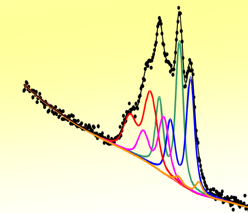
Comparative Study of Two Different Methods for Film Thickness Determination on Model and Real Systems Using Software UNIFIT 2010



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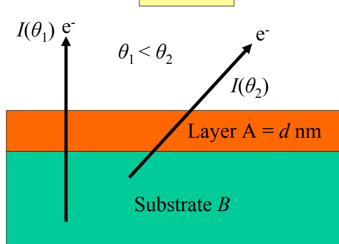
Abstract: The accuracy of thickness determination of laterally homogenous films by XPS in the range of few nm may be improved by using two different methods. The well established angle resolved photoelectron spectroscopy (**ARXPS**) for determining film thicknesses will be compared with the method using the relative quantification of **t**wo **d**ifferent photoelectron lines at different kinetic **e**nergies (**TDEXPS**) and the same emission angle. Only the substrate intensities were used. The advantages and disadvantages of both methods will be shown. The reliability and accuracy of the thickness determination by the two different methods is discussed for suitable model and real systems. The easy handling of the data analysis for estimating film thicknesses using UNIFIT will be demonstrated.

Theorie

ARXPS

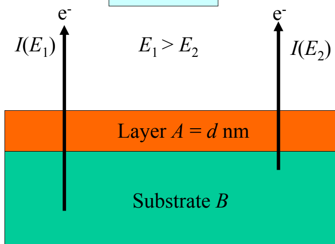


$$d = \lambda_A \cdot \cos\theta \cdot \ln\left(\frac{I_{B\infty}(\theta)}{I_B(\theta)}\right)$$

$I_{B\infty}$ - Intensity clean substrate B, I_B – Intensity covered substrate B
$\lambda_A(E)$ – Inelastic mean-free path layer A (or attenuation length *EAL*)
θ – Emission angle with respect to surface normal

Requirements:
- well calibrated angles
- stable excitation conditions
- stable spectrometer sensitivity

TDEXPS

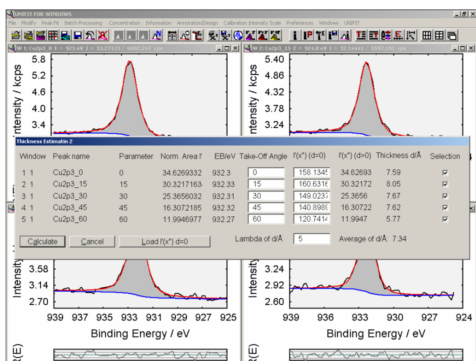


$$d = \cos\theta \cdot \frac{\lambda_A(E_1)\lambda_A(E_2)}{\lambda_A(E_1) - \lambda_A(E_2)} \cdot \ln\left(\frac{I'_B(E_1)}{I'_B(E_2)}\right) \quad [1]$$

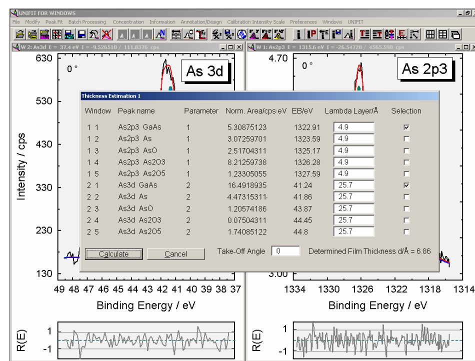
I'_B – Normalized intensity covered substrate B, $I'_B = I_B/SF$
$\lambda_A(E)$ – Inelastic mean-free path layer A (or attenuation length *EAL*)
SF – Sensitivity factor

Requirements:
- well known sensitivity factors $SF = \sigma \cdot T \cdot \lambda_B$ with:
  $T(E)$ - Transmission function, $\sigma(h\nu)$ – Photo ionisation cross-section,
  $\lambda_B(E)$ – Inelastic mean-free path substrate B
- Ratio of the normalized intensities of the clean substrate is unity $I'_{B\infty}(E_2)/I'_{B\infty}(E_1) = 1$

Input/Output Dialogs



Input: 1. Intensity substrate (peak area) with (I_B) and without layer ($I_{B\infty}$)
2. Take-Off angles θ, 3. Lambda parameter (*IMFP* or *EAL*)

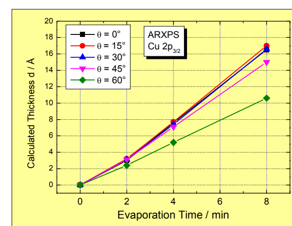


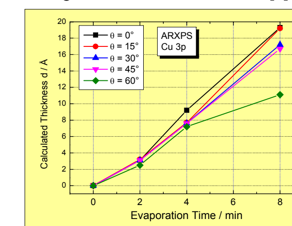
Input: 1. Normalized Intensity substrate (peak area) $I'_B(E_1)$ and $I'_B(E_2)$
2. Take-Off angle θ, 3. Lambda parameters (*IMFP* or *EAL*) of E_1 and E_2

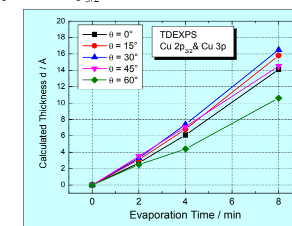
Lit: [1] S. Hofmann in: Ed. D. Briggs, M.P. Seah, Practical Surface Analysis by Auger and X-ray Photoelectron Spectroscopy, John Wiley & Sohn, Chinchester 1983, S. 168
[2] C.J. Powell, A. Jablonski, NIST Electron Effective-Attenuation-Length Database, Version 1.0, SRD 82, Gaithersburg, MD, 2001; A. Jablonski, C.J. Powell, Surf. Sci. Rep. 47, 33 (2002)

Example 1: Fe on Cu foil (ARXPS / TDEXPS)

Experimental conditions:
- Spectrometer: ESCALAB 220 iXL
- Lens mode: SAE 120, Mechanical Apertures: Field of View Lens: 4, Objective Lens: 5, Angle acceptance: 4°:
- Evaporator: Focus EVC 300, Evaporation time: 2 min, 4 min, 8 min
- Lambda values: Average EAL of the NIST software [2] for Cu 3p and Cu $2p_{3/2}$





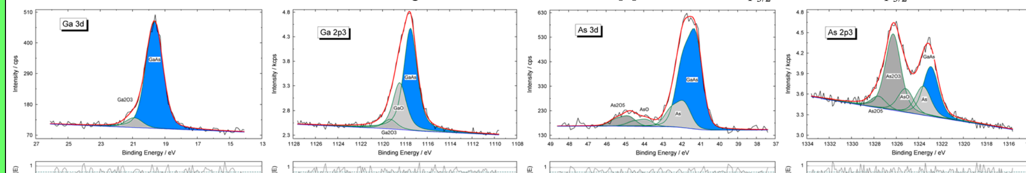


	d / Å		
Method /Sample	2 min	4 min	8 min
ARXPS Cu $2p_{3/2}$, Cu 3p	2.5, 2.5	7.5, 8.0	16.0, 18.0
TDEXPS, Cu 3p & Cu $2p_{3/2}$	3.0	7.0	15.0

Results:
- 60° data not useful here due to experimental problems
- potentially non-homogeneous sample at 2 min evaporation time,
- relatively good agreement between both methods

Example 2: Oxid on GaAs (ARXPS / TDEXPS)

Experimental conditions:
- Spectrometer: ESCALAB 220 iXL
- Lens mode: SAE 120, Mechanical Apertures: Field of View Lens: 5, Objective Lens: 7, Angle acceptance: 6°
- Oxide layer generated by Ozone, - Peak areas estimated by peak fit
- Lambda values: Average EAL of the NIST software [2] for Ga 3d, Ga $2p_{3/2}$, As 3d and As $2p_{3/2}$



ARXPS

	d / Å		
Lines /Angle	0°	30°	60°
Ga 3d / Ga $2p_{3/2}$	9.7 / 7.3	6.1 / 8.5	14.6 / 9.2
As 3d / As $2p_{3/2}$	11.4 / 8.6	7.4 / 6.5	10.2 / 7.0
Average of d / Å: 8.9			

Strong variations due to overlapping intensities of layer and substrate!
ARXPS: - Problems with clean substrate measurements (e.g. preferential sputtering)
TDEXPS - avoids clean surface data
➔ big advantage on complex samples

TDEXPS

	d / Å		
Lines Pair/Angle	0°	30°	60°
Ga 3d, Ga $2p_{3/2}$	7.4	9.8	7.7
As 3d, As $2p_{3/2}$	6.9	7.0	6.9
Average of d / Å: 7.6			

Summary (ARXPS / TDEXPS)

- **both methods, ARXPS and TDEXPS using substrate intensities (appropriate for layers <2,5 nm), give similar results,**
- **the choice of the used Lambda parameter (*IMFP*, Practical *EAL*, Average *EAL*, ...) has the largest influence of the calculated, absolute values of the layer thickness *d*,**
- **the calculated uncertainty of *d* estimated with TDEXPS is about two times larger compared with ARXPS,**
- **TDEXPS requires only one measurement, but ARXPS needs two,**

ARXPS:
- **the estimation of the layer thickness *d* using ARXPS is independent from sensitivity factors and is well established,**
- **stable spectrometer conditions have to be saved**

TDEXPS:
- **well calibrated spectrometers and appropriate photo lines are required,**
- **intensity of the clean substrate is not necessary**

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