Maximizing Speed and Sensitivity: Simultaneous High-throughput EDS-WDS Elemental Mapping in the SEM

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Background incl. aims

High-throughput Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDS) elemental mapping ensures the acquisition of multiple elemental maps simultaneously (hyperspectral mapping) within minimal timeframes. This process requires a high beam current on the SEM side to generate high input of X-ray counts, coupled with fast electronics of the Silicon Drift Detector (SDD) to operate at short pulse processing times, thereby efficiently converting incoming X-rays into output X-ray counts. However, shorter pulse processing times may lead to a low energy cut-off, as the noise level becomes comparable to that of the signal from low energy X-rays, presenting a challenge for high-throughput EDS mapping of light elements. While Wavelength Dispersive X-ray Spectroscopy (WDS) exhibits lower throughput than EDS at equivalent beam currents, its superior sensitivity and higher peak-to-background ratio enable better detection of light elements that may be difficult to observe using EDS alone. Therefore, simultaneous WDS mapping of light elements and high-throughput EDS mapping of higher-Z elements can facilitate rapid and accurate determination of element distribution for both light and higher-Z elements.

Methods

To demonstrate this capability, a boron steel sample was mapped using an EDAX Octane Elite Super EDS detector and an EDAX Lambda Plus WDS detector (Gatan inc.) in a Field Emission SEM. The beam current was set at 75 nA to achieve approximately 250,000 counts per second (cps) on the EDS detector. Capitalizing on advancements in SDD efficiency [1], a pulse processing time of 0.48 µs was used, resulting in an output of approximately 190,000 cps. An 80 Å diffracting crystal combined with polycapillary optics in the WDS detector were utilized to map B K. The region of interest was simultaneously mapped using EDS and WDS for a duration of 2 hours in the EDAX APEX 3.0 software (Gatan inc.). Drift corrections by the software were applied to compensate for sample drift induced by the high beam current.

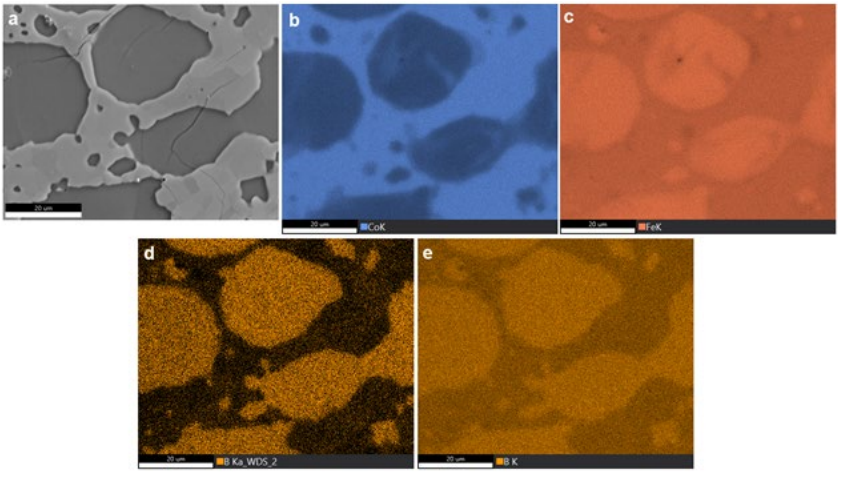
Results

The SEM image (Figure 1a) unveils grains clusters segregated by relatively large melt-like pockets ranging from 5 to 25 µm in size. Orientation contrast highlights individual grains approximately 5-10 µm in size, with melt-like features as small as 1 µm developed at triple junctions. The resulting elemental maps are 512x400 pixels. The Co K EDS map (Figure 1b) illustrates a higher Co concentration within the grains in comparison to the melt-like pockets, while the Fe distribution depicted by EDS (Figure 1c) displays an inverse correlation with Co levels. Leveraging the superior sensitivity and high peak-to-background ratio of WDS, the WDS map of B K (Figure 1d) distinctly reveals a significantly higher concentration within the melt-like pockets and a depletion within the grains. However, due to the superfast pulse processing time utilized, the EDS map of B K (Figure 1e) fails to show the pronounced concentration contrast between melt-like pockets and grains.

Conclusion

In summary, simultaneous EDS-WDS mapping at a high beam current successfully delineated the element distribution for both the light element boron and the transition metals in a boron steel sample. By harnessing the benefits of both techniques, simultaneous high-throughput EDS-WDS mapping enables the acquisition of high-quality X-ray map data within the shortest possible timeframe.

Graphic:



Keywords:

EDS-WDS, mapping, high-throughput, light elements

Reference:

[1] T Nylese and J Rafaelsen, Microscopy Today 25 (2017), p. 46. doi:10.1017/S1551929517000190