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Sample Preparation Considerations for X-ray EDS Analysis in the Physical Sciences

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The use of X-ray Energy Dispersive Spectroscopy (XEDS) in an electron microscope is perhaps the most utilized technique for the microanalysis of materials. X-ray detectors can be found on scanning electron microscopes (SEM), dual beam focused ion beam (FIB) systems, and transmission electron microscopes (TEM) for both the physical and biological sciences. With respect to using it for analysis, there are two basic assumptions that apply, 1) the volume analyzed is homogeneous and 2) the exiting surface for the X-rays is flat. The one exception to this is thin film on substrate analysis, but it is still assumed that the film layers and substrate are homogeneous and the sample is flat. Of course, additionally for analysis in the TEM, the sample must be thinned to electron transparency.

There are numerous methods for sample preparation for SEM and TEM. In the past, preparing a sample for XEDS in the SEM would be relatively straight forward; it would entail mechanically polishing the sample flat, coating it with carbon if it were non-conductive, placing it under an electron beam with sufficient overvoltage, and analyzing it. However, with the advent of the other characterization techniques in the SEM (or FIB), such as EBSD, STEM, t-EBSD, 3D tomography, Spectrum Imaging, etc., the sample may also need to be prepared with that technique in mind as well. Because of this, the same processes that have been traditionally used for TEM sample preparation might also be used for samples that are analyzed in the SEM by XEDS. With this in mind, the issues associated with these sample preparation techniques that lead to artifacts will be discussed. It would be prohibitive to discuss specific details of these different techniques and how they relate to XEDS analysis to cover all aspects. Instead, the goal of this paper is to discuss how artifacts from sample preparation may affect the analysis of the sample and how they relate to the two primary assumptions. Table 1 lists the sample preparation techniques to be discussed with a short description of the microscopy utility and potential artifacts affecting XEDS analysis. The effects that some of these artifacts would have on XEDS analysis are obvious, but others may be more subtle. For example, why would plasma cleaning, a necessary procedure for many samples to prevent hydrocarbon contamination, be listed as a potential source for contamination? Each artifact listed can be associated with one of the two basic assumptions. Topography changes, by differential sputtering or polishing violate the flatness assumption and will affect the path length of X-rays and thereby affect the absorption and fluorescence. Precipitation, contamination, re-deposition, oxidation violate the homogeneous assumption and modify the composition of the analyzed volume. The degree to which the specific sample preparation artifact has on the XEDS analysis due to the departure from these assumptions is strongly dependent on the analytical instrument. The key to good microscopy is sample preparation with a minimum of artifacts introduced. Although, in general, the sample preparation requirements for good analytical results with XEDS are less stringent, the microscopist should be aware of the potential for artifacts introduced during preparation that could influence the analytical results.

Table 1
Sample Preparation Artifacts

Preparation Technique	Microscopy Technique	Artifact
Mechanical Polishing (e.g. lapping, Tripod Polishing, dimpling)	SEM: XEDS analysis, cross section TEM: plan view, cross section	Smearing, differential polishing, edge rounding, scratched surfaces, embedded abrasives
Ion Milling	SEM: Ion Polishing for EBSD, slope cutting, Ion etching for phase and grain boundary contrast enhancement FIB: 3D Tomography (XEDS, EBSD), Cross section TEM: Final thinning, FIB cleaning	SEM: preferential sputtering leading to topography FIB: amorphization, curtaining, Ga incorporation, microstructure changes TEM: Topography, preferential sputtering leading to thickness variations within/between phases, chemical changes, phase changes, contamination, amorphization, hydride formation, re-deposition
Electropolishing/Chemical Polishing	SEM: Etching for phase and grain boundary contrast enhancement TEM: Final thinning	SEM: Differential polishing leading to topography, TEM: Differential polishing leading to variations in thickness or phase dropouts, surface re-deposition leading to thickness composition variations with thickness, hydrocarbon contamination
Ultramicrotomy	SEM: Serial block face imaging and 3D Tomography TEM: Final thinning	SEM: Surface topography TEM: ??
Cleave/Fracture	SEM: Cross section TEM: Plan view, Cross section, slivers	SEM: Particle Geometry TEM: ??
Crushing/Grinding	SEM: N/A TEM: Final thinning, Standards	SEM: Particle geometry TEM: Particle geometry that leads to variation in thickness for standards
Plasma Cleaning/Trimming	SEM: Cleaning hydrocarbon contamination TEM: Cleaning hydrocarbon contamination, removing amorphous surface layers	SEM: Oxidation, Contamination TEM: Oxidation, Contamination, re-deposition