New Horizons in Multi-Technique Auger Electron Spectroscopy: Nanoscale Surface Sensitive Chemical Imaging of Additive Manufacturing Materials

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Recent advances in Auger Electron Spectroscopy, including *in situ* FIB capabilities, put AES at the forefront of surface analysis techniques. With an average depth of analysis of 5 nm and lateral spatial resolution as small as 8 nm, together with the availability of multi-technique capabilities, AES is a robust analytical tool for nanoscale feature analysis. The quantitative elemental information AES provides from solid surfaces combined with focused ion beam tomography allows for *in situ* cross-sectioning and subsequent elemental characterization of solid materials. State of the art multi-technique AES is highlighted here via the analysis of an ever-growing area of research interest: materials for additive manufacturing. Specifically, an alloy powder material used in the laser-powder bed diffusion process of 3D printing will be analyzed for grain boundary diffusion.

In this work, CuCrZr alloy powder particles were cross-sectioned and analyzed using a PHI 710 scanning Auger nanoprobe equipped with a 25kV Schottky field emission electron gun and a coaxial Cylindrical Mirror Analyzer (CMA). We demonstrate the use of AES in conjunction with a focused ion beam (FIB) to produce site-specific imaging of grain boundary diffusion within the alloy.

The FIB cross-section of the particle reveals several regions of discoloration (Figure 1a) indicative of grain boundary diffusion. Further analysis of the cross-section with AES elemental mapping at 3 μ m field of view (Figure 1b) clearly indicates an increased concentration of oxygen, chromium, and zirconium in this region. In addition to *in situ* FIB cross-sectioning and elemental mapping with high spatial resolution, AES is also capable of elemental quantification of the grain boundaries.

A second region of high carbon intensity (Figure 2) was located with AES elemental mapping. This region, less than 200 nm in diameter, was selected for point analysis and shown to contain 46.3 atomic percent of carbon, a significantly higher carbon content than the bulk grain concentration. Accurate spatial and chemical characterization of these nanoscale features is of critical importance as they could negatively affect the mechanical properties of the final printed material. The multi-technique capabilities along with high spatial resolution chemical imaging and quantification capabilities, make AES a powerful tool for analyzing these types of nanoscale features.





Figure 1. SEM images (row a) of a FIB cross-section of a CuCrZr powder particle at 20 μ m, 10 μ m, and 5 μ m field of view and elemental Auger maps (row b) at 3 μ m field of view.



Figure 2. AES elemental maps of a freshly exposed grain boundary cross section at 5 μ m (left, carbon) and 1 μ m field of view (center, copper and carbon) with a point spectrum (right) extracted from a region of high carbon.