

TEM/APT Correlative Analysis on Multi-Layer Ceramic Capacitors (MLCCs)

A novel protocol for high-precision targeting, in which critical regions identified by STEM can be precisely converted into APT specimens.

Introduction

Multi-Layer Ceramic Capacitors (MLCCs) are essential components in modern electronics, delivering high capacitances in a compact form. They are ubiquitous in applications ranging from hand-held devices to automotive systems— a single smartphone can contain thousands of individual MLCCs. Despite their reputation for reliability, MLCCs can exhibit failure such as current leakage, aging, and thermal instability. Understanding how material properties influence long-term reliability is therefore critical.

The complex layered architecture and nanoscale features of MLCCs pose significant characterization challenges, particularly in quantifying subtle compositional variations that impact both performance and degradation. In this technical note, we present a correlative approach combining scanning transmission electron microscopy (STEM) along with atom probe tomography (APT) to enable targeted, high-resolution characterization of MLCCs.

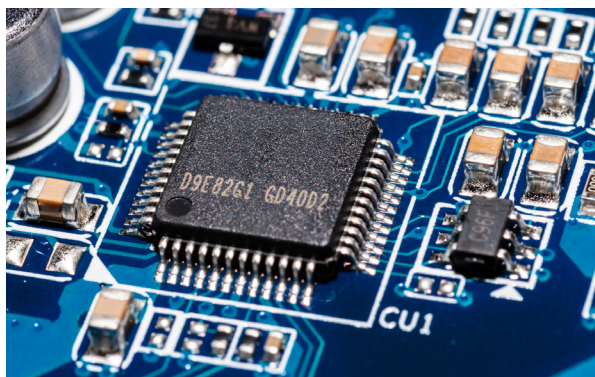


Figure 1. MLCCs on a motherboard

Characteristic Structure of MLCCs

MLCCs consist of alternating layers of dielectric ceramic and metal electrodes, as illustrated in the schematic below. They are classified by capacitance, operating voltage, and temperature performance. For this study, we examined a commercially available Grade 1 C0G MLCC rated for 27 pF at 50 V, recognized for its excellent stability across a wide temperature range. Although conceptually simple, the electrode layers and ceramic encapsulation exhibit complex polycrystalline microstructures and compositions.



MLCC cross-section diagram

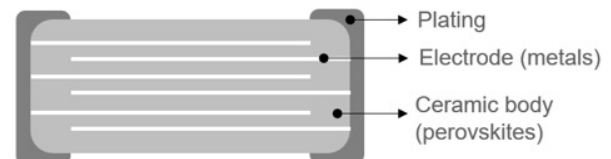


Figure 2. A class 1 compact MLCC purchased from the market and schematic diagram of a typical MLCC structure, highlighting the alternating dielectric and electrode layers.

Working Principle of Atom Probe Tomography (APT)

APT enables three-dimensional elemental and isotopic mapping with sub-nanometer spatial resolutions and exceptional chemical sensitivity. The technique relies on field evaporation of atoms from a needle-shaped specimen, followed by time-of-flight mass spectrometry for ion identification. This approach provides quantitative 3D chemical analysis, essential for understanding both matrix level elements as well as dopant/contaminant distribution in electronic materials.

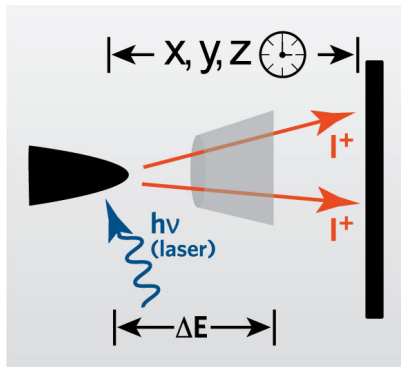


Figure 3. APT combines time-of-flight mass spectrometry and single ion detectability to investigate elemental distribution in 3D at the atomic-scale.

Working Principle of Scanning Transmission Electron Microscopy (STEM)

In STEM, a finely focused electron probe (80–300 kV) is rastered across an electron-transparent specimen. Scattered and transmitted electrons are collected point-by-point to construct high-resolution images. Additional detectors enable elemental analysis via energy-dispersive X-ray spectroscopy (EDS) and electronic structure characterization through electron energy-loss spectroscopy (EELS).

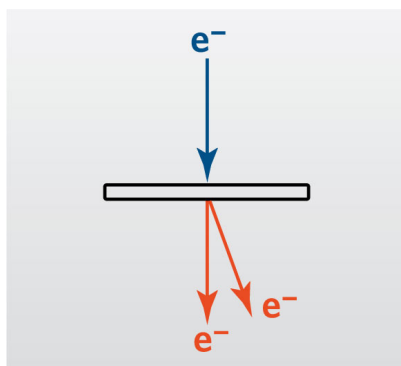


Figure 4. General schematic of scattered and transmitted electrons in STEM.

Correlative Analysis for Comprehensive Characterization

Each analytical technique offers unique strengths and limitations. APT delivers unparalleled compositional detail at the atomic scale, but is restricted to very small volumes. For heterogeneous samples, precise targeting becomes critical. At EAG, we employ a “digging down” strategy—progressively narrowing the area of interest using optical, electron, and ion microscopy. For MLCCs, STEM provides essential microstructural context, mapping grain structures and performing initial compositional screening at a larger scale. This step identifies key features such as interfaces and grain boundaries. We then apply a novel protocol to transform the same STEM lamella into a needle-shaped APT specimen, ensuring accurate targeting of critical regions. Without STEM guidance, APT sampling risks missing relevant features. This workflow enables true correlative analysis on the same feature using multiple techniques, including EDS, EELS, nanobeam diffraction, and high-resolution imaging.

Correlative Analysis of Ceramic Triple Junctions

The high-performance MLCC was first embedded and mechanically polished to expose its internal layered structure. SEM imaging revealed electrode layers (~2 μm thick) beneath a plating layer. Secondary ceramic phases were observed in a darker contrast both inside the electrode layers and within the ceramic body.

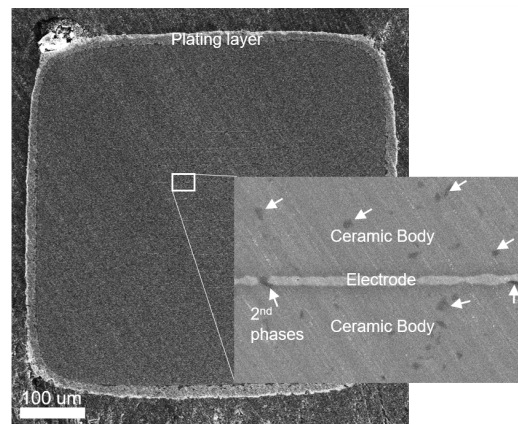


Figure 5. SEM image of mechanically polished cross-section revealing the layered structure in MLCC showing 2 μm thick electrode layers sandwiched between the ceramic body. The arrows in the inset indicate the secondary phases.

A STEM specimen was prepared from the electrode/ceramic interface using focused ion beam (FIB). STEM-EDS mapping revealed nanoscale Ni grains within the electrode layer and micrometer-sized Zr-rich oxide grains with sharp grain boundaries in the ceramic. A Si-rich secondary ceramic phase was also observed, exhibiting a smooth interface with the electrode and a rough interface with primary grains. Notably, the ceramic was not a conventional perovskite such as barium titanate or strontium titanate. No significant segregation was observed at grain boundaries which can be attributed to the limitations of EDS sensitivity and the morphology of interfaces in 3D.

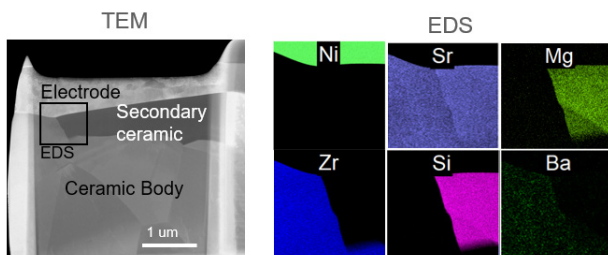


Figure 6. Detailed morphology shown by STEM with elemental distribution obtained using EDS indicating Zr rich primary ceramic grains and Si rich secondary ceramic grains.

To probe these interfaces at the atomic scale, APT was employed. Using our specimen preparation protocol, a wedge-shaped specimen was mounted on an APT-compatible half-grid then converted into a needle-shaped specimen targeting the triple junction identified by STEM.

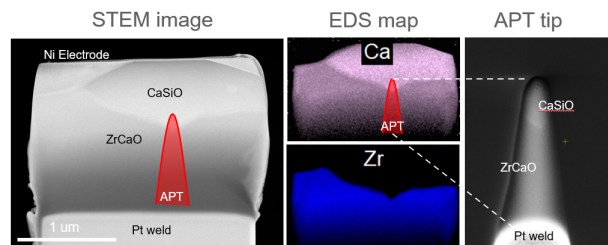


Figure 7. Our novel approach allows the conversion of STEM lamella into needle shaped APT specimen to target the triple junction.

APT confirms the Si enrichment in secondary grains and Zr enrichment in primary grains, along with trace elements such as Mn, Sr, Mg, Hf, and Ba. The 3D nature of APT data enables extraction of

compositional information revealing a $\text{CaZr}_{1.8}\text{O}_{8.2}$ primary phase and a $\text{Ca}_{2.6}\text{SiO}_{3.8}$ secondary phase. STEM like 2D contour maps were taken from the triple junction identifying ~ 0.8 at.% Al segregation and Si and Mn diffusion into the primary grain boundary.

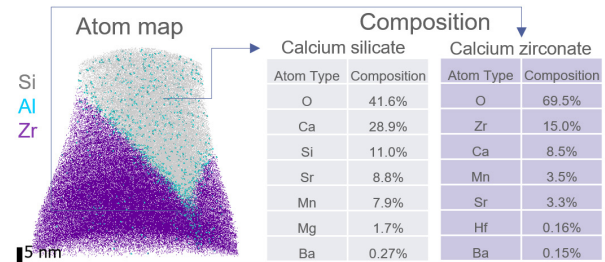


Figure 8. High quality atom probe data was acquired from the triple junction confirmed using STEM, showing a Si enriched secondary ceramic grain between two Zr enriched primary ceramic grains. Detailed phase compositions are listed in the table.

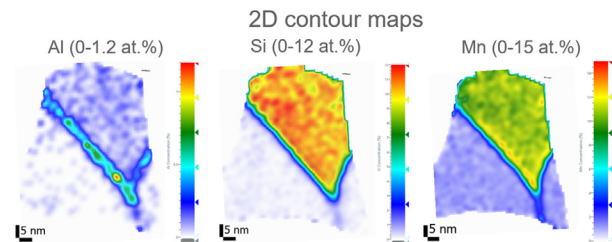


Figure 9. Detailed investigation on elemental segregation to phase boundaries and diffusion along triple junctions.

Conclusion

APT provides atomic-scale compositional insights with a sensitivity far exceeding STEM-EDS or EELS. This case study demonstrates a robust correlative workflow in which STEM-guided targeting enables precise APT analysis of critical regions. By integrating complementary techniques, EAG achieves a synergistic approach that significantly enhances characterization capabilities for complex electronic materials.

Contact us today to learn how we can help you with your next project.