Analytical and imaging improvements with recent FEG microprobe

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Wavelength dispersive spectrometry (WDS) microanalysis is a local chemical analysis technique widely used since the development of the device by Raimond Castaing during his PhD in 1951 [1]. Numerous technical developments have continued to advance the capabilities of electronic imaging and elemental analysis of this tool. We present here some of them through examples obtained in the framework of the renewal of a 1990's electron microprobe by one of last generation. This is the JEOL JXA-8530F/Plus microprobe, equipped with four wavelength dispersive spectrometers (WDS), an energy dispersive spectrometer (SDD) and a dedicated spectrometer for X-ray photons of very low energies (SXES).

The analytical capabilities of this kind of instrument are very closely related to the construction of WDS spectrometers, such as the Rowland circle diameter value, the nature and size of the diffracting crystals, as well as the type of the proportional counters. A great versatility of analysis has been obtained by combining each of these factors to allow maximum optimization, both from the point of view of the wavelength selectivity and from the point of view of the analytical sensitivity [2] (see fig. 1). For this third generation Schottky field emission gun, the maximum current value reaches 3 µA at 30 kV and has a high intrinsic current stability related to JEOL patented FEG design. Coupled with a spectrometer optimized for high counting rates, detection limits in local chemical analysis can be extremely low, down to tens of ppm.

In the field of very low energy photons, JEOL has developed a new type of ultra-high resolution spectrometer using diffraction gratings with varied-lines-spacing: the Soft X-ray Energy Spectrometer, SXES [3]. It opens the door to new topics of local studies by microprobe, such as the analysis of lithium micro-batteries with the JS50XL grating for the detection of the Li K line at 54 eV (see fig. 2). Another field of application is the study of chemical effects or the analysis of light elements in the presence of heavy elements. For example, when analyzing tungsten carbides, it is possible to overcome the interference between C K and W N lines with the JS200N grating, while these are not separated in a classical WDS spectrometer equipped with pseudo crystal such as LDE2 (2d = 98 Å).
Thanks to the FEG technology, a clear improvement in the spatial analysis resolution is visible. Submicron singularities in massive samples are clearly demonstrated as shown in figure 3 [2]. On a suitable preparation, it is even possible to visualize the distribution of a dopant such as zinc in nanoparticles of iron oxides (see fig. 4). Although this is not the primary function of such a device, the contribution of FEG technology on a microprobe makes it possible to get closer to the electronic imaging capabilities obtained on the FEG scanning electron microscopes.

[1]: R. Castaing PhD, O.N.E.R.A. Publication N°55, 1952, ISSN 0369-7622
[2]: "Chemical characterization in SEM and Microprobe - contribution of WDS spectrometry", Pedagogical days of GN-MEBA association, 2016, Paris (France), http://www.gn-meba.org/
[3]: "A Soft X-ray Emission Spectrometer with High-energy Resolution for Electron Probe Microanalysis", H. Takahashi et al., Microsc. Microanal. 16 (Suppl 2), 2010, p34-35

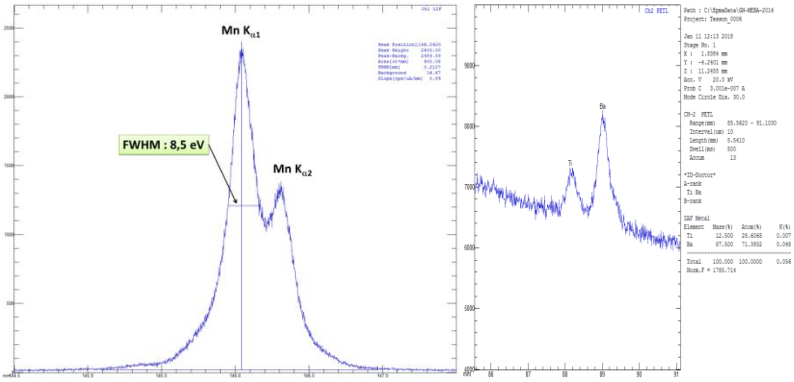

Figure 1: High energy resolution & high analytical sensitivity (700 ppm Ba, 100 ppm Ti) 2]

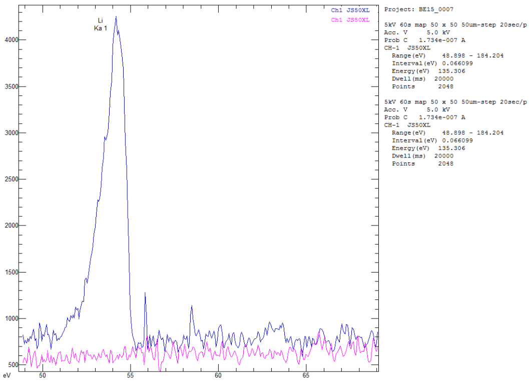

Figure 2: Li K detection on a lithium-based micro battery

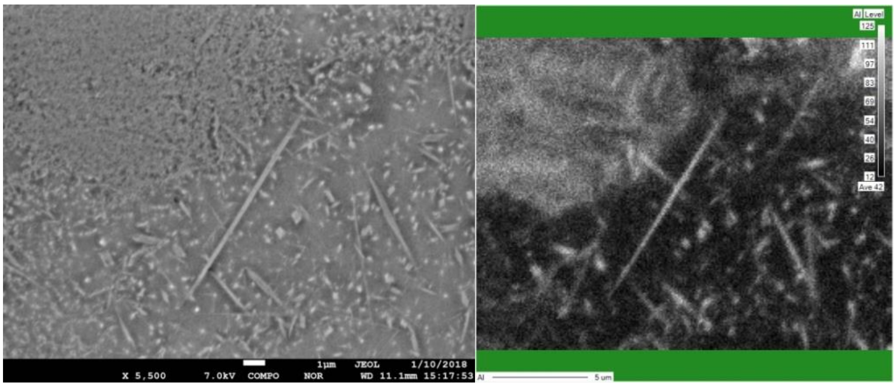

Figure 3: BEI reference image and Al K map showing needle-shaped alumina of less than 300 nm width in a bulk porcelain shard [2]

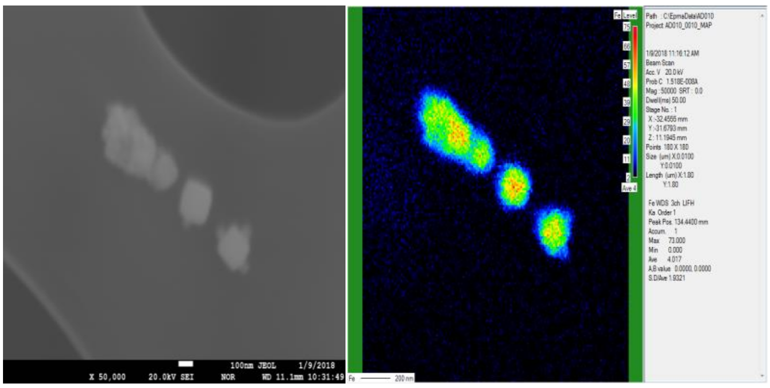

Figure 4: SEI reference image and Fe K map showing very high spatial resolution on magnetite nanoparticles