Spatially resolved EELS with an in-column Omega filter - characterisation of energy filter aberrations and their correction by image processing

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IMC2014 Prague, IT-5-P-2281

Introduction

Spatially resolved EELS (SR-EELS) [1] is a technique to preserve spatial information when recording EEL spectra. Essentially, many EEL spectra are recorded in parallel as a function of one spatial coordinate, perpendicular to the energy dispersive direction. This method is useful for investigating specimens like interfaces and layer systems. We apply SR-EELS in a TEM with an in-column Omega filter [2]. Remaining aberrations can be corrected by processing the recorded SR-EELS dataset, using the results of a previous characterisation measurement.

Workflow

The SR-EELS workflow involves the following steps:

- **Calibration measurement** of a uniform specimen
- **SR-EELS measurement** of the layer system
- **Characterisation** of the distortion
  - Using scripts for Gnuplot and ImageJ [3]
- **Correction** of the SR-EELS measurement
  - Using a plug-in for ImageJ [4]

SR-EELS measurement

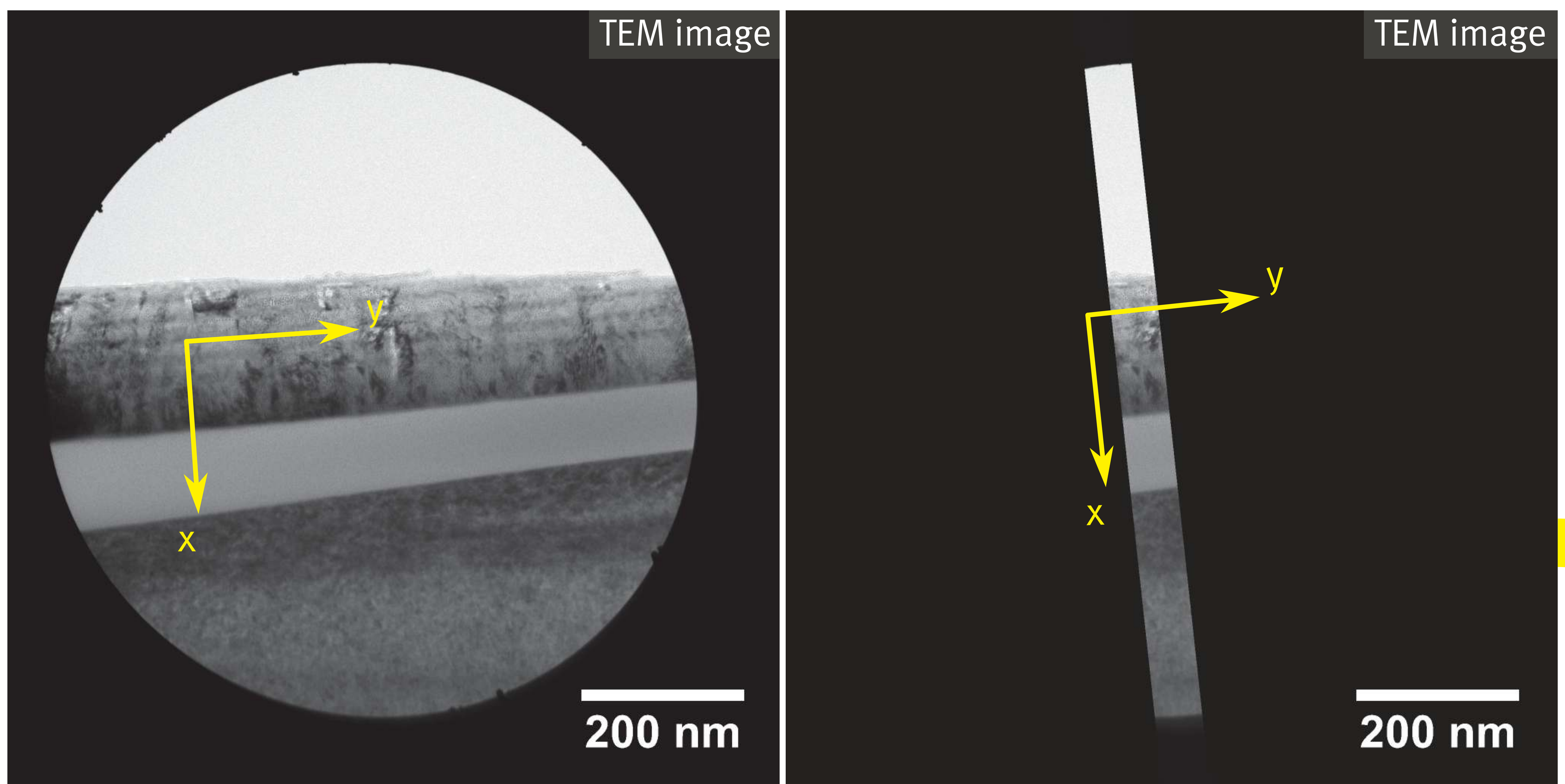


Figure 1: The specimen is an iron chromium layer system on a silicon wafer with silicon oxide surface. **Left:** A round aperture at the filter entrance plane is used. This setup is not preferable for SR-EELS as it results in a non constant aperture width (*Δy(x)*). **Right:** A slit aperture at the filter entrance plane that results in *Δy=const.*

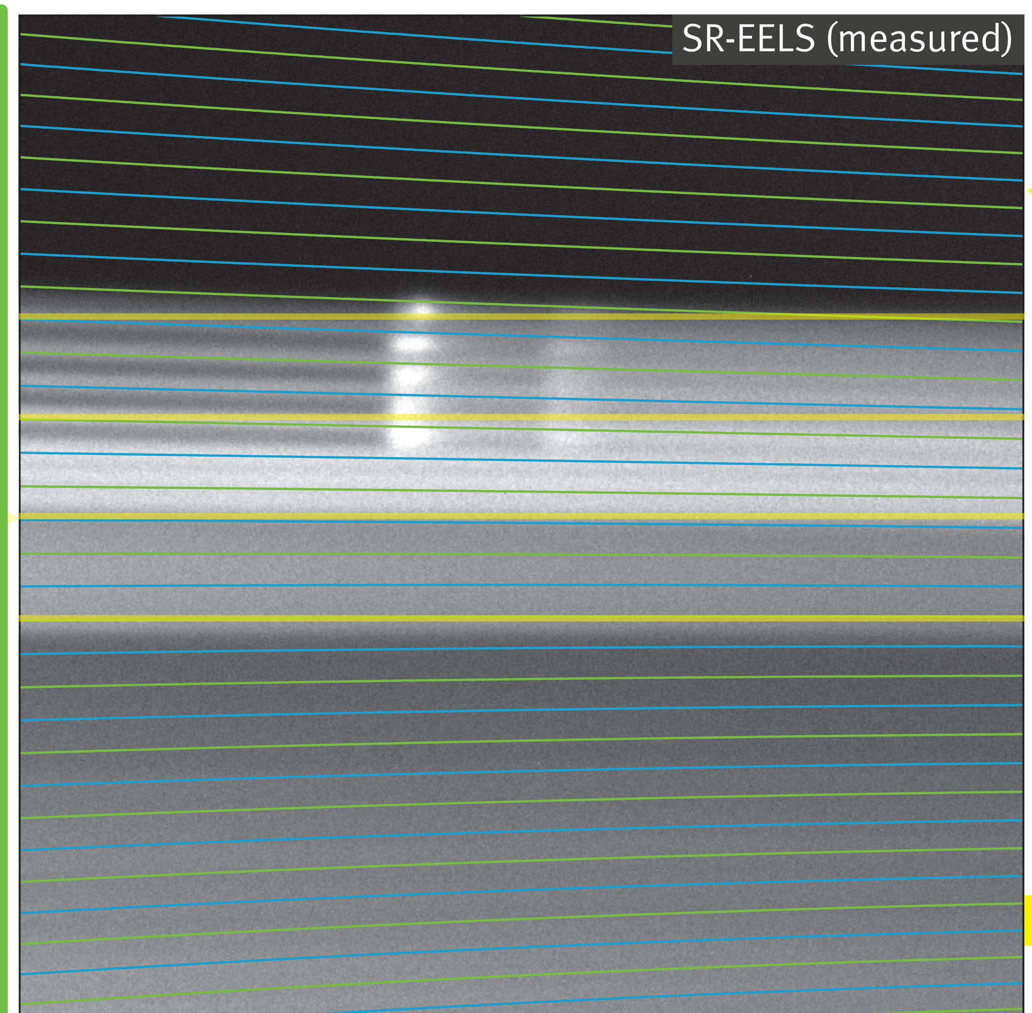


Figure 2: SR-EEL dataset (using QSinK7=-11%) that shows a slight distortion. The overlay shows the distortion that was calculated by evaluating the characterisation measurements.

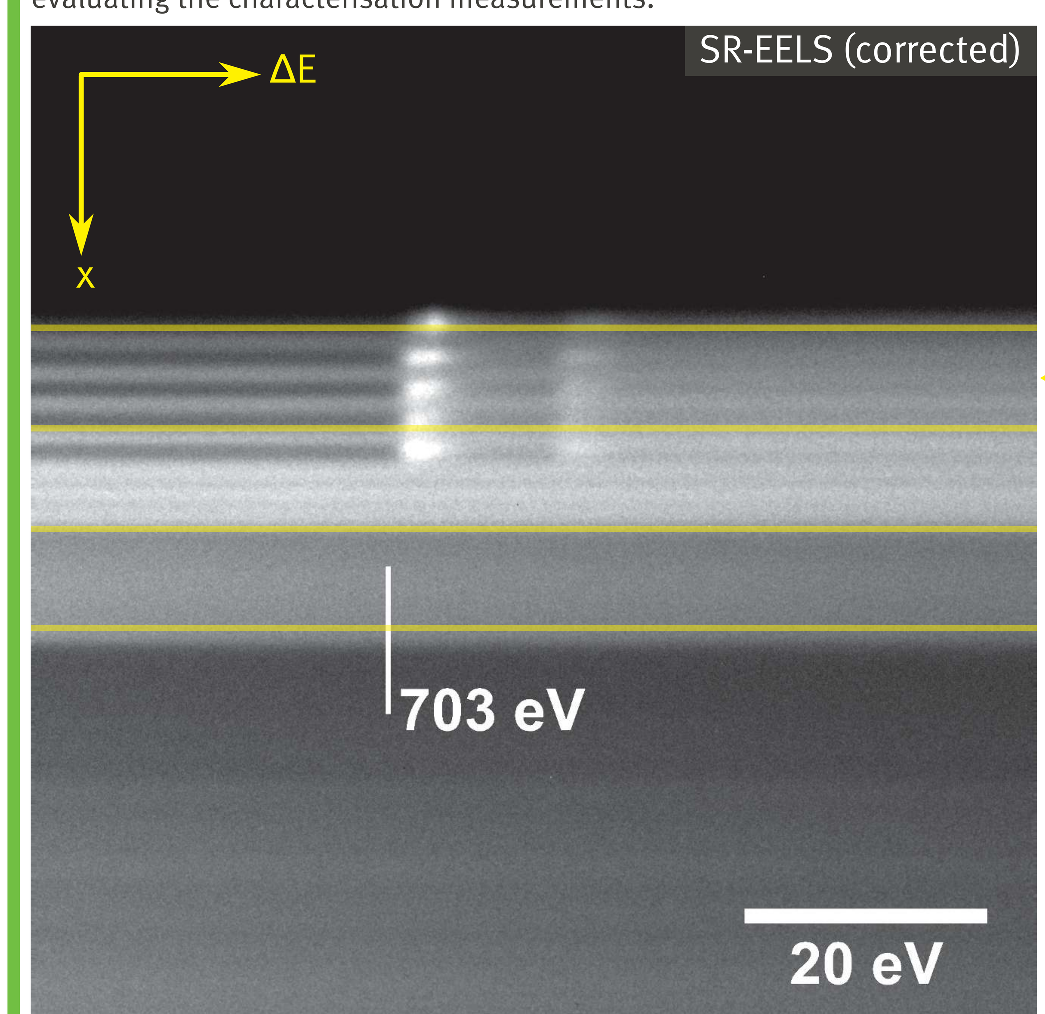
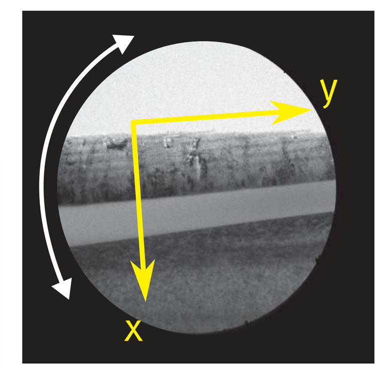
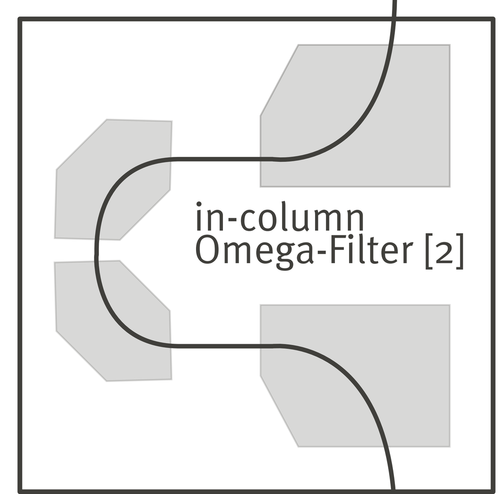


Figure 3: SR-EELS data, corrected by using the results of a characterization measurement. The horizontal lines are included to show the excellent result of the correction.

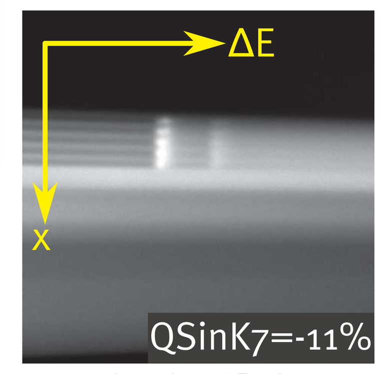
SR-EELS schematic

- Rotate the specimen to match the orientation of the imaging filter.
- Choose between a round aperture and a slit aperture to select the region of interest.





- By default there is only a small lateral dispersion in SR-EELS mode.
- The parameter QSinK7 increases the lateral dispersion (see figure 6).



Two out of three coordinate axes can be measured with a 2D detector (CCD camera). Using the SR-EELS mode, these two disperse coordinates are ΔE and x.

Mode	disperse coordinates	selective window
ESI	x, y	ΔW, $\Delta\Omega$
SR-EELS	x, ΔE	Δy, $\Delta\Omega$

ΔW: energy window
$\Delta\Omega$: solid angle (aperture)
Δy: width of slit parallel to x

Characterisation

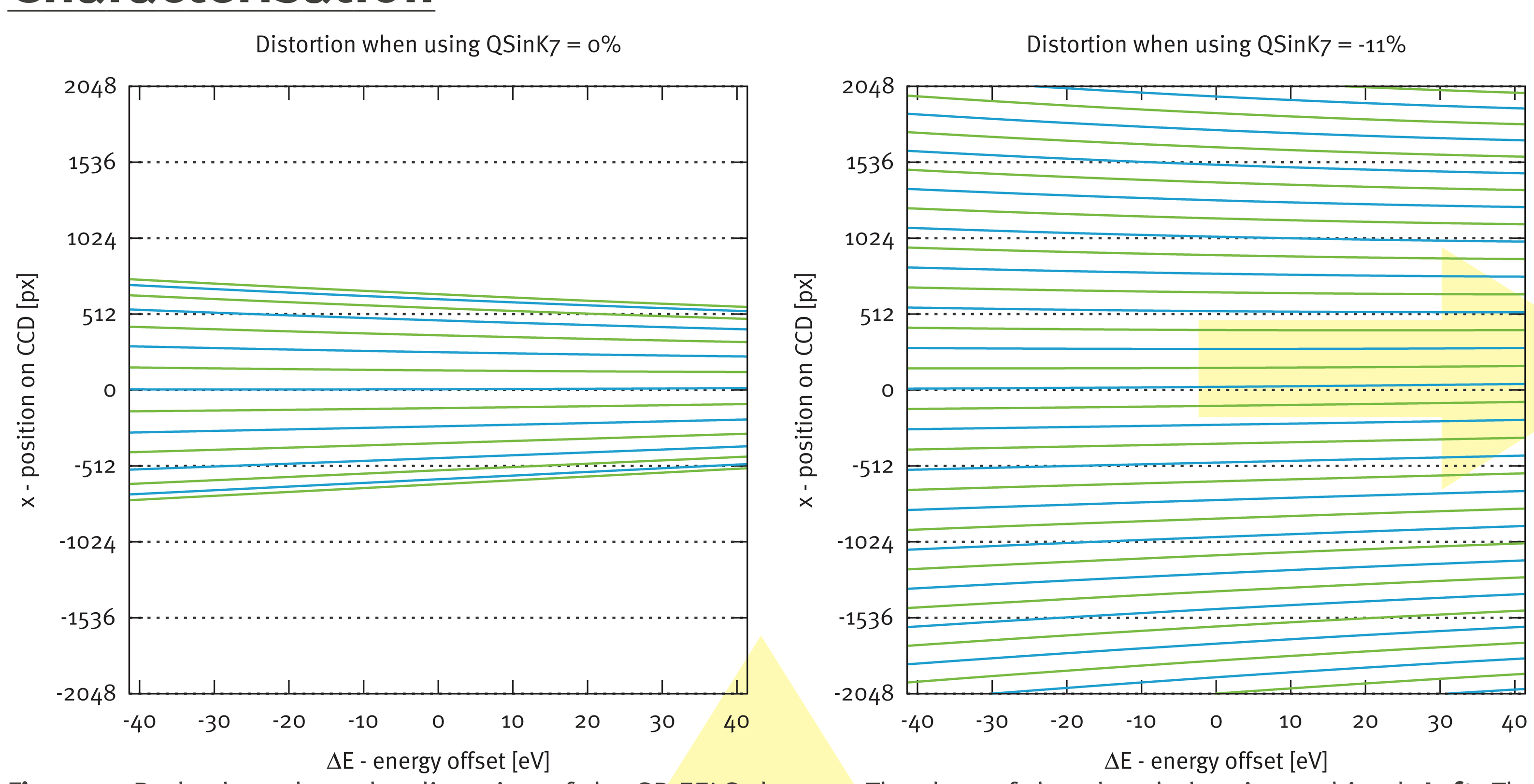


Figure 7: Both plots show the distortion of the SR-EELS dataset. The data of the plots below is combined. **Left:** The default settings of the energy filter are used. The lateral axis is clinched when moving away from the central axis. **Right:** The parameter QSinK7 increases the lateral extend of the SR-EELS dataset. It reduces the distortion. The lines are nearly equal in distance.

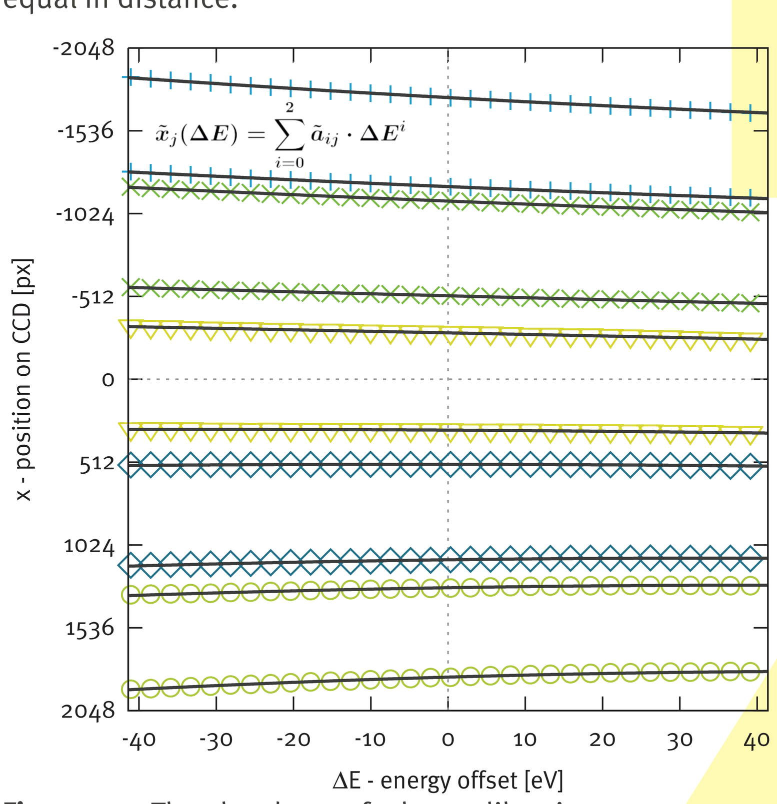


Figure 5: The borders of the calibration spectra are plotted. For each border the polynomial *$x_j(\Delta E)$* is fitted with the parameter *m=2*.

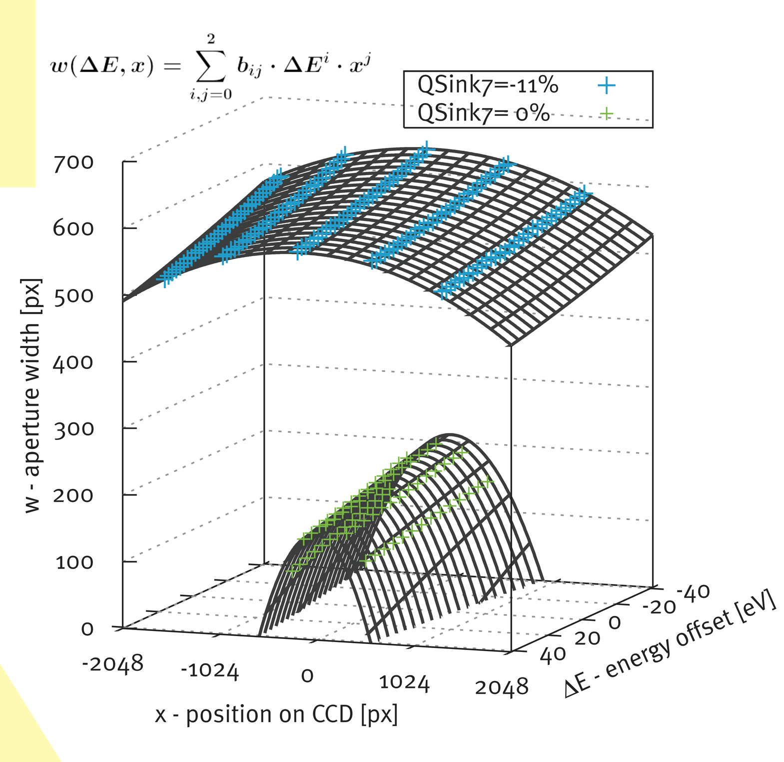


Figure 6: The width of the calibration spectra is plotted against the position of the spectrum centre. The function *w(ΔE,x)* is fitted to all data points.

The **calibration measurements** involve the following steps:

- Shift a small aperture at the filter entrance plane.
- Record a SR-EELS dataset for each position of the aperture.
- Perform the analysis shown at **figure 4**.

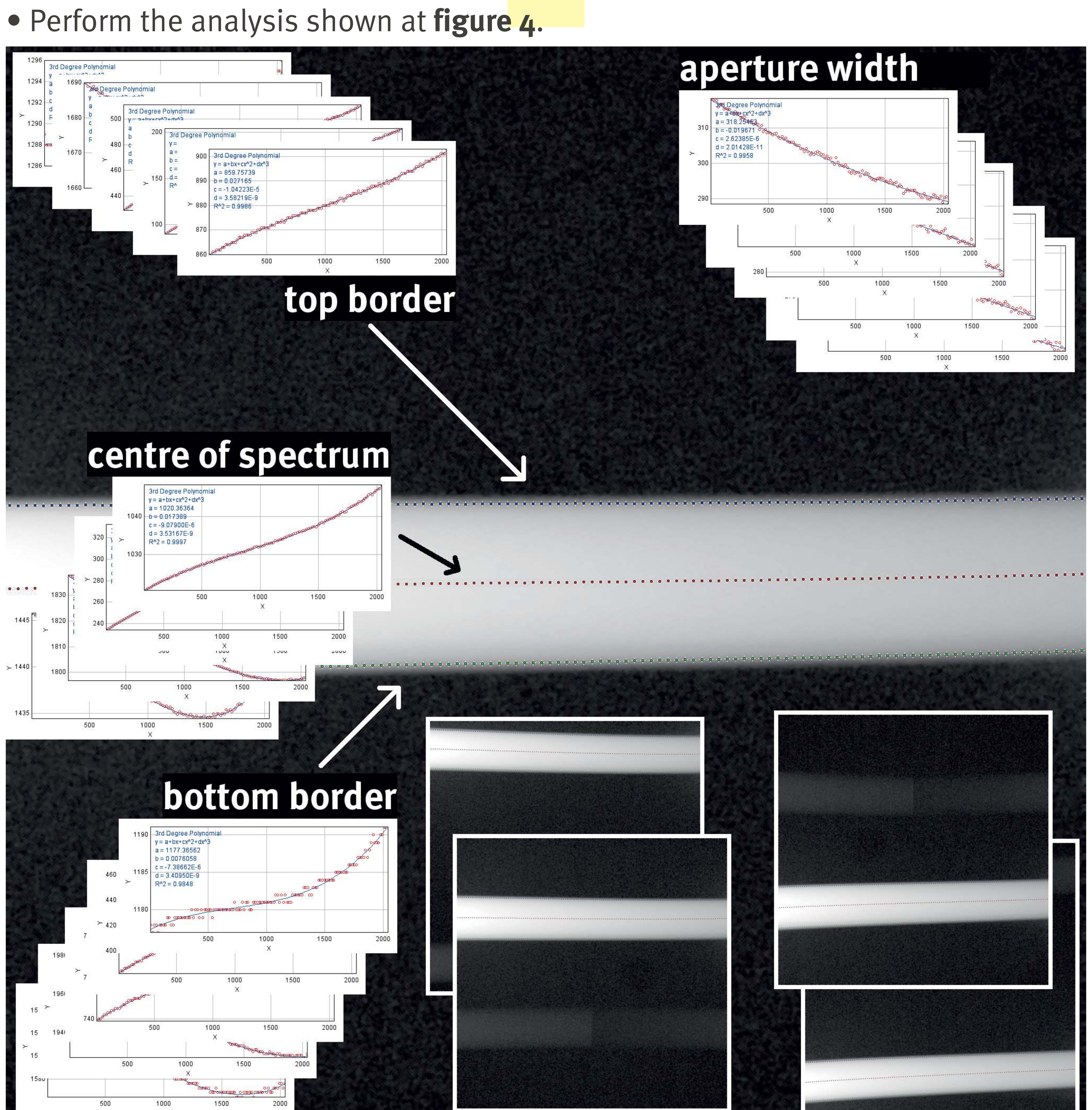


Figure 4: 5 spectra are shown that were recorded for different positions of the aperture at the filter entrance plane. For each spectrum, the shown data is extracted using a macro for ImageJ [3].

Correction

Each spectrum border can be described by:

$$\tilde{x}_j(\Delta E) = \sum_{i=0}^{m} \tilde{a}_{ij} \cdot \Delta E^i.$$

The parameters $\tilde{a}_{ij}$ vary by a polynomial, too. This results in

$$\tilde{x}(\Delta E, x_0) = \sum_{i,j=0}^{m,n} a_{ij} \cdot \Delta E^i \cdot x_0^j, \quad (1)$$

where x_0 is the position of the border at $\Delta E = 0$.
The width of the spectra is expressed by a 3D polynomial.

$$w(\Delta E, x) = \sum_{i,j=0}^{k,l} b_{ij} \cdot \Delta E^i \cdot x^j \quad (2)$$

The SR-EELS correction is a transformation of the curved axes – described by eq. (1) with $m = n = 2$ – to a Cartesian coordinate system, where $\tilde{x}_0(w)$ can be calculated using eq. (2):

$$\Delta\tilde{E}(\Delta E, x) = \frac{1}{\sqrt{a}} \mathrm{arcsinh} \frac{2a \cdot z + b}{\sqrt{4ac - b^2}} \Big|_{z=0}^{z=\Delta E} \quad \text{and} \quad (3)$$

$$\tilde{x}(\Delta E, x) = a_0 + a_1 \cdot \Delta\tilde{E} + a_2 \cdot \Delta\tilde{E}^2 \quad (4)$$

with
$$a = 4a_2^2,$$
$$b = 4a_1 a_2,$$
$$c = a_1^2 + 1,$$
$$a_0 = a_{00} + a_{01} \cdot \tilde{x}_0(w) + a_{02} \cdot \tilde{x}_0^2(w),$$
$$a_1 = a_{10} + a_{11} \cdot \tilde{x}_0(w) + a_{12} \cdot \tilde{x}_0^2(w) \text{ and}$$
$$a_2 = a_{20} + a_{21} \cdot \tilde{x}_0(w) + a_{22} \cdot \tilde{x}_0^2(w).$$

Implementation: `SR_EELS_CorrectionPlugin.java` [4].

Lateral profile of the Fe L_{23}-edge

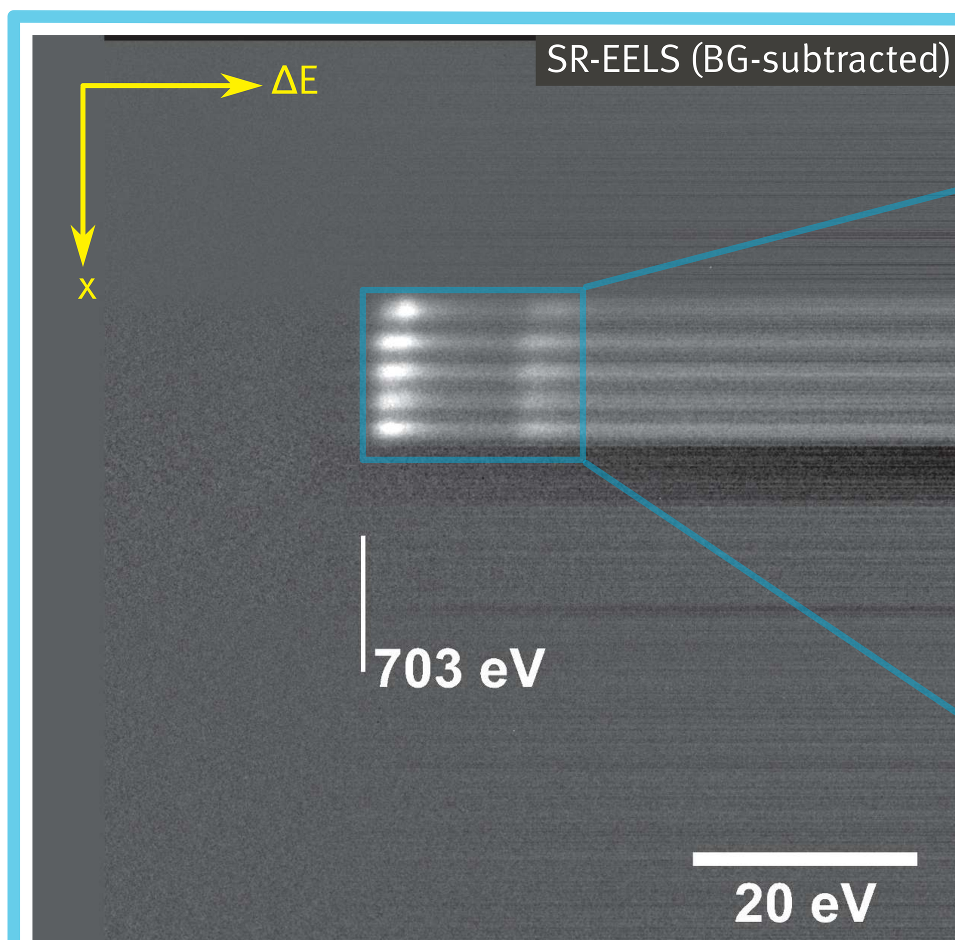


Figure 8: The Cornell Spectrum Imager (CSI) [5] has been used to remove the background and extract the iron signal.

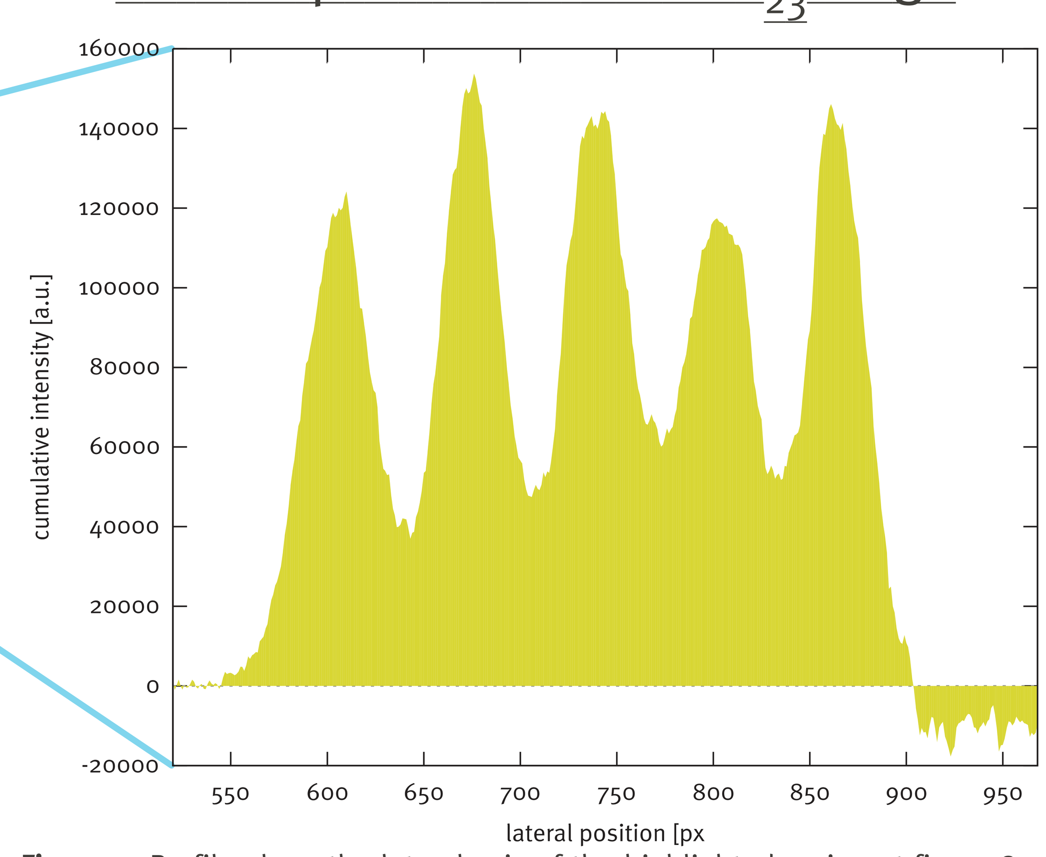


Figure 9: Profile along the lateral axis of the highlighted region at figure 8. All 5 layers show the same width, as expected. A calibration of the lateral axis has not yet been performed.

References

[1] L. Reimer et al.: Ultramicroscopy **24** (1988), 339-354.
[2] S. Lanio: PhD thesis (1986), TH Darmstadt.
[3] The code that has been used to perform the characterisation is available on GitHub:
`https://github.com/EFTEMj/EFTEMj/Scripts+Macros`
[4] The SR-EELS correction is part of the EFTEMj plugin:
`https://github.com/EFTEMj/EFTEMj/blob/master/EFTEMj/src/main/java/sr_eels/SR_EELS_CorrectionPlugin.java`
[5] P. Cueva et al.: Microscopy and Microanalysis **18** (2012), 667-675.