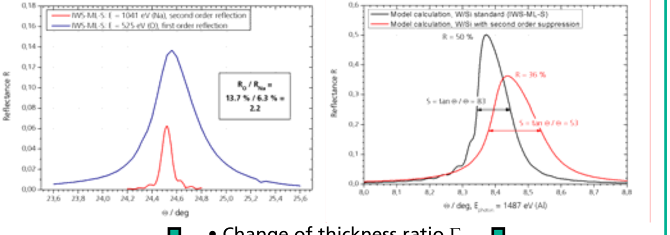
Consequences for multilayer fabrication:

- proper choice of layer materials, layer thickness, and number of periods
- strict periodicity of the layers in growth direction
- multilayers with sharp and abrupt interfaces
- multilayers with morphological smooth interfaces (no roughness)
- single layers with low absorption and high contrast to each other
- suitable capping layers to avoid oxidation and contamination

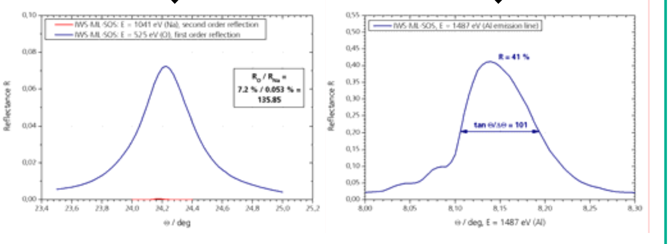
Higher order suppression

Standard multilayer monochromators with $\Gamma = d_{absorber}/d_p = 0.2 - 0.3$
=> Overlap of reflections of different emission lines can occur

Solution possible with a multilayer design change to $\Gamma = 0.5$
=> second order suppression
However: decreased reflectance and resolving power for all emission lines



- Change of thickness ratio Γ,
- Change of absorber



Multilayers for high selectivity

For high resolving powers, multilayers have to meet the following requirements:

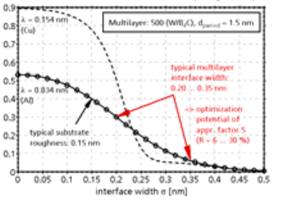
- **high number N of periods (up to N = 800)**
- **extremely thin single layers (d ~ 0.5 nm)**

Consequences:

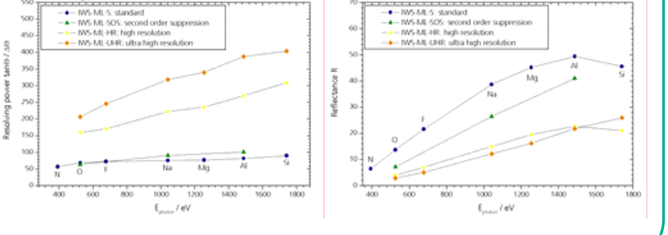
- Barrier layers not applicable
  => limited material choice (W/B_4C, Cr/Sc, Mo/B_4C, …)
- Deposition process has to be very long-term stable
- Reflectance highly sensitive to interface width

Small d-spacing multilayers
=> Soft X-ray reflectance much more sensitive to interface width

Improvement of 500 % possible if interface width can be reduced from 0.35 nm to 0.20 nm



Synchrotron measurements at BESSY



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