# SPECTROSCOPIC EUV REFLECTOMETRY FOR CHARACTERIZATION OF THIN FILMS AND LAYERED STRUCTURES

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### INTRODUCTION

Spectroscopic reflectometry with Extreme Ultraviolet radiation (EUV, 5-40 nm) offers a possibility of non-destructive study of surfaces. As a photon-in/photon-out technique, EUV reflectometry offers probing depth up to 50-100 nm, which is exceeding that of electron-out techniques, where the probing depth is limited to single nanometers due to short mean free paths of the ejected electrons. Because of the strong and element specific interaction of EUV with matter the method shows high sensitivity and chemical contrast to buried thin interlayers. High reflectivity in grazing-incidence geometry (up to 10°-20° to the surface) makes possible surfaces sensitive analysis of ultrathin film systems and determination of layer parameters such as thickness, roughness, elemental composition and material density.

#### PENETRATION DEPTH OF EUV RADIATION

For the EUV radiation of given wavelength  $\lambda_{c}$  the estimated penetration depth  $z_{0}$  into the interface depends on the grazing incidence angle  $\theta_{r}$  and can be found as follows:



Near-edge penetration depth  $z_0$  for SiO<sub>2</sub> (left) and LaLuO<sub>3</sub> (right) at various incidence angles, calculated for 1/e decay (k=1). Optical constants from *http://henke.lbl.gov/optical\_constants/* 

#### NEAR-EDGE EUV REFLECTOMETRY

Presence of an absorption edge in the reflectivity spectra allows for direct elemental sensitivity. Near-edge fine structure in reflectivity spectra, which arises from multiple scattering of excited electrons by neighboring atoms, contains information about the local site symmetry and chemical bonds and can be used for quick qualitative characterization of structures (spectral "fingerprints").



Reflectivity spectra of natural (left) and thermally grown (right) silicon oxide with near-edge fine structure allow to determine silicon oxidation states in the compound and distinguish between crystalline and amorphous phases. Blue line – laboratory tool measurements, red and black lines – literature data for comparison.

[11] M. Banyay, Surface and Thin Film Analysis by Spectroscopic Reflectometry with EUV Emitting Laboratory Sources, PhD thesis, RWTH Aachen (2011 [\*1]E. O. Flatova, A. A. Sololov, E. Vu. Taracheva, I. V. Bagrov, Technical Physics Letters 35 (1), 70–75 (2009) [\*1]E. O. Flatova, A. S. Sholakov, and V. A. Lukyanov, Phys. Solid States 4(7), 1327-1240 (1596)

## DETERMINATION OF OPTICAL CONSTANTS

The combination of spectrally and angularly broadband measurements offers high precision of the analysis, which exhibits low parameter cross-correlation and allows determination of refractive index.



(Left) Measured and fitted reflectivity spectra for grazing incidence angles from 2° (top curve) to 44° (bottom curve) in 3° steps. (Right) Measured (determined from fit results) and calculated (from atomic scattering factors) refractive index n=1- $\delta$ +i $\beta$  of a high-k dielectric highlighting the sensitivity of EUV optical constants to material crystallinity.



LAB-BASED TOOL AND APPLICATIONS



- Plasma-based EUV source
- Kr/Xe working gas mixture
- Multi-angle: 2-15°
- Broadband: 9.5-17 nm
- Single pulse measurements
- Real-time reflectivity display
- Spatially resolving
  Relative-to-angle and absolute reflectivity measurements

Photo of the Polychromatic Angle-resolving Non-destructive Tool for High-speed Extreme ultraviolet Reflectometry (PANTHER). Inset: operation scheme of the reflectometer.

Si sample, contaminated in a LPP-source chamber was investigated. Absolute reflectivity was measured with stepwise movement across the separation area of two contamination regions. The reflectivity map and 2D plots from different positions are shown below.



Si sample with two contamination regions (left) and its reflectivity map showing transition between contaminated regions (middle). Reflectivity spectra (blue) and the fit results (red) at different X positions on the sample, from top to bottom: 3 mm, 0.8 mm, 0.4 mm, 0 mm (right). The central part of the silicon wafer (X = 0 mm) shows the presence of a crystalline silicon oxide layer (4 nm). In the contaminated region (X = 3 mm), the best fit result has 0.7 nm of carbon and 10 nm of amorphous Al2O3 topping the oxidized silicon wafer. Two minima (in the top curve) at 15.7 and 16.2 nm correspond to the near-edge feature of the deposited material – amorphous aluminum oxide [\*\*\*\*]. Blaws, Kneis, M. Mende, L. Jensen, and D. Ritau, Proc. SPIE 3237, 33371' (2014)





(Left) EUV emission spectra of pure Xe (blue) and Xe/Ar mixture (red) utilized for the photoemission electron microscopy. Bandwidth of the combination of two ML-mirrors is shown in black. (Right) Section of the photoelectron spectrum measured at 13.7 eV photon energy. Electron binding energies of the materials that fit within the peak are highlighted.

#### LLO FILMS OF DIFFERENT CRYSTALLINITY

Different crystalline modifications of LaLuO<sub>3</sub> (LLO) have been characterized by EUV reflectometry. Measurements were performed at ELETTRA synchrotron facility (BEAR beamline).



Reflectivity spectra of LaLuO<sub>3</sub> (LLO) samples measured at  $5^{\circ}$  (left) and  $20^{\circ}$  (right) grazing incidence angle. Insets show zoomed La absorption edge region.



Reflectivity spectra of orthorhombic LLO at different incidence angles: comparison with PANTHER measurements (left) and IMD best fit results (right).



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