Resolving X-ray peak overlaps for elemental analysis with ChemiSEM Technology

Introduction

Energy-dispersive X-ray spectroscopy (EDS) is a powerful analytical technique widely used for the elemental characterization of materials. Characteristic X-rays are emitted by different elements in the material of interest when bombarded with a high-energy electron beam. By detecting and analyzing those X-rays, EDS provides valuable information about the material composition and distribution of the elements within the analyzed area.

The correct identification of EDS peaks is the first and most crucial step in the analysis of any sample. If this qualitative analysis is incorrect, all subsequent quantitative analyses will be impacted, distorting point analyses, line scans, and X-ray maps. Samples that contain multiple different elements can be particularly challenging, especially when there are overlaps between these peaks. It can be difficult for some EDS systems to correctly assign these peaks and separate the overlaps, leading to incorrect results. With Thermo Scientific™ ChemiSEM™ Technology, the process of assigning peaks and resolving overlaps is completely automated and reliable, so you can be confident in your results. All the steps of background subtraction, peak fitting, and separation are run live and without user interaction. With synthetic spectrum and background visualization tools, you can also compare measured data with your expected results.

Peak overlaps and deconvolution

Overlaps occur when the separation of the characteristic X-ray peaks is less than the resolving power of the EDS spectrometer. The process of separating these peaks is called deconvolution, and is necessary in order to accurately determine the contributions of each individual element to the overall peak profile.

Peak deconvolution iteratively refines the fit by considering the reference peak shapes, the energy resolution, and any background contributions. Accurate peak deconvolution is a prerequisite for reliable quantitative and qualitative analysis and improves the detection of trace elements, especially in the presence of peak overlaps.

Peak deconvolution with ChemiSEM Technology

ChemiSEM Technology uses iterative, automatic peak identification to resolve overlapping peaks through an adaptive digital filtering process that performs background removal and linear least-squares fitting. The individual peak contributions are separated, producing pure net counts that are further processed with matrix correction routines, which account for absorption, fluorescence, and atomic-number variations. Throughout the data collection process, results are continually modeled and refined using a range of statistical validation routines, providing you complete confidence in the analysis.

Additionally, peak assignment and fitting can be continuously checked through the use of spectrum simulation. This automatically validates elemental identification and quantification based on exact microscope settings. Background and synthetic spectra take into account the instrument's accelerating voltage, working distance, and beam current. Spectrum simulation can be performed live or applied to any previously acquired spectrum, validating all quantitative analysis.

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Application examples

Tantalum capacitor

A cross section through a tantalum capacitor was inspected for quality control purposes, showing both the backscattered electrons (BSE) image and ChemiSEM quantitative image (Figure 1). The ChemiSEM image highlights the complex chemistry of the sample, composed of multiple elements.
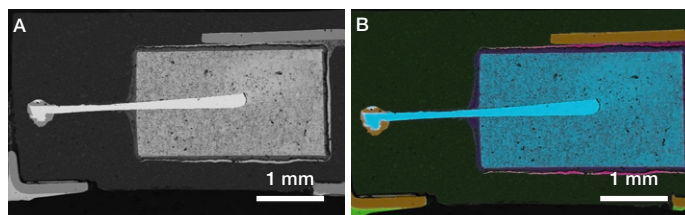

Figure 1. Backscattered electron image of a tantalum capacitor cross section (A) with a ChemiSEM image of the same area of interest (B).

The ChemiSEM live chemical image shows quantitative data, with the intensity of each color related to the concentration of the assigned element. In this sample, there were two peak overlaps; one between copper and tantalum (at ~8 keV) and the other between silicon and tantalum (at ~1.7 keV) as shown in Figure 2.
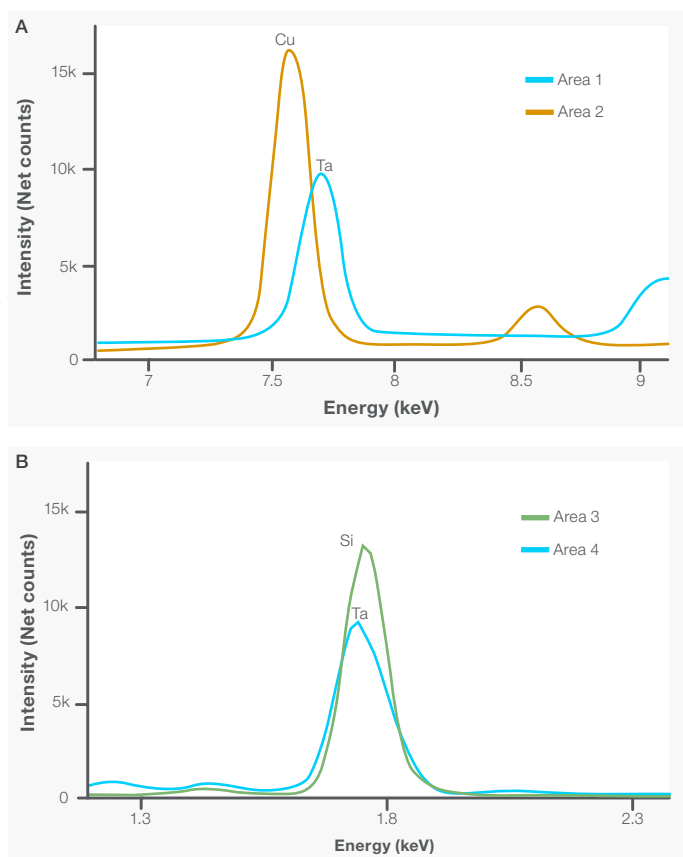

Figure 2. A) Copper and tantalum peak overlaps; copper has a peak at 8.046 keV while tantalum has a peak at 8.145 keV. B) Silicon and tantalum peak overlaps; silicon has a peak at 1.74 keV and tantalum has a peak at 1.71 keV.

Traditional EDS count maps show the absolute number of counts within each region of interest. However, this approach can lead to incorrect results, owing to peak overlaps and background noise (Figure 3).
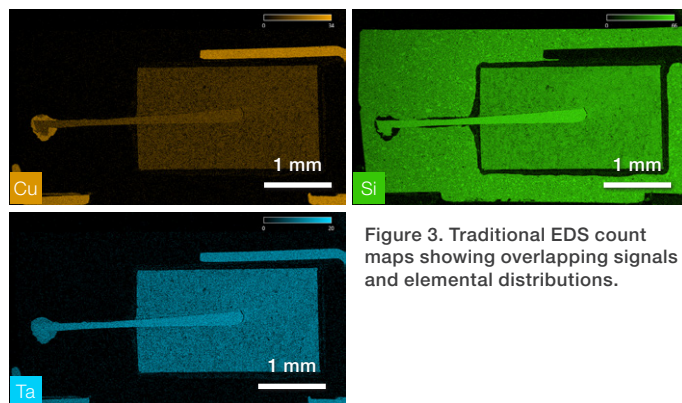

Figure 3. Traditional EDS count maps showing overlapping signals and elemental distributions.

Here, the count maps are producing misleading results due to the previously mentioned elemental overlaps. ChemiSEM live quantitative maps solve this problem, providing a full peak deconvolution for every pixel. The data processing is designed to provide artifact-free quantitative maps.

As seen in Figure 4, the localization of the copper electrode, cathode, and anode is now clearly visible, with spurious background signals eliminated. The tantalum is shown to be localized, and erroneous silicon overlaps are removed.
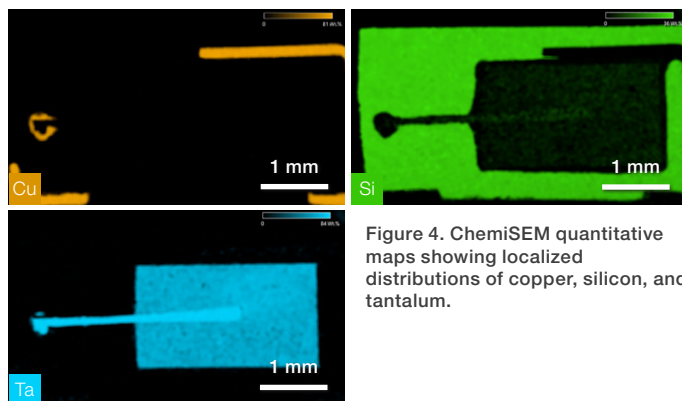

Figure 4. ChemiSEM quantitative maps showing localized distributions of copper, silicon, and tantalum.

Characterization of geological sections

Geological sections are often complex, presenting significant challenges for EDS analysis. Overlapping peaks, in particular, are a common problem, making reliable peak deconvolution necessary for the collection of meaningful information. The following examples show two sectional analyses. The first sample features several different minerals including barium sulfide (BaS) and ilmenite ($FeTiO_3$).
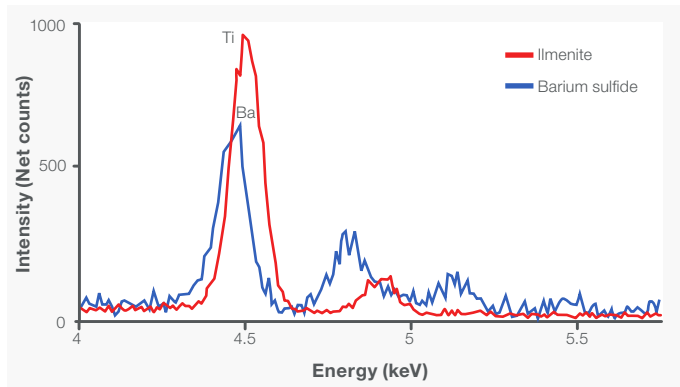

Figure 5. Barium and titanium peaks with overlap at ~4.5 keV.

The barium Lα line (at 4.465 keV) overlaps with the titanium Kα line (at 4.508 keV). Their gross count maps are generated using this overlapped peak, resulting in near identical elemental distributions that could easily be misinterpreted, leading to incorrect conclusions regarding material composition. With ChemiSEM Technology, barium and titanium peaks are correctly deconvolved, providing increased certainty in the analysis.
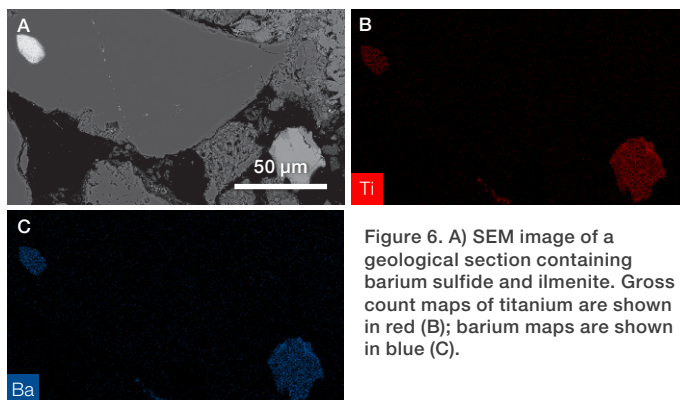

Figure 6. A) SEM image of a geological section containing barium sulfide and ilmenite. Gross count maps of titanium are shown in red (B); barium maps are shown in blue (C).
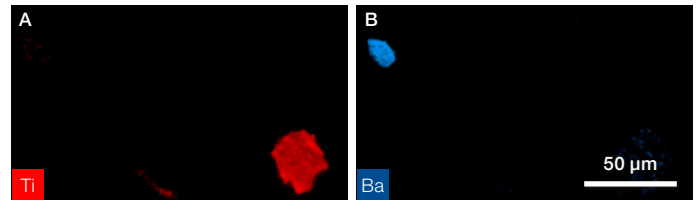

Figure 7. ChemiSEM maps of titanium (A) and barium (B).

Another geological section is shown in Figure 8, featuring large monazite (($Ce,La,Th)PO_4$) and zircon ($ZrSiO_4$) inclusions.
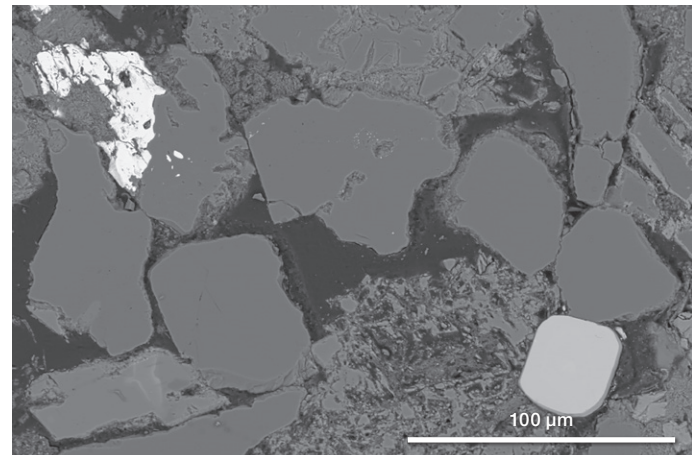

Figure 8. SEM image of a geological section, showing a monazite (brighter feature in the top left area of the image) and a zircon (brighter squared feature in the bottom right area of the image).
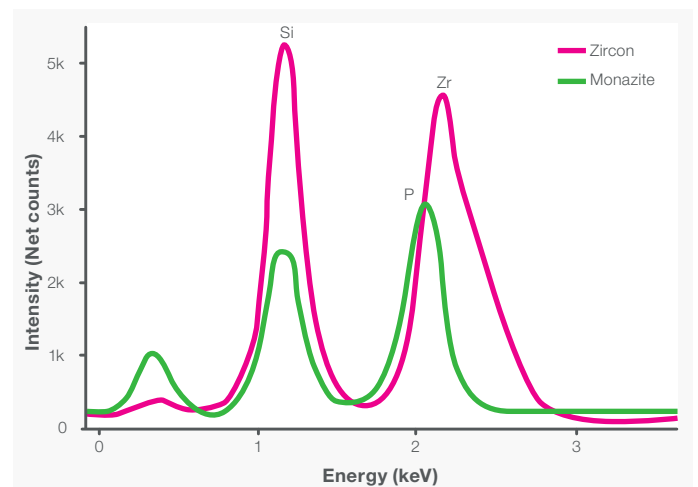

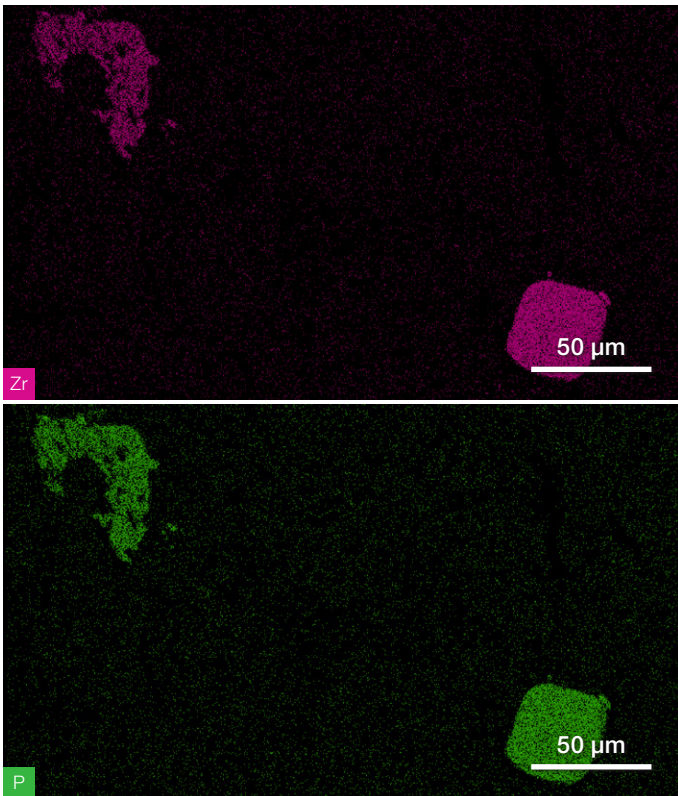Figure 9. Phosphorous and zirconium peaks with overlap at 2.013 keV and 2.042 keV, respectively.

Figure 10. Zirconium and phosphorous gross count maps for the geological section in Figure 8.
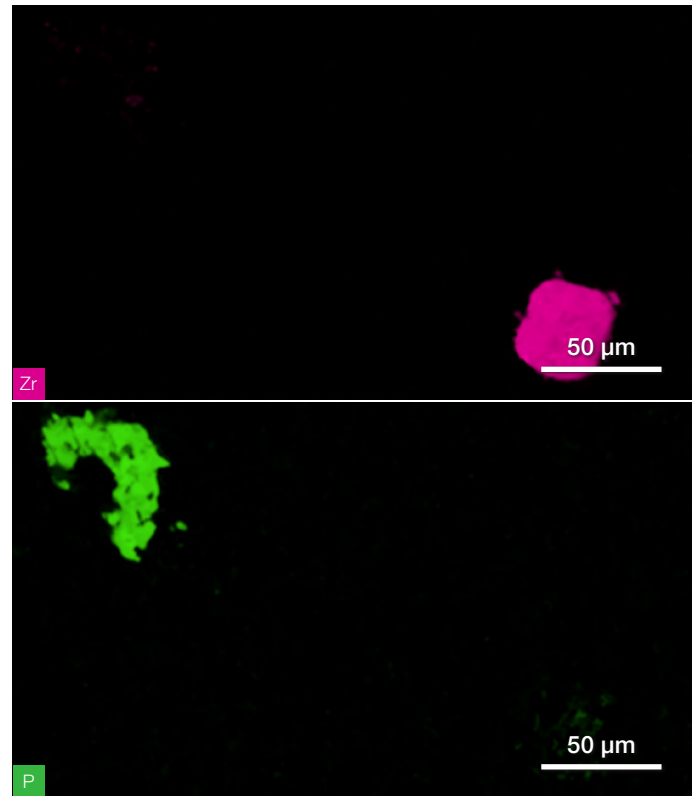

Figure 11. ChemiSEM maps of zirconium and phosphorous in a geological section.

The Kα line of phosphorous (at 2.013 keV) overlaps with the Lα line of zirconium (at 2.042 keV), resulting in identical gross count maps with traditional EDS analysis (Figure 10).

Using the ChemiSEM peak deconvolution algorithm, the signal is correctly deconvolved and assigned to, respectively, zirconium and phosphorous (Figure 11).

Conclusions

The presence of overlapping peaks represents a significant challenge for EDS analysis. Correct peak identification and deconvolution is an essential prerequisite for reliable, meaningful results. ChemiSEM Technology incorporates robust, automated deconvolution algorithms that can assign the right elements regardless of SEM operating conditions, bringing you peace of mind with accurate elemental analysis.

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