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Enhanced Compositional Mapping on the SEM Through Combined EDS-WDS Mapping in AZtecWave

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Combining energy dispersive spectrometry (EDS) and wavelength dispersive spectrometry (WDS) on a scanning electron microscope (SEM) provides a powerful and flexible tool for investigating spatial compositional variations in solid samples. EDS is highly sensitive and can quickly generate element maps over large sample areas. With large area silicon drift detectors (SDD) and advanced data processing, EDS X-ray mapping is so rapid that live chemical imaging (the generation of real-time compositional maps as the user moves around the sample) has become a reality [1]. Where EDS mapping reaches its limitations is when there is a need to investigate the spatial distribution of trace elements, as typically the detection limit of EDS is greater than ~1000 ppm (depending on element). Challenges with compositional mapping via EDS also arise when X-ray lines of more than one element are closely spaced, and therefore overlap in the EDS spectrum making them difficult to differentiate. This is where WDS comes in. WDS has a higher spectral resolution in comparison to EDS and consequently, higher peak to background ratios and lower detection limits (<100 ppm for many elements). Therefore, with WDS it is possible to map the distribution of trace elements, as well as produce accurate and representative maps for elements affected by X-ray peak overlaps in EDS.

To demonstrate this, here we present an example EDS-WDS dataset collected from a steel sample containing inclusions with a range of compositions [2]. Some of the inclusions have an internal structure, and compositional variations that can simply be observed through BSE imaging. EDS point analysis on the inclusions reveal that some of the inclusions contain Ce, La, and other rare earth elements (REE). Due to the significant number of overlaps of REE peaks it is difficult to positively identify exactly which REE are present based on the EDS spectrum, especially those present in trace concentrations, and therefore are severely impacted by surrounding major element peaks. Performing a WDS scan on the same inclusion type using the Wave spectrometer and AZtecWave reveals the presence of low concentrations of Pr and Gd (Fig. 1). To investigate the spatial distribution of these elements in the inclusions we have conducted combined EDS-WDS mapping. Using AZtec Tru-Q EDS spectrum processing, element maps are accurately displayed for the major-minor elements (e.g., Mn, S, Ce), and the distribution of Pr and Gd in the inclusions can be detected and observed in the WDS maps (Fig. 2). The corresponding Pr and Gd maps collected by EDS show that these elements are below the EDS detection limit.

Recently we have been developing combined EDS and WDS mapping in our latest software, AZtecWave. This brings the possibility of simultaneously collecting EDS and (multiple) WDS maps in an optimized and time-effective manner (e.g. for the same duration). AZtecWave automatically selects the most appropriate X-ray line and diffracting crystal for the WDS element map(s), and EDS and WDS are run with the same dwell time per pixel. Guided analytical workflows and assistance in choosing suitable magnification for WDS mapping (to avoid beam defocusing), enables analysts of all experience levels to achieve successful mapping results. Whilst most element mapping requirements can be adequately addressed with EDS alone, the ability to easily set up to collect WDS maps for one or more

elements in combination with EDS, expands the analytical capability of the SEM for instances where microanalysis is more challenging (e.g. the requirement for trace element mapping). This enables the distribution of major, minor and trace elements to be determined on a single and flexible instrument. Applications for combined EDS-WDS mapping on the SEM are varied and wide-ranging, and examples from the fields of geology, metallurgy, and ceramics will be presented [2].

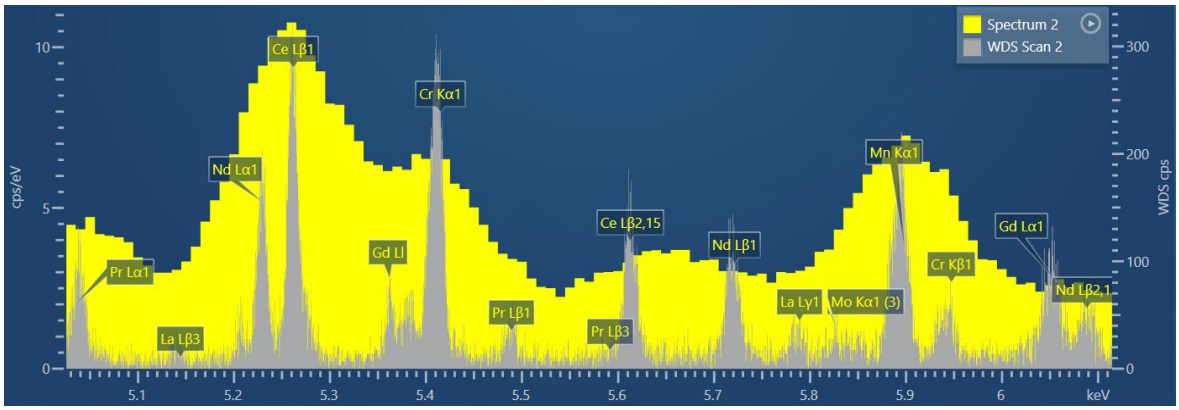

Figure 1. A WDS scan (grey) and concurrent part of the EDS spectrum obtained from a point on the REE-containing inclusion. Due to the higher spectral resolution of WDS it is possible to positively identify Pr and Gd peaks.

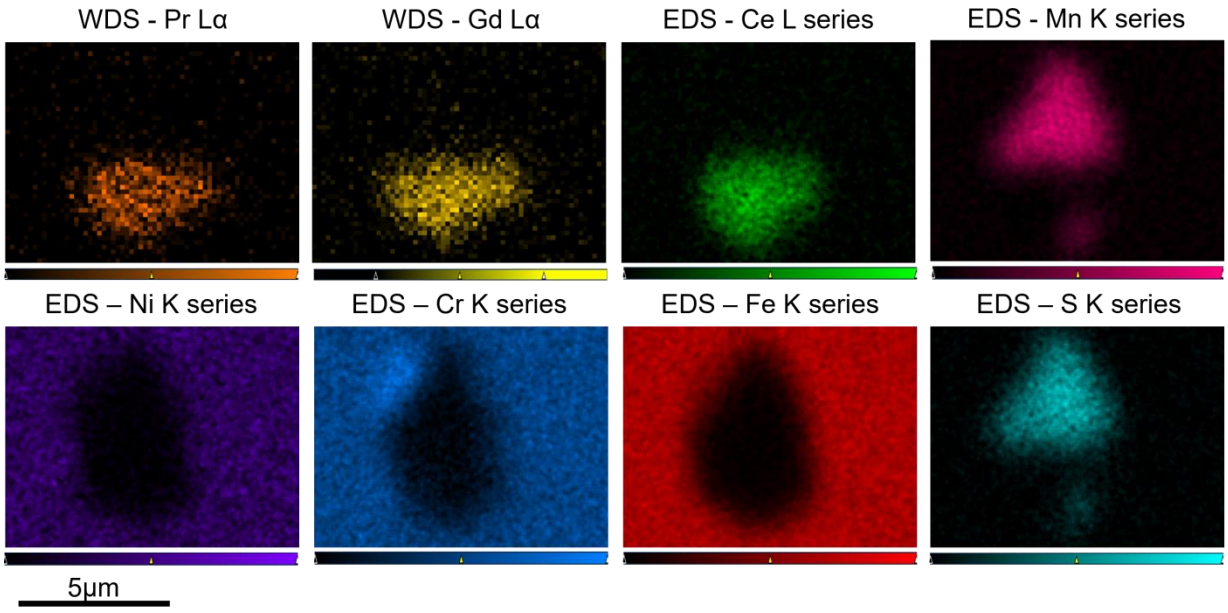

Figure 2. EDS and WDS element maps collected from a steel-hosted inclusion made up of multiple phases. Low concentrations of Pr and Gd were mapped via WDS using the LiF crystal, and each acquired for 100 frames. This data (and Fig. 1) was collected on a JEOL 5900 SEM operating at 20 kV.

References:

[1] S Burgess et al., Microscopy and Microanalysis **27**(S1) (2021), p. 1840. doi:10.1017/S1431927621006723
[2] The authors acknowledge Geoff West (Warwick Manufacturing Group) and John Seifert (Electrical Power Research Institute) for providing the steel sample