

Cathodoluminescence and energy dispersive X-ray spectral data sets – Processing the data

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Whole spectrum mapping captures all the data using the available detectors at each successive pixel. However, while no assumptions are made about the elements present one has to choose accelerating voltage, beam current, energy step size, beam resolution, step size and acquisition time per pixel. When collecting whole cathodoluminescence spectra additional variable choices are made for spectrometer resolution and sensitivity range. Any number of these variables can lead to significant changes in the quality and applicability of the data. One difficulty with whole spectrum mapping is extracting elemental or spectroscopic relationships from the dataset. In particular, finding multi-dimensional relationships and resolving them from edge effects and solid solutions. The key advantage to whole spectra data is that it enables the user to review and process the data off-line[1,2], and that post processing software can improve detection of minor phases or minor element levels.

Examining all the discrete channel images is often the first pass of data inspection. If energy dispersive x-ray data is collected, then the cumulative sum spectra can aid in identification of major elements and phases. A RGB image is a powerful way to overlay maps to see correlations. By selecting the two major components, from the sum spectra, and assigning the third component to a rolling selection of other discrete channel images one can reveal subtle correlations in the data. One important mechanism for showing correlations is to have a dynamically calculated auto-scaling which removes outliers when setting the colour palette. A similar strategy can be employed for checking cathodoluminescence major associations. Combining cathodoluminescence major channels with elemental associations can reveal minor elements or structural changes in phases. The nature of these phases can be identified by selecting an area in a phase of interest and summing the spectra. If the energy dispersive spectra are available then the summed spectra can be processed through to quantification using modern matrix corrections. Significant improvements in the counting statistics of the phase determination can be made if scatter plots are used to select all pixels within phases. This strategy attempts to assign all points in a cluster to a phase and is simple for well defined clusters but difficult for minor clusters or ones not easily statistically separated. Auto-clustering techniques have a clear advantage in assigning all pixels to clusters[3]. An alternative strategy is principal component analysis which finds the underlying relationships between frequencies or energies[4].

Another helpful visualisation procedure for spectral data involves selecting a line across the mapped area. The width of the line can be arbitrarily set and is used to average data in the “width” direction removing minor statistical fluctuations. The line of “width” averaged intensity versus distance is represented as a profile in Figure 1. Intensities of single or multiple channels within the data can be shown along this line. An additional step is to produce a plot of energy versus distance with intensity displayed by a colour palette, as shown in Figure 2. This overview of all the spectral data reveals key spectral signatures within the data. By changing the thresholds within the intensity plot subtle features can be inspected revealing minor components, both of which may have been overlooked if only XY maps at various energy or wavelength slices were inspected.

References

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 [3] N. C. Wilson, et al., *The 16th Australian conference on Electron Microscopy, Canberra, (2000)* 46
 [4] P. Kotula et al., *Microsc. Microanal.* (2003) 9, 1

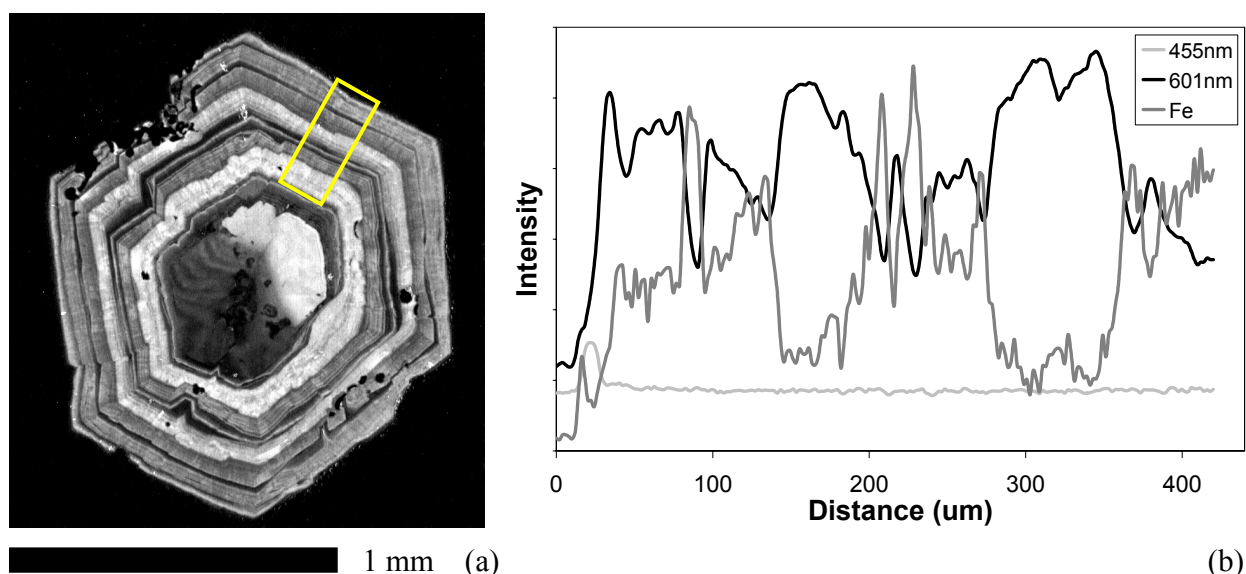


Figure 1. (a) Cathodoluminescence map of an Apatite grain at 455 nm. (b) Profile starting from outer edge to inner part of grain, averaging in the direction parallel to growth zones.

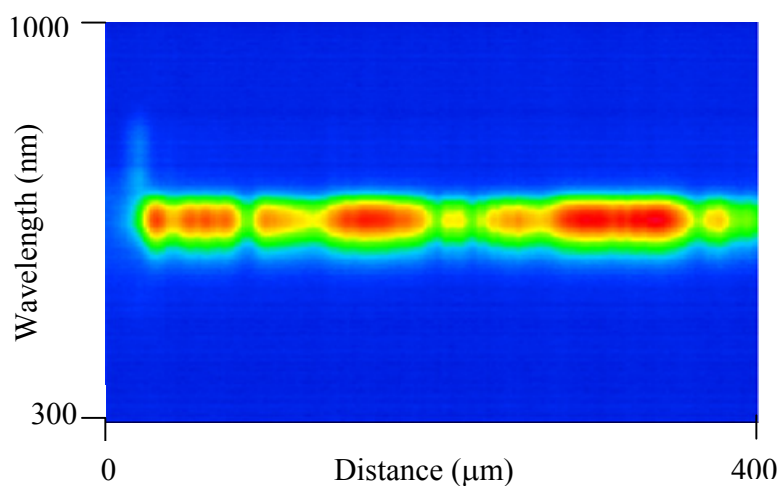


Figure 2. Cathodoluminescent wavelength versus distance intensity plot extracted from the line profile in fig. 1a.