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Data Analysis Methods for Modern Analytical Electron Microscopy

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The vast amounts of data furnished by modern analytical EM experiments demand increasing levels of sophistication in their treatment if all the information they contain is to be successfully recovered. In this presentation we will give an overview of the ways in which the data can be mined, ranging from the standard techniques, through more modern approaches, towards as yet untested methods. The emphasis will be on EELS rather than EDX, although the consequences of the recent striking technical progress in the latter technique will be briefly reviewed.

Perhaps the most fundamental expectation from such experiments is the reliable quantification of the sample's local elemental composition. Despite vastly improved equipment, better understanding of the electron-matter interactions and the introduction of advanced models for fitting to the data, routinely reliable quantification across the periodic table has proved surprisingly and stubbornly elusive, particularly in the case of EELS. We will compare the two main approaches to this problem; the "Egerton" method [1] and curve fitting or model-based quantification [2,3]. Each will be shown to have its domain of usefulness, although the latter is usually preferable, since it can more readily furnish an estimate of the confidence in the results obtained. The future of EELS as a basic quantification method will be analysed in view of the current and expected future EDX capabilities.

Nowadays, the data acquired in a typical analytical EM experiment is usually in the form of a spectrum-image or EFTEM dataset, rather than an individual spectrum. This has a number of consequences, some of which may not yet have been realised, or at any rate, taken into account or exploited. One area which has been partially explored is the use of multivariate analysis (MVA) methods, the best known of which in the EELS community is principal components analysis (PCA) which is now quite routinely used for noise reduction purposes. However, other powerful methods from the field of blind source separation (BSS) are starting to reveal their interest, in particular independent components analysis (ICA), which is often capable of providing a more physically meaningful breakdown of the various processes occurring in the sample and contributing to the spectrum [4]. In favourable instances this can be an alternative approach to the quantification processes cited above [5,6]. In other cases it may reveal very subtle (invisible in the raw data) changes in the spectral shapes which enable the mapping of fine structure variations with greatly improved sensitivity. The example in figure 1 shows a map of two components found to be contributing to the Ti-$L_{2,3}$ edge in an ion-beam-damaged $SrTiO_3$ (STO) layer. One component exhibits the crystal field splitting typical of octahedrally coordinated Ti^{4+} ions and its map confirms the regular arrangement of the atoms as would be expected in STO. The other displays a greatly reduced crystal field splitting effect, and indeed the map of its distribution displays considerably less crystalline order. The titanium is thus shown to be present in two distinct states, a fact which was indiscernible in the raw (and indeed in the PCA treated) data. However, in cases like this, the relative intensities of the different components found by the analysis are arbitrary, and their quantification thus remains a considerable challenge. Some possible approaches will be discussed.

Optimisation of data analysis procedures may have other potential benefits that have not yet been considered in detail by the EELS community, including partial solutions to the increasingly severe problem of beam damage (ever more intense beams, ever more sensitive materials). For example, PCA has in effect reduced the time and the electron dose required to perform an experiment with a given signal-to-noise ratio. In a similar way, other smart data mining approaches may further reduce the number of initial observations necessary. Another part of the EM community is already starting to use methods which exploit prior knowledge about the sample to reduce the number of observations required in tomography experiments [7]. We will discuss a few of the possible ways in which similar knowledge might be used to limit dose and thus damage in EELS/EDX mapping measurements.

References
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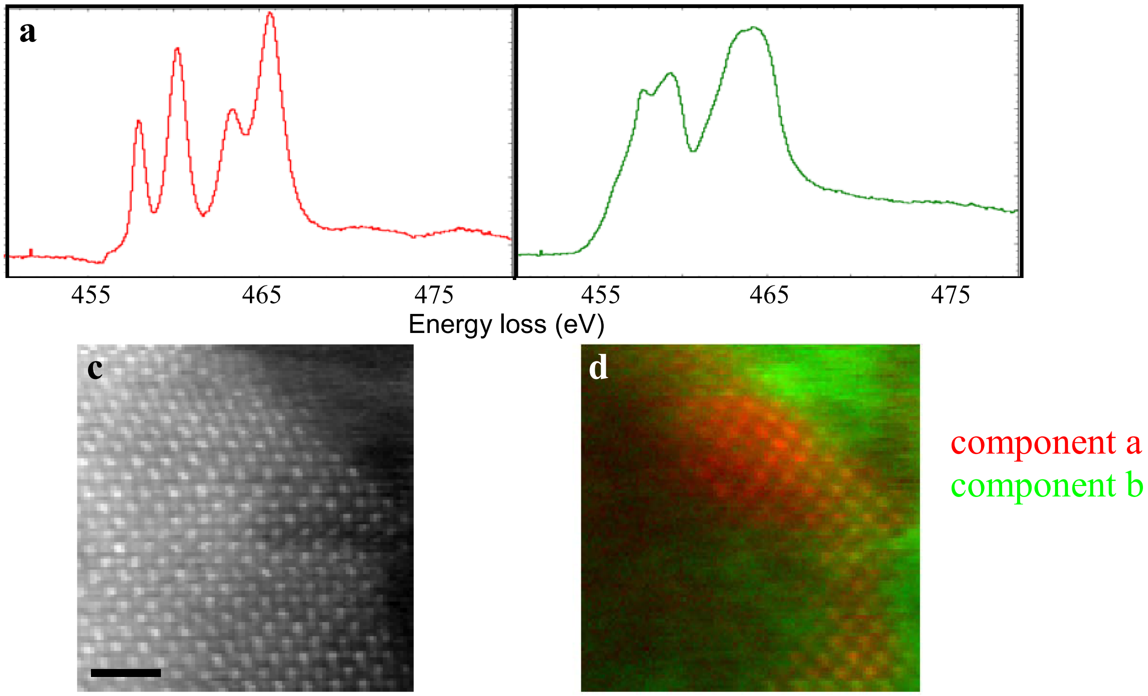

FIG. 1. ion-damaged STO sample: a) and b) two independent components found for the Ti L edge, with and without strong crystal field splitting respectively, c) HAADF image of the zone, d) map of the two component intensities. *In collaboration with the Department of Electronics, Barcelona University and the Barcelona Materials Science Institute*