TEM, STEM and T-SEM EDS Quantification at its Best



Webinar, June 2019



TEM, STEM and T-SEM EDS Quantification at its Best



Speaker:

Meiken Falke,

Global Product Manager TEM-EDS,

Bruker Nano GmbH, Berlin

Host:

Max Patzschke

Application Scientist,

Bruker Nano GmbH, Berlin





Outline



- Intro: TEM, STEM and T-SEM EDS
- Geometry Considerations
 - collection / take off angle
 - Sample position ... tilt, mounting, shape
- TEM EDS Quantification
- TEM EDS Quantification and Display of Results:
 - ESPRIT implementation
 - ESPRIT SW use for TEM-EDS explained in simple steps
- Problems, Tricks and Tips

EDS from Electron Transparent Samples





- TKD specimen holder
- Commercial STEM holders
- Home made versions

Solid Angle Correction for Flat SDD in 2D





The solid angle of A_{D_Flat_vertical}

$$\Omega = A_{\rm corr} / (r + \Delta r)^2$$

Smaller detector closer to specimen enables

- Larger A_{corr}
- Larger solid angle

A large take-off angle above the sample is needed to avoid shadowing and stray radiation > use small area!

Small detector areas can be positioned higher above the sample due to geometric constraints in TEM.





How to calculate geometric solid angle:

N. Zaluzec, on solid angle Microsc. Microanal., 15 (2009) 93;

Also check Nestors web page!

How to measure solid angle using standard:

R. F. Egerton, S. C. Cheng, Ultramicroscopy 55 (1994) 43-54;

Also see formulas on later slides!































































Geometric Limitations (TEM): Solid and take-off angle!!





Geometric Limitations (TEM)





Inverse solid angle! ... how much of the surroundings do we see? A small collimator opening is better to avoid system peaks.

Specimen Mounting







- Thinnest part of specimen must point towards EDS detector
- The specimen holder must have a cut out towards EDS detector
- Make sure that the path for X-rays is not obstructed by sample or grid parts or holder
- Beware: most holders are turned 180° during insertion

Specimen Mounting; View along column







Specimen Mounting; View along column





TEM EDS Quantification; R. Egerton 1994, line intensity for a particular element line / transition



 $I_{\rm x} = N_{\rm A} \, \sigma_{\rm A} \, \omega_{\rm A} \, (\Omega/4\pi) \, \varepsilon \, N_{\rm e} = n_{\rm A} \, t \, \sigma_{\rm A} \, \omega_{\rm A} \, (\Omega/4\pi) \, \varepsilon \, N_{\rm e}$

Cliff and Lorimer:

 $k_{AB} C_B$; k_{AB} can be determined experimentally or theoretically I_{B} I_{x} number of X-ray photons in a characteristic peak of species A Ν number of atoms per unit volume n t number of atoms per unit area times thickness ionization cross section (Casnati et al., 1982, Bote et al., 2009) σ fluorescence yield (Hubbell et al., 1994, Krause, 1979) ω $\Omega/4\pi$ solid angle / geometrical collection efficiency detection quantum efficiency 3 N number of incident electrons + absorption

Semiconductor Structure; Standard STEM, 30mm2 SDD, ~ 0.1sr





Semiconductor Structure; First View Standard STEM, 30mm2 SDD, ~ 0.1sr





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Finder H He F1 F2 F3 F4 F5 F6 F7 F8 Li Be Inputs B C N O F Ne I1 I2 I3 I4 I5 I6 I7 I8 Na Mg Al Si P S Cl Ar K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn Fr Ra Ac Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No Lr Lines Fe Clear all Auto ID -Dynamic lines New element Regions

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Semiconductor Structure; First View: Specimen region of interest for quant. Check composition / condition variations





Semiconductor Structure; First View: Specimen region of interest; Check composition / condition variations





For Cliff-Lorimer quantification: calculate theoretical Cliff-Lorimer factors for the specific geometry and conditions



,Standards' menu:



EDIT CLIFF-LORIMER FACTORS

? ×



Factor

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Import



Legend:

- 0.000 Undefined data
- 1.234 Calculated data from theory
- 1.234 Calibrated data

AN	El.	K	L	L M	
13	Al	0,985	0,000	0,000	
14	Si	1,000	0,000	0,000	
15	Ρ	1,043	0,000	0,000	
16	S	0,990	0,000	0,000	
17	Cl	1,039	0,000	0,000	
18	Ar	1,129	0,000	0,000	
19	К	1,065	0,000	0,000	
20	Ca	1,136	104,791	0,000	
21	Sc	1,167	44,596	0,000	
22	Ti	1,185	31,685	0,000	
23	V	1,234	12,196	0,000	
24	Cr	1,254	8,398	0,000	
25	Mn	1,336	5,012	0,000	
26	Fe	1,378	2,962	0,000	
27	Со	1,490	2,359	0,000	



Titanium

Reset

Se	Ref.	Standard
К		
L		
М		

Reset all

Cancel

OK



Semiconductor Structure; First View



Quantification setup





Element Mapping Results; theoretical C-L factors








T-SEM with radially symmetric EDS; fluorescence







T-SEM with radially symmetric EDS; fluorescence, pseudo color





Preview Capture ~ Acquire	✓ ○ QMap ✓ EDS ⊕ TEM XRF ⊕ XRF	Loaded: C:\Qua	ntax User\Edx\Data\chip 20kV	60min.bcf 🗲
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		200 mm		$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
	Result types	P EDS STEM Loaded: C:\Quantax User\Edx\Data\chip 20kV 60min.bcf Map Phases Charts Line scan Spectrum 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 92 <p92< p=""></p92<>		
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39

T-SEM with radially symmetric EDS; fluorescence >system peaks







For Cliff-Lorimer quantification: calculate theoretical Cliff-Lorimer factors for the specific geometry and conditions



,Standards' menu:



Qmap setup









24.09.2019

Display quant result in at% for Ti





1

Display quant result in at% for W





24.09.2019

Quant result with 4x4 pixel binning: net counts, now check of 2 ROI







Add to standards

OK



OK



Density: 4,00 g/cm³

OK



Add to standards

OK

EDS for Catalysis, Quantification Pt-Pd Core Shell Particles



mass%, 30 mm², 0.12 sr (Standard EDS); Cs-corr. STEM



24.09.2019

EDS for Catalysis, Quantification Pt-Pd Core Shell Particles





BRUKER

Data courtesy: Dogan Ozkaya, Johnson Matthey Technology Center

EDS for Life Science at 0.1sr Malaria Parasite: *Plasmodium* in erythrocyte treated with Chloroquine







TEM EDS Quantification; R. Egerton



TEM EDS Quantification; R. Egerton

24.09.2019

TEM EDS Quantification Cliff Lorimer / Zeta-factor ... + EELS!





G. Kothleitner et al., Microsc. Microanal. **20** (2014) 678 Towards assumptionless analysis ...



682 Gerald Kothleitner et al.



Figure 3. Relative thickness map (t/λ) (a), and model of the thickness of the focused ion beam milled cone (b). The inverse of (a) multiplied by (b) yields a map displaying the inelastic mean free paths λ of the sample, used for further thickness calibrations (c).

G. Kothleitner, Micr. Microanal. 2014

+ G. Kothleitner, Micr. Microanal. 2014



TEM Quantification in Esprit

Zeta Factor Method to Retrieve Thickness and Composition, EELS used here only as Reference



TEM Quantification in Esprit 2.1 Zeta Method: GaP wedge







24.09.2019

Experimental C-L or Zeta factors:



InAs Nanorods with P-rich layers: Quantified Linescan, 30mm² SDD, SLE window, 0.12sr









Respective data set view; see Ni-stray radiation from Ni grid: no/low Ni-L line!





Quantification: exp. Cliff-Lorimer factors



QUANTIFICATION - 1



Background regions Element lines Dynamic lines

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Phosphorus	15	0	0,00	0,00	0,00	0,00	0,00
Arsenic	33	311	39,30	39,30	50,20	2,69	6,85
Nickel	28	522	0,00	0,00	0,00	0,00	0,00
Gold	79	11	2,25	2,25	1,09	0,86	38,32
Carbon	6	148	0,00	0,00	0,00	0,00	0,00
Indium	49	551	58,45	58,45	48,71	6,68	11,43
Copper	29	23	0,00	0,00	0,00	0,00	0,00
		Sum	100,00	100,00	100,00		

Density: 6,71 g/cm³



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Quantification: exp. Cliff-Lorimer factors, Change the less well known factors: (if overlapps, low energy, L-lines)



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escription	
eal time [s]: 8,489	VALIDATION, LAST STEP
e time [s]: 8,489	Confirm assignment and certification values
ecification in O Mass-% Atomic-% O Stoich-% 	Standardy
	Description:
ement Atom conc. [%] Error [%]	Mass
osphorus 0,00	Assign Element concentration Error Currently assigned to sta
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Solution Solution sel 0,00 g 0 \$ pon 0,00	Standard cannot be used for Phi(RhoZ) quantification, because spectrum does not contain system calibration data. You should calibrate your system in this case.
enic 50 kel 0,00 ld 0 \$ bon 0,00 ium 50	Standard cannot be used for Phi(RhoZ) quantification, because spectrum does not contain system calibration data. You should calibrate your system in this case.
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enic 50 kel 0,00 d 0 \$ bon 0,00 ium 50 iper 0,00	Standard cannot be used for Phi(RhoZ) quantification, because spectrum does not contain system calibration data. You should calibrate your system in this case. Check value of Cliff-Lorimer factors: Assign Element Reference New factor Old factor Arsenic 2,43329 2,43329 4,81637

Quantification: Cliff-Lorimer factors, Experimentally determined factor in red





Zeta-factor method: Need beam current and Standard with known c, t, ρ can be calculated or known



×

EDIT STANDAR	RD PROPERTIES X
Name	1
Description	
Real time [s]:	8,489
Life time [s]:	8,489
Specification in	O Mass-%
Element	Atom conc. [%] Error [%]
Phosphorus	0,00
Arsenic	50
Indium	50
Nickel	0,00
Gold	0
Carbon	0,00
Copper	0,00
Oxygen	0,00
Sum of concor	trations [%]: 100 00%
Sum of concern	
Thickness [nm	1] 30
Density [g/cm	³] 6,62
Beam current	[pA] 110 🗘
	Add to standard library
Ad	ld as temporary reference

VALIDATION, LAST STEP

Confirm assignment and certification values.

Standard: 1

Description:

Assign	Element	Mass concentration	Error	Currently assigned to standard
V	Arsenic	39,49%		
Image: A start of the start	Indium	60,51%		
	Gold	0,00%		

Check value of Zeta factors:

Assign	Element	New factor	Old factor	
	Arsenic	1691,50363	2,43329	
Image: A start of the start	Indium	2158,37022	2,90644	
	Gold	0,00000	4,81637	

OK

Back



Get all Zeta-factors from the C-L factors

EDIT ZETA FACTORS

- Legend:
- 0.000 Undefined data
- 1.234 Calculated data from theory
- 1.234 Calibrated data

AN	El.	К	L	М	-
32	Ge	2,229	2,052	0,000	
33	As	1691,504	2,129	0,000	
34	Se	2,742	2,144	0,000	
35	Br	2,994	2,100	0,000	
36	Kr	3,431	2,254	0,000	
37	Rb	3,860	2,220	0,000	
38	Sr	4,420	2,259	0,000	
39	Y	5,045	2,329	0,000	
40	Zr	5,862	2,343	0,000	
41	Nb	6,858	2,302	0,000	
42	Мо	8,129	2,341	0,000	
43	Тс	9,614	2,497	0,000	
44	Ru	11,567	2,470	0,000	
45	Rh	13,759	2,596	0,000	
46	Pd	16,713	2,710	0,000	
47	Ag	19,909	2,775	0,000	
48	Cd	24,367	2,908	0,000	
49	In	29,324	2158,370	0,000	
50	Sn	35,841	3,266	0,000	
51	Sb	43,374	3,382	0,000	
52	Те	53,774	3,534	0,000	
53	1	63,148	3,516	0,000	
54	Xe	76,943	3,609	0,000	
55	Cs	92,331	3,635	0,000	
56	Ba	112,726	3,745	256,315	
57	La	134,270	3,734	36,943	
го	60	150 600	2 724	21 001	

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Fit to Stds

Import

Cadmium Se.. Ref. Standard

Reset

K L M

Left counts, right quantified; Line Scan > Line Profile





Left C-L, right Zeta-factor method: C-L underestimates P content





- The Zeta-factor method is particularly helpful in case of light/heavy element mixtures
- Cliff-Lorimer also offers some absorption correction suitable for K-lines
- Both are precise within few at% depending on materials
 - C-L measures relative within one sample series under same conditions
 - Zeta measures absolute values, beam current must be stable and standard well known

T-SEM EDS; Semiconductor Structure: Line Scan as well







10 nm lateral resolution of Ni and Ti distribution maxima

Extracted linescan from the map data Effective measurement time of lineprofile: 8 s



^{24.09.2019}

M. W. Chu et al., Phys. Rev. Lett. 104, 196101 (2010)

Columns: easier than single atom ID: EDXS Mapping of $Bi_6Ti_xFe_vMn_zO_{18}$





TCD (Trinity College Dublin) Nion UltraSTEM200XE 200 kV with Bruker 100 mm² X-Flash SD detector, 100 mm² windowless SDD; 0.7 sr collection angle.

432x225 pixels, 4.1 msec/pix => 400 sec for map.

No drift correction. Bi = green, Ti = blue.

courtesy Lynette Keeney, Clive Downing and Valeria Nicolosi. TCD, Ireland.

Atom column EDS needs simulation for correct interpretation B. D. Forbes et al., PHYSICAL REVIEW B **86**, 024108 (2012)



- Electron transparent samples can be analysed in TEM and SEM.
- Geometry must be determined completely to understand or avoid, if possible, systematic errors.
- Careful analysis allows to determine most systematic errors.
- The Zeta-factor method delivers absorption correction and thickness determination, density and thickness are still difficult to disentangle > combine with EELS.
- Atom column EDS needs theory support to account for channeling and cross talk between columns.
- Use EDS to it's full potential, just element ID is not satisfactory!
EDS Quantification of electron transparent samples in TEM, STEM and T-SEM







Innovation with Integrity