Composition and thickness of buried nano-structures by spectrum separation: from interfaces, precipitates to amorphous composites

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Buried nano-structures, such as interface and precipitate, are traditional interests of microscopists and spectroscopists. However, imaging is limited to 2D and often restricted to uniformity through the thickness. AEM analysis can get partially quantitative information for the buried structures but it complicates with the probe characteristics. An indirect EELS methodology, “spectrum separation” developed based on “spatial difference” technique, is suitable to explore the full chemical information of the buried nano-structures, benefiting from the inherent connection of structure and composition of any phase from the atomic scale on [1].

With sufficient information of ELNES behavior for the probed element, spectral contributions of a buried interface and the surrounding matrix can be separated to a precision related to a combination of energy and spatial resolutions. Quantification of the separated signals brings out both concentration and space information of the related element, in addition to the exclusive ELNES of the buried structure, which can be further extended to reveal chemical composition. ~1 nm thick amorphous film covering grain boundary in ceramics was fully analyzed by the “spectrum separation” method, various unique characters of the buried thin film were revealed [2]. Atomic plane of interface could also be probed in such an indirect way [3]. The method is in principle similar to the basis-searching methods such as the Artificial Neural Network and the Multiple-variant Statistic Analysis, but anchored with the bonding and/or electronic characters of the element to make the procedure simple and reliable.

The “spectrum separation” methodology is also extendable to buried nano-particles or clusters inside a matrix, be it a grain, an amorphous body or a nano-structure itself. For precipitation within a crystalline grain, not only the full composition is retrievable, but the thickness of precipitates can also be estimated. This method is especially powerful in inter-penetrated nano-composites, when combined with the EELS “spectrum imaging” technique. In precursor-derived Si-based ceramics which exhibit often an apparently amorphous structure, “phase” separation at near 1 nm scale is able to be characterized by such an approach, hence expanding the scope, and pushing the limit, of the spatially-resolved analysis in an unconventional way.

References