Three-Dimensional Subnanometer Compositional Analysis of Radiation Damaged Materials with Atom Probe Tomography – Technology and Practical Considerations

Robert M. Ulfig, Ty J. Prosa, Yimeng Chen, Katherine P. Rice, Isabelle Martin, David A. Reinhard, P.H. Clifton, Brian P. Gieser, Edward Oltman, Dan R. Lenz, Joseph Bunton, Thomas F. Kelly and David J. Larson

CAMECA Instruments, Madison, USA
Corresponding author: Robert Ulfig >Robert.ulfig@ametek.com>

Over the past 15 years, the number of peer reviewed publications referencing the use of atom probe tomography (APT) has grown by nearly a factor of five (Larson et al., 2013). The performance of the typical atom probe today is orders of magnitude better than the early systems in terms of data collection rate, field of view, mass resolving power, reliability and software. The availability of easy to use laser pulsed systems coupled with the changes in performance and availability of FIB-SEM sample preparation has dramatically opened the array of applications that can be analyzed.

Modern local electrode atom probe systems (LEAP®), can identify subnanometer, spatially resolved, 3D information of any element or isotope with up to 80% detection efficiency at millions of atoms per minute with a field of view (FOV) that can exceed 250 nm and an achievable sensitivity limit in the low parts per million range. The imaging, time-of-flight mass spectrometer, of the LEAP 5000™ from CAMECA® includes improvements in FOV and uniformity, multi-hit sensitivity, faster data acquisition, especially in voltage-pulsed mode, and a new control software platform that improves yield and the speed at which a user can optimize run conditions. Additionally, a new atom probe platform, the EIKOS™ (Larson et al., 2018), offers dramatic improvements in the simplicity of design and operation, including an over 50% reduction in the cost of ownership while still providing high-performance voltage/laser pulsed APT data.

In addition to these instruments performance developments, it is now possible to complete specimen transfer at ultra-high vacuum (UHV) levels with the ability to keep the specimen at cryogenic temperatures to enable new applications such as water-based systems, fast oxidizers like lithium, and the ability to analyze the distribution of hydrogen/deuterium in materials. Vacuum transfer systems can also be used to safely move radioactive specimens. Also, new sample preparation techniques and software developed in cooperation with EDAX® to combine transmission electron backscatter diffraction (t-EBSD) and APT to provide synergistic information (see Figure 57.) and improve APT data reconstruction (Rice et al., 2016).

Although the most common APT applications remain metallurgical in nature, these hardware and software improvements continue to open new applications by enabling sufficiently high yield and data quality to provide novel information. Recent new applications include, failure analysis of FinFET devices, 3D printed alloys, high entropy materials, rapid oxidizers, zeolites, cryogenically preserved biomolecules, H/D distribution in materials as well as traditional analysis of metals, metal oxides and protective coatings. Examples of APT in the analysis of nuclear structural materials and fuel will be reviewed.

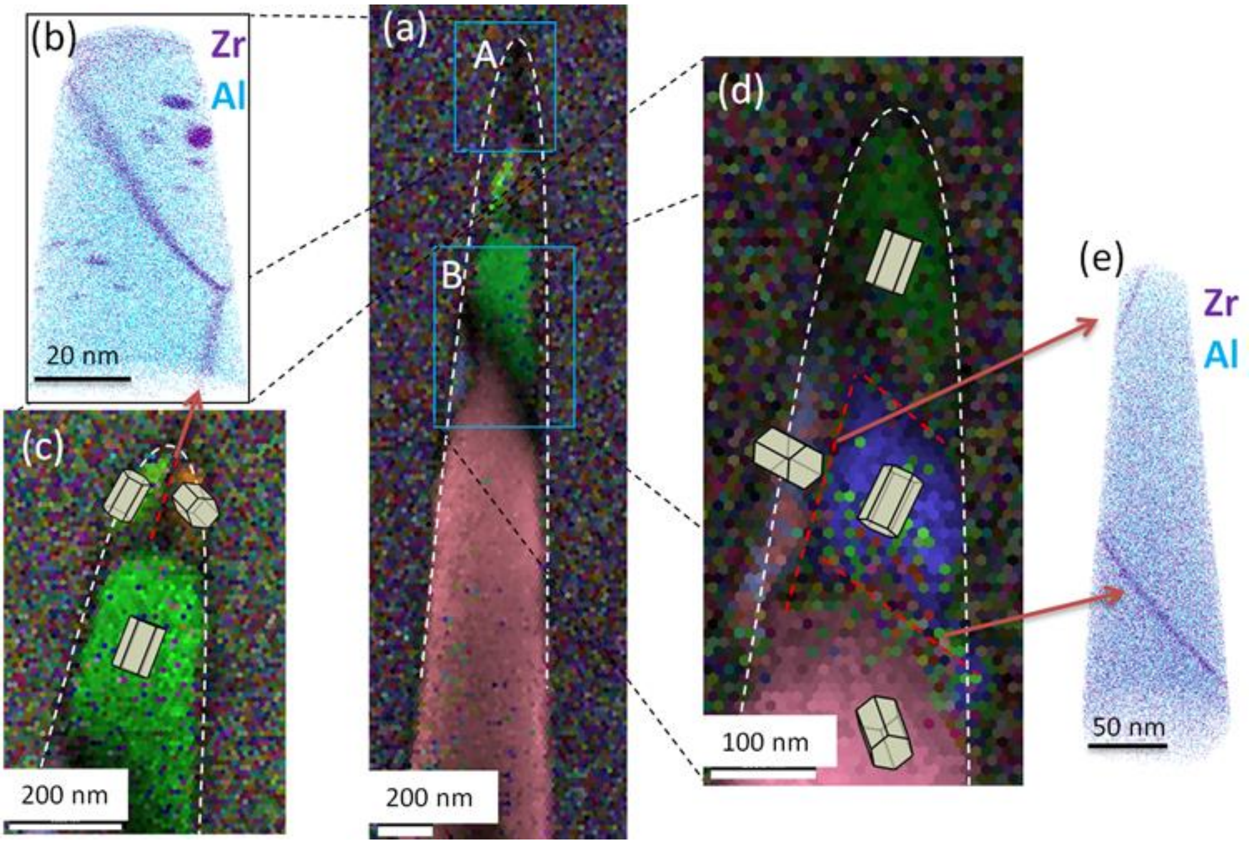

Figure 57: t-EBSD maps of alumina grains and correlative APT results from an alumina scale contraining zirconium.

References

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