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Phase Differentiation Based on X-ray Energy Spectrum Correlation with an Energy Dispersive Spectrometer (EDS)

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Knowing the spatial distribution of the different phases in a specific material or alloy is of primary importance to relate its mechanical properties to its microstructure. The most common technique to achieve this purpose is based x-ray maps obtained with an energy dispersive spectrometer (EDS) attached to a scanning electron microscope (SEM) [1]. Quantitative processing of each energy spectrum of the recorded hyperspectral image [2] is done by applying the ZAF or the PhiRoZ quantitative methods [3] combined to principal component analysis [4] or other segmentation techniques to produce a phase map where each pixel is allocated to a specific chemical phase. However, the assignment of the necessary thresholds to separate phases can be a difficult task especially because of the inherent statistical noise of the EDS detection.

In this presentation, we describe a different path to conduct x-ray microanalysis for producing realistic phase maps. Contrary to the conventional microanalysis technique where only the characteristic line of each element present in the sample is being used for quantification, the correlation technique described here uses the energy spectrum as a whole. At each pixel of the hyperspectral image, the experimental spectrum is correlated with a library of possible phase candidates and the phase with the highest normalized Pearson product-moment correlation coefficient is assigned to the pixel (Figure 1). The advantage of this approach is to combine not only all the characteristic peaks available for each element but also the background components of the spectrum, all these components being the fingerprint of a specific phase.

This method is robust regarding to the statistical noise in the standard spectra library (Figure 2) as well as in the hyperspectral image, which is a huge advantage because the number of counts per pixel is in general very small in hyperspectral images, even at high counting rates. The technique is particularly suitable to low microscopy phase mapping which suffers from very low counting rates as well as severe overlaps in the soft X-ray region where realistic spectrum deconvolution techniques [5] are difficult to apply. The possibility of replacing standards by synthetic spectra generated using analytical models or Monte Carlo modeling was investigated and produced very similar phase maps. This opens a very promising path to characterize the distribution of phases in alloys where intermetallic phases are not available as standards. Multiple examples will be shown to demonstrate the potential use of this method and a discussion of its future development will be initiated.

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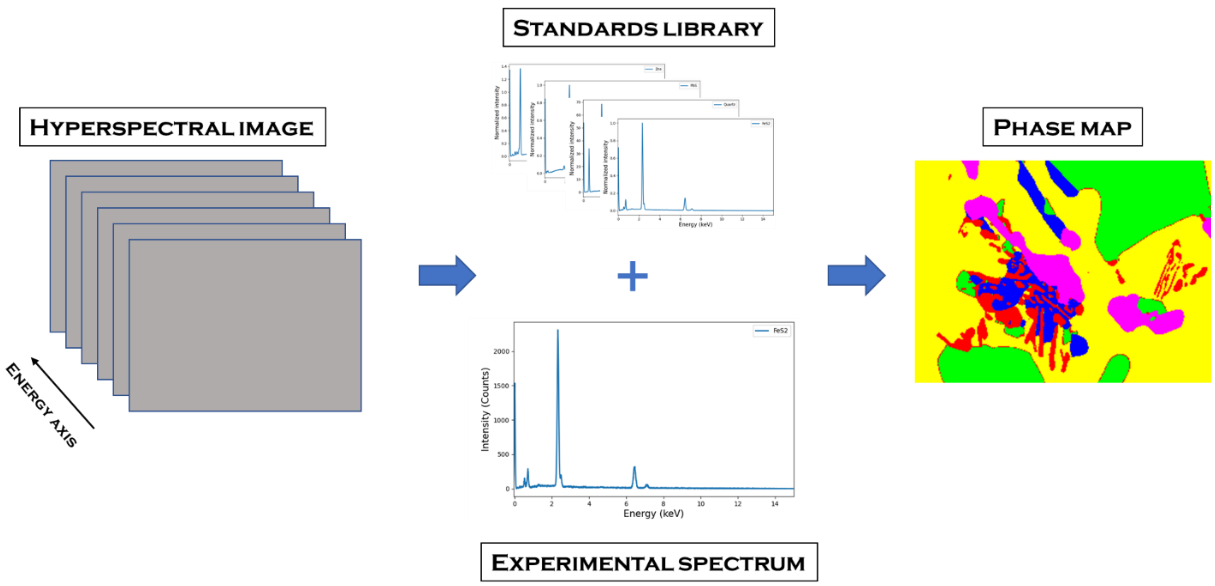

Figure 1. Principle of the correlation method for phase map analysis using EDS raw count datasets.

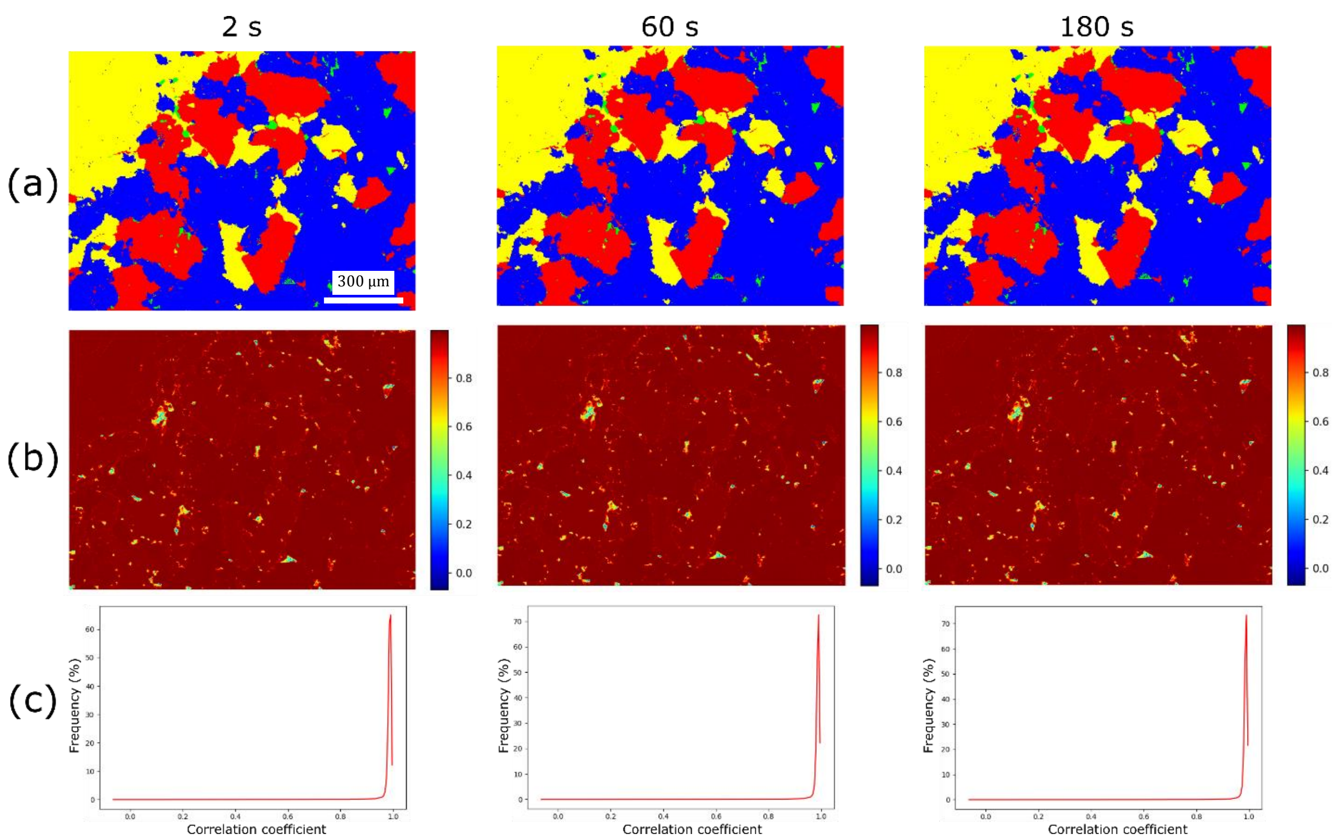

Figure 2. Phase maps obtained using the correlation technique from the same EDS spectrum image using standards acquired with 2 s, 60 s and 120 s live times. (a) Phase maps, (b) maps of the correlation coefficient for the best candidate in the correlation process and (c) the corresponding best correlation coefficient histogram of (b).

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