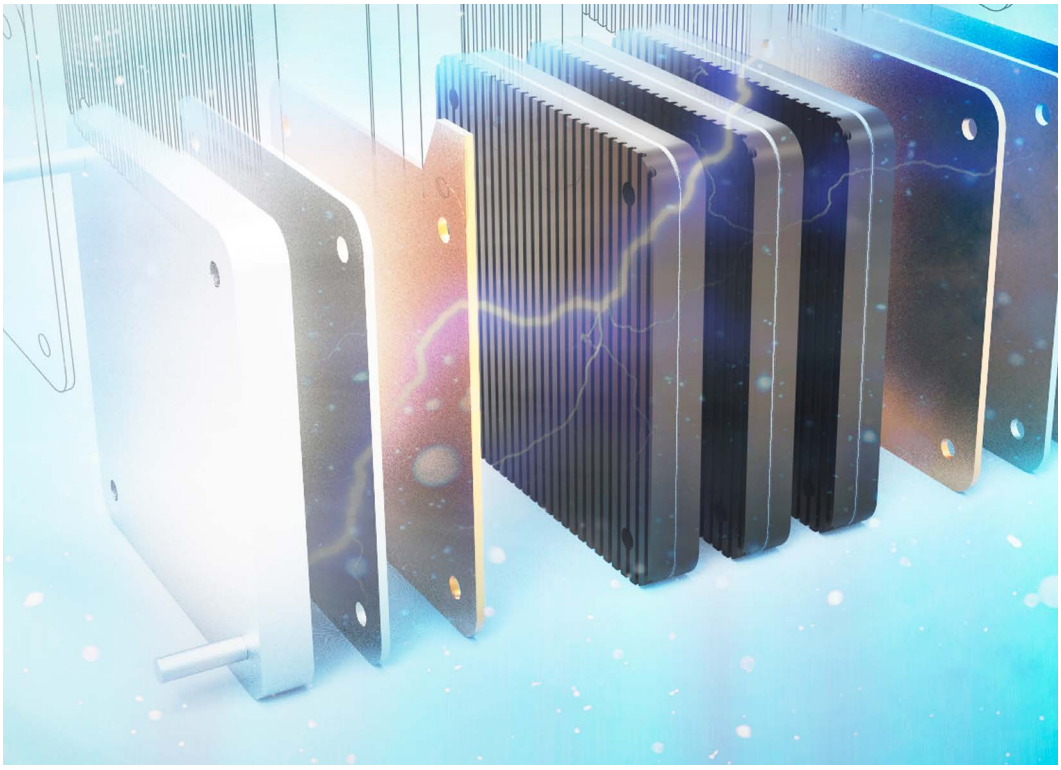
# NANO-CHARACTERIZATION AND THE FUTURE OF FUEL CELLS



## An overview of R&D opportunities using Atom Probe Tomography.

From hydrogen to novel SOFC ceramic materials.



According to experts, we're well on our way to using fuel cells to generate clean and efficient electrical power for a wide range of applications — from transportation to material handling to backup power. The growing focus on environmental issues, coupled with government initiatives and private-sector investment, are expected to drive the fuel cell market from USD 2.83 billion in 2019 to USD 7.12 billion by 2026. [1]

### Fuel cells

Fuel cells are devices that use a fuel source, often hydrogen, and an oxidant, often oxygen, to create electricity from an electrochemical process. A key advantage of fuel cells is their ability to operate at higher efficiencies than conventional combustion engines and convert chemical energy to electrical energy with efficiencies of up to 60%, according to the U.S. Department of Energy (DOE). [2]

Fuel cells are a preferred source not only for clean energy, but auxiliary and distributed power generation where grid power is unavailable, according to the DOE. Ty Prosa, senior research scientist and applications development team manager at CAMECA explained that many types of fuel cells exist that differ primarily by the kind of electrolyte they use. Many include those currently under development, each with its own applications and advantages.

Fuel-cell conversion processes typically involve hydrogen, which is expected to be a major energy carrier in future energy systems. The need to understand performance, durability and other barriers to fuel cell commercialization is critical for government,

**RESEARCHERS ARE USING APT TO BETTER UNDERSTAND HIGH-TEMPERATURE OXIDE MATERIALS AND CONNECTING MATERIALS, INCLUDING SPECIALTY STEELS USED TO CHAIN CELLS TOGETHER.**

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university and industry R&D, and demonstration and deployment.
This need has resulted in a surge in the nanoscale study of hydrogen distribution in materials and many other areas. Atom Probe Tomography (APT) is fast becoming the advanced microscopy technique of choice for providing 3D maps of atom arrangements within sub-micron volumes of matter. [3] (See sidebar for more on APT).

## APT R&D

Developers are exploring a variety of fuel cell technologies for new alternative energy systems. Many experts believe solid oxide fuel cells (SOFCs) are among the most promising systems due to their fuel flexibility, system efficiency and scalability.

A range of SOFC technologies are being developed, and APT applications for them are expected to be ideal depending on material pore variables such as density and size, CAMECA's Prosa, an APT specialist, said. "There are extensive reports about researchers using APT to better understand both high-temperature oxide materials and connecting materials, including specialty steels used to chain cells together." [4]-[10] Ceria and zirconia are SOFC ceramic materials that are among the most popular studied using APT, Prosa added. "APT can provide information on sub-micron phase characterization, analysis of grain boundaries, locations of dilute and mobile species at the surface, precipitate or cluster characterization and more."

"These are important measurements for understanding evolution, and the control of materials structure and properties for active fuel cell elements operating at high temperatures," he said. "When the APT specimen survivability is high, this can be a standard tool."

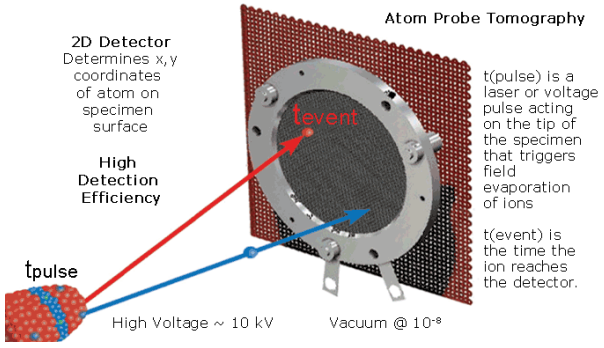
One excellent demonstration of APT SOFC characterization involves zirconia, using a model yttria-stabilized-zirconia (YSZ) system to make it easier for correlative APT and Transmission Electron Microscopy (TEM) analysis. (See figure.) "APT analysis is an ideal supplement to the STEM analyses when studying alloys and nanoscaled precipitates less than 10 nm, as the elemental distribution of all atomic species, namely, Ni, Zr, Y, and oxygen can be quantitatively determined with atomic resolution," stated J. Szász et al. [8]

### ATOM PROBE TOMOGRAPHY

Atom Probe Tomography (APT) is a powerful microscopy technique. It is the only method that offers both 3D imaging and chemical composition measurements at near-atomic scale (about 0.1-0.3nm resolution in depth and 0.3-0.5nm laterally).

APT has contributed to major advances in materials science, from alloy design to semiconductor analysis to fuel cell characterization. The CAMECA APT product line comprises the LEAP 5000 and EIKOS families.

Since the turn of the century, the user base of APT has expanded from a handful of researchers to thousands — and growing. The surge in publications featuring atom probe work is testament to its increasing popularity. For more, see CAMECA, "APT Tutorial." [16]






*The LEAP® 5000XR is CAMECA's atom probe microscope with unmatched 3D sub-nanometer analytical performance. The instrument contributes to R&D across a wide range of applications — from metals to semiconductors to interconnect materials. Its detection efficiency is more than 40% extra atoms per nm3 analyzed.*

J. Szász et al. added that a reverse-current treatment procedure was used to increase triple-phase boundary densities between YSZ, Ni grains and pores, i.e., hydrogen. The process induced a layer of nanoscaled Ni, YSZ and pores, and increases anode performance by 40%.
From an APT application perspective, this process is compatible with the porous YSZ structure because the pores are substantially filled in with Ni. This clearly improves the structural integrity for survivability.

"The APT characterization of hydrogen-oxygen fuel cell materials provides key 3D chemical information at near-atomic scale. Precise measurement of sub-micron volumes and nanoscale features in 3D is the hallmark of the technique," Prosa said.

## Conclusion

Research has long been underway in areas such as the study of separation membranes and catalysis, which is essential to manufacturing processes. [11], [12] APT also shows potential for providing near-atomic chemistry information for photoelectrode development. [13]

Other R&D has focused on methods for analyzing carbon-nanospheres, which are materials with great potential for energy development [14]. APT also has been used to help understand a magnesium alloy and hydrogen storage, a necessity for some fuel cell technologies. [15]

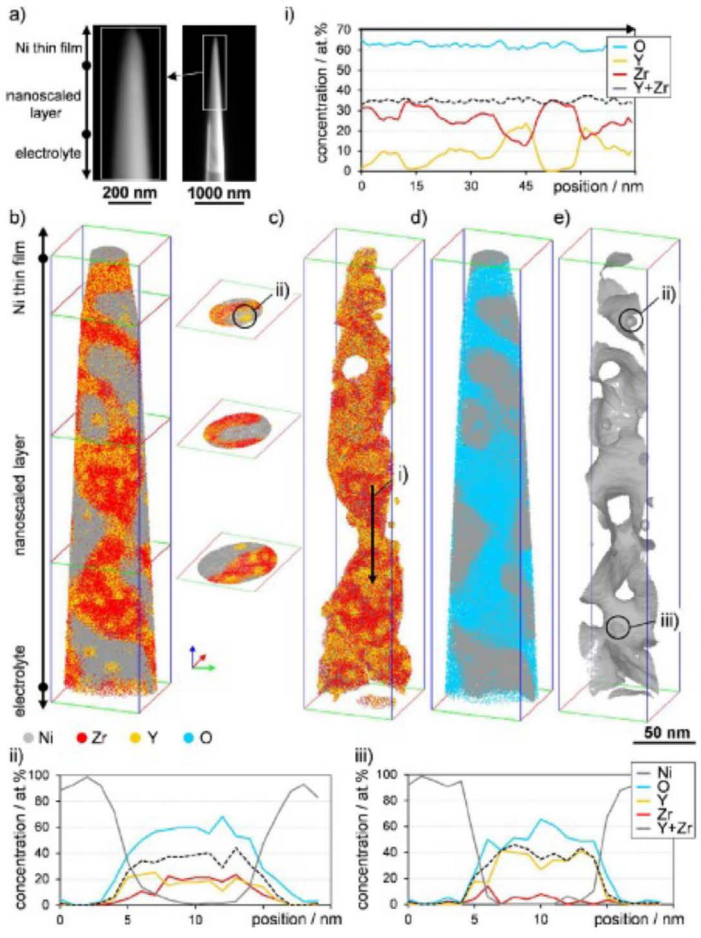



*Figure showing randomized controlled trial process and resulting APT data: (a) Needle-shaped sample tip (left) with the part analyzed by APT with a volume of 830,531 nm3. (b)–(d) Elemental atom mappings of (b) Ni (gray), Zr (red), Y (yellow), with disk-shaped cutouts showing Y-rich precipitates embedded in the Ni phase with (ii) and (iii) exemplary concentration profiles at positions, encircled in (e). (c) The (Y, Zr)-oxide (i) with the concentration profile of Y, Zr and O inside a cylinder indicated by the black arrow in (c). (d) Ni, O (blue) signal. (e) The interconnected isosurface at 40 at. % Ni.* [8]

**APT IS FAST BECOMING A KEY TECHNIQUE IN THE SOLID OXIDE FUEL CELL NANO-CHARACTERIZATION TOOLKIT.**

After years of APT analysis of steels, it's been clearly demonstrated that the technique is ideal for grain-boundary, phase-boundary, and precipitate analysis in SOFC interconnect materials development, Prosa said.
With specimen preparation procedures similar to TEM, the measurement of elemental migration through the microstructure during cell lifetime is possible with APT, according to experts.
For many demonstrated reasons, "APT is fast becoming a key technique in the solid oxide fuel cell nano-characterization toolkit," Prosa said.

CAMECA is a global supplier of microanalytical and metrology instrumentation for research and process control. CAMECA, a business unit of AMETEK, provides a diverse range of advanced characterization technologies that measure elemental and isotopic composition in materials down to atomic resolution, and equip government, academic and high-tech industry labs around the world.

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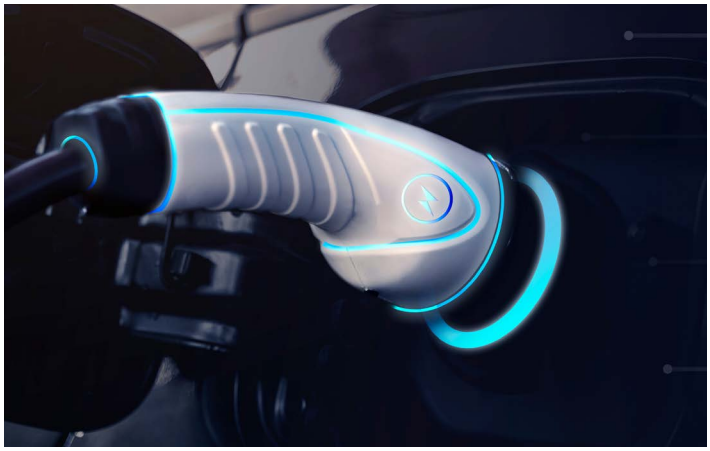
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