

High spatial resolution, low voltage and ultra-fast energy dispersive X-ray spectroscopy on a scanning electron microscope

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Energy Dispersive X-Ray Spectroscopy (EDX) on a Scanning Electron Microscope (SEM) is a surface chemical analysis technique. When using this technique, surface means a volume of typically a few cubic microns under the real sample surface¹. In order to acquire real surface EDX spectra, SEM operators have to work at very low electron beam energies to minimize electron beam penetration on the sample, thus reducing the analysis volume and, in turn, decreasing the signal that reaches the EDX detector. The resulting low signal spectra are not useful for semi-quantitative analysis or mapping/linescan analyses.

We have recently attached an X-Max Ultim Extreme EDX detector (Oxford Instruments) to our extreme high resolution Thermo Fisher Magellan 400L FESEM. With this detector we have been able to perform low energy EDX analysis with high energy resolution and high signal inputs, allowing us to do real surface analysis and to detect low energy peaks from elements that are typically detected at higher energies. Thanks to the high surface detecting area of the Ultim Extreme detector, mappings and linescans are done in a much shorter time than before, allowing us to acquire high signal mappings or linescans in just a few minutes, both in SEM and STEM modes, and with nanometre spatial resolution.

Here we present different analysis examples performed on our state-of-the-art EDX system, including high resolution STEM-EDX maps and linescans of multicomponent nanoparticles, such as Fe₃O₄/Mn₃O₄ nanocubes, Ni/ZnO catalyst or AgCeO₂ heterodimers, where features of around 10 nm can be easily resolved (see Figure 1). We will also show the capabilities for light element analysis by the detection of boron in metal-organic framework (MOF) nanosheets.

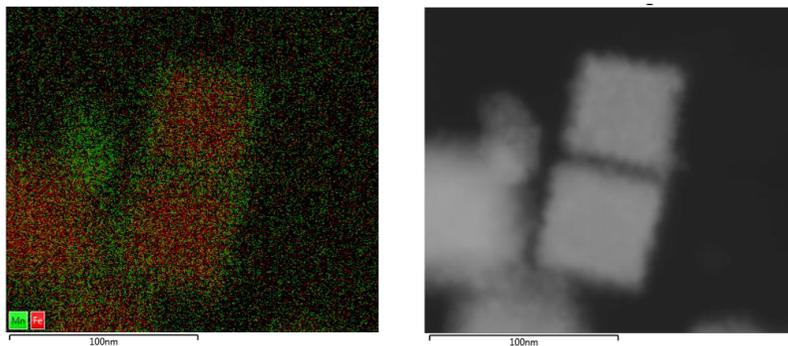


Figure 1. $\text{Fe}_3\text{O}_4/\text{Mn}_3\text{O}_4$ nanocubes high resolution STEM-EDX mapping. Left: STEM-EDX map showing spatial localization of Mn and Fe. Right: the corresponding SEM image.

References

1.- Goldstein J. (2003). Scanning electron microscopy and X-ray microanalysis, Springer, ISBN 978-0-306-47292-3

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