Correlative microscopy and techniques with atom probe tomography: Opportunities in materials science

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In the last decade, the applicability of atom probe tomography (APT) has been strongly extended from highly conductive materials such as metals and alloys to semiconductors and insulators as well as to more sophisticated systems. However, atom probe tomography can only provide information about composition for most of these complex materials, while the correlation between composition and other material properties such as structural, functional, and mechanical properties remains challenging to be analyzed by APT alone. Therefore, various groups worldwide have put notable efforts recently in combining APT with other microscopy methods and techniques ex situ and in situ with the goal to understand the composition–property interrelationships at the same position of the sample. Hence, the present work not only provides a short overview of such works, but also describes three short examples of possible opportunities in materials science when using correlative microscopy and techniques with atom probe tomography.

Introduction

It has been demonstrated over the years that atom probe tomography (APT) is a powerful technique that is able to provide access to composition and sometimes to structure of materials in three dimensions and down to subnanometer level. Moreover, in the last decade, the applicability of APT has been extended from the highly conductive materials such as metals and alloys to semiconductors and insulators and even to frozen liquids, biological materials, bones, catalytic materials, and minerals. However, for most of these complex materials, APT can only provide information about composition, raising questions on the role of the other material properties such as structural, functional, and mechanical properties relevant for the design of structural and functional materials.

The idea of combining APT with other microscopy techniques started out of necessity. The design of the next generation of advanced and sustainable materials requires understanding of the entire spectrum of material properties such as chemical, structural, optical, electronic, and mechanical properties as well as their interrelationship. This can only be achieved if APT can be combined with the existent microscopy, spectroscopy, and other techniques allowing a cross-characterization on the same position of the sample and sometimes even over several length scales. For example, considerable efforts have been done by various groups worldwide to enable the combination of APT with (scanning) transmission electron microscopy—(S)TEM, scanning photoemission electron microscopy—SPEM, transmission electron backscatter diffraction—(t)EBSD, or in situ photoluminescence—PL. Although outstanding, these studies had allowed to determine the correlation between the composition (from APT) and only one of the material properties (i.e., either structural or optoelectronic properties). Therefore, very recently, substantial efforts have been put forth to reveal if a correlation or a rather coincidence relationship exists between the composition and several materials properties, such as optical/electrical and structural properties. These efforts have led to novel methodologies called correlative EBIC-EBSD-APT, where EBIC stands for electron-beam-induced current.

One other benefit of correlative microscopy with APT is to improve the understanding of the field evaporation process and thus facilitating advanced reconstruction methodologies, which will be critical to push the capability of APT to analysis of challenging heterogenous materials. Even though APT has been used extensively by many researchers over...
several decades, there is still active research aimed toward understanding the field evaporation mechanisms of oxides, nitrides, and heterostructures, which can benefit by careful correlative microscopy. In particular, as semiconductor devices are shrinking in size and individual layers are becoming few-atoms thick and arranged in complex geometries in 3D, characterizing composition of such 3D structures is challenging for most conventional microscopy methods. Successful APT analysis of these devices is a high priority for many semiconductor manufacturers. In these cases, there is a clear value added by correlative microscopy with APT and computational modeling of APT field evaporation in revealing the field-evaporation mechanisms and reconstruction artifacts inherent to current APT reconstruction algorithms.

The scope of this article “Correlative microscopy and techniques with atom probe tomography: Opportunities in materials science” is not only to review the existing APT-based correlative methods, but also to introduce new perspectives that will allow reaching new horizons in materials science.

**APT-based correlative microscopy and techniques**

Simple APT-based correlative microscopy methods such as (BSE-)SEM-APT studies (BSE in SEM stands for backscattered electron imaging) are enough to answer complex questions taking place at internal interfaces. For example, dendrite formation at the Li-anode/LLZO (Li$_2$La$_3$Zr$_2$O$_{12}$)-solid electrolyte interface and dendrite propagation inside the LLZO bulk lead to short circuit in a solid-state battery. But before finding solutions to suppress these dendrites, we need to know the elements, which are involved in the formation of these dendrites, and their exact content. Therefore, APT is considered to be a suitable technique to investigate these dendrites due to its 3D capabilities and possibility to determine the composition down to the subnanometer level.

This task cannot be easily accomplished by other techniques due to the difficulty to map and quantify Li, which is the least dense metal and the lowest atomic mass solid element available in nature. For example, direct imaging and quantification of Li ions by standard analytical techniques such as energy-dispersive x-ray spectroscopy (EDXS) in combination with (scanning) transmission electron microscopy ((S)TEM) is extremely challenging because the energy resolution of standard EDXS detectors is insufficient for Li detection (Li-K line at 55 eV). Moreover, Li detection by electron energy-loss spectroscopy (EELS) in combination with STEM is a substantial challenge because the preparation of electron-transparent specimens is difficult and the acquisition of EELS spectra requires a sufficient electron dose that may change the original Li distribution in LLZO. In contrast, beside its 3D capabilities and near-atomic resolution as described above, the APT detects both light and heavy elements with equal probability.

Yet, the dendrites, which are supposed to be formed at the Li/LLZO interface, cannot be successfully characterized by APT without placing very precisely this interface inside the tiny APT needle, closer to its apex. Thus, the precise location, extraction, and placement of the interface within a needle-shaped specimen can only be done by employing the dual-beam FIB-SEM microscope equipped with a BSE detector. Figure 1a shows a (BSE-)SEM image of the Li anode and LLZO solid electrolyte as well as their interface under low electron-beam energy of 5 kV. The backscatter yield is related to the standard Rutherford scattering cross section $R_{SCS}$ such that $R_{SCS} \sim (Z/E)^2$, where $Z$ is the atomic number of the probed sample and $E$ is the electron-beam energy. It becomes clear that the backscatter yield increases with increasing the $Z$ number and decreasing $E$ value. This is why in this study we used a low $E$ value of 5 kV. Yet, this was not sufficient to obtain a BSE image for Li (but only for LLZO) because the atomic number of Li is too low ($Z=3$). Hence, the Li anode appears completely black because Li does not elastically backscatter the electrons or if it does, the energy of the backscattered electrons is so low that they cannot reach the BSE detector.

Nevertheless, this (BSE-)SEM image is extremely helpful to precisely localize the Li/LLZO interface and to continue with the lift-out procedure schematized in Figure 1b. A 20-μm-long wedge-shaped lamella containing the Li/LLZO interface is prepared and lifted out using a micromanipulator. Part of this lamella (chunk) is mounted on the Si-support tip and an annular milling procedure is applied. A continuous imaging of the specimen during milling and a careful positioning of the annular pattern are mandatory to successfully place the Li/LLZO interface within a tiny needle of 100 nm in diameter. The APT needle obtained is shown in Figure 1c where the Li/LLZO interface is clearly visible.

APT investigations from Figure 1d clearly reveal not only the Li/LLZO interface, but also the presence of a 5-nm-wide dendrite formed already in the as-deposited Li/LLZO/Li cell, without performing any charging process. Moreover, the composition of this dendrite contains mainly 51 at.% Li and 23 at.% O and differs strongly from the composition of LLZO (i.e., 30 at.% Li, 48.6 at.% O, 10.2 at.% La, 8 at.% Zr, and 1.5 at.% Ta, which is close to the expected nominal composition). Besides Li and O, C strongly accumulates at this dendrite too with a value of 6.8 at.%, whereas no C was found inside the LLZO. In fact, it is well known that Li is very sensitive toward exposure to ambient air, which quickly leads to the formation of lithium hydroxides and lithium carbonates. Therefore, the sample was kept under Ar atmosphere to prevent the formation of these unwanted phases. Moreover, the surface of the sample was polished just before inserting it in the FIB-SEM microscope for the imaging and preparation of the APT tips.

For the moment, it is not clear if this dendrite grew from the Li anode inside the LLZO bulk or along a grain boundary, because LLZO is a polycrystalline material with an average grain size of about ~2.5 μm. It is indeed thermodynamically favorable to grow Li dendrites from the Li anode along the LLZO grain boundaries, since our recent study...
proved strong Li accumulation at LLZO grain boundaries already during synthesis. Yet, the growth of these dendrites from the Li anode toward the LLZO bulk cannot not be negligible because the Li isocomposition surface from Figure 1d (built using a Li isocomposition value of 42.5 at.%) clearly proves the presence of many protrusions directing toward the LLZO bulk. Last but not least, the chemical roughness of Li-bulk/LLZO heterointerface of about 15 nm is much superior than the typical roughness of less than 5 nm detected in typical semiconductor heterostructures.

Furthermore, the design of the next generation of green energy materials requires understanding the relationship between composition, structure, and electronic properties down to the interface level. This had not been possible to achieve until recently. Cojocaru-Mirédin and team had initiated at RWTH Aachen University a novel “correlative techniques” approach based on the combination of APT with EBSD with PPMS (physical property measurement system), which allows to correlate the chemical properties with the structure and transport at the exact same position of the sample. This expertise is essential to answer key questions linked with, for example, grain-boundary effect on thermoelectric properties (such as electronic conductivity $\sigma$) of $n$-type PbTe (Ag-doped) thermoelectric material.

A good TE material should possess a large Seebeck coefficient ($S$), high $\sigma$, and low thermal conductivity ($k$). These coefficients are embodied in the dimensionless TE figure of merit $ZT = S^2 T \frac{\sigma}{k}$ where $T$ is the temperature. The total thermal conductivity $k_{\text{tot}}$ consists essentially of electronic thermal conductivity $\kappa_e$ and lattice thermal conductivity $\kappa_l$, which shows that these parameters (e.g., $\sigma$ and $k$) are interwoven in a complex way making the improvement of $zT$ challenging. To enhance the $zT$, some effective strategies, such as phonon engineering, nanostructuring, and band engineering, have been developed. However, in all these existing studies, the true impact of each individual nanostructure on the thermoelectric properties could not be determined. This is simply because determining these thermoelectric properties locally at the nanostructure level remains impossible.

Therefore, the novel “correlative technique” approach described in Figure 2 represents a first try toward understanding these thermoelectric properties such as electronic conductivity $\sigma$ down to the grain-boundary level. First, in Figure 2a, the EBSD is used to determine the local crystal structure (i.e.,
the crystal orientation from which the grain-boundary type between adjacent grains (see Figure 2b) is derived. Second, the PPMS is used to study the electronic transport properties of the Ag-doped PbTe thermoelectric material in an environment where the temperature is well controlled from 2 to 400 K. For the PPMS investigations, a Hall-bar structure shown in Figure 2d is micromachined, which allows to determine the electronic properties of the grain boundaries and their neighboring grains. By using the Hall effect and the van der Pauw method integrated within the PPMS, the resistivity \( \rho \) (\( \rho = 1/\sigma \), where \( \sigma \) is the electronic conductivity), the charge-carrier concentration \( n \), and the mobility \( \mu \) are determined (Figure 2e–g) given that these parameters are interrelated such as \( \sigma = q \mu n \) (where \( j = e, h \) subscripts represent quantities related with electrons and holes, \( q \) is the electron charge, and \( n \) is the density of charge). Third, the needle-shaped specimen containing the grain boundary of interest (see methodology in Reference 21 for the sample preparation) is investigated by APT quantifying the Ag content at the boundary as well as the compositional changes of the matrix elements (Pb and Te).

Ideally, during the operation conditions (\( T > 300 \) K), the grain boundaries in PbTe thermoelectric material should not scatter the electrons to preserve the electronic conductivity \( \sigma \) of the bulk material and to improve the Seebeck coefficient and \( zT \) value. For the first time, we prove that this requirement is fulfilled at one the selected random high-angle grain boundary decorated by Ag. More precisely, no electron scattering effect is observed at the grain boundary (see Figure 2e–g) at temperatures above 150 K. Yet, at temperatures below 150 K, a clear electron scattering phenomenon takes place at the grain boundary (green curves) when comparing with the bulk material (red curves).

**Correlative microscopy with APT: Understanding APT-related aberrations**

Direct correlation of APT with TEM and field evaporation simulations can provide insights that can be used to improve the APT reconstruction fidelity. One study that highlights this is the direct correlation of APT with aberration-corrected STEM and finite element-based field evaporation simulations for understanding local magnification artifact during the analysis of Au nanoparticles embedded in MgO. The Au nanoparticles are nucleated within MgO single-crystal oxides by implantation and annealing. Standalone APT analysis reveals an atomically abrupt interface between Au and MgO. Hence, the APT and HRSTEM results were not consistent, motivating the application of directly correlated STEM and APT. Figure 3a shows the STEM image of the APT needle before and after field evaporation in the APT along with the APT reconstruction in (b). The bright contrast regions in the STEM image of needles (a) are the Au nanoparticles. Au is shown in yellow in the reconstruction in (b) and Au nanoparticle regions are highlighted using a 10 at.% Au isocomposition surface in (c). From the APT results, it was found that the maximum Au composition at the center of these nanoparticles was still below 25 at.% and the ions of MgO and Au were overlapping directly, which...
is a clear signature for artificial ion intermixing (Figure 3d–e). This motivated an analysis of the surface of the needle after APT, which revealed that Au nanoparticles were protruding from the surface of the needle. This can only be explained by the large difference in the evaporation rate between Au and MgO (Figure 3f). Finite element field evaporation simulations validated the experimental observations that indeed such artificial ion intermixing could occur if there are significant differences in evaporation field between the Au nanoparticles (evaporation field of Au is estimated to be 53 V/nm) and the MgO oxide matrix (evaporation field of MgO is estimated to be between 22 and 25 V/nm) (Figure 3g–h). Such insights can help improve the interpretation of APT data and help to better understand limitations of APT and current reconstruction algorithms, which will be crucial as the material systems analyzed by APT become highly heterogenous and complex.36,37

**Perspectives: Efforts to develop in situ combined instruments**

All the instruments involved for the correlative microscopy and techniques studies previously described are combined ex situ. More specifically, the sample is transported ex situ between the instruments prior investigation. Yet, over the last several years, there are efforts to develop in situ combined instruments.

Given the ultrahigh vacuum inside the APT analysis chamber, additional microscopy and spectroscopy methods can be integrated within the APT instrument as schematically shown in Figure 4. Specifically, the column of a scanning transmission electron microscope integrated within the APT analysis chamber can provide not only the initial needle specimen shape before APT experiments, but also provide information on how the specimen shape would evolve during experiment.38,39 Additionally, photoluminescence spectroscopy probes can be integrated with the laser system used in APT to obtain in situ optoelectronic properties of functional materials.23 Such efforts in the past to integrate an APT instrument with other microscopy and spectroscopy tools are discussed in more detail next.

A first example is the in situ combination of a TEM or an SEM column with an APT ion optics and detection system.38–40 One ambitious concept was put forth by Kelly et al.39 for the atom scope, which aimed to combine a transmission electron microscope with an APT sample and ion optics with a position-sensitive detector, all built within the same microscope. The schematic of the instrument proposed by Kelly et al.39 is given
Another notable advancement was in directly combining a 30 KeV STEM column on an APT by researchers at Colorado School of Mines. They demonstrated an ability to collect STEM images and diffraction patterns of APT specimens. The first realization of a combined TEM/STEM-APT instrument is the target of the TOMO project of the Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (ER-C) at Forschungszentrum Jülich. Project TOMO is part of the overarching project ER-C 2.0, which is funded by the German Ministry of Education and Research (BMBF). The instrument will be built by Thermo Fisher Scientific with Cameca as the partner for the APT components. The final TOMO instrument will become part of the user facility at the Ernst Ruska-Centre and will certainly offer new opportunities for both APT and TEM users.

A second example that is also described in detail within this issue of *MRS Bulletin* is the *in situ* combination of the photoluminescence spectroscopy with the APT. This setup was developed by the GPM Group at Rouen University and is called photonic atom probe. More details can be found in the next section as well as in the article “Correlative atom probe tomography and optical spectroscopy: An original gateway to materials science and nanoscale physics” by Di Russo and Rigutti in this issue.

**In this issue**

This issue of *MRS Bulletin* is dedicated to highlight many crucial contributions of correlative microscopy with APT to fields ranging from material synthesis/
processing–microstructure–property relationships and material degradation of structural and functional materials as well as understanding inherent complexities in extraterrestrial materials.

Sasaki et al. highlight specific contributions of correlative STEM and APT for analyzing solute segregation to interfaces in Fe–P–B–Cu soft magnetic alloys, (NdDy) FeB, and Sm(Fe0.8Co0.2)12B0.5 hard magnetic alloys, which in turn helped better understand the magnetic properties. In addition, they also discuss key contributions made by complementary STEM and APT for studying GP zone precipitates in Mg–0.5Ca–1.6Zn–0.3Zr alloy. One example of revealing solute segregation to dislocations in Mg–1.2Al–0.8Zn–0.5Mn–0.5Ca by directly correlative TEM–APT is also discussed. Finally, the value of correlative STEM–APT in revealing solute clustering in functional materials is discussed by using an example from Mg-implanted GaN.

McCarroll et al. discuss the value of correlative microscopy with APT for nanoscale isotopic and compositional analysis of precious samples from extraterrestrial sources such as sample return missions from the moon, comets, and asteroids. The application of APT for geological research and planetary research has been growing rapidly. Use of correlative microscopy with APT really allows a comprehensive understanding of these precious samples and the low sample volume needs for APT make it an ideal technique for analyzing these materials, as discussed by McCarroll et al. The procedure of sample preparation of such extraterrestrial materials for correlative microscopy is discussed in addition to key examples that revealed solute segregation to interfaces in lunar apatite. They close their article with a perspective on how the future developments in correlative microscopy and APT can make it an even more powerful method for robustly measuring composition, trace element composition, and isotopic variations, including those important for chronology.

Material degradation by corrosion is another key area where correlative microscopy with APT can make a significant impact. Rodenkirchen et al. discuss the contributions made by correlative microscopy and APT in the area of understanding high-temperature corrosion of Ni-based superalloys used in turbine engines. Specific examples of use of directly correlated STEM and APT in analyzing stress-corrosion cracks in steel are discussed. Complementary SEM, EBSD, SIMS, TEM, and APT for analysis of interfaces within the microstructure, solute segregation to dislocations, and compositional partitioning between the alloy microstructure and oxide scales are also discussed.

The study of phenomena taking place at surfaces and subsurfaces is key for electrocatalysts because the design of a reliable green hydrogen technology requires the understanding of subtle processing–structure–property relationships. Therefore, Gault et al. propose an innovative way of combining the APT with typical instruments used in surface science such as x-ray photoelectron spectroscopy (XPS) but also with electron-based spectroscopies such as electron energy-loss spectroscopy (EELS) in STEM. With this innovative concept, Gault et al. could study for the first time the oxygen isotope exchange experiment on iridium oxide during the oxygen evolution reaction within different sets of atomic planes that form the end surface of the needle. This study is a coming back to the original roots of APT (a surface science technique), but also provides a novel correlative concept necessary to tackle the challenges in electrocatalysis.

It is without any doubt that in situ combined instruments represent an important future development in materials science and, if successful, have big chances to result in a novel commercial instrument. This is the case for the photon atom probe (PAP) instrument that is not yet commercially available, but has opened new avenues in science for the study of nanoscale physical phenomena driven by field, stress, or sample shape. This PAP instrument has been developed by the GPM Group at Rouen University, France, under the guidance of Lorenzo Rigutti. The latest developments as well as the high-end sample characterization are explained in detail within this issue.

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Data availability
The data sets generated and analyzed during the current study are available from the corresponding authors on reasonable request.

Conflict of interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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