A laboratory setup for angle resolved X-ray fluorescence analysis – characterization and applications

M. Spanier[1], D. Grötzsch[1], C. Herzog[1], F. Kramer[1], I. Mantouvalou[1], J. Lubeck[2], J. Weser[2], W. Malzer[1], B. Beckhoff[2] and B. Kanngießer[1]

Introduction

A novel laboratory UHV setup for the investigation of elemental depth profiles in the nm-regime with the help of grazing incidence (GIXRF) and grazing emission (GEXRF) X-ray fluorescence is installed at the Berlin Laboratory for innovative X-ray Technologies (BLiX) at the Technical University Berlin.

This is realised by the use of a small power micro focus X-ray tube in combination with a polycapillary optic as the beam shaping element and a windowless silicon drift detector.

The setup was developed in cooperation with the research group X-ray and IR Spectrometry at the Physikalisch-Technische Bundesanstalt (PTB) [1].

Transmission Function

The excitation spectrum is affected by the polycapillary optic's transmission characteristic. This can be determined by measuring a scatter spectrum with and without the polycapillary optic [2].

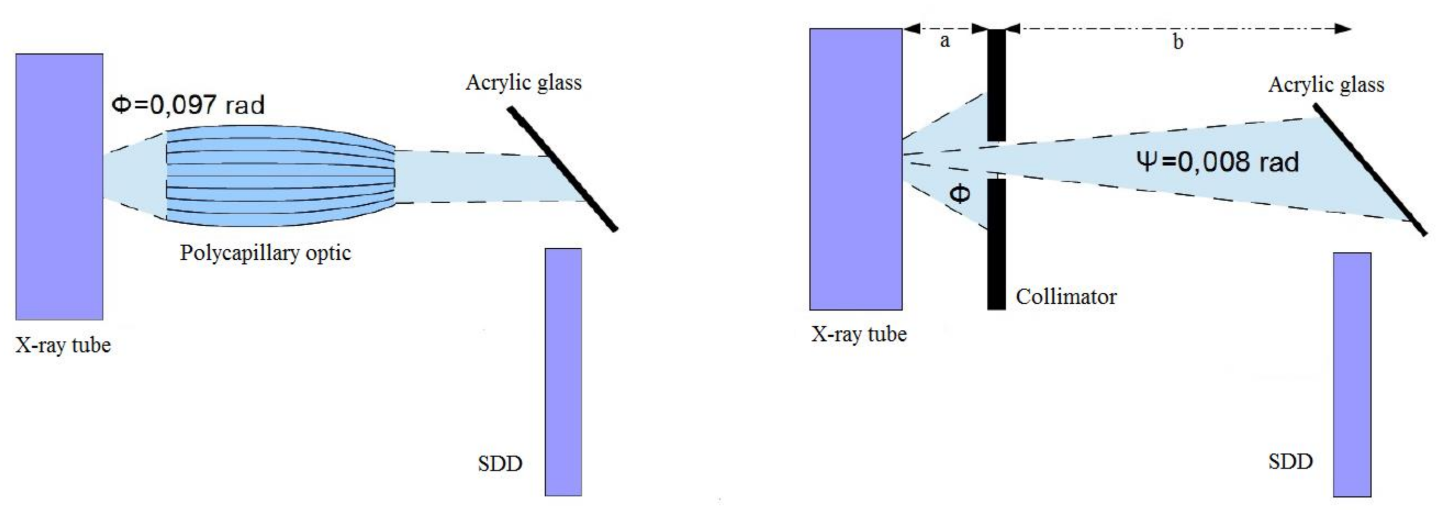

Figure 1: Geometry for the determination of the polycapillary optic's transmission function. Scatter spectra measurement with (left) and without optic (right) [3].

The transmission function is defined as: $T(E) = \frac{t_2 \cdot \Omega_2}{t_1 \cdot \Omega_1} \cdot \frac{d^2}{a^2 \cdot \Phi^2} \cdot \frac{N^{det}_{sca,1}(E)}{N^{det}_{sca,2}(E)}$

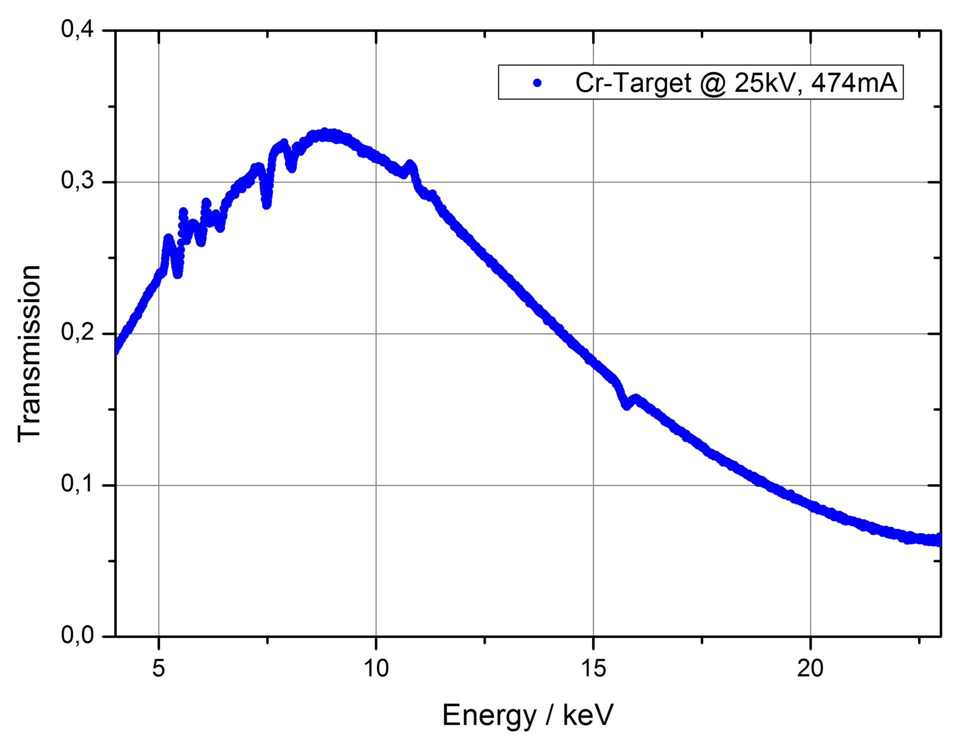

Figure 2: Polycapillary optic's transmission function for a small power micro focus X-ray tube with Cr-target @ 25 kV, 474 mA

- Polycapillary optic's transmission function maximum of around 33% reached at around 8 keV.
- Artifacts resulting from fluorescence lines from the sample holder.
- Compton scattering is neglected.
- Estimated error bars in the range of 20%.

FP based forward calculation

- Fundamental parameter based XRF analysis software [4], in-house development
- Implementation of a fluorescence line dependent calibration factor to overcome the plenty of not yet exactly known experimental influence quantities ⟹ geometry dependent calibration factor needed
- Transmission function is used for the simulation of the excitation spectrum

Challenges: geometry factor including solid angle of detection, beam divergence and incidence angle offset, FPs

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GIXRF Measurements and simulations

Single layer sample system

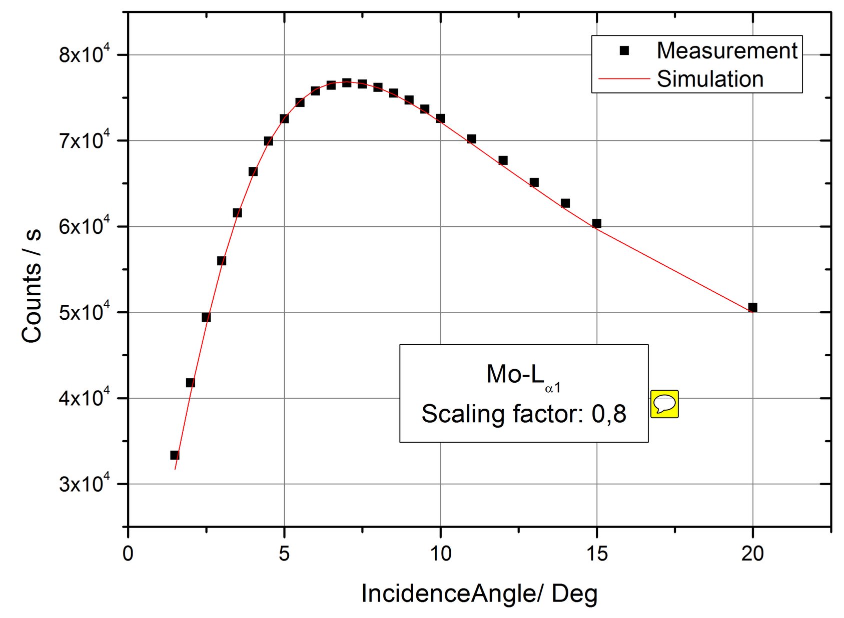

Figure 3: Fluorescence intensities for Mo $L_{\alpha1}$ from 570nm Mo / SiO_2

Simulated curve is in good agreement with the measurement. Adaptation of the absolute fluorescence intensities by a calibration factor of 0.8.

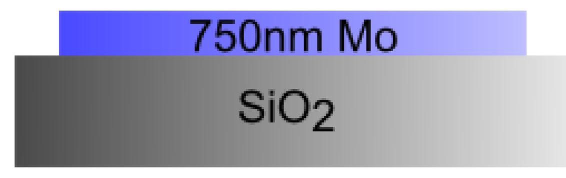

Figure 4: The single layer sample system

Multi layer sample system

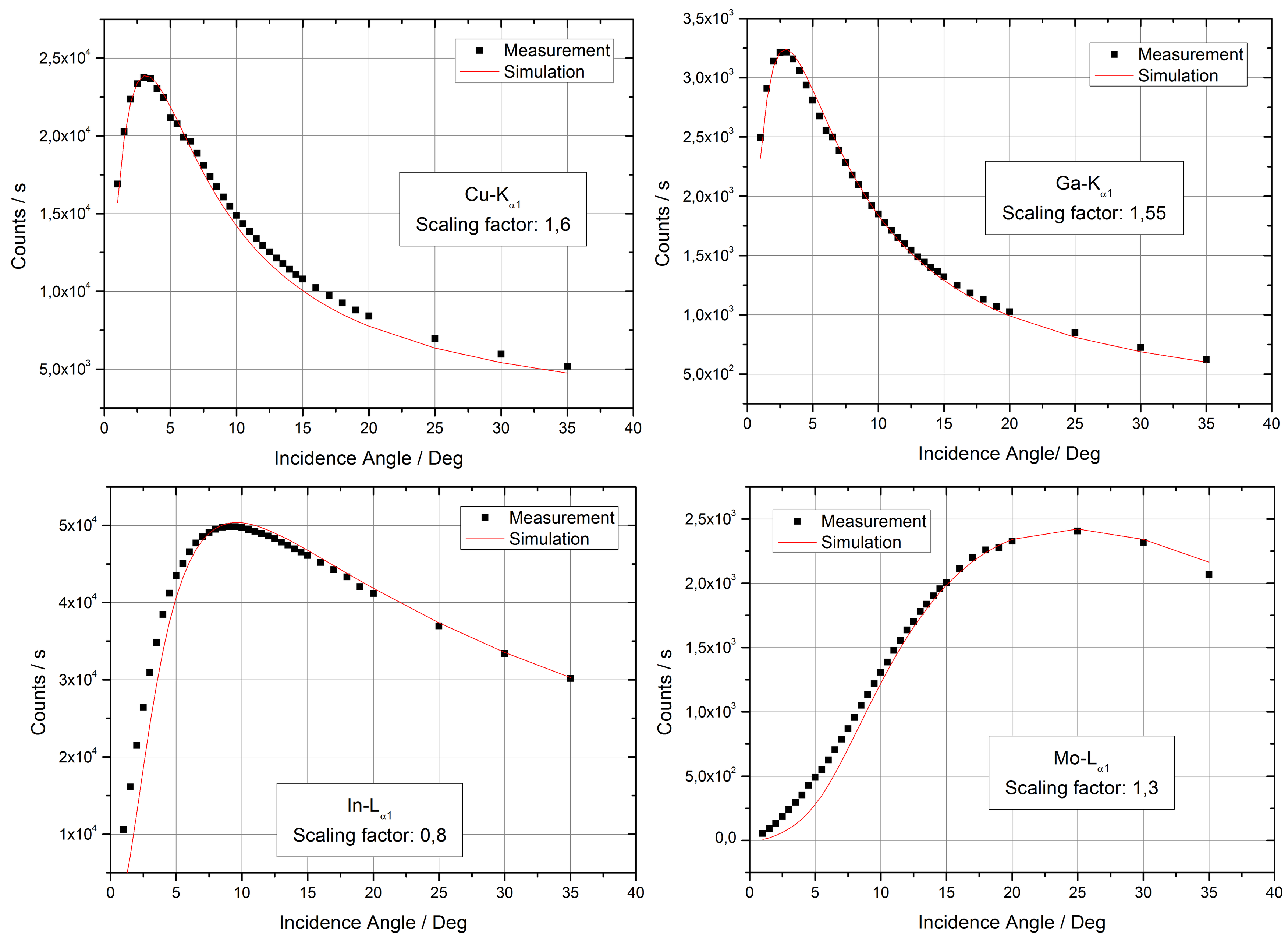

Figure 5: Fluorescence intensities for Ga $K_{\alpha1}$, Cu $K_{\alpha1}$, In $L_{\alpha1}$ and Mo $L_{\alpha1}$ from sample 300 nm CuGa / 850 nm In / 570nm Mo / SiO_2

Also good agreement between simulated curves and measurement data but different calibration factors needed. Especially for the buried layers and small incidence angles deviations become evident. This could be caused by secondary fluorescence effects which are not included yet.

Variances between estimated and real layer thicknesses or formation of diffusion gradients in between could be another explanation.

Further investigations and adaptions are planned.

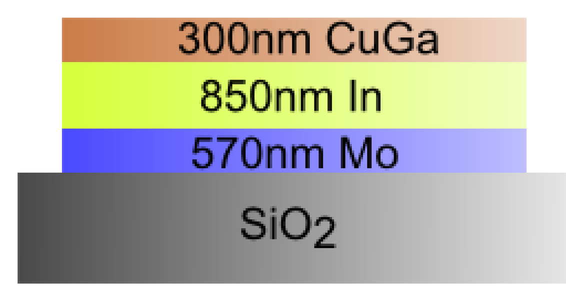

Figure 6: Principle texture for the multi layer sample system

Perspectives

Goal: a suitable laboratory setup for the investigation of elemental depth profiles e.g. in thin film solar cells or diffusion layers in the nm-regime

- Further development of forward calculation analysis
- Adaption and enhancement for already existing backward calculation algorithm for monochromatic excitation on laboratory setup conditions

Contact

Malte Spanier, PhD Student
mspanier@physik.tu-berlin.de

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[1] Institute for Optic und Atomic Physics, Technical University Berlin, Hardenbergstr. 36, 10623 Berlin, Germany
[2] Physikalisch- Technische Bundesanstalt, X-ray and IR Spectrometry, Abbestr. 2-12, 10587 Berlin, Germany