

# INTERVIEW

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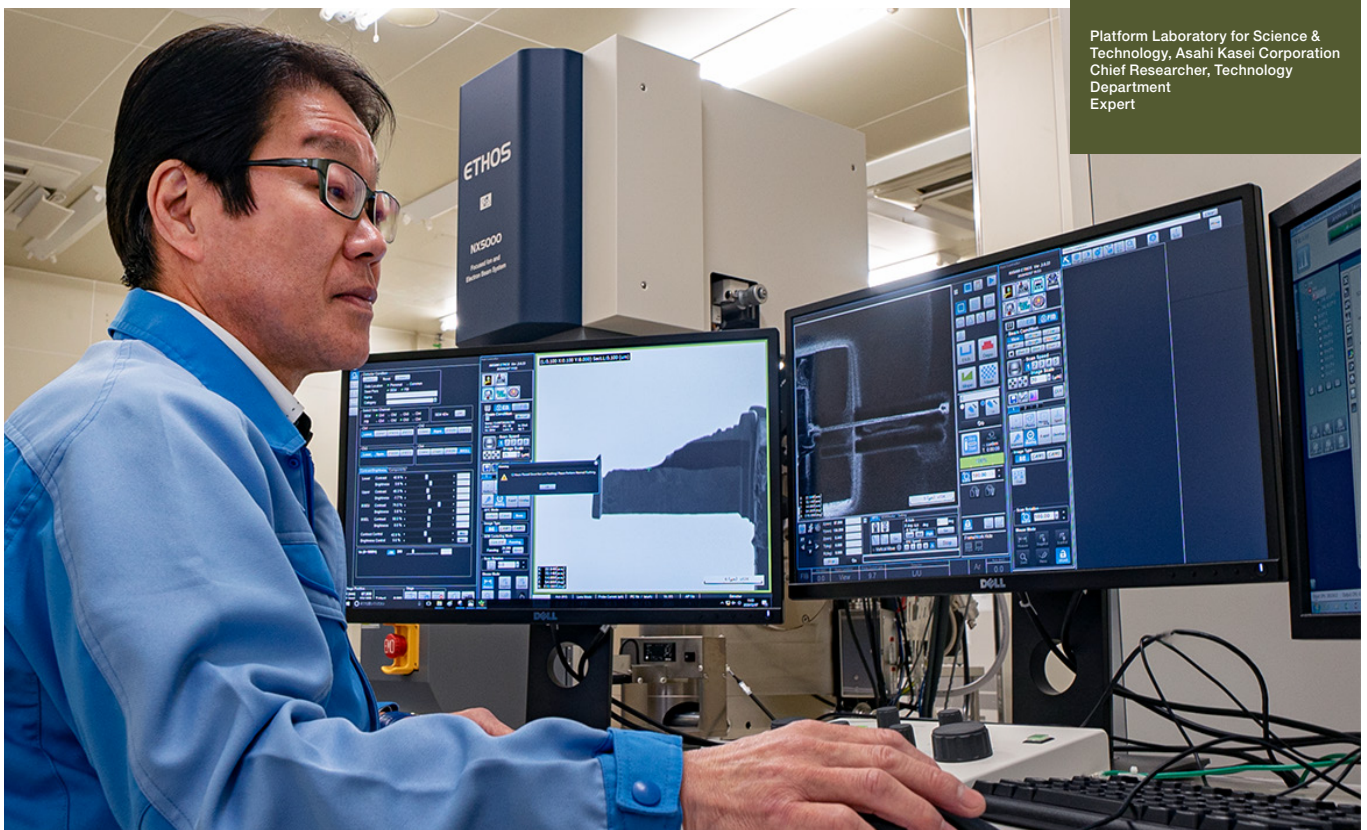
In October 2019, Asahi Kasei Corporation Honorary Fellow Dr. Akira Yoshino won the Nobel Prize in Chemistry. Dr. Yoshino was awarded for his achievements in perfecting the basic principles of lithium-ion batteries (LIBs), which are now so indispensable to our everyday life. Hirohide Otobe, Expert at Asahi Kasei's Platform Laboratory for Science & Technology, was one of those who supported the Yoshino team in advancing the practicality of LIBs. This time, I visited Otobe to talk to him about the role of electron microscopes in Asahi Kasei product development over his nearly 30 years supporting company research and development.

Developing Various Observation Methods to Solve In-House Developer Challenges

## Supporting Development by Making the Unseen Visible

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# 1. In Pursuit of a Practical Lithium-Ion Battery

LIB research began in 1981 when Dr. Yoshino started researching polyacetylene, a conductive polymer. Yoshino found that polyacetylene could be used as an electrode for rechargeable batteries and moved forward with research on negative electrodes in particular. Then, in 1985, Yoshino's team switched to lithium cobaltate (a metal oxide with lithium ions) for the positive electrode and from polyacetylene to carbon-based material for the negative electrode, completing the basic structure of the world's first lithium-ion battery (LIB).

There were still many challenges left before LIBs would reach the market, however. One that was considered the most important was safety measures to prevent issues such as ignition. For LIBs to be mounted in green vehicles and other mobility applications in addition to mobile IT devices, they would have to be kept from igniting or exploding in the event of an impact.

Positioned between the LIB positive and negative electrodes, a separator serves both to allow lithium ions to

flow between the two electrodes and to prevent short circuits. For the separator, Asahi Kasei used a polyolefin microporous membrane with a thickness of 20  $\mu\text{m}$  or less. In the case of a thermal runaway in the LIB, the separator melts to fill the micropores and works as a fuse to stop battery function. Asahi Kasei was the first company in the world to mass-produce the separator, establishing it as the de-facto standard. This technology was important in ensuring the safety of LIBs and gaining social acceptance. Demonstrating his skills in structural analysis and evaluation of these separators was none other than Otobe.

Otobe reflects on his experiences at the time. "We analyzed the 3D structure of the separator using a FIB-SEM (focused ion beam (FIB) scanning electron microscope (SEM)), and measured the diffusion of lithium ions using nuclear magnetic resonance (NMR) spectroscopy. We ran simulations based on the results. What was particularly memorable for me in separator development was examining what structures led to the best battery properties."

# 2. Technology Built on the Achievements of Their Predecessors

While ion beams are capable of performing multi-slicing of porous material, cross-sectional 2D images are not possible as depth is visible with SEM. Without a 2D binary image, the 3D structure cannot be reconstructed. Therefore, Otobe's team pre-filled the porous holes with resin. However, just filling the holes was not enough to achieve sufficient contrast. "I kept wondering what I could do to get a complete 2D binary image, and what I came up with was electronic staining. I think the point of that work was to apply the company's know-how regarding electronic staining."

Thus, using FIB-SEM, Otobe's team performed repeated slicing and observation to reconstruct the structure from the large number of SEM images produced. By doing so, they were the first in Japan to successfully visualize the 3D structure of a separator.

They presented the results at the Electrochemical Society

of Japan's Battery Symposium in Nagoya. The scale of the impact was apparent when Otobe finished his lecture: attendees from companies in the analytical sector were lined up to trade business cards with him.

Otobe remains modest. "Again, it was all due to staining technology that our company has been working on for the last 30 to 40 years. We inherited generations of know-how built on the efforts of our pioneering predecessors working with microscopy and NMR. The way I see it, we achieved what we did by repeatedly taking on new challenges on the backs of their efforts."

Currently, the company manufactures Hipore as wet membrane separators and Celgard as dry membrane separators, both with greatly improved battery safety. Still, they make daily progress on their separators, making them stronger and thinner in pursuit of higher energy density.





## 6. Using AI and MI in Pursuit of the Ideal Structure

For my closing question, I asked Otake about the future of PLST. “Our goal is to propose an ideal structure to the developer. In pursuit of that goal, we look to leverage AI technology and MI (materials informatics) based on Big Data to calculate parameters that demonstrate a correlation between the 3D structure and features of the products. Developers are always chasing more functionality. From an analysis standpoint, we always wind up probing for what the developers want, asking with every proposal, ‘Is this it?’” I also think handing the developer models printed on a 3D printer would be an improvement.”

In efforts to accelerate and streamline technological development, things are expected to revolve more and more around data science. However, in Otake’s opinion, data are just numerical values. “Computational chemistry certainly is a larger factor than before; this is true. However, proposals will only come to life when computational chemistry is tied into the actual product.” He closes emphatically: “There’s absolutely no way to get around detailed observations of fine-grained structure in real space.”

(Interview/article: Toshinari Yamaguchi)



### Editor’s Notes

Throughout the interview, Otake repeatedly described microscopy as “fun.” It’s easy to imagine the joys of that first time you think, test, and then get to actually observe something that no one else has ever seen. I asked Otake about his current challenges. “Every day we’re expected to produce ever finer details. That’s the everyday struggle. I’d love to reconstruct 3D images of nanoscale polymer materials at resolutions smaller than 10 nm.” In terms of batteries, Otake is also interested in operando measurements performed in the actual operating environment. “I wonder how we can observe a battery with the positive and negative electrodes, separator, and electrolytic solution all in place.” Apparently, his intellectual curiosity knows no bounds. Solutions obtained using electron microscopes will revolutionize our society. It is my sincere hope that the technological innovations of Otake’s team will improve people’s lives someday.

