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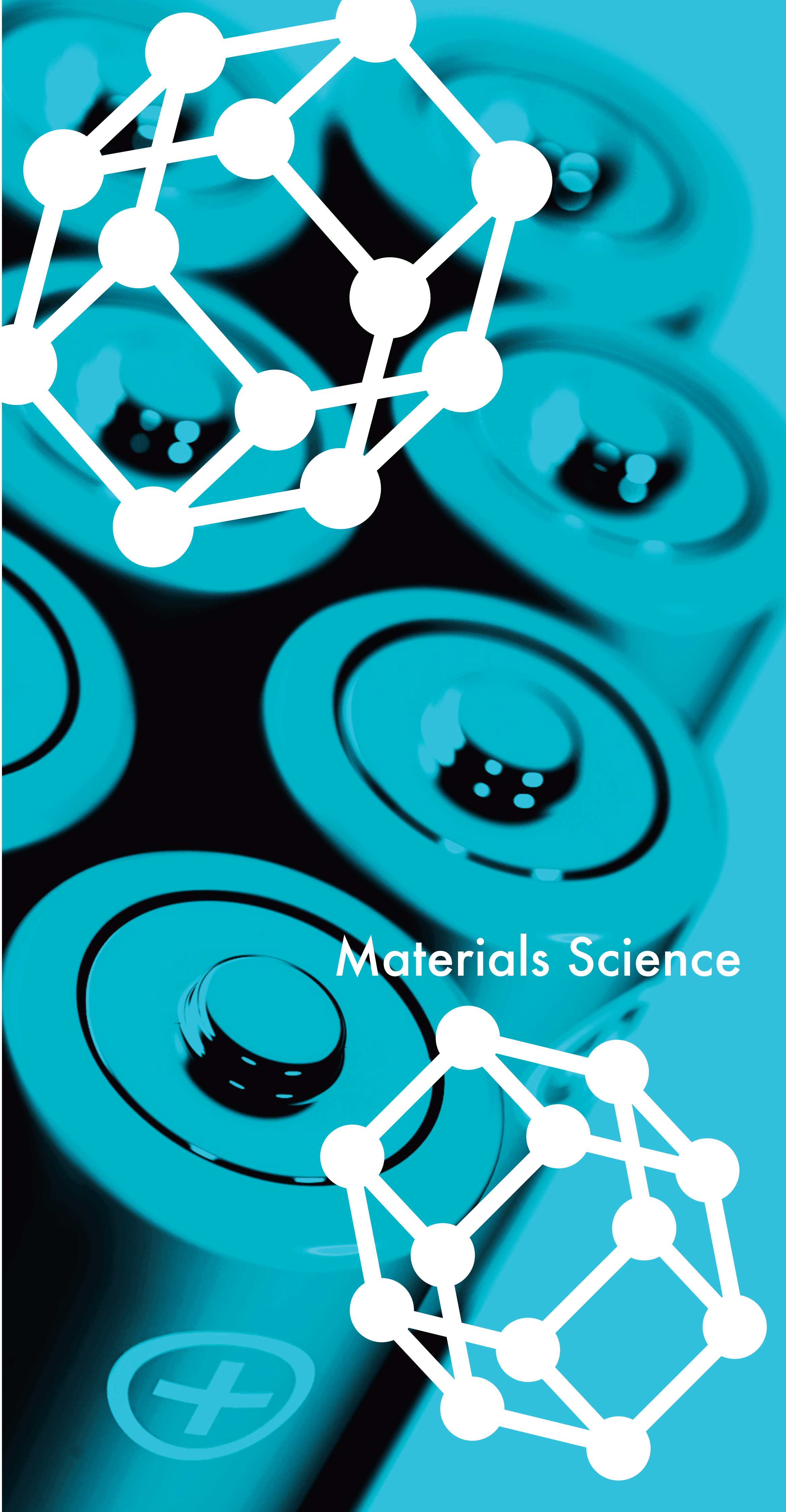
Shimadzu Journal



Vol 10
ISSUE1

SJ23_0101 ISSN 2188-0484

Dear Readers,

Yoshiaki Mase
General Manager,
Analytical & Measurement Instruments Division

As a company working with partners worldwide, Shimadzu aims to create solutions that address societal issues and promote the implementation of these solutions in society. Along with the ongoing effects of COVID-19, the global economy remains uncertain due to soaring raw material prices and shortages of materials, as well as the protracted situation in Russia and Ukraine. As the movement of people and goods changes worldwide and interest in, and commitment to, the global environment grows, the fields in which analytical and measuring instruments are used and how they are used are changing.

Amid these dramatic societal changes, this issue features a special section on "Materials Science," which is at the root of every industry in the world. Focusing particularly on lithium-ion batteries (LiBs), which continue to evolve toward a sustainable society, we introduce researchers working on the front lines and highlight applications that make full use of cutting-edge technologies.

First, Dr. Sascha Nowak, head of the Analytics & Environment division of the MEET battery research center in Münster, talks about qualitative and quantitative evaluation of electrolyte aging, verification of recycling methods, toxicological testing, and other topics. Next, Mr. Keiji Sumiya of Showa Denko Materials Co., Ltd. speaks about the importance of visualization technology in the development of LiB materials and his company's initiatives in this area.

In addition to the interviews, we introduce applications on analyzing LiBs from different angles using our latest technologies. These include examples of chemical state analysis of cathode active materials using new wavelength-dispersive X-ray fluorescence spectrometers, measurement of electrolyte solutions using FT-IR spectroscopy, nondestructive observation of the internal structure of cylindrical LiBs using X-ray CT, and observation of small areas of electrode materials using SPM and evaluation of their physical properties. In addition, we introduce some recent initiatives related to material science, our new organizational structure, a new center for promoting open innovation, and a unique new product (MIV-X ultrasonic optical flaw detector).

Despite society changes and challenges, Shimadzu is realizing its corporate philosophy of "Contributing to Society through Science and Technology," embracing change rather than fearing it, and creating an even better society. We look forward to working with you on innovative research and challenging projects and hope that this journal will provide you with inspiration for your own work.

Yours Sincerely,

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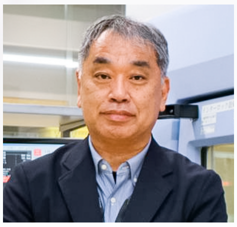


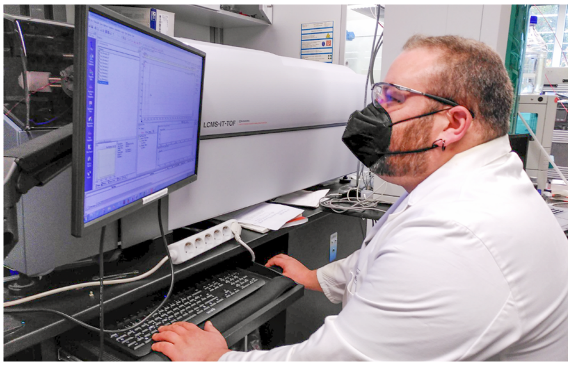
Mr. Keiji Sumiya

Interview 1 Interview with Dr. Sascha Nowak

"Very reliable, professional and friendly."

We interviewed Dr. Sascha Nowak, the head of the division Analytics & Environment of the MEET battery research center in Münster. Münster Electrochemical Energy Technology (MEET) at the University of Münster is one of the foremost battery research center in Germany.

Dr. Sascha Nowak, thank you very much for spending some time for this interview. At first, could you outline the research and let us know what discovery and achievement have been made so far?



We focus on (electrolyte) aging, transition metal migration and surface investigations, recycling and 2nd life as well as toxicological aspects with regard to working safety. With many newly developed methods, we could discover and verify aging products and mechanisms. Furthermore, all applied methods were transferred to the recycling of lithium-ion batteries to ensure the identification of the recycling components and quality control during the recycling processes.

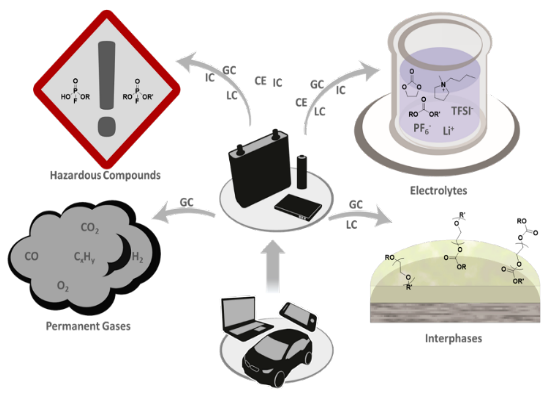
Could you introduce the research that has been particularly successful among your many advanced researches?

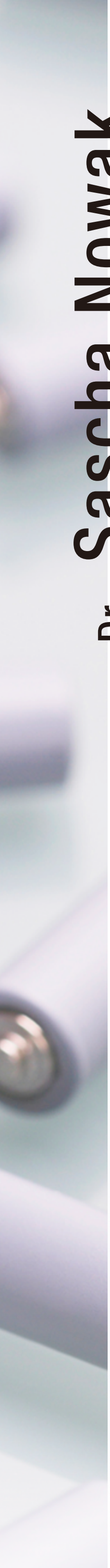
The competence field electrolyte aging characterizes electrolytes in their various states of operation. Aging effects are observed in terms of qualitative and quantitative investigation using different analytical methods. This also includes the synthesis of substances as standards that are not commercially available. In addition, recycling methods like extracting the electrolytes from cells are analytically developed and validated. Supercritical gases with and without the addition of solvents are being tested for the extraction of electrolyte components from spend batteries. The extraction is carried out either in an autoclave or in commercially available extraction units. To characterize the electrolyte, a wide variety of methods that focus on identification and quantification of the different degradation compounds are used. Reaction schemes are concluded from these measurements that provide a deeper understanding of the manifold processes taking place inside a battery.

Furthermore, migration of metals into the electrolyte and deposition on the electrodes is investigated. Here, the focus is not only to determine total amounts, but also to examine species of migrated metals and the factors influencing this process. This helps to combine electrolyte degradation mechanisms with the aging processes of electrodes.

The methods used in electrolyte aging to identify and quantify the aging/decomposition products are also applied to evaluate and determine the risk given by hazardous substances in cooperation with toxicologists. This also includes an overall evaluation of the electrolyte to meet safety criteria.

Another focus of toxicological examination is the investigation of degradation products that may be formed during /origin from the extraction, processing and further storage of recycled material. These are analyzed for their possible toxicity. The results are then included in an evaluation of the entire recycling process and of the individual steps in this process with regard to their specific potential risk.





Dr. Sascha Nowak

How are Shimadzu instruments helping you in your research?

We are mainly using GC instruments with different detectors (TCD, BID, MS and FID) and sampling possibilities like HS and SPME [cf. Leißing et al, (2020)]. The analysis of the electrolyte is the focus with regard to the aging mechanisms. Additionally, we implemented a pyrolysis system to investigate high boiling compounds like binder and separators [cf. Stenzel et al, (2019)]. As a second step, we added a LCMS-IT-TOF™ to our portfolio, which further helped us broadening our application range [cf. Henschel et al, (2019a)].

Could you tell us why you chose Shimadzu as your partner when you started this project?

We got some recommendations and the possibility to use a fully equipped robotic autosampler on an GC instrument having different detectors installed in one setup was helpful in the beginning of our research. For example, we had the opportunity to include several detectors like FID and MS with a two-column setup, AOC-5000 autosampler and an optic injector as additional inlet system in one GC setup.

Furthermore, the robustness of the systems was quite impressive with regard to our application, since we handle quite complex and difficult samples, including a highly concentrated fluorinated salt, which can release HF and other fluorinated compounds.

What are Shimadzu's strengths compared to other vendors?

Since the application was quite new, even unusual request with regard to instrumental setups could be discussed and realized which really helped us to advance further with our methods and understanding.

Finally, could you share any requests that you have with respect to analytical and measuring instrument vendors?

In general, quantitative information is always needed. However, since standard and reference materials are not available so far, speciation is the method of choice now. Therefore, further advancements in this area, whether automated coupling or robust and interference-free detection are welcome.

It was significant to know what you think of us and our collaboration. We will strive to meet your expectation more than ever. Thank you very much.

Dr. Sascha Nowak

Recent publications using Shimadzu instruments

Henschel J, Schwarz L, Glorius F, Winter M, Nowak S. 2019a. 'Further insights into structural diversity of phosphorus-based decomposition products in lithium ion battery electrolytes via liquid chromatographic techniques hyphenated to ion trap - time of flight mass spectrometry.' Analytical Chemistry 91, Nr. 6: 3980-3988. doi: 10.1021/acs.analchem.8b05229.

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Henschel J, Peschel C, Günter F, Reinhart G, Winter M, Nowak S. 2019c. 'Reaction Product Analysis of the Most Active "Inactive" Material in Lithium-Ion Batteries—The Electrolyte. II: Battery Operation and Additive Impact.' Chemistry of Materials 24: 9977-9983. doi: 10.1021/acs.chemmater.9b04135.

Horsthemke F, Friesen A, Ibing L, Klein S, Winter M, Nowak S. 2019. 'Possible Carbon-Carbon Bond Formation During Decomposition? Characterization and Identification of New Decomposition Products in Lithium Ion Battery Electrolytes by Means of SPME-GC-MS.' Electrochimica Acta 295: 401-409. doi: 10.1016/j.electacta.2018.08.159.

Stenzel YP, Börner M, Preibisch Y, Winter M, Nowak S. 2019. 'Thermal profiling of lithium ion battery electrodes at different states of charge and aging conditions.' Journal of Power Sources 433: 226709. doi: 10.1016/j.jpowsour.2019.226709.

Peschel C, Horsthemke F, Leißing M, Wiemers-Meyer S, Henschel J, Winter M, Nowak S. 2020. 'Analysis of carbonate decomposition during the interphase formation in isotope labeled lithium ion battery electrolytes – Extending the knowledge about electrolyte soluble species.' Batteries & Supercaps 3, Nr. 11: 1183-1192. doi: 10.1002/batt.202000170.

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Henschel J, Peschel C, Klein S, Horsthemke F, Winter M, Nowak S. 2020. 'Clarification of decomposition pathways in a state-of-the-art lithium ion battery electrolyte through 13C-labeling of electrolyte components.' Angewandte Chemie International Edition 59, Nr. 15: 6128-6137. doi: 10.1002/anie.202000727.

Henschel J, Horsthemke F, Stenzel YP, Evertz M, Girod S, Lürenbaum C, Kösters K, Wiemers-Meyer S, Winter M, Nowak S weniger. 2020. 'Lithium ion battery electrolyte degradation of field-tested electric vehicle battery cells - A comprehensive analytical study.' Journal of Power Sources 447: 227370. doi: 10.1016/j.jpowsour.2019.227370.

Leißing M, Horsthemke F, Wiemers-Meyer S, Winter M, Niehoff P, Nowak S. 2021. 'The Impact of the C-rate on Gassing during Formation of NMC622 II Graphite Lithium Ion Battery Cells.' Batteries & Supercaps 4, Nr. 6: 1344-1350. doi: 10.1002/batt.202100056.

Leißing M, Peschel C, Horsthemke F, Wiemers-Meyer S, Winter M, Nowak S. 2021. 'The origin of gaseous decomposition products formed during SEI formation analyzed by isotope labeling in lithium ion battery electrolytes.' Batteries & Supercaps 4, Nr. 11: 1731-1738. doi: 10.1002/batt.202100208.

Kösters K, Henschel J, Winter M, Nowak S. 2021. 'Development of a fast online sample preparation for speciation analysis of lithium ion battery electrolyte decomposition products by liquid chromatography hyphenated to ion trap-time of flight-mass spectrometry and to inductively coupled plasma-sector field-mass spectrometry.' Journal of Chromatography A 1658: 462594. doi: 10.1016/j.chroma.2021.462594.

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Dr. Sascha Nowak

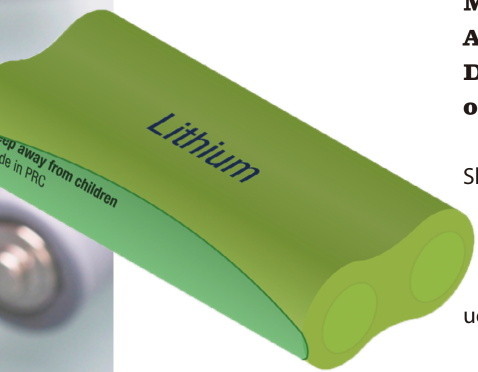
▶ Sascha Nowak studied chemistry at the University of Münster and got his PhD in Analytical Chemistry. After his doctorate, he joined the working group of Prof. Winter at the MEET Battery Research Center in 2009 as a postdoctoral researcher where he established the analytical department. From 2010-12 he was the head of the competence areas Analytics and Recycling and since 2012 he holds a position as scientific staff at the MEET Battery Research Center at Münster University as the head of the division Analytics and Environment, which mainly focuses on electrolyte aging, transition metal migration and surface investigations, recycling and 2nd life as well as toxicological investigations.

Interview 2 Interview with Mr. Keiji Sumiya

"Understanding and trusting relationships based on the idea of contributing to each other"

We interviewed Keiji Sumiya from the Japanese chemical manufacturer Showa Denko Materials Co., Ltd. Showa Denko Materials is a global company that offers one-stop solutions for a variety of challenges in fields ranging from advanced materials to technical services. For many years, Shimadzu Corporation has provided support for all sorts of analytical processes involved in R&D work at Showa Denko Materials.

Mr. Sumiya, thank you very much for spending some time for this interview. At first, would you give us a brief overview of the main businesses of Showa Denko Materials, the mission of your organization, and your personal area of expertise?



Showa Denko Materials is a Japanese chemical manufacturer of semiconductor and electronics-related materials, mobility materials, functionally engineered polymer materials, and life sciences-related products. The company was originally a chemical products division of Hitachi, Ltd. It was spun off in 1962 to establish the former Hitachi Chemical Co., Ltd. Throughout our long history, we have continued to develop and release advanced functionally engineered materials in various fields.

I currently work in the analysis department, where we use sophisticated analytical technologies to fulfill our mission of providing strong support for product development and intellectual property strategies for functionally engineered materials.

My specialty is in inorganic materials (crystalline and battery materials) and corresponding data analysis. In the past, I was engaged in developing detector elements for PET cancer imaging systems and worked with the National Institute for Materials Science (NIMS) on joint research to develop a fully solid-state QPM device. That project resulted in successfully developing the world's first fluoride SHG short wavelength laser oscillator. Later, in 2009, I was involved in a project to develop materials for rechargeable lithium-ion batteries (LiB) used in electric vehicles, where I pioneered technologies for identifying and visualizing functions and mechanisms that were critical for material development. Currently, I am

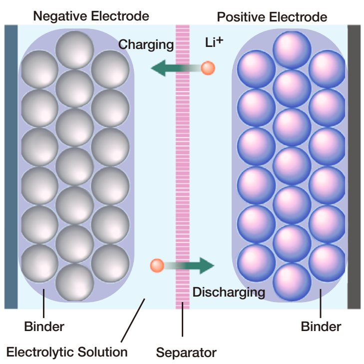
Mr. Keiji Sumiya

using my experience from analyzing such a wide variety of materials to consider battery fields from a broad perspective.

In your diverse development experience, what are some of the key points for your team from pioneering such innovative new technologies for LiB material development?

To overcome analytical challenges in developing LiB materials, we have been focusing on visualizing the 2D and 3D distribution of compounds. LiBs have a relatively simple structure, but they still involve many factors that need clarification. Basically, LiBs are a combination of positive electrodes (inorganic powder), negative electrodes (inorganic powder), separators (organic membranes), and electrolyte solution (organic liquid); consequently, the components of LiBs consist of mixed materials: solid, liquid, inorganic and organic.

Cell Cross Section



In particular, the powders in positive and negative electrodes can be formed into any shape to control their specific surface area, but a binder (a non-conductive organic material) is required to form the layered electrode structure. The surface and interface status of these binders and particles have a major effect on battery properties and are key factors in battery development.

What factors make it difficult to analyze batteries in such a simple structure?

The main positive- and negative-electrode materials in LiBs consist of particles that are 10 to 20 µm in diameter and form a bumpy surface layer. LiBs are very difficult to analyze due to a charge-discharge reaction affected by a complex interaction between electrical, structural, chemical reaction, and other factors, and internal materials that change properties if the battery is unsealed and the materials are exposed to oxygen, for example.

Furthermore, given that batteries were invented about 210 years ago and practical applications developed about 160 years ago, they have a longer history than even analytical instruments. Due to high reproducibility, batteries can be made with almost identical properties by manufacturing them with the same conditions. Their relatively high reliability means battery R&D and product development can be accomplished to some extent without sophisticated analytical instruments. Considering the history and background of battery technology in combination with a general tendency to be not overly dependent on analysis, it is difficult to claim that progress in battery R&D and product development cannot be achieved without an extensive assortment of analytical instruments.

Despite that historical background, why does Showa Denko Materials actively continue battery R&D based on analysis?

A wide variety of batteries are being developed by manufacturers around the world, but even if a new battery technology is established, it is difficult to achieve widespread adoption unless the technology is broadly recognized. The current popularity of LiBs was triggered by their use in mobile phones, without which the current widespread adoption might never have occurred. Even if great products are created, unless they are used by manufacturers, many of these battery products will disappear from the marketplace due to low awareness. In those circumstances, analytical technologies for visualizing the chemical reactions occurring throughout a sealed battery provide development engineers an important tool for verifying that specific reactions are actually occurring inside the battery. At the same time, visualization technology provides peace of mind and confidence to battery users (by verifying functionality) and also serves as a tool for communication between battery developers and selectors.

Mr. Keiji Sumiya

We heard that you have a unique story about your promotion for broader acceptance of battery analysis…?

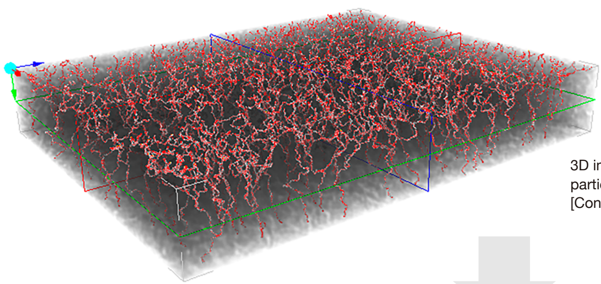
At the time, I was wondering how I could explain the benefits of battery analysis to a manager responsible for coordinating development but who put little focus on battery analysis. Then, I consolidated the key points of combined multifaceted analytical data onto both sides of an A3 size piece of paper and left it on his desk without saying anything. That was the trigger that flipped the development coordinator's attitude from "We do not need analysis" to "Wow! That is really amazing! If I gather everyone together, would you give a presentation today?" After that, we gave similar presentations to customers, resulting in reactions such as "We have never seen data like this before!" It converted the images that battery developers had only been imagining in their minds into visual data they could actually see. Rather than simply analyzing batteries, it provided the opportunity to build trust by presenting analytical data based on anticipating customer expectations 2 or 3 steps ahead and generating reactions such as "We had no idea you had thought so deeply about it."

How have Shimadzu analytical and measuring instruments been useful for that visualization process?

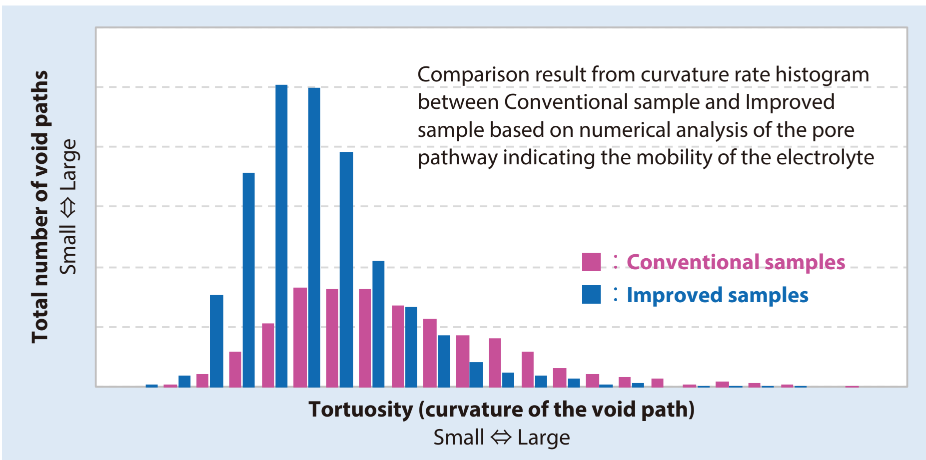
Our company uses a variety of Shimadzu instruments, such as X-ray CT, SPM, LC, GPC, and GC-MS systems, depending on the analysis target and the purpose.

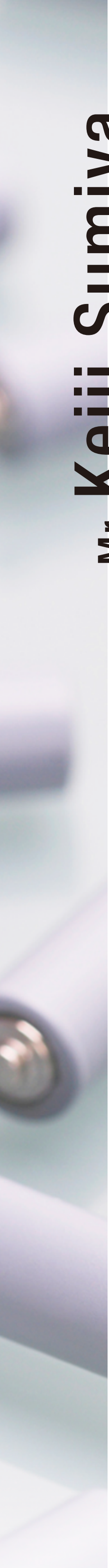
For example, Shimadzu instruments are used to analyze both the negative-electrode structure and reactions at the surface of negative-electrode materials, which have a major effect on battery performance.

A Shimadzu X-ray CT inspection system is typically used for structural analysis of negative electrodes. Improving performance requires controlling the voids between particles, which function as the pathways for lithium ion movement. Since the X-ray CT inspection system enables non-destructive observation of electrode structures, the electrolyte penetration pathways can be visualized by identifying the voids formed between negative-electrode material particles. Visualizing the voids to compare how pathways are connected in a product being developed versus a standard product resulted in determining that development samples with superior performance have more pathways and lower pathway curvature.



3D image visualizing the pore pathway among anode material particles based on the X-ray CT image analysis
[Consideration of electrolyte movement from void pathway]





Mr. Keiji Sumiya

A Shimadzu scanning probe microscope (SPM) with a variety of measurement modes is typically used to analyze negative-electrode material surfaces by a combination of multiple methods. The LiB charging and discharging process forms a layer of reaction products, called solid electrolyte interface (SEI), on negative-electrode material surfaces that can affect battery performance. To analyze the factors that influence the effectiveness of heat treatment on the negative-electrode materials intended to improve battery performance, standard and heat-treated samples were compared. The results visually showed a combination of information, including that the heat-treated samples tended to have a thinner and smoother SEI layer, the presence of hard precipitates visible in phase-contrast images, and differences in surface potential values. Such combined information is considered difficult to obtain with SEM or TEM systems.

Those analytical results enabled understanding the connection between battery properties, the reaction state and electrode structure of negative-electrode materials, and material parameters, which contributed to a major improvement in battery properties and differentiating our products from competitors.

People tend to assume "visualization" refers to simply observation, but it has also enabled new imaging methods not possible by regular observation, such as visualizing the hardness properties, electric potential, material composition, distribution of chemical reactions, or internal structures, so that entire phenomena can be evaluated from multiple perspectives.

What do you think are Shimadzu's strengths compared to other analytical instrument manufacturers?

The fact that Shimadzu offers such a broad range of analytical instruments. it is quite rare for an analytical instrument manufacturer to offer such a diverse line of instruments and Shimadzu has a completely different market presence than other broad-line instrument manufacturers. Showa Denko Materials is organized based on having "analytical generalists" that can operate a variety of instruments depending on the given objectives and giving each analyst the freedom to use all the instruments. That means analysts can personally analyze phenomena from multiple perspectives. Shimadzu can offer total solutions for comprehensive analysis based on a variety of approaches. Another attractive aspect of Shimadzu is its ability to propose new products or other solutions from a broad perspective, rather than from a particular specialized field, which has stimulated us as well. In addition, Shimadzu responds efficiently to questions or requests for advice. This spirit of seeking mutual understanding and making a contribution during that interaction process has resulted in a relationship of trust.

Thank you again for your time today. Finally, could you share any requests that you have with respect to Shimadzu or analytical and measuring instruments manufacturers.

I want Shimadzu to offer solutions that only Shimadzu can offer, but from different perspectives than others. If Shimadzu intends to further capitalize on its strength as a broad-line analytical instrument manufacturer, I sincerely hope you realize that goal. For example, I think it would be great if Shimadzu offered a variety of analytical instruments that could be interlinked.

Mr. Keiji Sumiya

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- Captures sharp images with optical microscopy to SPM microscopy modes

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- High-throughput observation and fast physical property mapping

Learn more

▶Keiji Sumiya is General Manager of Materials Analysis, Advanced Technology Research and Development Center, Innovation Promotion Headquarters, Showa Denko Materials Co., Ltd. Since joining the company, he has been engaged in research and development of functional materials (about 17 years) and material function analysis (about 18 years). The products involved include semiconductor die-bond materials, radiation detection elements (scintillators) for cancer detection PET, and negative electrode materials for Li-ion batteries for EVs. Since 2013, as the head of the Materials Analysis Division, he has vigorously promoted business contributions through support analysis of all R&D divisions of the company. The Materials Analysis Division takes a pivotal role in developing the functional materials products in Showa Denko Materials' diverse group.

Showa Denko K.K. (SDK) (Tokyo: 4004) announces its integration with Showa Denko Materials Co., Ltd. (SDMC) effective on January 1, 2023 to establish “Resonac”.

More information: Showa Denko Press release

Materials Science

Polychromatic Simultaneous WDXRF Spectrometer — Application to Valence Analysis of Cathode Materials of Lithium-Ion Batteries

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[1]X-ray/Surface Analysis Business Unit, Analytical & Measuring Instruments Division, Shimadzu Corporation
[2]Spectroscopic Analysis Group, Advanced Analysis Unit, Technology Research Laboratory, Shimadzu Corporation

Abstract A polychromatic simultaneous wavelength-dispersive Xray fluorescence (PS-WDXRF) spectrometer has an ability to measure the valence changes of 3d transition metals with high precision in the laboratory. Adjustment and maintenance of the drive mechanism are unnecessary, and high-precision measurements are possible in a short time because the optical system has no moving parts and is compact.

In this article, an application of the PS-WDXRF spectrometer to a valence analysis of Mn, Co, and Ni, which are the 3d transition metals used as cathode materials in Li-ion batteries (LIBs), is reported.

Introduction

Electric vehicles (EVs) are attracting attention worldwide as a means to solve the problems of global warming and oil depletion. The spread of EVs depends on the development of high-performance secondary batteries. Li-ion batteries (LIBs) are currently the preferred option for EVs due to their high energy density, rapid charging, long lifetime, and low cost. In particular, Ni-rich materials with a high proportion of Ni instead of expensive Co are suitable for achieving high energy density with low cost.[1–3] Knowing the valence changes of the 3d transition metals in the LIB cathode materials during charging and discharging is important for understanding the redox mechanism and improving the performance of LIBs.[4–8]

A polychromatic simultaneous wavelength-dispersive X-ray fluorescence (PS-WDXRF) spectrometer[9] is an extremely useful device for the development of LIB cathode materials because it can measure simultaneously the valence changes of 3d transition metals with high precision in the laboratory. Adjustment and maintenance of the drive mechanism are unnecessary, and high-precision measurements are possible in a short time because the optical system has no moving parts and is compact. A schematic of PS-WDXRF is presented in Figure 1. It is composed of an X-ray tube, a slit, a flat analyzing crystal, and a one-dimensional position-sensitive X-ray detector. In this optical system, fluorescent X-rays from a specimen pass through the slit and are dispersed by the flat analyzing crystal. They are simultaneously detected by different channels of the detector corresponding to different energies. Because fluorescent X-rays spread and are incident on the crystal at different angles, only X-rays that satisfy the Bragg condition at each angle are diffracted and incident on the detector. In this approach, all the X-rays corresponding to Bragg angles ranging from θ_L to θ_H can be measured simultaneously. Furthermore, the rotation mechanism of the specimen eliminates the position dependence of the measurement point, and the spatially averaged measurement data can be obtained.

The developed PS-WDXRF spectrometer is capable of measuring simultaneously the three main elements in LIB cathode materials: Mn, Co, and Ni. In this report, an application to a valence analysis of these 3d transition metals is shown.

Application to valence analysis of LIB

Peak energy of fluorescent X-ray emitted from a material shifts along with a change in the chemical state of the element. Figure 2 shows an overlay of Mn Kβ spectra of MnO_xs and $LiMnO_x$s normalized by peak intensity. The inset shows the enlarged view of the top of $K\beta_{1,3}$. It is clear that the peak energy shifts according to the nominal valence of Mn. In addition to that, the change in the peak intensity of Kβ′ is also observed.

By plotting the peak energy as a function of nominal valence of Mn, it was found that the Mn $K\beta_{1,3}$ peak energies of the MnO_x specimens are negatively proportional to the valence, and those of the $LiMnO_x$ specimens showed a similar relation, see Figure 3(a).

The dependence of the peak intensity ratio $K\beta'/K\beta_{1,3}$ on the nominal valence is shown in Figure 3(b), which indicates that the peak intensity ratios of the MnO_x and $LiMnO_x$ specimens were also negatively proportional to the valence and displayed a similar dependence as that of the peak energy.

These suggest that the changes in the peak energy of Mn $K\beta_{1,3}$ and the peak intensity ratio $K\beta'/K\beta_{1,3}$ can be used for valence identification.

For other elements, Co, and Ni, out of three important elements in LIB cathode materials, it had been reported that they also showed similar behavior in terms of the relationship between the peak energy shift and the nominal valence of the element.

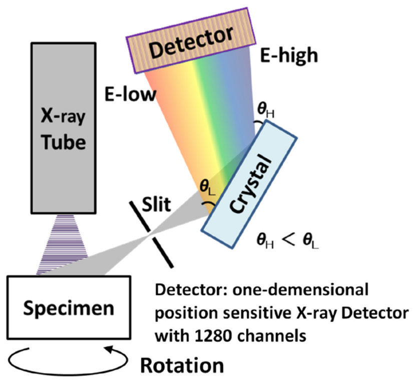

Figure 1. Schematic of PS-WDXRF. Fluorescent X-rays from the specimen pass through the slit and are dispersed by the flat analyzing crystal. They are simultaneously detected by different channels of the detector corresponding to different energies. The rotation mechanism of the specimen eliminates the position dependence of the measurement point, and spatially averaged measurement data can be obtained.*1

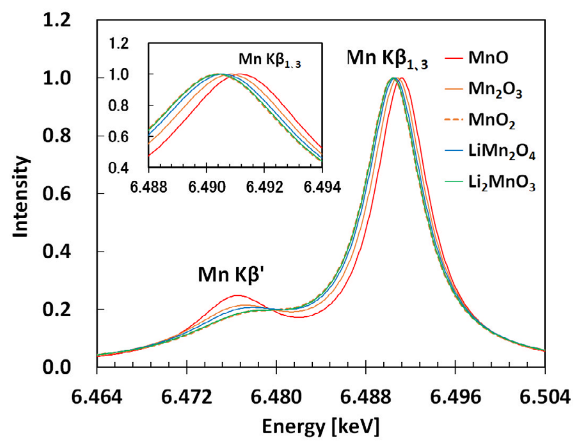

Figure 2. Overlay of the Mn Kβ spectra of MnO_xs and $LiMnO_x$s fitted by the Lorentz function, normalized by peak intensity. The inset shows the enlarged view of the top of $K\beta_{1,3}$. It is clear that the peak energy shifts, and the peak intensity of Kβ′ changes according to the valence of Mn.*2

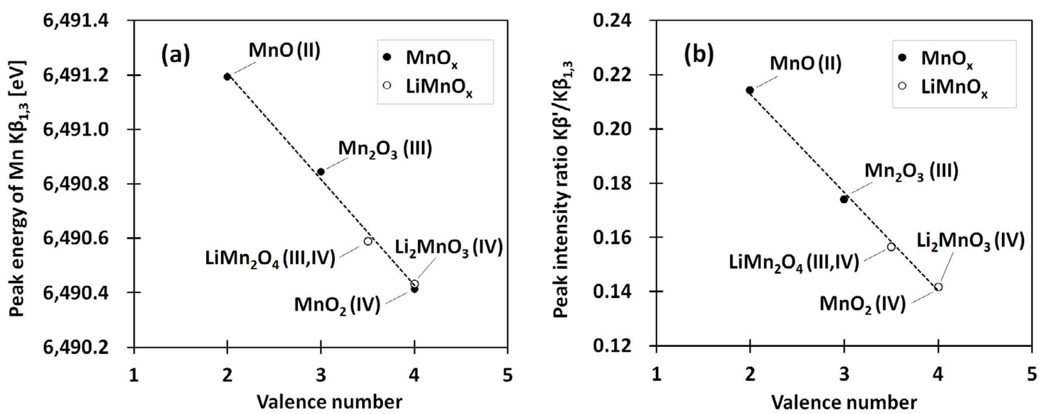

Figure 3. (a) Dependence of the Mn $K\beta_{1,3}$ peak energy on the valence number for MnO_xs and $LiMnO_x$s. The Mn $K\beta_{1,3}$ peak energies of MnO_xs are negatively proportional to the valence, and those of $LiMnO_x$s show a similar relation. These results indicate that the presence of Li ions does not affect the relation between the Mn $K\beta_{1,3}$ peak energy and the valence number. (b) Dependence of the peak intensity ratio $K\beta'/K\beta_{1,3}$ on the valence number. The peak intensity ratio $K\beta'/K\beta_{1,3}$ shows a similar dependence as that of the peak energy.*3

 ▸ For more information

Specimens

In this study, three different valence manganese oxides (MnO_xs), MnO (II), Mn_2O_3 (III), and MnO_2 (IV), and two different valence lithium manganese oxides ($LiMnO_x$s), $LiMn_2O_4$ (III, IV) and Li_2MnO_3 (IV), were prepared for use in the valence identification of Mn in LIB cathode materials. CoO (II) and NiO (II) as well as $LiCoO_2$ (III) and $LiNiO_2$ (III) were prepared for use in the valence identification of Co and Ni in LIB cathode materials. The use of CoO and NiO was unavoidable because divalent Li oxides of Co and Ni could not be obtained.

Ten pieces of $LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$ (NCM523)-based LIB cells were prepared to compare five different charge/discharge states and to confirm reproducibility with two cells in each case. Each cell consisted of a cathode, a Li foil anode, a 1 M $LiPF_6$ electrolyte dissolved in 1:1 (vol %) ethylene carbonate/diethyl carbonate solvent, and a microporous separator. The cathode was a 50 µm thick mixture of 90 wt % $LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$, 5 wt % acetylene black conductive material, and 5 wt % poly(vinylidene difluoride) binder. In the second cycle, after the first activation cycle, the procedure was paused for both cells in different half-steps of charge/discharge states at a current rate of 0.1 C (10 h of charging and discharging at 303 K, cutoff voltage ranging from 3.0 to 4.2 V). Each charge state is expressed as SOC-# (#% state of charge), and each discharge state is expressed as DOD-# (#% depth of discharge). Then, each cell was disassembled, washed, and sealed with a water-vapor barrier transparent film to prepare each LIB cathode specimen.

Results and discussion

The valence changes of Mn, Co, and Ni during charging and discharging are shown in Figure 4. The length of each error bar equals one standard deviation for five measurements. The results for Mn obtained using $K\beta'/K\beta_{1,3}$ are also shown in thick green in Figure 4. The valence changes of Ni and Co in the NCM523 LIB cathode are 0.68 (from 2.90 to 3.58) and 0.19 (from 3.00 to 3.19), respectively. The valence of Mn remains approximately 4.4 or 4.0 and hardly changes. Consequently, the composition of Li is estimated to change between 0.63 and 0.25 (or 0.75 and 0.37 if the valence of Mn is 4.0) during charging and discharging at cutoff voltage ranging from 3.0 to 4.2 V. Thus, Mn does not contribute to the redox process, and the contribution of Co is small. In contrast, Ni contributes the most to the redox mechanism. These results indicate that increasing the Ni content instead of the Co content in LIB cathodes improves energy density and reduces cost. Although the absolute value of the valence is ambiguous because of the difference depending on the reference material and the way of referring to the X-ray fluorescent spectrum, it is certain that the valence change can be accurately measured.

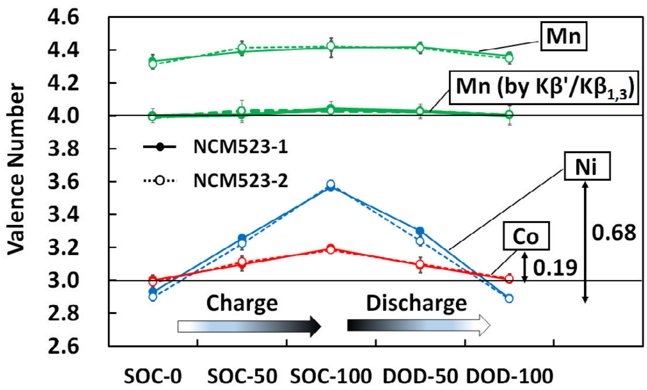

Figure 4. Valence changes of Mn, Co, and Ni in the NCM523 LIB cathode during charging and discharging. Each charge state is expressed as SOC-# (#% state of charge), and each discharge state is expressed as DOD-# (#% depth of discharge). The length of each error bar equals one standard deviation for five measurements.*4

*4 Reproduced with permission from [Sato 2020] *Copyright 2020 American Chemical Society. ▸ For more information

Conclusions

With a capability of observing energies of X-ray fluorescent peaks with high precision, the PS-WDXRF spectrometer can be employed to measure the valence changes of Mn, Co and Ni which comprise the cathode material of LIBs.

To confirm its ability, NCM523-based LIB cathodes were analyzed, and we found that the valence changes of the 3d transition metals in NCM523 during charging and discharging were 0.68 (from 2.90 to 3.58) for Ni, 0.19 (from 3.00 to 3.19) for Co, and no change from approximately 4.4 or 4.0 for Mn. These results indicate that Ni contributes the most to the redox process in NCM523-based LIB, Co contributes slightly, and Mn does not contribute. Therefore, increasing the Ni content instead of the Co content improves energy density and reduces cost. The results were obtained simultaneously in a short time in the laboratory. Thus, PS-WDXRF is expected to be a useful tool for the development of LIB cathode materials.

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This report is based on a research article;

Sato, K., et. al., "Evaluation of Analytical Precision of Polychromatic Simultaneous WDXRF Spectrometer and Application to Valence Analysis of Cathode Materials of Lithium-Ion Batteries":
***Analytical Chemistry*, 92, 758-765 (2020).**

Materials Science

Measurement of Electrolyte Solutions for Lithium-Ion Secondary Batteries with IRSpirit Glove Box System

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User Benefits
- IRSpirit can be controlled via wireless communication.
- Installing the IRSpirit in a glove box enables sample measurements in an environment purged with inert gas.
- Anaerobic samples such as cell components of lithium-ion secondary batteries can be analyzed with FTIR.

Introduction

When cell components of lithium-ion secondary batteries are affected by water vapor and oxygen in the atmosphere, the battery characteristics are significantly affected. It is therefore desirable to handle and characterize the cell components under an atmosphere not affected by water vapor or oxygen.

The IRSpirit, a compact FTIR, can be installed in a glove box, thus enabling the evaluation of cell components in a high purity argon atmosphere with low dew point and low oxygen concentration.

This article introduces an example of the measurement of organic electrolyte solutions, commonly used in lithium-ion secondary batteries.

IRSpirit Glove Box System

Since IRSpirit can be controlled wirelessly, sample can be measured while the FTIR unit is installed in a glove box (custom order required). Communication is established via a wireless converter and a wireless router. Fig. 1 shows the schematic diagram of IRSpirit glove box system.

Fig. 2 shows an example of a system where IRSpirit is installed in a flow-type glove box (Glovebox Japan, GBJA100). With this system, FTIR analysis can be conducted under an atmosphere of argon, nitrogen, etc.

Fig. 3 shows the inside of the glove box. In the glove box, the IRSpirit unit, a wireless converter, and if necessary, a cooler (fan) and a mouse for controlling the PC are installed. The PC is placed outside the glove box, and analysis is performed via wireless communication between the IRSpirit and PC.

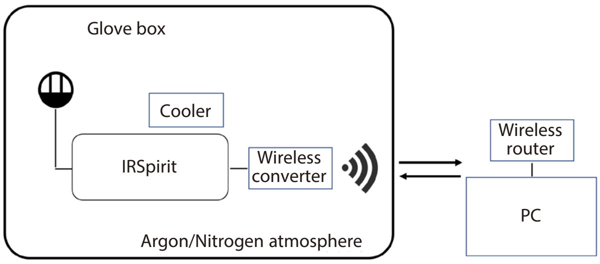

Fig. 1 Schematic Diagram of IRSpirit Glove Box System (Cooler installed as necessary)

Note that since thermal conductivity is different between argon and air, the heat exhaust mechanism of the instrument may not function properly, thereby preventing acquisition of correct infrared spectra. To avoid this, we used a cooler to enhance heat exhaustion in this analysis, and controlled the dew point in the glove box to -70 ˚C (moisture content of 2.58 ppm) or lower, and the oxygen concentration to 0.3 ppm or lower. Analysis in a glove box thus enables highly accurate measurements without being affected by water vapor or oxygen.

Fig. 2 Example of IRSpirit Glove Box System

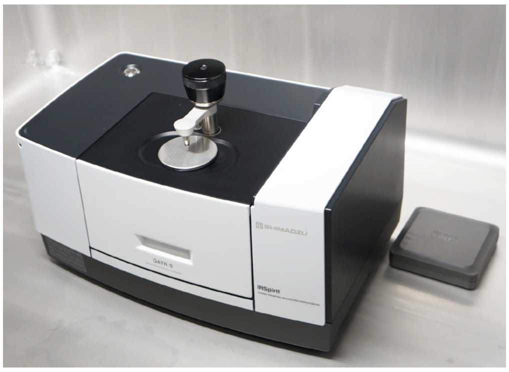

Fig. 3 IRSpirit (Left) and Wireless Converter (Right) in Glove Box

Effect of Atmospheric Gases on Measurement of Electrolyte Solutions for Lithium-Ion Secondary Batteries

An electrolyte of EC (ethylene carbonate) + DEC (diethyl carbonate) (3 : 7) solution containing 1M $LiPF_6$ (lithium hexafluorophosphate), an organic electrolyte solution commonly used in lithium-ion secondary batteries, was measured under air and argon atmospheres. Fig. 4 shows the measured infrared spectra and Fig. 5 shows the enlarged region between 2800 and 4000 cm^{-1} in Fig. 4.

Although the optical properties of argon and air are very different, Fig. 4 shows that the infrared spectra acquired under both atmospheres were almost identical, appearing to be unaffected by the difference of atmosphere.

Note that in Fig. 5, the broad absorption of symmetric and antisymmetric OH-stretching vibrations from water molecules were observed between 3400 and 3700 cm^{-1} under the air atmosphere. Meanwhile, the influence was negligible in the infrared spectrum under the argon atmosphere.

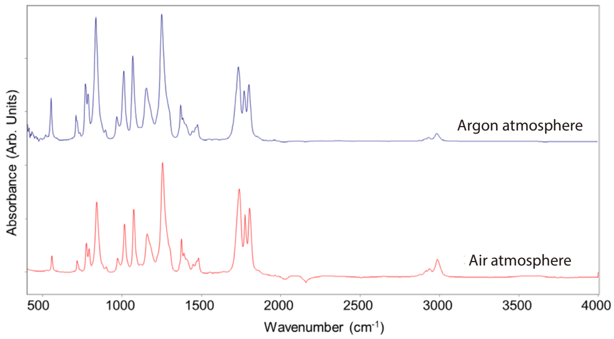

Fig. 4 Infrared Spectra of EC+DEC (3 : 7) Electrolyte Solution Containing 1M $LiPF_6$

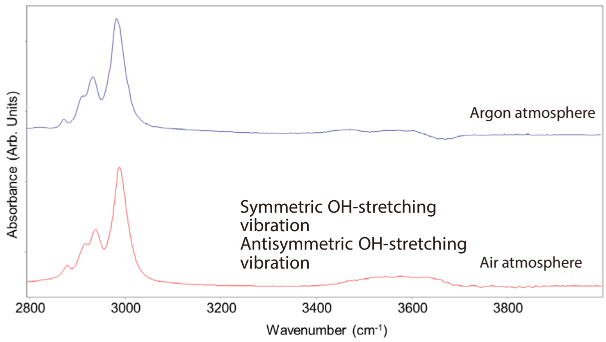

Fig. 5 Infrared Spectra of EC+DEC (3 : 7) Electrolyte Solution Containing 1M $LiPF_6$(Enlarged)

Measurement of Electrolyte Solution for Lithium-Ion Secondary Battery in Inert Atmosphere

Organic electrolyte solutions are used as the electrolyte solution for lithium-ion secondary batteries, and their moisture content is strictly controlled. Such an electrolyte solution was measured by FTIR in an argon atmosphere.

Fig. 6 shows the infrared spectra of EC+DEC (3 : 7) electrolyte solution with 1M $LiPF_6$ and EC+DEC (3 : 7) solution without 1M $LiPF_6$. The only difference between the two samples is the presence or absence of $LiPF_6$.

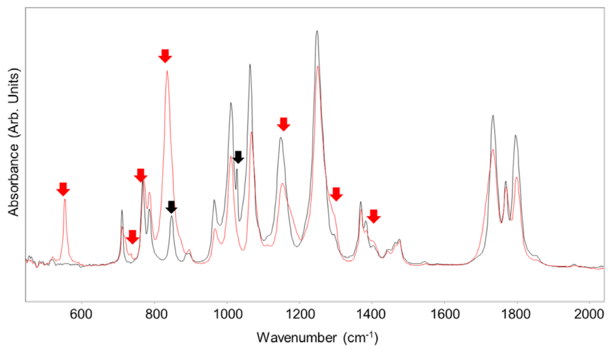

Fig. 6 Infrared Spectra of EC+DEC (3 : 7) Electrolyte Solution Containing 1M $LiPF_6$ (red) and EC+DEC (3 : 7) Solution (black)

* In this application, measurements were performed under specific conditions and environments since glove boxes and argon atmospheres reduce the heat exhaust function and may prevent proper instrument operation. Please contact for details.

The absorptions indicated with red and black arrows in Fig. 6 are characteristic to each component. To clarify the difference between the two spectra, Fig. 7 shows the difference spectrum where the black line in Fig. 6 was subtracted from the red line.

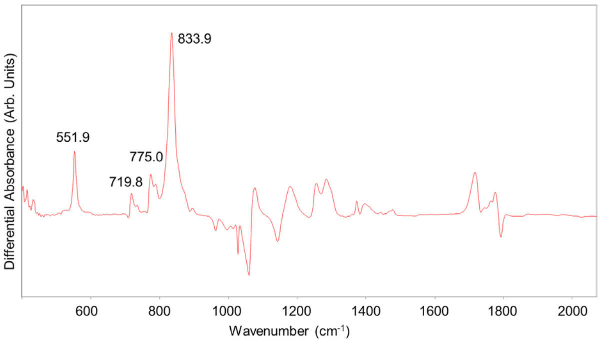

Fig. 7 Difference Spectrum between EC+DEC (3 : 7) Electrolyte Solution Containing 1M $LiPF_6$ and EC+DEC (3 : 7) Solution

We can see in Fig. 7 that the differences in absorption intensity appeared at 551.9 cm^{-1}, 719.8 cm^{-1}, 775.0 cm^{-1}, and 833.9 cm^{-1}. These absorptions cannot be explained simply by the vibrational mode of $LiPF_6$. Given that characteristic vibration modes of solvation between either EC or DEC and lithium ions appear in the 700 cm^{-1} to 1000 cm^{-1} frequency range[1]), the four absorption lines shown in Fig.7 are probably characteristic absorption lines of lithium ions solvated by EC or DEC.

Conclusion

We introduced an application example of the IRSpirit glove box system. Although FTIR measurements are usually performed in air, the IRSpirit can also handle anaerobic samples such as cell components of lithium-ion secondary batteries by installing the instrument in a glove box for measurements under inert atmosphere. In addition, even though the measurement wavenumber region of FTIR includes the infrared absorption of water vapor and carbon dioxide, such influences effected by air can be reduced by measurement under inert atmosphere using a glove box.

Acknowledgment

The data shown above was provided by Associate Professor Takashi Ito at Frontier Research Institute for Interdisciplinary Sciences, Tohoku University. We would like to take this opportunity to express our sincere gratitude. Please also see the article in FTIR TALK LETTER Vol. 35.

Reference

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Materials Science

Analysis of the Cylindrical Lithium-Ion Battery by X-Ray CT System and Introduction to the Charge/Discharge Device Attached System

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Solutions COE, Analytical & Measuring Instruments Division, Shimadzu Corporation

User Benefits

- Microfocus X-ray CT system enables the observation of internal structure in lithium-ion batteries.
- The distance between the electrodes can be measured, and the junction of the positive/negative terminal leads can be observed.
- The charge/discharge device attached system*1 enables changes in the internal structure of the battery to be observed after charging and discharging.

Introduction

Lithium-ion batteries (LIB) are secondary batteries in which Li ions move between the cathode and anode to charge/discharge, and are classified into three types, cylindrical, square, and laminated, depending on the shape of the cell. Especially, the cylindrical LIB is widely used in mobile batteries, notebook computers, etc. because of its low manufacturing cost and high energy density. In recent years development for electric vehicles has been remarkable, and research and development is progressing in pursuit of higher performance such as higher output and large capacity.

On the other hand, LIBs may have manufacturing defects such as short circuits due to foreign matter mixed in the electrodes and unwinding, and such defects may result in combustion and explosion accidents. Therefore setting the manufacturing conditions for development prototypes and their inspection are important. The X-ray CT system (Fig. 1) is an effective tool for analyzing the inside of batteries non-destructively.

This article introduces analyses of a cylindrical LIB using an X-ray CT system and the charge/discharge device attached system.

*1 The charging/discharging device must be provided by the customer.

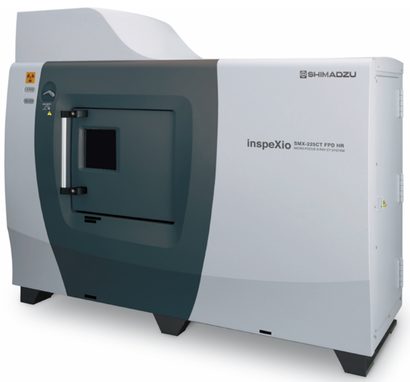

Fig. 1 Microfocus X-Ray CT System inspeXio™ SMX™-225CT FPD HR Plus

Fluoroscopy of the Cylindrical LIB

Fig. 2 shows a fluoroscopic image of a 21700*2 type LIB cell. The contrast in a fluoroscopic image is determined by the relative difference of X-ray absorption of the materials. Fluoroscopy is used for simple inspection because it can be observed in short time. As shown in Fig. 3, electrodes are composed of cathodes, anodes, and separators, which are arranged alternately.

*2 Battery model diameter : 21 mm, full length : 70 mm

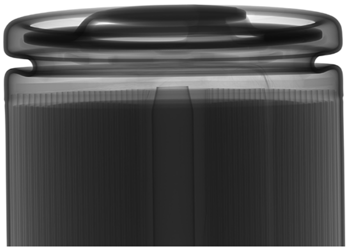

Fig. 2 Fluoroscopic Image of the Positive Terminal in a 21700 Type LIB Cell

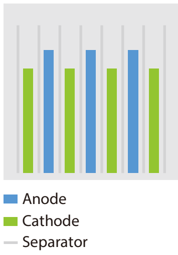

Fig. 3 Schematic Diagram of the Electrode Structure in a LIB

X-Ray CT Scan of a Cylindrical LIB

The X-ray CT scan is effective when more detailed observation/analysis is required. Fig. 4 shows a cross-sectional image of the 21700 type LIB cell, and Fig. 5 shows its three-dimensional image.

In the cross-sectional image, cathodes and anodes and the current collector tab, which are overlapped in fluoroscopic images, are clearly distinguished. In addition, a three-dimensional image can be displayed by stacking cross-sectional images, and the internal structure is observed by partially cutting or selectively displaying only specific materials.

Fig. 6 shows an image of the unrolled cylinder cross-sectional image of the 21700 type LIB cell. The unrolling position is indicated by the purple line in Fig. 6-A, and the unrolled cylinder cross-sectional image is shown in Fig. 6-B. Here, the current collector tab is shown, and by enlarging it the structure of its joint can be observed. In addition to vertical and horizontal cross-section observation, observation along a cylindrical surface is also possible, so, the electrodes can be observed as they were before they were rolled.

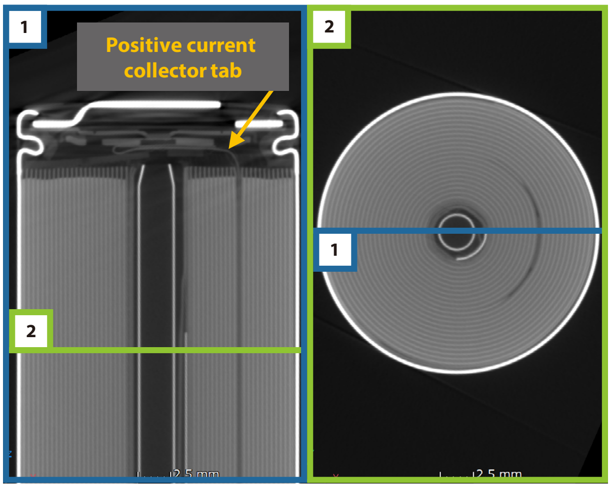

Fig. 4 Cross-Sectional Image of the 21700 LIB Cell

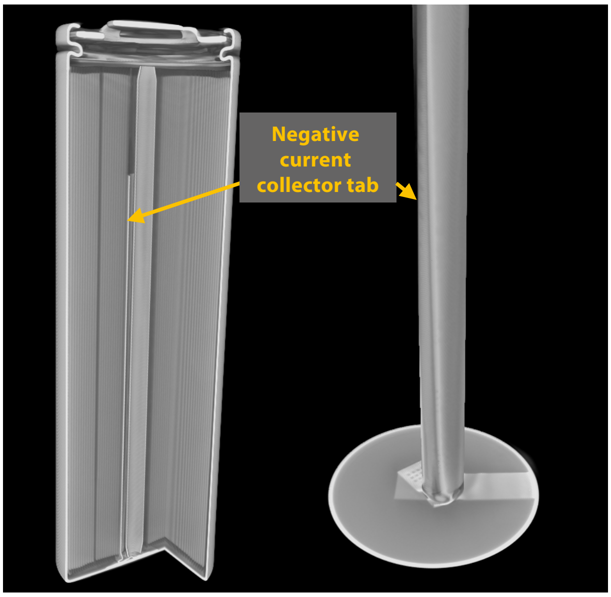

Fig. 5 Three-Dimensional Image of the 21700 LIB Cell

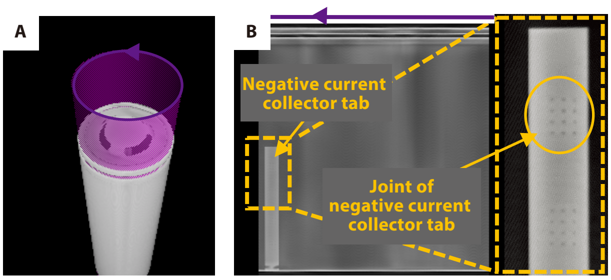

Fig. 6 Unrolled Cylinder Cross-Sectional Image of the 21700 LIB Cell
-A Indicates unrolling position -B Unrolled cross-sectional image

Distance Measurement between Electrodes

Fig. 7 and Table 1 show the results of distance measurement between electrodes. In addition to measuring lengths on the cross-sectional image, the pitch of the cathode and anode are also measured from the line profile of the gray value as shown in Fig. 8.

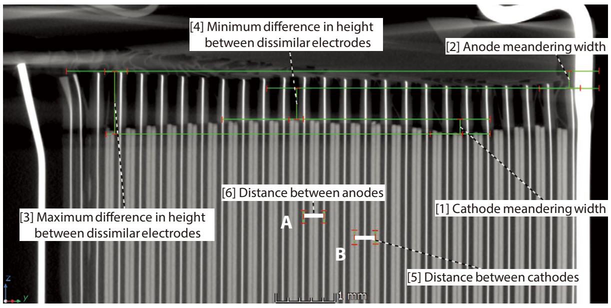

Fig. 7 Result of the Distance Measurement between Electrodes

Table 1 Result of the Height and Width Measurement between Electrodes

Measurement points	Result (mm)
[1] Cathode meandering width	0.248
[2] Anode meandering width	0.293
[3] Maximum difference in height between dissimilar electrodes	1.067
[4] Minimum difference in height between dissimilar electrodes	0.532
[5] Distance between cathodes	0.356
[6] Distance between anodes	0.348

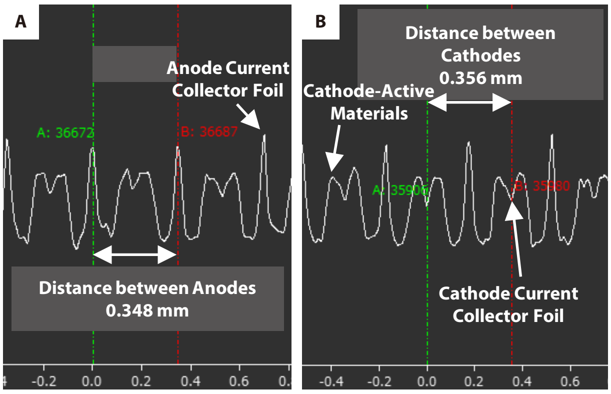

Fig. 8 Result of Distance Measurement with Line Profile of Gray-Value
-A Line profile of line A -B Line profile of line B

Charge/Discharge Device Attached System

It is known that the electrodes expand and contract during charging and discharging of LIBs, and the battery deteriorates due to volume changes and chemical changes. In deterioration evaluation and development for longevity, there is an increasing need to observe the internal structure of the battery when charging and discharging are repeated.

This system enables CT scanning while charging and discharging the lithium-ion battery inside the device*3 (Fig. 9). It can be connected to a charge/discharge device prepared by the customer.

*3 The main specifications of the X-ray CT are the same as those of the standard inspeXio SMX-225CT FPD HR Plus.

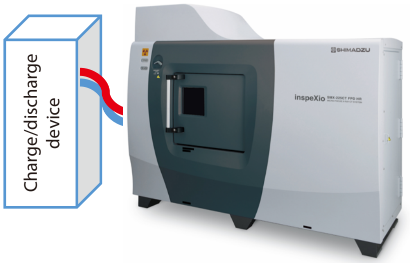

Fig. 9 inspeXio™ SMX™-225CT FPD HR Plus with Charge/Discharge Attached System (Illustration)

Fig. 10 shows a cross-sectional image of a 18650 type LIB cell before and after the charge/discharge test. In this cross-sectional image after the test, deformation of the electrodes can be seen in the part indicated by the red frame. Even if the battery has no problem in appearance, the internal electrodes may be deformed. It is possible to observe the inside non-destructively by using the X-ray CT system.

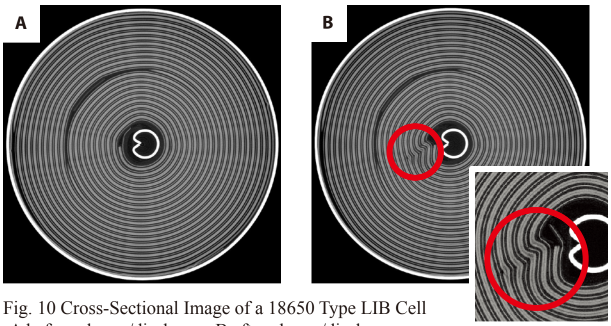

Fig. 10 Cross-Sectional Image of a 18650 Type LIB Cell
-A before charge/discharge -B after charge/discharge

Conclusion

This article introduces analyses of a cylindrical LIB using an X-ray CT system and a charge/discharge device attached system. X-ray fluoroscopy is suitable for simple observation and inspection in a short time, but X-ray CT is effective for more detailed observation and analyses. By using the X-ray CT system, it is possible to measure the distance between electrodes and observe the joint of current collector tab. In addition, by using the custom product charge/discharge device attached system, deformation of electrodes can be observed in-situ.

In this way, the CT system can be used in situations such as setting manufacturing conditions for development prototypes and sampling inspection of manufactured products.

Materials Science

SPM (AFM) Measurements of Cathode and Anode Materials from All-Solid-State Lithium-Ion Batteries without Exposure to Air

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[1]Solutions COE, Analytical & Measuring Instruments Division, Shimadzu Corporation
[2]X-Ray/Surface Business Unit, Analytical & Measuring Instruments Division, Shimadzu Corporation

User Benefits

- SPM (AFM) observations and measurements can be performed without exposure to air.
- The cathodes and anodes of all-solid-state lithium-ion batteries can be observed and measured.
- Shapes can be observed at the microscopic scale, and current and electrical forces can be visualized.

Introduction

Further improvements to battery performance are called for due to policies promoting electric vehicles (EV). This includes expectations for the commercialization of all-solid-state lithium-ion batteries because of their advantages including safety, high energy density, and resistance to degradation. One issue in the practical utilization of such batteries is the difficulty of reducing the interface resistance between the electrodes and the solid electrolyte. However, it is believed that shape observations and measurements of current at the microscopic scale will lead to a better understanding of interface reaction mechanisms leading to the resolution of this issue. This article introduces examples of surface observations of cathode and anode materials and measurements of current in all-solid-state lithium-ion batteries using SPM.

SPM-8100FM and Glove Box

These measurements were performed using the SPM-8100FM high-resolution scanning probe microscope [SPM (AFM)], and a gas flow type glove box (Fig. 1). The features of this equipment are as follows.

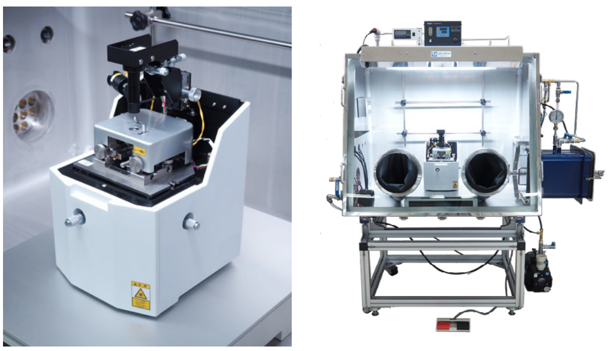
SPM

(1) It can operate in frequency modulation (FM) in addition to amplitude modulation (AM).

(2) ZXY measurement enables the acquisition of 3-dimensional data, including current and electric forces (hereafter: ZXY data).

Glove Box

(1) Oxygen and water vapor concentrations are both 1 ppm or less.



(a) SPM-8100FM (b) Gas Flow Type Glove Box

Fig. 1. SPM-8100FM and Glove Box

All-Solid-State Lithium-Ion Batteries and Related Issues

Fig. 2 is a schematic of an all-solid-state lithium-ion battery (hereafter: All-solid-state LiB). All-solid-state LiBs use a solid electrolyte, so they are safer than LiBs that use a normal combustible liquid electrolyte. However, the contact between the solid electrolyte and the electrodes is poor, so the interface's electrical resistance is large, making high-speed charging and discharging difficult. The SPM can measure the distribution of current and electric forces at the microscopic scale, so it holds promise as one method for evaluating the interface between solid electrolytes and electrodes.

In this article, sintered compacts of commercially available electrode material were observed and measured, in anticipation of the analysis of electrodes in contact with a solid electrolyte. The materials used were lithium cobaltite ($LiCoO_2$: hereafter LCO) for the cathode and lithium titanate ($Li_4Ti_5O_{12}$: hereafter LTO), both of which are used in all-solid-state LiBs. Fig. 3 shows an overview of the observations. Note that the electrode material used here was in its initial state, beforechargingor discharging.

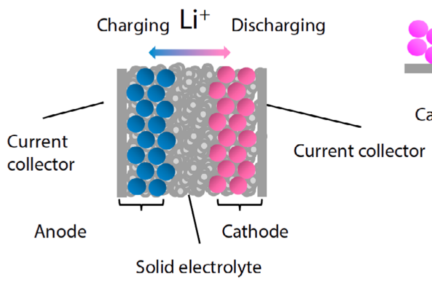


Fig. 2. Schematic Diagram of All-Solid-State LiB

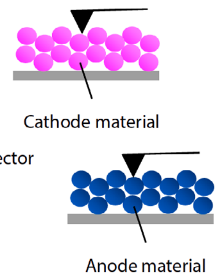
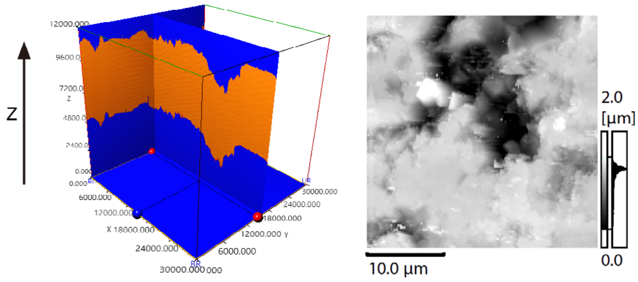


Fig. 3. Observation of a Sintered Compact of Commercially Available Electrode Material

ZXY Current Measurements and Image Construction

Observations and measurements were performed by measuring the ZXY-current. Fig. 4 shows a typical height image and ZXY data for LCO measurements, and Table 1 shows the measurement conditions. Normal shape measurements use planar data with Z information for each point with X and Y coordinates. However, the ZXY measurement method is based on force curve measurements, in which there is data for all three axes, Z, X, and Y. As a result, a variety of images can be constructed including height images and adsorption force images, based on the acquired ZXY data.[1), 2), 3), 4)]



(a) ZXY Data (b) LCO Height Image

Fig. 4. LCO ZXY Data and Constructed Height Image

Table 1 Measurement Conditions

Instrument:	Scanning Probe Microscope SPM-8100FM
Scanner:	Deep type scanner (maximum operation range XY: 55 µm, Z: 13 µm)
Holder:	Micro current holder Measurement range: ±10 nA
Observation Mode:	ZXY measurement
Pixel Number:	Z: 1024 X: 256 Y: 256
Purge Gas:	Argon
Atmosphere:	Oxygen 0.7 ppm or less, water vapor 0.75 ppm or less

Image Construction and Inspection

The images in Fig. 5 (a) to (j) were constructed from ZXY data in the measurement range from XY: 30 µm Z: 12 µm to XY: 5 µm Z: 6 µm.

(1) Height Images

The 30 µm LCO height image (a) and the 30 µm LTO height image (c) showed irregularities on the order of 2 µm, and the roughness (Sa) analysis showed surface roughness at 341.5 nm and 333.6 nm. Several gaps in the LCO were also found. In contrast, no gaps were found in the LTO, but residual marks from sample formation were evident on the samples in the vertical direction.

The 5 µm LCO height image (e) showed that the grains in the electrode material were about 1 µm in size, and that there were small gaps between them. There were also grains several hundred nm in size as indicated by the arrows. The LTO height image (h) showed plate-like crystal structures as indicated by the arrows.

(2) Current Images

In the 30 µm LCO current image (b), the current distribution was not uniform, and the current was detected over 41.7 % of the area (as analyzed using particle analysis software). In the 30 µm LTO current image (d), no current was detected, but this was likely because of the high resistance of the LTO in the uncharged state.

In the 5 µm LCO current image (f), it was found that the direction of current flow was different on the left and right sides of the yellow dotted line. On checking the 5 µm LCO height image (e), the yellow dotted line was found to be the boundary of a crack. In addition, it was evident that there was no current flow at the grains several hundred nm in size, indicated by the arrows.

As in the 30 µm LTO current image (d), no current was detected in the 5 µm LTO current image (i).

(3) Attractive Force Images

Normally, attractive forces in force curves are said to be caused by meniscus forces, van der Waals forces, or electrical forces due to water adsorption on the surface of a sample. In these measurements, however, the water vapor concentration was 75 ppm or less, so the effect of meniscus forces was likely small. Taking the above into consideration, it was found that the 5 µm LCO attractive force image (g) was correlated with the distribution in both the 5 µm LCO height image (e) and the 5 µm LCO current image (f). It was also found that the 5 µm LTO attractive force image (j) was correlated to the distribution of plate-like crystals in the 5 µm LTO height image (h) (arrows). It is therefore likely that the attractive force images represent van der Waals forces or electrical forces, and that these indicate the compositional distribution of the electrode material.

From the information above, it is likely that the LCO current distribution reflects the compositional distribution of the material, and that the path of the current is due to cracks or gaps between the grains. Regarding the LTO, for which current images could not be obtained in this case, there are plans to measure the anode of an actual battery after charging.

Conclusion

The surfaces of all-solid-state LiB electrode materials LCO and LTO were measured without exposure to air, and height images, current images, and attractive force images were obtained. It is expected that such measurements will be applicable to the resolution of issues with resistance at the interface in all-solid-state LiBs.

References

1. Application News No. S 47 New SPM Measurement Method: ZXY Scanning, Overcoming the Difficulties of Conventional SPM Measurement
2. Application News No. S 49 Visualization of Spatial Distribution of Magnetic Force by ZXY Measurement
3. Application News No. S 57 Visualization of Distribution of Electrostatic Force in Electrolyte Clarifying Corrosion and Cell Reactions
4. Application News 01-00256-EN Visualization of Current Distribution by ZXY Measurement: Current Measurement of Graphite Sample

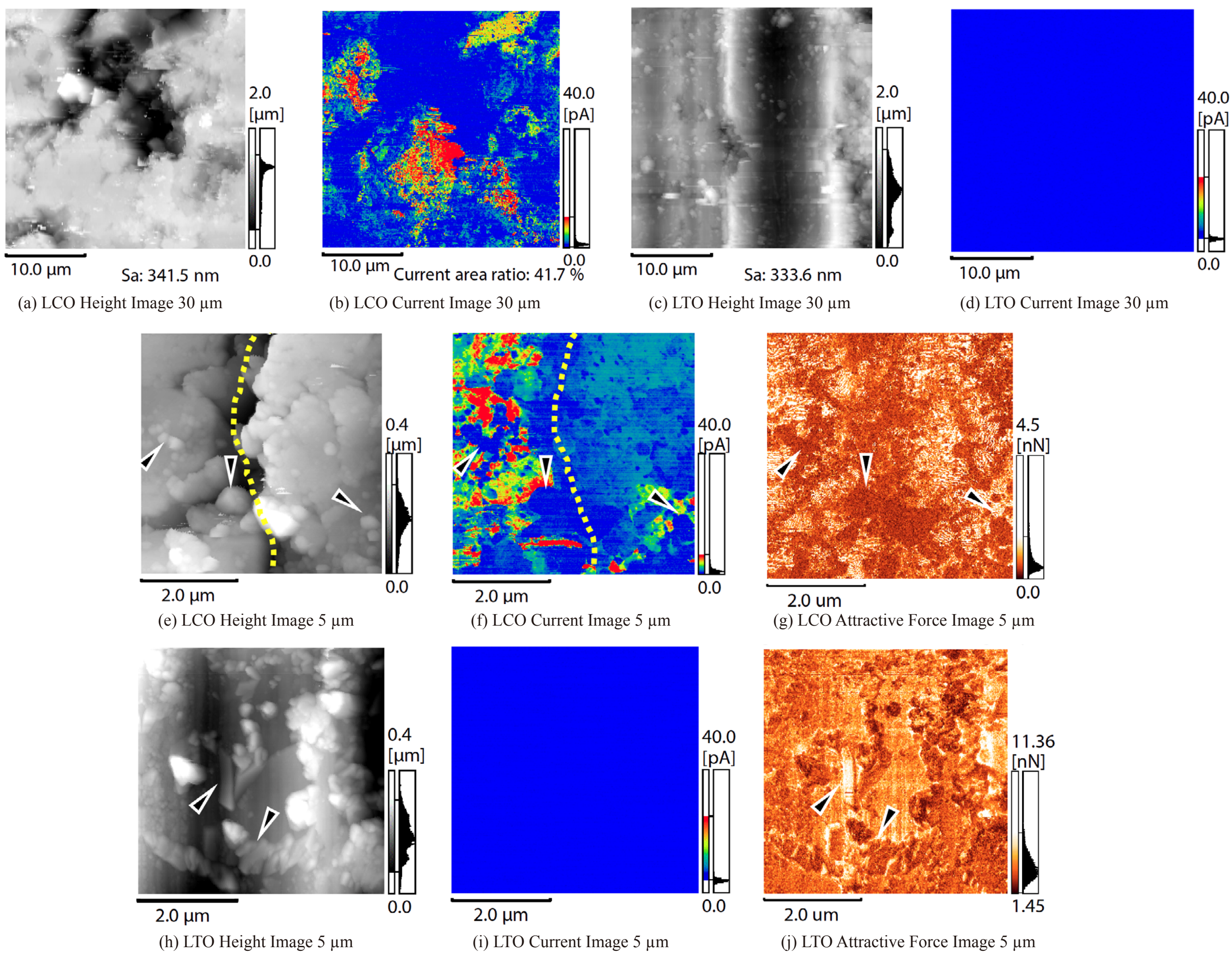


Fig. 5. Height Images, Current Images, and Force Curve Attractive Force Images

Shimadzu Selection

This is a selection of articles by Shimadzu relating to Materials Science analysis and evaluations. From posters presented at the international conferences to the website updates, they include everything from the latest applications to the latest publications. In these articles, you will find a variety of applications of our instruments as well as cutting-edge technologies. Please obtain the articles of your interest through the links on the titles.

Guide to Lithium-ion Battery Solutions

Demand for lithium-ion Battery is expected to expand further in the future, driven by demand for electric vehicles, which are supported by policies in various countries around the world, and demand for PC, smartphone, and tablet device, which are driven by digital transformation (DX). In addition, companies and research institutes around the world are actively engaged in research and development to commercialize all-solid-state batteries as next-generation battery. This guide book provides total solutions for each evaluation or material of Lithium-ion Battery development.

Multilateral Evaluation of Positive and Negative Electrodes in Lithium-ion Batteries

In recent years, the applications of lithium-ion battery have expanded dramatically, and research on increasing capacity, extending life, reducing cost, and improving safety has been actively conducted. The components of lithium ion are roughly divided into positive electrode, negative electrode, separator, and electrolyte solution. In particular, positive electrode and negative electrode are important components for improving performance. This document introduces the analysis technology for each manufacturing process.

Analysis of MEA (Membrane Electrode Assembly)

Electrochemical devices in which electrode catalyst layers are bonded on a solid polymer electrolyte membrane, which is a hydrogen ion conductor, are called membrane electrode assemblies (MEA). Among their uses are fuel cells, water electrolysis technologies, hydrogen production technologies, and dehumidification. Using a Shimadzu EPMA-8050G EPMA electron probe micro analyzer, this article presents an example of a comparative evaluation of a new MEA before use and a product with deteriorated performance.

Analysis of Ultraviolet-Degraded Plastic by Plastic Analyzer

Plastic materials are one of the various types of contaminants that occur in production lines. Although qualitative analysis of most plastic contaminants is possible by using a commercial database, correct identification of plastics that have been damaged by UV radiation or heat is difficult due to changes in the pattern of the infrared spectrum. The libraries included in Shimadzu FTIR-based Plastic Analyzer contain infrared spectra of plastics that have been damaged by UV radiation and heat and enable highly accurate qualitative analyses which reflect the state of degradation.

Evaluation of PC/ABS Plastics with Different Blend Ratios

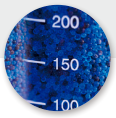
PC/ABS plastics combine the respective advantages of polycarbonate (PC) and ABS. With amorphous plastics such as PC/ABS plastics, thermal properties such as the linear expansion coefficient, specific heat, and thermal conductivity have an inflection point at the glass transition temperature. In addition, with injection molding, it is important to assess the glass transition temperature as a guide to the solidification temperature. This article introduces an example of the evaluation and comparison of samples using two instruments, a differential scanning calorimeter, and a fourier transform infrared spectrophotometer.

Solutions for Cellulose Nanofibers

Cellulose nanofibers (CNF) are plant-derived carbon-neutral materials that are light-weight and strong, yet they also exhibit high elasticity and resistance to heat. With recent concerns on global warming and climate change, there is growing attention regarding CNF playing a major role in sustainable bio-based materials and may be one of the fundamental solutions to various environmental problems. This solutions book help for research and development to expand the use of CNF.

Analysis of Components Emitted during Photodegradation of Bionylon with Light-Induced Hydrolysis

As the issues of marine plastic waste become more serious, there is a need to develop plastics that exhibit sufficient performance in use but quickly decompose in the marine environment. The authors have developed a high-performance nylon resin from itaconic acid that can start to decompose in sunlit or water environments. The nylon has a pyrrolidone ring in the main chain and exhibits light-induced hydrophilicity, but the structural changes and mechanisms involved have not been clarified. Therefore, the components emitted from samples due to light irradiation were analyzed using high-sensitivity GC-MS.



Analytical Solutions for Analysis of Polymer Additives

Although plastic products are used in a wide range of fields, including as packaging containers for pharmaceuticals and food, and as materials in transportation equipment and home appliances, the adoption of SDGs by the United Nations and the promotion of a circular economy is leading to an increased emphasis on recycling. Polymer additives are an important element in the recycling of plastic products and ensuring the quality of recycled goods. This application book example analyses of polymer additives performed using Shimadzu products.

Analysis of Super Elastic Alloys and Shape-Memory Alloys

Titanium materials possess desirable mechanical properties such as corrosion resistance and heat resistance and are widely used, including in the medical field, as they have excellent biocompatibility. Research and development of low-toxicity titanium alloys are underway. Orthodontic wire with a super elastic property is used in the treatment of dental malocclusions. Orthodontic wires with different transformation points are used appropriately depending on the patient's paradental condition and sensitivity to pain. In this article, three types of orthodontic wires with different transformation points were analyzed using an EPMA-8050G electron probe microanalyzer.

Polymers & Plastics

Plastics and polymers are widely used in various products that play an important role in our daily life. To develop new products, chemical and physical properties like heat-dependent characteristics and degradation paths need to be investigated using various analytical instruments. On this webpage, we introduce Shiamdzu's solutions for resins and plastics testing.

Carbon Neutrality

On this webpage, we introduce how Shimadzu contributes to technological development and quality control in fields such as hydrogen fuels, biofuels, wind power generation, and other renewable energies, as well as automobiles and storage batteries, which are all indispensable for achieving carbon neutrality.

Latest topics 1

Appointment of New President and CEO

The Shimadzu Group welcomed spring with a new organizational structure in April 2022. We are pleased to announce Yasunori Yamamoto as Shimadzu's new President.

President Yamamoto joined Shimadzu with an extensive background in physics, including elementary particles, fields of force, and laser fusion, and has been involved in the technology development of testing machines in the analysis and measurement division. After that, he held significant positions such as President of Shimadzu Europe GmbH (Germany), as well as Managing Executive Officer, Director, and Senior Managing Executive Officer (CFO) of the headquarter.

"Nothing happens unless first a dream." These words by the poet Carl Sandburg are his cherished motto. He says, "it is precisely now when our social environment is undergoing such startling changes that we must lend an ear to our global customers and face the issues with our technology. In addition, I would like to put our efforts into both the requirements of our current era and what will be needed in the era to come." For the earth for the future.

Top Message

Using Science and Technology to Innovate and Solve Societal Challenges Together with Customers from Around the World

Shimadzu Corporation has been doing business based on our corporate philosophy, "Contributing to Society through Science and Technology," since being founded almost 150 years ago.

The analytical and measuring instruments, industrial machinery, and aircraft equipment that we provide are used in a wide range of industries, where they play a role in protecting the safety and security of society and improving convenience of life through our customers' businesses. In addition, the Shimadzu products used at healthcare facilities for diagnosis, treatment, or measuring health, and the equipment used to support the development of new drugs, serve an important role in supporting the desire of people to live healthy lives.

I believe we have been able to continue contributing to society, despite our limitations, because we have always taken on the challenge of solving issues that our customers face. Consequently, we have steadily improved our technologies to "separate and visualize" gases, liquids, solids, genes, proteins, and other substances to determine their properties. We have also repeatedly taken on the challenge of developing new devices and creating new technologies that are key to manufacturing. Those efforts have resulted in a treasure trove of diverse technologies able to quickly solve customer challenges.

Today, society continues to change at an unprecedented pace. In the face of an endless stream of new challenges, including the pandemic, global warming, declining birth rates, and aging populations, we remain committed to actively confronting these challenges with renewed determination. By listening carefully to the silent voices of society and the earth, we will rise to solve those challenges and work with customers around the world to create innovative solutions. That is truly the mission expressed by our corporate philosophy, "Contributing to Society through Science and Technology."

Shimadzu will continue to use the technologies and wisdom inherited from our past to transition to a higher level than ever before. Therefore, please look forward to more great things to come from Shimadzu in the future.

Yasunori Yamamoto,
President and CEO

Latest topics 2

Shimadzu Tokyo Innovation Plaza, a New Center for Analytical & Measuring Instruments Business has been completed

Accelerating Open Innovation at a Convenient Location near Haneda Airport

In October 2022, Shimadzu completed the building of Shimadzu Tokyo Innovation Plaza, a center for the development of technology for analytical applications and the provision of solutions to customers. It is located at KING SKYFRONT* (Kawasaki, Japan), a hub for open innovation promoting new industrial development in the life sciences and environmental fields which are expected to grow globally.

KING SKYFRONT is an open innovation hub that creates new industry from the world's highest standard R&D, that plays an important role in Japan's growth strategy by contributing solutions to a variety of issues including health, medicine, welfare, and the environment while also creating global businesses in these fields. It is located in the Tono-machi region of Kawasaki ward, across the Tamagawa River from Haneda Airport, a 10-minute drive away.

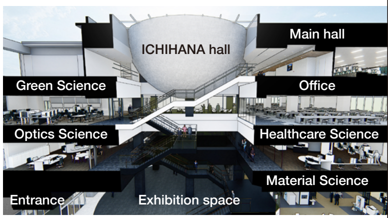
Learn more about KING SKYFRONT

Making use of this convenient location, Shimadzu Tokyo Innovation Plaza aims to promote the development of cutting-edge analytical techniques, the provision of solutions, and collaborative research together with global customers and neighboring research agencies. It further aims to be a space to exchange ideas and create new knowledge by engaging in international academic meetings, conferences, and seminars.

Experience with Analytical Instruments/Laboratory Environments in an Advanced Laboratory

At this new center, in addition to performing demonstration measurements to introduce the functionality of Shimadzu's leading equipment and usage methods, laboratory designs and experimental environments will be proposed. Analysis laboratories are opened using glass walls, enabling visitors to observe analyses from a walkway.

Exhibition areas, halls, lounges and conference rooms are available in addition to the analytical laboratories which are the core of this new center. Hands-on exhibits incorporating the five senses, as well as demonstration measurements and seminars, are among the events planned.



Adopting a Floor Design that Accelerates Open Innovation

*KING is an acronym for Kawasaki Innovation Gateway and reflects the meaning of "tono" (feudal lord) as found in the name of the nearby town Tono-machi. SKYFRONT refers to its location across from Haneda Airport, which connects Japan to the rest of the world.

Latest topics 3

A Shimadzu Group American Sales Subsidiary Recognized as a "Great Place to Work"

Shimadzu Scientific Instruments, Inc. (SSI), a Shimadzu Group company in the United States, was recognized as a "Great Place to Work" by the Great Place to Work Institute in April. This was the second year in a row that it received this recognition.

This agency conducts surveys and analyses regarding great places to work in about 60 countries worldwide. Using the media in each country, it announces companies and organizations recognized that have achieved a certain benchmark.

SSI, which was established in 1975, provides sales and service for analytical and measuring instruments primarily from its headquarters in Maryland, USA as well as 11 offices across the nation. In recent years, the Innovation Center was established for the development of applications for analytical instruments. The company strives to contribute to the resolution of social issues in the healthcare, food products, and environmental fields.

This survey evaluated the attitudes of 75 % of the SSI employees who said the company is a great place to work, as well as any points they were proud of in their work at the company.

Office Locations

SHIMADZU SCIENTIFIC INSTRUMENTS, INC.

President: Yoshiaki Maeda
Headquarters address: 7102 Riverwood Drive, Columbia, Maryland 21046, U.S.A.
Other branches: Columbia, MD/Boston, MA/Chicago, IL/Houston, TX/Durham, NC/Somerset, NJ/Kansas City, KS/San Francisco, CA/Carlsbad, CA/Tampa, FL
Number of employees: 533 (as of March 31)

65th GC ANNIVERSARY

Latest topics 4

Celebrating 65 Years of Gas Chromatography Innovation

2022 marks the 65th anniversary of the first GC (GC-1A) released by Shimadzu Corporation. To commemorate the occasion, Shimadzu is planning a variety of activities throughout the year to March 2023.

Celebrating 65 Years of Gas Chromatography Innovation

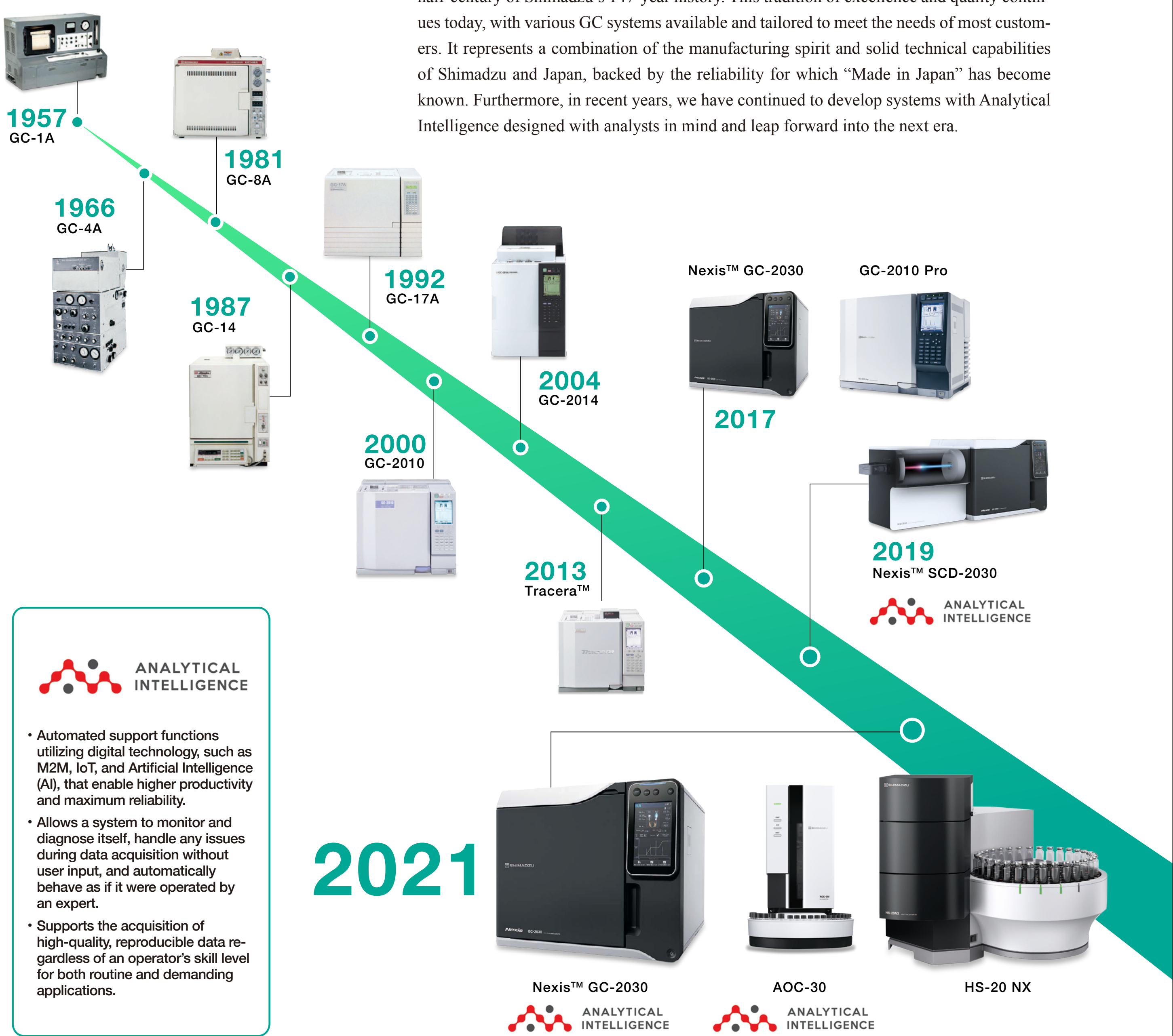
Shimadzu Corporation celebrates its 65th anniversary in 2022 when it began manufacturing and selling mass-produced gas chromatographs (GC) in 1957. The history of Shimadzu GC began with its use in petrochemical research applications in Japan. Shimadzu GC has expanded its product lineup to include automated sampler systems, gas chromatographs, and gas chromatograph mass spectrometers (GCMS). Customers use them in various markets worldwide, including petroleum, chemical, food, environmental, pharmaceutical, and analytical industries. We have celebrated our 65th anniversary only because we have continued to be chosen by our customers by steadily responding to various analytical requests and challenging analytical problems with the latest technologies that meet the needs of the times. In recent years, analytical laboratories that require higher productivity have faced various challenges, such as the generational change of skilled analysts and the problem of helium supply, which is an essential gas for GC/GCMS.

Shimadzu has been solving these issues by incorporating state-of-the-art Analytical Intelligence technology into its GC systems. We will continue to refine our latest analytical technologies and support our customers in solving their problems so that we can reach the next milestone, the 70th anniversary of Shimadzu GC.

Ryo Takechi
Product manager of GC

A History of Cutting-Edge Innovation

Since then, Shimadzu has continued to develop gas chromatography products for over a half-century of Shimadzu's 147-year history. This tradition of excellence and quality continues today, with various GC systems available and tailored to meet the needs of most customers. It represents a combination of the manufacturing spirit and solid technical capabilities of Shimadzu and Japan, backed by the reliability for which "Made in Japan" has become known. Furthermore, in recent years, we have continued to develop systems with Analytical Intelligence designed with analysts in mind and leap forward into the next era.



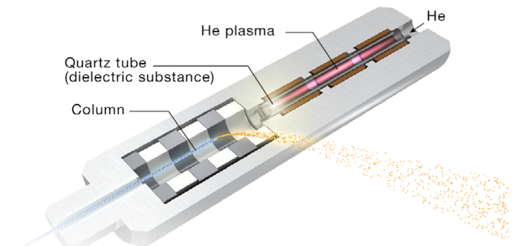
ANALYTICAL INTELLIGENCE

- Automated support functions utilizing digital technology, such as M2M, IoT, and Artificial Intelligence (AI), that enable higher productivity and maximum reliability.
- Allows a system to monitor and diagnose itself, handle any issues during data acquisition without user input, and automatically behave as if it were operated by an expert.
- Supports the acquisition of high-quality, reproducible data regardless of an operator's skill level for both routine and demanding applications.

Contribute to Carbon Neutrality

We contribute to quality control and technological development in fields such as artificial photosynthesis, hydrogen fuels, biofuels, and other renewable energies, as well as storage batteries, which are all indispensable for achieving carbon neutrality using the latest gas chromatography technology.

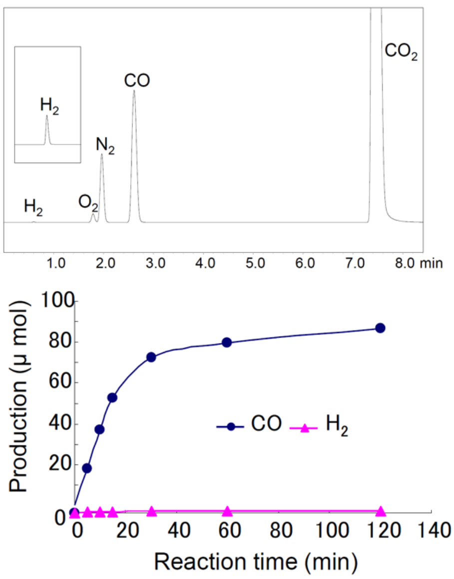
Shimadzu's proprietary technology has been adopted for the BID (Barrier Discharge Ionization Detector), which incorporates ionization via a new dielectric barrier discharge plasma. BID is a new universal detector that can detect any compound except Ne and He.



Barrier Discharge Ionization Detector BID-2030

Nexis™ Technologies – BID

The BID can detect carbon monoxide and hydrogen, as well as inorganic gases and hydrocarbons with high sensitivity, including formic acid and formaldehyde, produced in the reduction reaction of carbon dioxide using a photocatalyst.



Chromatogram of Substances Generated in a Photochemical Carbon Dioxide Reduction and Transition of Production Amount

Shimadzu's GC system is also used for quality control of hydrogen fuels, biofuels, and analysis to ensure the safety of lithium-ion batteries.

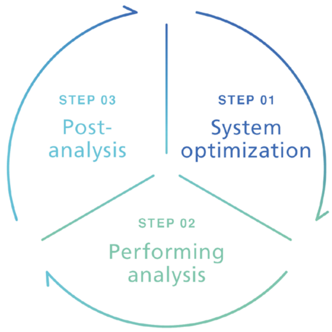
High-Sensitivity Analysis System for Inorganic Gases

Solutions for Biofuels

Analysis System for Gases in Rechargeable Lithium-Ion Batteries

Designed with the Analyst in Mind

In today's environment, results are needed regardless of whether the analyst works in the laboratory, the office, or at home. With the flexibility to accommodate a variety of applications, Shimadzu's GC supports the analyst's procedures beyond the laboratory, at virtually any location.

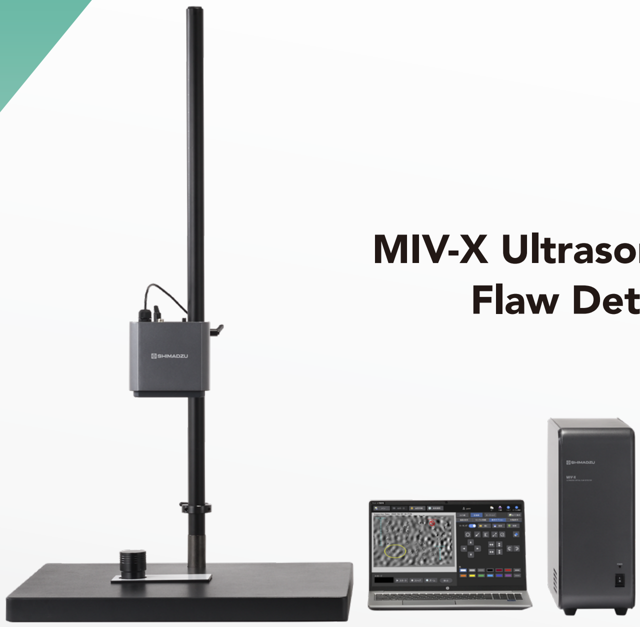


The Shimadzu's GC features Analytical Intelligence to help achieve improve productivity. Clean Pilot, a system conformity test, and LabSolutions™ can provide an automated workflow together with remote operation and monitoring from instrument startup to analysis completion. Automated workflows incorporate the work-style habits of experienced analysts. The result is reliable data collected over extended periods and higher productivity.LabSolutions Direct is a remote access tool used to control or monitor GC systems via a simple user interface on a commercially available smartphone or tablet. Consequently, analyses can be performed while monitoring the status of instruments from locations away from the laboratory. In addition, Shimadzu's GC is designed to enable even beginners to perform reliable analysis with various functions that reduce the burden of GC maintenance, such as self-diagnosis, automatic carrier gas leak check, and click-tech that makes it easy to install columns and maintain inlets.

We will continue to contribute worldwide by providing reliable quality GC globally.

Nexis™ GC-2030 - Features

Proposed Initiatives for Conserving Electricity



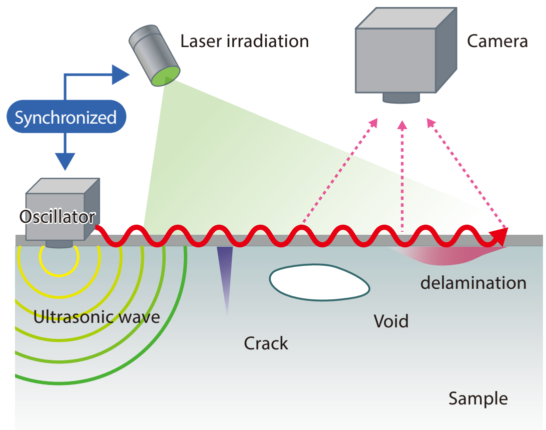
Latest topics 5

Ultrasonic Optical Flaw Detector

Shimadzu Corporation announces the release of the MIV-X ultrasonic optical flaw detector. This product visualizes and digitizes flaws, such as peeling, cracks, and voids, in parts and multi-materials in aircraft, automobiles, and electrical and electronic equipment using our proprietary ultrasonic optical flaw detection technology*, which captures images of the ultrasonic propagation on the surface of an object. While inheriting the basic functions of its predecessor–the MIV-500, which could inspect an area up to 400 mm by 600 mm in approximately 20 seconds, The new MIV-X offers significant advancements that contribute to the evolution of a highly functional nondestructive inspection system. The advancements include a noise removal function that enables more precise imaging, a flaw size measurement function, and an increase in resolution from the minimum detection size of 1 mm to 0.5 mm. This complements the conventional hammering test flaw-checking process, while simultaneously providing automatic logging of the measurement results.

Ultrasonic optical flaw detection is a nondestructive inspection technology that uses ultrasound and light. The surface of the test object is excited with ultrasound, and the tiny changes in the surface caused by these vibrations are detected with laser irradiation and a camera. If peeling, cracks, or other flaws are present, ultrasound discontinuities (propagation disturbances) are detected. Ultrasonic optical flaw detection enables the visualization of internal flaws (approximately 1 mm deep) that are hard to find using conventional ultrasonic flaw testing. It can easily detect flaws in joints and bonding surfaces in a multi-material R&D process that combines different materials to increase strength and reduce weight. Shimadzu is marketing the MIV-X ultrasonic optical flaw detector to manufacturers and contract analysis companies of transportation equipment and machinery, raw materials, and construction, as well as research agencies engaged in the development of multi-materials and other new materials.

*This technology is patented in Japan, China, and the United States.



Principle behind Ultrasonic Optical Flaw Detection

Learn more about MIV-X Ultrasonic Optical Flaw Detector

MIV-X Ultra-sonic Optical Flaw Detector Instruction Video

RELEASED

New Products

IR-Xross

ANALYTICAL INTELLIGENCE

Fourier Transform Infrared Spectrophotometer

The IRXross offers the high performance and easy operation that meets the demands of the pharmaceutical and chemical industries. IRXross provides an S/N ratio of 55,000:1, the highest in its class. It is also compliant with data integrity regulations required in the pharmaceutical field. The IRXross offers the optimal solution for a new era with diverse application requirements.

Learn more

XSeeker 8000

ANALYTICAL INTELLIGENCE

Microfocus X-Ray CT System

This product is equipped with a high-output X-ray generator and a high-resolution flat panel detector. Despite its compact size, it has a high X-ray output of 160 kV, enabling clear observations of molded plastic parts as well as aluminum die cast parts and other metal parts. In addition, the newly developed XSeeker control software provides high operability and the highest throughput to date.and quality evaluation to inspections at machining sites.

Learn more

AA-7800

Atomic Absorption Spectrophotometer

The Shimadzu Atomic Absorption Spectrophotometer AA -7800 Series is versatile enough for a variety of analytical applications (Any Application), safe and easy to use even for beginners (Any User), and offers continuous analysis using autosamplers and remote data analysis via network connections to increase the flexibility of the analysis operator's work style (Any Location).

Learn more

MIV-X

MAIVIS Ultrasonic Optical Flaw Detector

Ultrasonic optical flaw detection enables the visualization of internal flaws (at a depth of about 1 mm) that are hard to find using conventional ultrasonic testing. The MIV-X visualizes and digitizes flaws (peeling, cracks, and voids) in parts and multi-materials in aircraft, automobiles, and electrical and electronic equipment.

Learn more

TRAPEZIUM SATELLITE

Testing Machine Remote Monitoring System

TRAPEZIUM SATELLITE is a remote monitoring system for testing machines. It consists of a monitoring device, a USB camera, and software for remotely monitoring the operating status of fatigue testing machines using a web browser. The operating status of equipment can be checked while away from the laboratory, which heightens the efficiency of testing work and reduces workloads.

Learn more

LCMS-9050

Quadrupole Time-of-Flight Liquid Chromatograph Mass Spectromet

The LCMS-9050 is a quadrupole Time-of-Flight (Q-TOF) mass spectrometer system with the highest mass accuracy stability levels available. The stable positive/negative high-speed polarity switching technology enables the simultaneous analysis of positive ions/negative ions, contributing to new applications development and heightened analysis efficiency.

Learn more

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Shimadzu Corporation

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Printed in Japan, November, 2022