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Meeting-report

Structural Analysis Enabled by the Invizo 6000® Large Field-of-View Atom Probe

Yimeng Chen[1], Isabelle Martin[1], Ty Prosa[1], Robert Ulfig[1], Katherine P. Rice[1], David J. Larson[1], David A. Reinhard[1], Dan Lenz[1], Nicholas R. Brewer[1], Michael Holman[1], Jacob Hanna[1], Gard Groth[1], Avnish Singh Pal[2], and Joysurya Basu[2]

[1]*CAMECA Instruments Inc., Madison, WI, United States*
[2]*National Electron Microscope Facility, Department of Metallurgical Engineering, IIT (BHU), Varanasi, India*

*Corresponding author: Yimeng.Chen@ametek.com

Since the first one-dimensional atom probe was invented in 1967 by Erwin Müller, one primary goal in the development of the technique has been to approach a larger field-of-view (FOV) [1]. The earliest three-dimensional capability was enabled in 1972 by John Panitz in his Imaging Atom Probe [2]. A revolution in atom probe tomography (APT) with true three-dimensional capability was developed in 1988 at the University of Oxford [3]. The improvement in data collection speed to hundreds of atoms per minute and wide FOV enlarged to ∼15 nm was spectacular for that era. Inspired by the idea of a Scanning Atom Probe proposed by Nishikawa in 1993 [4], Kelly and Larson et al. pushed the development further, aiming for higher data collection rates and larger fields-of-view [5]. The local-electrode geometry implemented in the Local Electrode Atom Probe (LEAP®) system allowed millions of atoms per hour to be collected from the center 40-60% of the apex area.

In 2021, CAMECA introduced the 6000 series of atom probe instruments that bring the benefits of deep -UV laser pulsing to commercial APT systems for the first time, improving success rates and data collection rates on certain applications. Beyond that, the new Invizo 6000 instrument features a novel counter electrode design, allowing dual laser beam illumination and an extremely large field-of-view (FOV) that captures nearly the entire volume of the specimen [6]. The large FOV provides more careful control of field evaporation, easier targeting for specimen preparation, and early detection of the regions of interest as they approach from the edge of the field of view. The ability to incorporate more information per data acquisition also greatly improves the fidelity of the APT reconstruction. In addition, a larger FOV instrument enables materials science research by allowing the interaction of precipitate structures to be observed more easily, or additional phases to be captured in a single analysis.

In this work, the microstructure of CoFeMn oxide was analyzed using the LEAP 6000 XR and the Invizo 6000 atom probes for comparison. This class of spinel-forming ceramic is known for a self-assembled chessboard-like nanostructure that evolves from a complex process of twinning and subdomain formation though pseudo-spinodal decomposition and coalescence [7]. The kinetics of these processes are important for the predictive design of such a complex structure. Obtaining chemical information using APT to help understand the full picture of structural evolution in three dimensions is the primary goal of this work.

The bulk material was prepared by a series of grinding and sintering processes on high purity precursor powders to obtain mixed oxide pellets of $Co_{0.6}Fe_{0.8}Mn_{1.6}O_4$ in composition. Atom probe specimens were prepared using the standard lift-out and FIB method mounted on electropolished tungsten wire substrates. Data acquisition was performed with a LEAP 6000 XR first in laser pulsing mode. Figure 1a shows the reconstructed volume where 15 at.% Fe isosurfaces were drawn showing the boundaries between the $CoFe_2O_4$ and $CoMn_2O_4$ spinel phases. The phase separation leads to a plate-like microstructure with an interval distance of between 5 to 10 nm, as observed in Figure 1b which was taken from the last 20 nm of the reconstructed data in Figure 1a. To compare the performance of larger FOV, data acquisition was resumed on the Invizo 6000 system by matching the equivalent acquisition conditions, including the base temperature, Mn^{++}/Mn^{+} charge-state ratio, and areal evaporation rate. The plate-like structure continued in the Invizo data as illustrated in Figure 1c from the side view. The top view clearly illustrates the benefit of a larger FOV where the formation of the above-mentioned chessboard structure has already merged from the detector outer area on the left. This work is used as a demonstration for the benefits of having very large FOV APT data. We have been successful in showing that with a limited FOV the relationship between plate-like and chessboard-like nanostructures would have been missed, providing valuable insights into the growth mechanism for these alloys. Detailed comparison on data quality and chemical analysis will be provided.

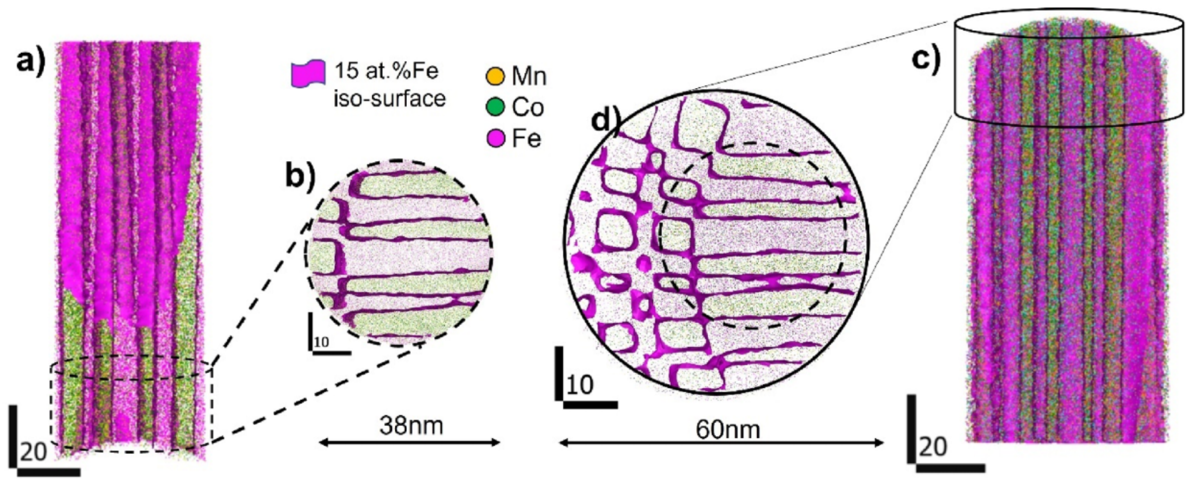

Fig. 1. a) Reconstructed volume of the spinel CoFeMnO mixed oxide acquired using a LEAP 6000 XR. The plate-like phase separation is evident in Figure 1b which is taken from the last 20nm of the data in Figure 1a. c) Reconstructed volume from the same tip acquired on an Invizo 6000. d) The radial FOV of Invizo is 1.6X larger than LEAP 6000 XR allowing the chessboard structure to be viewed in the left-hand side of the detector. The improvement in FOV translates to 2.5X in areal FOV.

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