Westfälische
Wilhelms-Universität
Münster

Spatially resolved EELS of FeCr layers using an in-column energy filter

M. Epping and H. Kohl

Physikalisches Institut und Interdisziplinäres Centrum für Elektronenmikroskopie und Mikroanalyse (ICEM), Westfälische Wilhelms-Universität Münster,
Wilhelm-Klemm-Straße 10, 48149 Münster, Germany

http://www.uni-muenster.de/Physik.PI/Kohl/ michael.epping@uni-muenster.de

Workshop on EELS, Uppsala 2012

Introduction

Spatially Resolved EELS (SR-EELS) is a powerful technique to analyze interfaces and layer systems with a high resolution at short acquisition times. In this work we apply SR-EELS in a TEM with a corrected in-column Omega filter to a FeCr layer system. We present two different operation modes and outline the requirements for both of them.

The data from measurements need further processing to obtain quantitative information. At the L-edges of Fe and Cr one needs to extract the background signal to get the net elemental signal. We process the SR-EELS data to generate 1D energy filtered images. Afterward we use a program for Energy Filtered TEM (EFTEM) to calculate 1D elemental maps from this data.

SR-EELS operation modes in detail

There are two operation modes to create spatially resolved spectrum images with an in-column energy filter. Both modes have different requirements.

1. Using the EEL spectrum mode

Requirements:
- a fully corrected energy filter
  (e.g. a corrected Omega filter)

An uncorrected in-column energy filter will result in a caustic at the spectrum image - a curved deformation of the lateral plane generated by the second-order aberration [1]. The hexapole elements of the corrected Omega filter suppress the axial image error [2] and the resulting caustic.

2. Using the image mode

Requirements:
- a slit aperture at the filter entrance plane
- control over the second projector system

The slit has to be orientated perpendicular to the energy dispersive plane. At each point of the slit an EEL spectrum is obtained at the energy dispersive plane. By defocussing the achromatic image plane one can tune the energy dispersion and observe the resulting SR-EELS image on the fluorescent screen or record it with a camera [1,3].

Our Zeiss Libra 200FE is equipped with a corrected in-column Omega filter that allows us to record SR-EELS images at the EEL spectrum mode. Additionally the microscope has a slit aperture at the FEP but by default it is not possible to control the second projector system manually. Without free lens control we cannot use the image mode to do SR-EELS.

Correction of the geometrical aberration

The Omega filter of the Zeiss Libra 200 FE is fully corrected but there is, however, still a distortion. With increasing energy loss the spatial extension of the spectrum image decreases. This distortion results from a geometrical aberration and is corrected by processing the recorded SR-EELS image. Fig. 1 shows an unprocessed SR-EELS image.

We implemented a plug-in for the open source image processing program ImageJ to correct the geometrical aberration. The correction is done in two steps. The first step is to detect the border of the spectrum. In the second step each energy channel is fitted to the same spatial extension.

A linear fit is not adequate to describe the borders of the spectrum. A more complex approach has to be used. Therefore Fig. 2 shows a flow chart of the border detection algorithm. A median filter is used to remove image noise. Only one image direction is of interest, that's why a derivation in x-direction is necessary as an edge detection filter. The steepest edge might not be the border of the spectrum, especially when a layer system is analyzed. To solve this problem the algorithm picks the N steepest edges and finally marks the left most one and the right most one as border of the spectrum. This procedure is repeated at every energy channel (line) of the SR-EELS image. An image of the borders is created from this data. The border image can be displayed as an overlay at the SR-EELS image to evaluate the precision of the border detection. At the left side of Fig. 3 such an overlay is shown.

The optimal value for the number of considered maxima N depends heavily on the structure of the processed image. The program can calculate the spectrum border for different values of N and automatically selects the value that yields to the best result.

A diagram of the spectrum correction algorithm is shown in Fig. 3. All lines of the recorded spectrum image are scaled to the same width. Only the part of the image between the detected borders is considered. The overall intensities at each line has to be preserved.

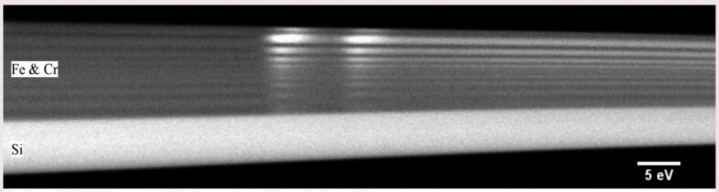

Fig. 1. SR-EELS image of a FeCr layer system with varying layer thicknesses..
The decrease of the spatial extension results from a geometrical aberration.

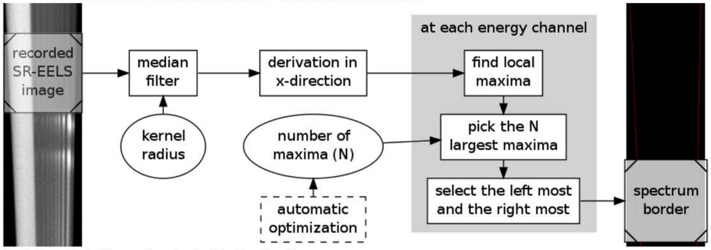
recorded SR-EELS image
median filter
kernel radius
derivation in x-direction
number of maxima (N)
automatic optimization
at each energy channel
find local maxima
pick the N largest maxima
select the left most and the right most
spectrum border

Fig. 2. Flow chart of the spectrum border detection algorithm.

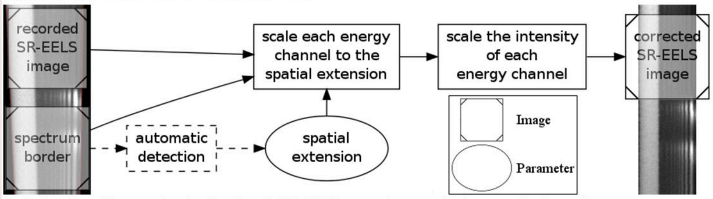
recorded SR-EELS image
spectrum border
automatic detection
scale each energy channel to the spatial extension
spatial extension
scale the intensity of each energy channel
corrected SR-EELS image
Image
Parameter

Fig. 3. Flow chart of the correction algorithm.Recorded SR-EELS image and spectrum border are combined at one image.

One dimensional elemental maps

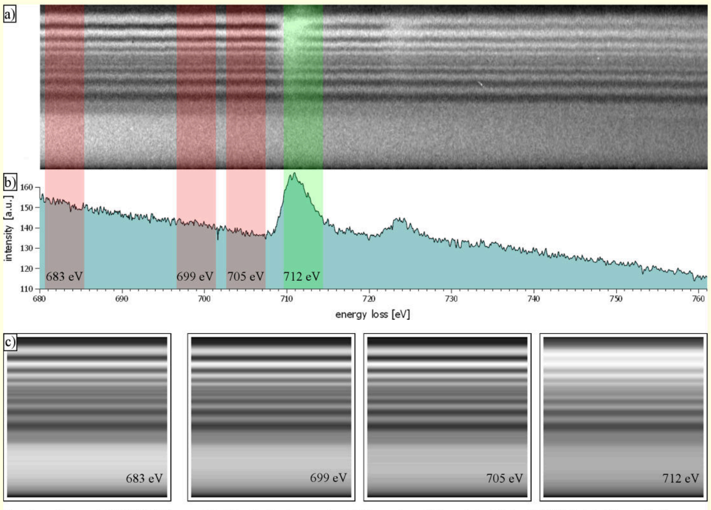

Fig. 4. A corrected SR-EELS image (a) of the Fe L-edges and an EEL spectrum (b) created with the SR-EELS data.The marked energy loss intervals were used to create one dimensional images (c). The images are expanded to quadratic shape.

Two effects contribute to the recorded signal in the SR-EELS images of the FeCr layers. First there is the signal from the L-edges of either Cr or Fe. Additionally there is a background signal. At the Fe L-edges (starting at 708 eV) a significant background signal of the Cr L-edges (starting at 575 eV) is present. Fig. 4a shows a SR-EELS image of the Fe L-edges and mainly the Cr signal is visible. To extract the Fe signal we use a program that calculates elemental maps using the 4 window method - 3 pre-edge windows and 1 post-edge window [4]. This method has been used to analyze the same specimen with Energy Filtered TEM (EFTEM) in previous studies [5]. We chose 4 energy loss intervals of 5 eV width (red and green areas at Fig. 4) and summed all energy channels at each interval. The resulting one dimensional images are shown at Fig. 4c. The x-direction is expanded to obtain a quadratic image - the used program requires quadratic images.

Fig. 5 shows the resulting one dimensional elemental maps for Fe and Cr. The images are rotated by 90° compared to Fig. 4 to allow a better comparison between Cr and Fe. The Fe layers are clearly recognizable at the 719 eV elemental map. This result demonstrates that elemental mapping routines can be used to analyze SR-EELS images. Due to problems with our microscope we were not able to obtain measurements at optimal conditions. To evaluate the resolution limit of this method further measurements are necessary.

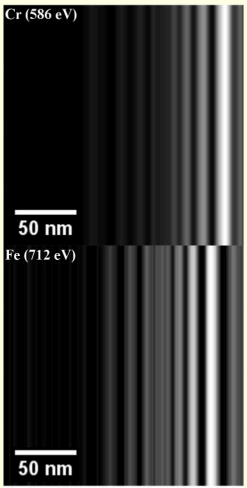

Fig. 5. Elemental maps of cromium (top) and iron (bottom).

Conclusion & Outlook

To acquire SR-EELS images we used the EEL spectrum operation mode. It is easier to use than the image mode, but a fully corrected energy filter is necessary. The geometrical aberration that is affecting the SR-EELS images can be corrected by processing the recorded images. We presented a method for automatic correction. Some optimizations are still necessary to improve the quality of the results.

By using the 4 window method to create one dimensional elemental maps we achieved good results. Unfortunately, the lateral resolution is limited by the poor quality of the recorded SR-EELS images, the 2 nm layers can not be resolved. Further measurements are necessary to evaluate the limits of the method used.

To describe the influence of instrumental parameters additional simulations of elemental maps [6] are necessary. A comparison to Energy Filtered TEM will be used to evaluate the results of SR-EELS. Furthermore this comparison will help to point out possible advantages of SR-EELS.

References & Acknowledgements

[1] L. Reimer et al., Ultramicroscopy **24** (1988) 339-354.
[2] S. Lanio, PhD thesis (1986), TH Darmstadt
[3] L. Reimer et al., Ultramicroscopy **46** (1992) 335-347.
[4] T. Heil, et al., Ultramicroscopy (2012), http://dx.doi.org/10.1016/j.ultramic.2012.04.009
[5] T. Heil & H. Kohl, Ultramicroscopy **110** (2010) 748-753.
[6] R. Knippelmeyer & H. Kohl, Journal of Microscopy **194** (1999) 30-41