Experimental Determination of the Solid Angle of EDX-Detectors

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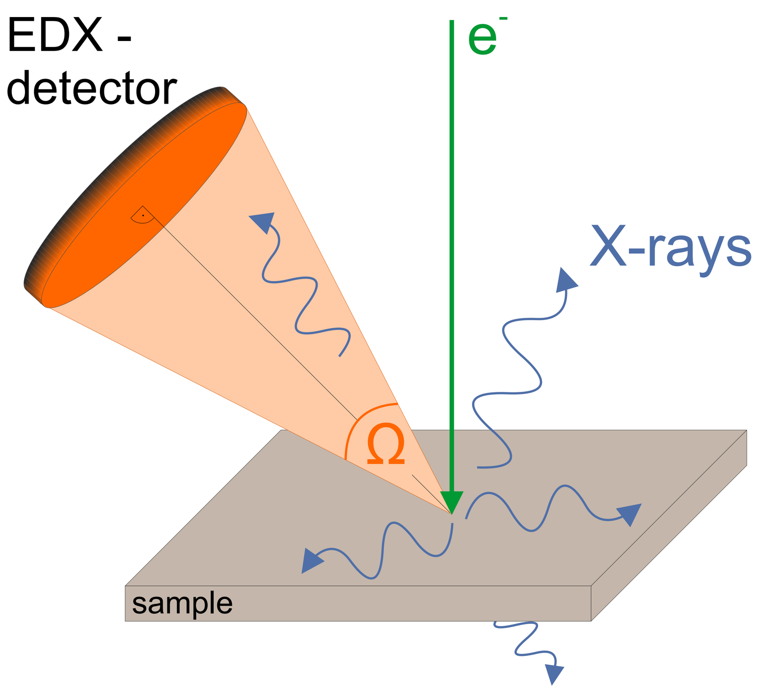
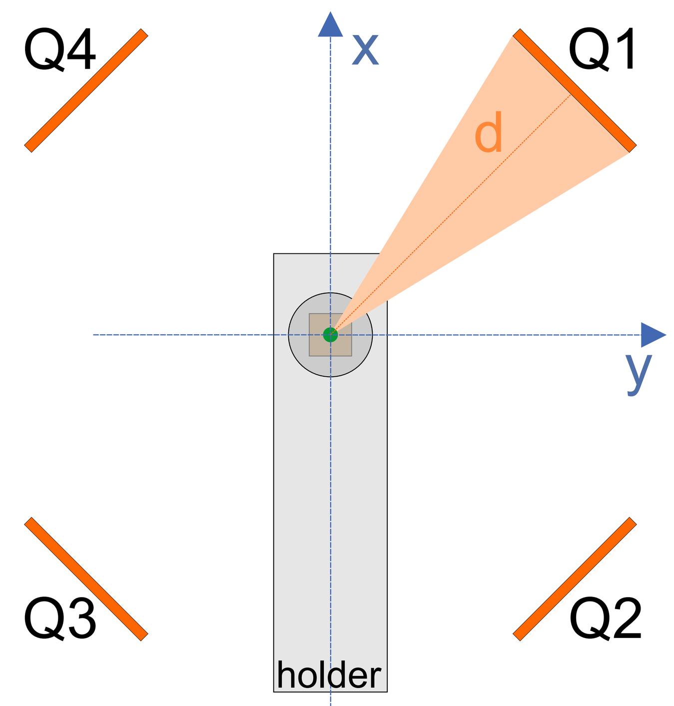
Introduction



Characteristic X-rays are emitted in all directions, but they are only detected within the detector solid angle Ω.

The detector solid angle is a limiting parameter in energy-dispersive electron spectrometry (EDXS) in transmission electron miscroscopy (TEM). A larger solid angle (and therefore an increase of detected X-ray intensities) leads to a higher signal to noise ratio, giving the possibility of: faster measurements, using a lower current to analyse more sensitive specimens, a better detection sensitivity, etc.
In this work we present a reliable method to experimentally determine the detector solid angle extending the method proposed by Egerton et al. [1] .

Used microscopes:
- Titan^3 (300 kV): Super-X detector with four silicon drift detectors (30 mm^2 each), windowless
- Tecnai F20 (200 kV): Si(Li) detector (30 mm^2), ultrathin polymer window



The Super-X detector: four detector-quadrants placed at a distance *d* to the sample.

Approach

The **solid angle Ω** can be determined using the intensity *I* of a certain X-ray line (e.g. K_α) for the element *A* when neglecting fluorescence and absorption via:

$$\Omega = \frac{1}{Q\omega a}\,\frac{I_A^X}{D_e t}\,\frac{AW \cdot 4\pi}{N_{AV} c_A \rho \varepsilon_A}$$

Therefore values for all occuring parameters are necessary. Known by using standard materials: Avogadro's number N_{AV}, atomic weight AW, mass fraction c_A, density ρ, detector efficiency ε_A

State-of-the-art data from literature research

Q … ionization cross section → Database NIST 164 [2]
ω … fluorescence yield → Database EADL [3]
a … X-ray line intensity ratio → Software MA Table [4]

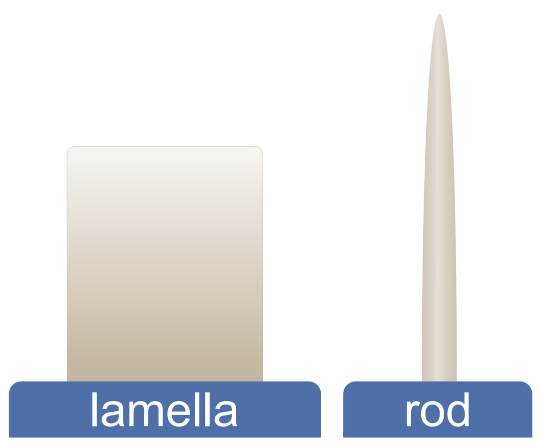
What needs to be measured to determine Ω?

I_A^X … intensity of a single X-ray line
D_e … total electron dose
t … thickness of the specimen

Measurements

Materials
Al
Al_2O_3
Si
SiO_2
GaAs
$SrTiO_3$

Two shapes
The lamella is used for EDX measurements; the rod is needed to determine the inelastic mean free path, thus the thickness of the lamella.

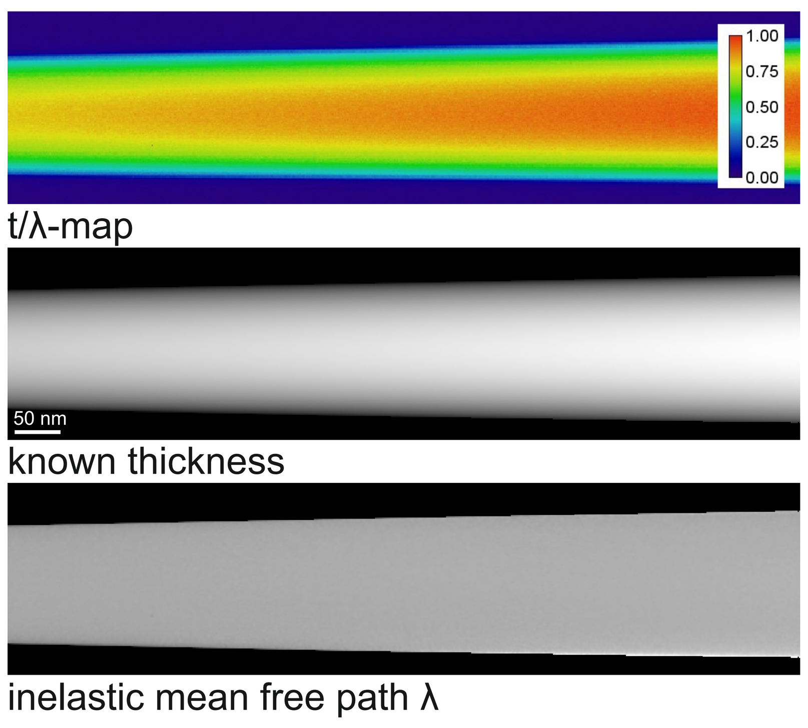


X-ray intensity
- EDX spectrum of the lamella: Gaussian fits to peaks
- K_α-lines from six elements: Al, Si, Ti, Ga, As, Sr

Current (to determine the total electron dose)
- Calibrated CCD (Tecnai F20)
- GIF Drift Tube (Titan^3)

Thickness of the specimen
t/λ-maps with EELS from the rod and the lamella [5]

- Use the rotationally symmetric shape of the rod
- Get the inelastic mean free path λ
- Determine the absolute thickness of the lamella



Results

Super-X Quadrant 1: solid angle Ω [sr] for each element

Ga	0.19	Sr	0.15	Al	0.14	Si	0.15
As	0.17	Ti	0.16		0.15		0.15

Using six different elements (see table above) as well as reliable databases, we are able to determine the solid angle of each detector quadrant with high quality.

	Detector	Ω [sr]	Total Ω [sr]	Detected fraction [%]	Nominal value [sr]
TITAN^3	Super-X Q1	0.16±0.01	0.64±0.03	5.1%	0.7
	Super-X Q2	0.15±0.01			
	Super-X Q3	0.16±0.01			
	Super-X Q4	0.17±0.01			
TF20	Si(Li)		0.06±0.01	0.5%	

References

[1] Egerton et al., Ultramicroscopy 1994, 55, 43–54
[2] Llovet et al., J. Phys. Chem. Ref. Data 2014, 43, 13–112
[3] Perkins et al., LLNL Report 1991, UCRL-50400, vol. 30
[4] Eggert, Microchim Acta 2006, 155, 129–136
[5] Kothleitner et al., Microsc. Microanal. 2014, 20, 678–686

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