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

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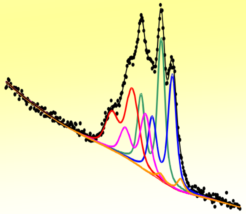
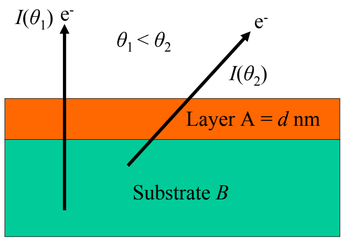


Abstract: The accuracy of thickness determination of laterally homogenous films by XPS in the range of few nm may be improved by using two different methods. The well established angle resolved photoelectron spectroscopy (**ARXPS**) for determining film thicknesses will be compared with the method using 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



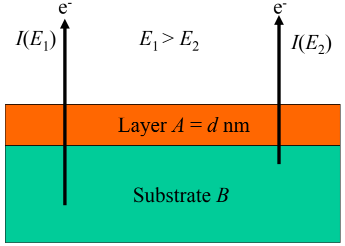
$$d = \lambda_A \cdot \cos\theta \cdot \ln\left(\frac{I_{B\infty}(\theta)}{I_B(\theta)}\right)$$

$I_{B\infty}$ - 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



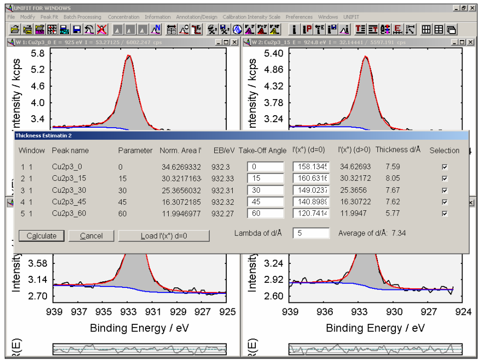
$$d = \cos\theta \cdot \frac{\lambda_A(E_1)\lambda_A(E_2)}{\lambda_A(E_1)-\lambda_A(E_2)} \cdot \ln\left(\frac{I'_B(E_1)}{I'_B(E_2)}\right) \qquad [1]$$

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

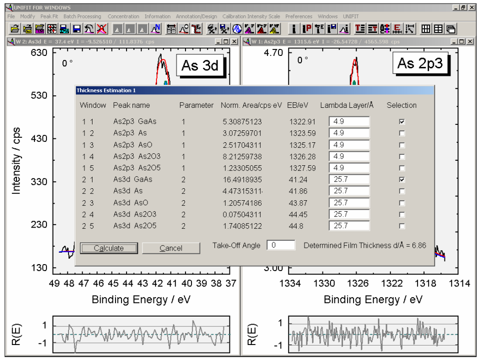


Thickness Estimatin 2

Window	Peak name	Parameter	Norm. Area I'	EB/eV	Take-Off Angle	I'(x°) (d=0)	I'(x°) (d>0)	Thickness d/Å	Selection
1 1	Cu2p3_0	0	34.6269332	932.3	0	158.1345	34.62693	7.59	☑
2 1	Cu2p3_15	15	30.3217163	932.33	15	160.6316	30.32172	8.05	☑
3 1	Cu2p3_30	30	25.3656032	932.31	30	149.0237	25.3656	7.67	☑
4 1	Cu2p3_45	45	16.3072185	932.32	45	140.8989	16.30722	7.62	☑
5 1	Cu2p3_60	60	11.9946977	932.27	60	120.7414	11.9947	5.77	☑

Lambda of d/Å: 5
Average of d/Å: 7.34

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*)



Thickness Estimation 1

Window	Peak name	Parameter	Norm. Area/cps·eV	EB/eV	Lambda Layer/Å	Selection
1 1	As2p3 GaAs	1	5.30875123	1322.91	4.9	☑
1 2	As2p3 As	1	3.07259701	1323.59	4.9	☐
1 3	As2p3 AsO	1	2.51704311	1325.17	4.9	☐
1 4	As2p3 As2O3	1	8.21259738	1326.28	4.9	☐
1 5	As2p3 As2O5	1	1.23305055	1327.59	4.9	☐
2 1	As3d GaAs	2	16.4918935	41.24	25.7	☑
2 2	As3d As	2	4.47315311	41.86	25.7	☐
2 3	As3d AsO	2	1.20574186	43.87	25.7	☐
2 4	As3d As2O3	2	0.07504311	44.45	25.7	☐
2 5	As3d As2O5	2	1.74085122	44.8	25.7	☐

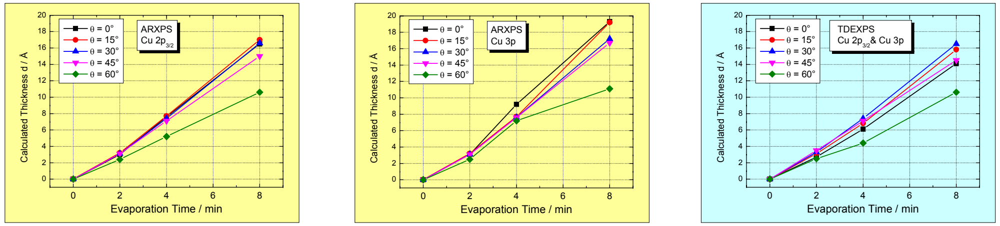
Take-Off Angle: 0
Determined Film Thickness d/Å = 6.86

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)

ARXPS — Example 1: Fe on Cu foil — 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 $2p_{3/2}$

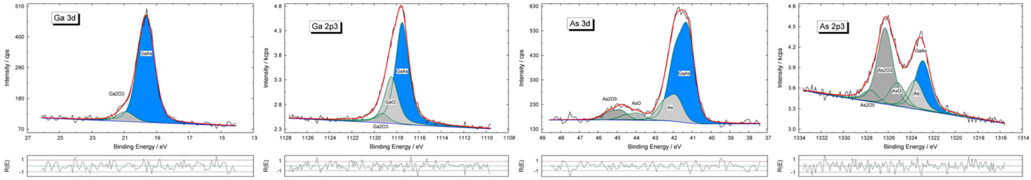


	d / Å		
Method /Sample	2 min	4 min	8 min
ARXPS Cu $2p_{3/2}$, Cu 3p	2.5, 2.5	7.5, 8.0	16.0, 18.0
TDEXPS, Cu 3p & Cu $2p_{3/2}$	3.0	7.0	15.0

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

ARXPS — Example 2: Oxid on GaAs — 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 $2p_{3/2}$



	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

	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			

ARXPS — Summary — 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,**

- **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**

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

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