Application Note: 51188

Resolving Overlapping Peak Problems with NORAN System 7 Spectral Imaging Software

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Key Words

- COMPASS
- EDS
- Low Voltage
- NORAN System 7
- Overlapping Peaks
- Quantitative Mapping
- X-ray Mapping
- X-ray Spatial Resolution

Introduction

Energy Dispersive X-ray Spectroscopy (EDS) has traditionally had a difficult time analyzing severe peak overlaps. Among these are the combination of the Si-K and Ta-M lines, and the combinations of O-K and Cr-L lines. These overlaps can be a significant problem in many EDS analyses, from correct peak identification to elemental quantification to elemental mapping. Furthermore, these combinations appear frequently in the electronic industry.

This application note compares various EDS methods of analyzing materials where peak overlaps are problematic, beginning with traditional peak count X-ray mapping, and then moving to more advanced methods available on the Thermo Scientific NORAN System 7 X-ray microanalysis system.

TEM Microanalysis with Typical Overlapping Peak Problems

Figure 1 shows an electron image collected using Spectral Imaging software of a semiconductor material containing layers of chromium, tantalum oxide, and silicon. The data for this analysis was collected on a Topcon EM-002BF transmission electron microscope (TEM) at 200 kV accelerating voltage. The analysis magnification is 300 kX.

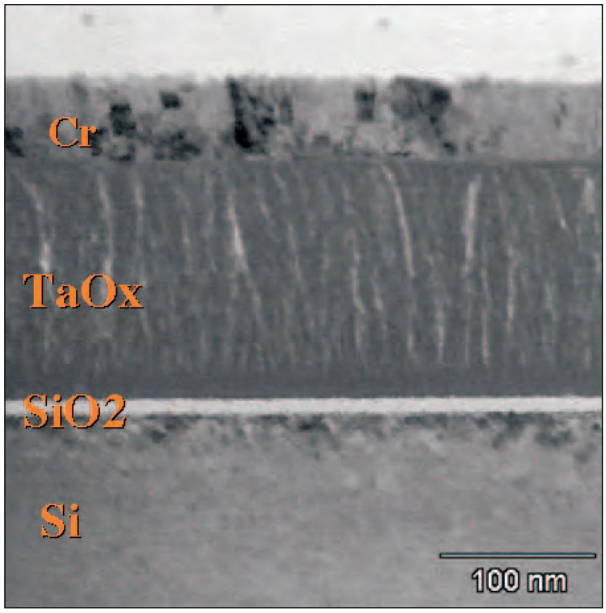

Figure 1: Electron image of multi-layer semiconductor material (Cr/TaO/Si substrate)

The spectra in Figure 2 show the peaks overlaps for the O-K line and Cr-L line near 0.5 keV, and the Si-K line and Ta-M line near 1.7 keV.

The X-ray peak count maps in Figure 3 show the simple summation of X-rays in a given energy range displayed for each pixel in the Spectral Imaging dataset. However, these counts are not only affected by the local composition, but are also perturbed by the local thickness and background counts. Here we see evidence of silicon and oxygen in the tantalum region, and evidence of oxygen in the chromium region, which we believe to be incorrect or misleading.

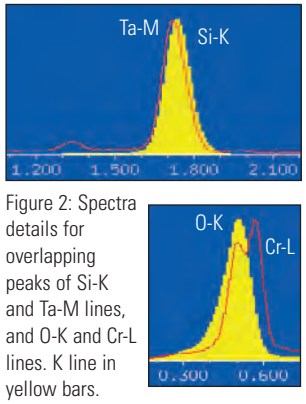

Figure 2: Spectra details for overlapping peaks of Si-K and Ta-M lines, and O-K and Cr-L lines. K line in yellow bars.

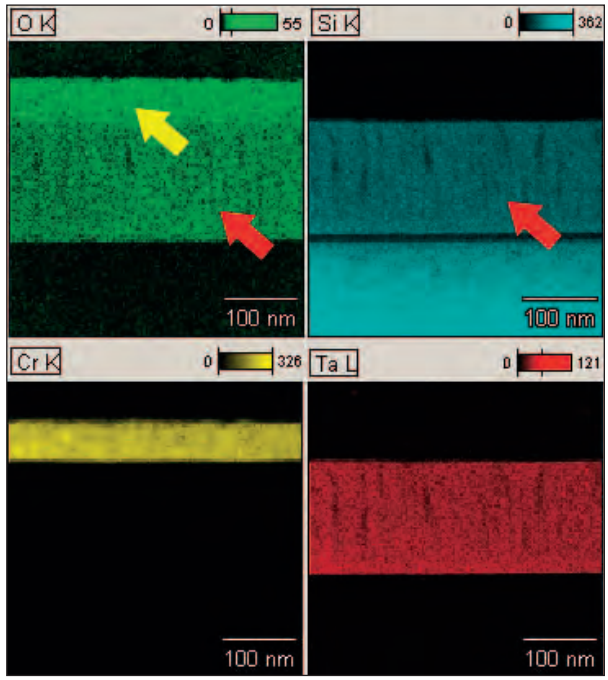

Figure 3: X-ray peak count maps showing the effects of overlapping peaks, with Si and O artifacts appearing in the Ta region (red arrows), and O appearing in the Cr region (yellow arrow)

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The quantitative map function of the NSS software removes the background and separates the individual peak contributions to produce pure net counts which are further matrix processed to provide composition results. The quantified maps (Figure 4) clearly show the correct locations of the oxygen, chromium, tantalum, and silicon. Silicon is not evident in the tantalum region, and oxygen is not shown in the chromium map but is localized in a previous undefined area of silicon dioxide.

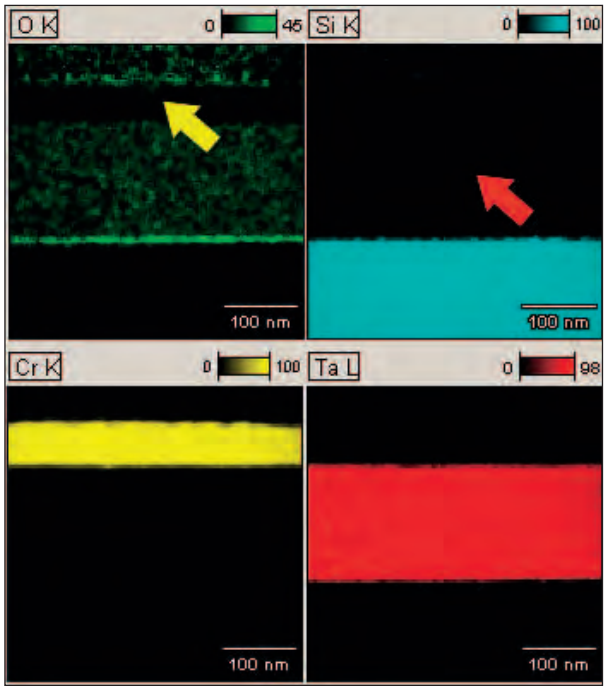

Figure 4: Quantitative X-ray maps from the same data set. Note the absence of Si in the Ta region (red arrow), and the absence of O in the Cr region (yellow arrow)

Low Voltage, High Magnification Analysis of the Same Material

Using peak deconvolution, low energy lines can be used for the analysis, such as Cr-L, and Ta-M. With Ta-Ma X-rays having an energy of 1.710 keV, we can use a beam voltage of 5 kV or less and still excite the required X-ray lines. The advantage of this operating condition is the reduction of the X-ray spatial resolution (Reed 1966), which is shown in Figure 5. Modern Field Emission Scanning Electron Microscopes (FESEM) are designed to provide superior resolution and adequate beam current under these conditions.

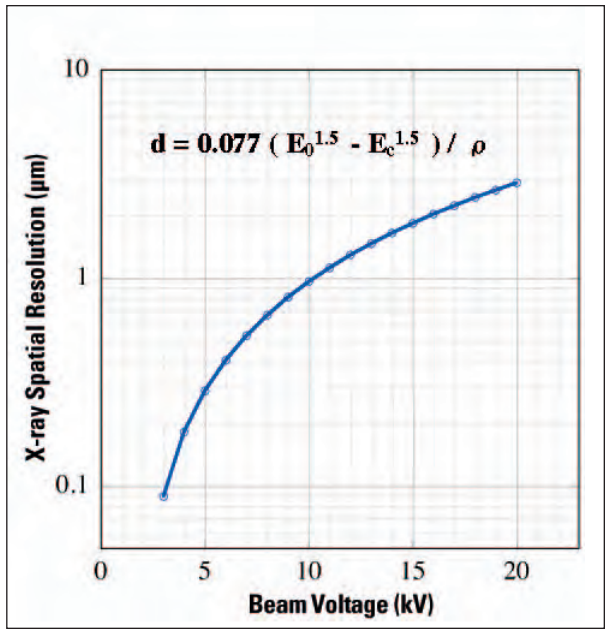

Figure 5: Curve showing the relationship between accelerating voltage and X-ray spatial resolution for a bulk sample. S.J.B. Reed, in *X-ray Optics and Microanalysis, 4th International Congress on X-ray Optics and Microanalysis*, eds. R. Castaing, P. Deschamps, and J. Philibert, Hermann, Paris, p. 339.

The same sample was also analyzed in an FESEM with an accelerating voltage of 3 kV, and a magnification of 80,000x. The EDS mapping analysis collection time was 60 minutes.

A typical spectrum (Figure 6) shows significant peak overlaps of the expected elements: the O-K and the Cr-L peaks, the Si-K and the Ta-M peaks.

As expected, the X-ray peak count maps of the Si and the Ta and of the O and the Cr appear to be the same, as Figure 7 illustrates.

With the elemental quantitative maps, however, the maps of the O and the Cr, and the Si and the Ta are clearly separated (Figure 8). The quantitative elemental map function of the NSS software removes the influence of the peak overlap and is shown to be very effective for use at low beam voltage and at high magnification on bulk samples.

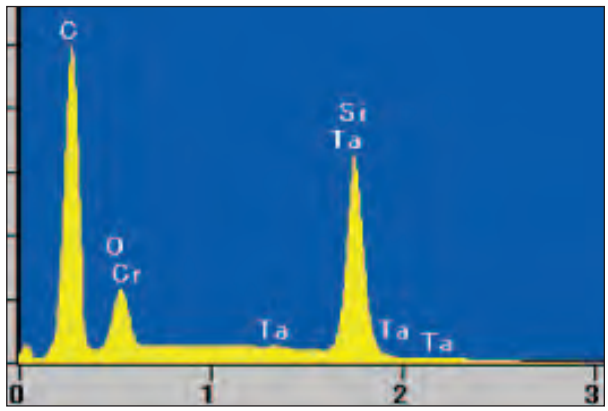

Figure 6: SEM/EDS analysis of multi-layer semi-conductor material show typical peak overlaps for oxygen and chromium, and silicon and tantalum

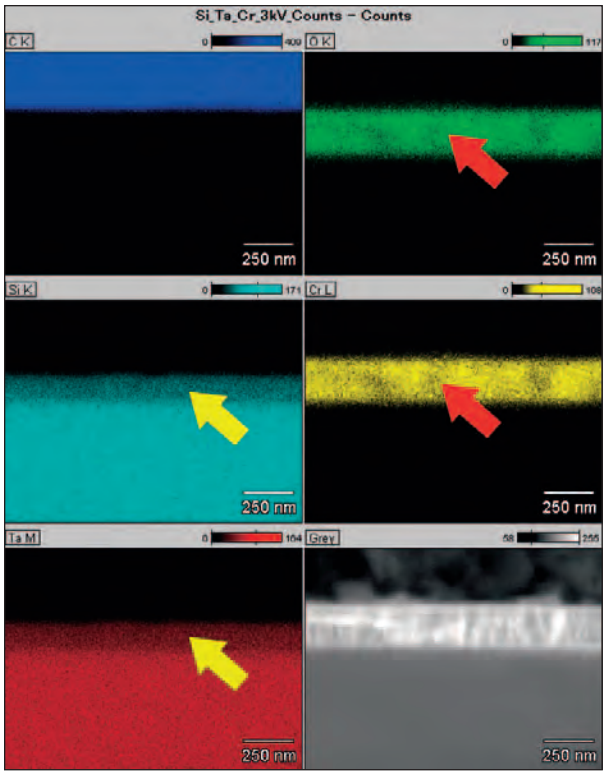

Figure 7: X-ray peak count maps showing inclusion of Si in the Ta region (yellow arrow) and O in the Cr region (red arrow)

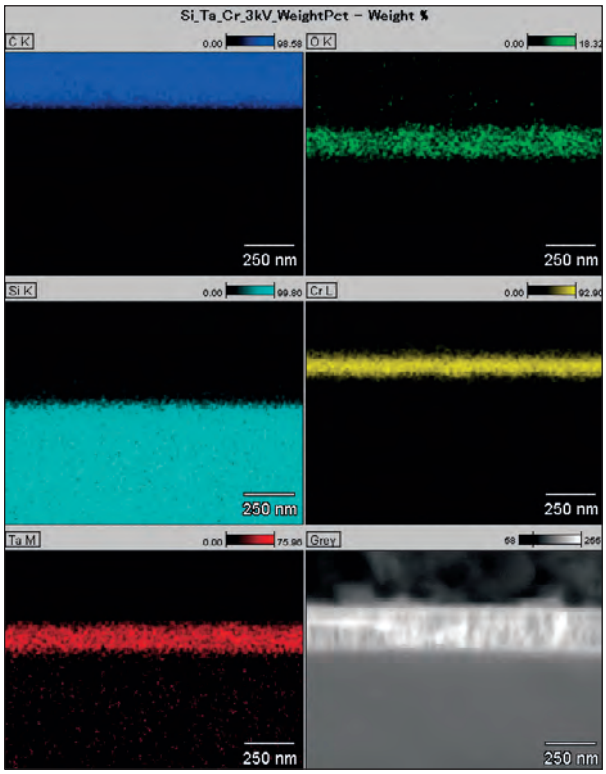

Figure 8: Quantitative maps showing clear separation of O and Cr, and Si and Ta

To understand the X-ray spatial resolution better, a line scan extraction was performed on the spectral imaging data set (Figures 9 and 10). Line profiles for Si-K and Ta-M were plotted across the common boundary. The resolution was measured from the 15% to 85% Ta amplitude locations as being approximately 45 nm. This value illustrates the extremely high X-ray spatial resolution that is possible using a FESEM at low beam voltages.

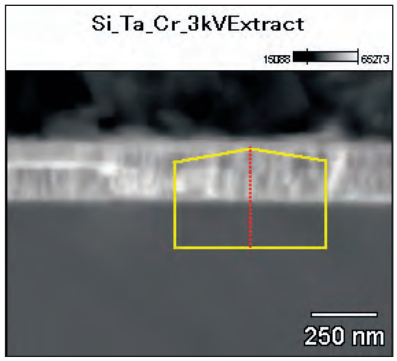

Figure 9: Linescan extraction of Spectral Imaging data

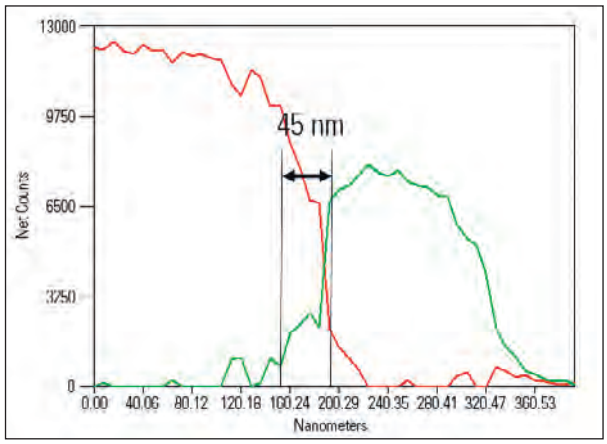

Figure 10: Linescan data showing the separation of the 15% to 85% amplitude locations as being approximately 45 nm an indication of the X-ray spatial resolution

COMPASS Analysis of the Same Material

In addition to extracting quantitative information from NORAN System 7 Spectral Imaging data sets, the analyst can perform a multivariate statistical analysis via the COMPASS software option. With no user inputs, COMPASS analyzes the complete spectral imaging data set looking for similarities. These similarities produce a set of components which are described by a spectra and location map.

COMPASS analysis of this data set (Figures 11 to 13) produced components which describe a silicon, a tantalum oxide, and a chromium region. Note that the signal-to-noise ratio of the images exceeds that of the quantitative maps. These results indicate that COMPASS analysis is effective for data collected at low beam voltage and high magnification.

They also illustrate that COMPASS can both spatially and spectrally deconvolute the data set. Furthermore, COMPASS eliminates analyst assumptions such as the inclusion of tantalum, which could easily have been overlooked due to the presence of silicon. Here COMPASS automatically presents a component map and spectra that locates the TaO_x region with no initial inputs.

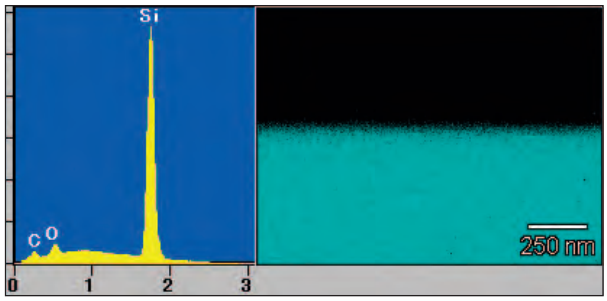

Figure 11: COMPASS-produced component spectrum and map of Si region

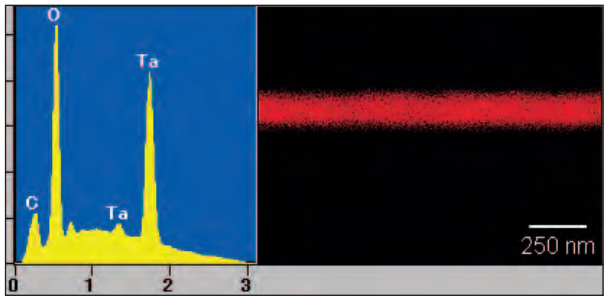

Figure 12: COMPASS-produced component spectrum and map of TaO_x region

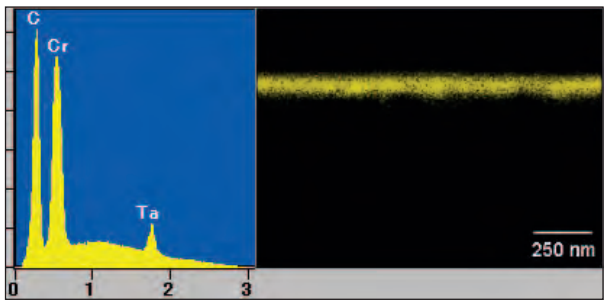

Figure 13: COMPASS-produced component spectrum and map of Cr region

Conclusions

Overlapping peaks can present significant problems in many EDS analyses, including X-ray mapping. Analyzing a multi-layer material that includes some classic overlapping peak combinations, we see how X-ray mapping by peak counts yields misleading results. Quantitative mapping introduces a more robust method of calculating elemental compositions by separating individual peak contributions – even at low beam voltages and high magnification. Our COMPASS multivariate analysis can further improve the analysis by presenting even higher signal-to-noise ratios. The software routine also eliminates user bias in selecting which elements to map.

Acknowledgment

TEM data courtesy of Topcon Corporation. SEM data courtesy of JFE Techno-Research Corporation.

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AN51188_E 03/08M