

Introduction of Automated Experimental Equipment for Exploration of Production Process Optimization in Fuel Cells

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Fuel cells are an important technology for the realization of a carbon-neutral society, and not only the research and development of their performance, but also the production process to ensure a stable supply to society is an extremely important technology. However, the parameters involved in the production process are enormous and social implementation requires a great deal of time. In order to contribute to the production process, Horiba has developed an automated experimental device that simulates the fuel cell production process. This apparatus was designed and fabricated with a focus on the fuel cell coating and drying processes, and is capable of fabricating fuel cell samples and performing basic evaluations. Here we introduce the overall concept and functions of the apparatus.



Introduction

In the domains of research, development, and manufacturing, the acquisition of high-quality data is crucial for accurate understanding of the target state and for enabling data-driven decision-making and management within the context of Digital Transformation (DX). HORIBA, Ltd. possesses a variety of analytical instruments, which, for example, allow for the collection of diverse data necessary to ascertain the status of each process throughout the battery lifecycle (Figure 1).

Although Japanese fuel cell technology leads the world, optimal designs that simultaneously achieve power generation efficiency, durability, and productivity have not necessarily been realized. In particular, production technologies, which involve a vast number of process parameters and complex physical phenomena, still have significant room for development. To leverage high-performance new materials developed through research in real-world applications,

it is essential to adapt these materials to mass production processes, specifically by scaling up and accelerating the processes, so as to maintain performance while achieving target costs. Traditionally, optimization of these process conditions has relied on repeated trial-and-error based on intuition, experience, and expertise, or exhaustive experimental combinations of numerous process parameters, both of which require substantial time and financial resources^[1].

The New Energy and Industrial Technology Development Organization (NEDO) has formulated and published a roadmap for fuel cell and hydrogen technology development, with the aim of promoting long-term collaborative efforts among industry, academia, and government. HORIBA, Ltd. has participated since fiscal year 2023 in one of NEDO's projects, titled "Collaborative Research and Development for Solving Common Issues toward the Dramatic Expansion of Fuel Cell Utilization," which is an industry-academia-government initiative. The objective of this project is to construct a common platform for

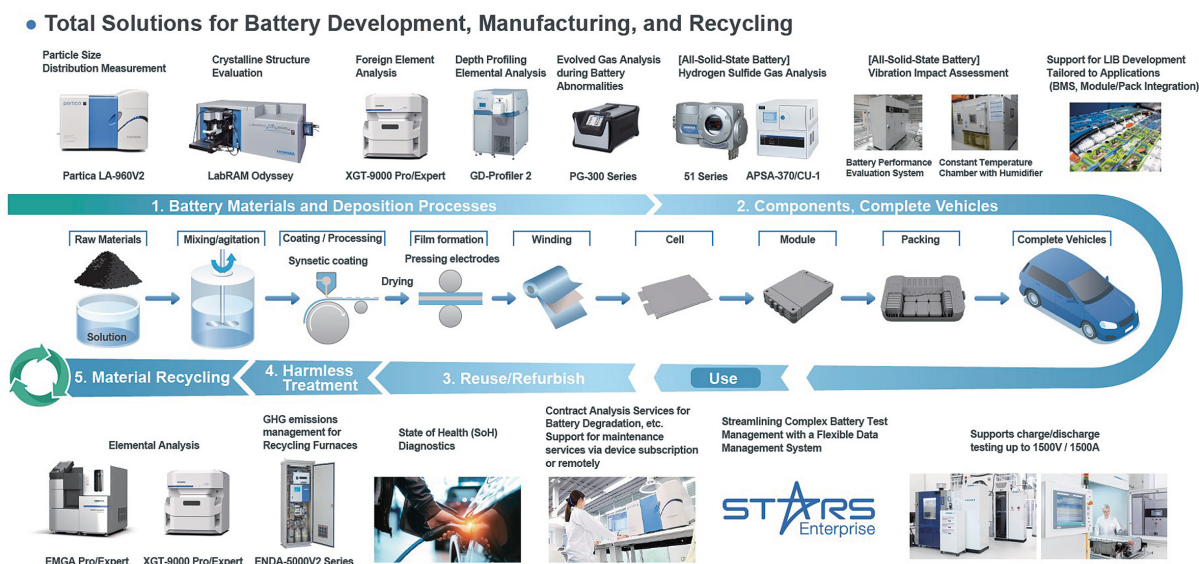


Figure 1 Battery life cycle and related analytical equipment.

“Process Informatics,” based on an automated experimentation and autonomous exploration system utilizing AI robots, in order to dramatically accelerate the development of fuel cell production processes. Within this project, the University of Tokyo is responsible for standard process development, Kanazawa University for prototype production of standard inks, Kyushu University for the development of evaluation methods, and HORIBA, Ltd. for elemental development, including the fabrication of an automated experimental apparatus simulating the production of fuel cell catalyst layers.

Concept of the Device in the Project

The apparatus developed in this project focuses on the aging process of the catalyst layer, which significantly affects production time and performance, in response to the demand for shorter fuel cell production times. Specifically, it is equipped with functions for “coating” and “drying,” as well as the capability to automatically conduct “evaluation” related to these processes. As a KPI, the project aims to achieve an experimental speed ten times faster than manual experimentation. With future practical deployment in mind, the design concept incorporates three major requirements, as described below.

1. Size Suitable for Installation in Existing Buildings (Elevator-Compatible)

Fuel cell production equipment is typically designed for mass production, with roll-to-roll (RtoR) systems applying battery materials to sheets extending hundreds of meters. The drying processes involve proprietary technologies, with parameters

such as temperature, airflow, duration, and flow patterns optimized during process development to improve yield. As a result, coating and drying units alone often exceed 10 meters in length, making it impractical to introduce such large equipment solely for process development. Therefore, the newly developed apparatus has been miniaturized to fit within the dimensions of a standard elevator, enabling installation without the need for building modifications.

2. Application of Conditions to Actual Production Lines and Low-Cost Operation

To minimize changes in conditions when scaling up to actual production, the apparatus incorporates a multi-stage drying furnace that simulates zone heating via hot air, as used in production equipment. To reduce the consumption of expensive fuel cell materials, a die-coating mechanism has been adopted. Depending on the coating thickness, the system is designed to operate at low cost, with a target of coating 100 samples per 1 mL of ink, each with a coated area of 1 cm².

3. Modularization of Measurement Units for Customizability

Following the fabrication of fuel cell catalyst layer samples via the coating and drying apparatus, performance evaluation is conducted. The current system evaluates surface roughness, image analysis, and electrical conductivity, with each measurement unit modularized to allow for future expansion and diversity in evaluation methods as data accumulates.

Introduction of the Device

Overall Overview

A photograph of the entire apparatus is shown in Figure 2. The system comprises two primary functions: the fabrication process and the evaluation process. By moving sample holders within the apparatus, the system enables the fabrication and evaluation of thin films for fuel cells. An external stocker unit is provided to store up to 108 sample holders, allowing for daily exchange and continuous 24-hour operation, thereby enabling testing of approximately 162 conditions. Based on previous experience, manual testing typically covers about 10 conditions per day, thus the apparatus achieves the KPI of a tenfold increase in testing speed compared to manual methods.



Figure 2 Automatic testing equipment.

A schematic diagram of the data exchange between each unit is presented in Figure 3. Sample fabrication is performed based on instruction data, and the conditions for the next fabrication process are determined from the measurement data obtained during evaluation, with this process repeated iteratively. The conditions are determined using a Bayesian optimization algorithm developed by the University of Tokyo. The following sections introduce each unit in detail.

Sample Holder and Transport

To facilitate sample transport, storage, and changes in film formation conditions, a dedicated sample holder was designed. The holder can accommodate sheet thicknesses of 50 μm and is compatible with various sheet materials of similar thickness, including the resin sheets used in this evaluation. PPS resin (polyphenylene sulfide), a type of super engineering plastic, was selected for its resistance to the hot air in the drying furnace. The apparatus is equipped with internal transport routes and a robotic arm to ensure smooth movement between units and to the stocker. These features enable miniaturization and versatility in coating materials, compared to conventional RtoR production equipment.

Coating

To minimize ink consumption and enable various evaluations, coating is performed at the minimum required size of 1 cm², consistent with the standard cell of the FC-Cubic research consortium. The die-coating method, driven by a plunger pump, is used for single-sheet coating, employing

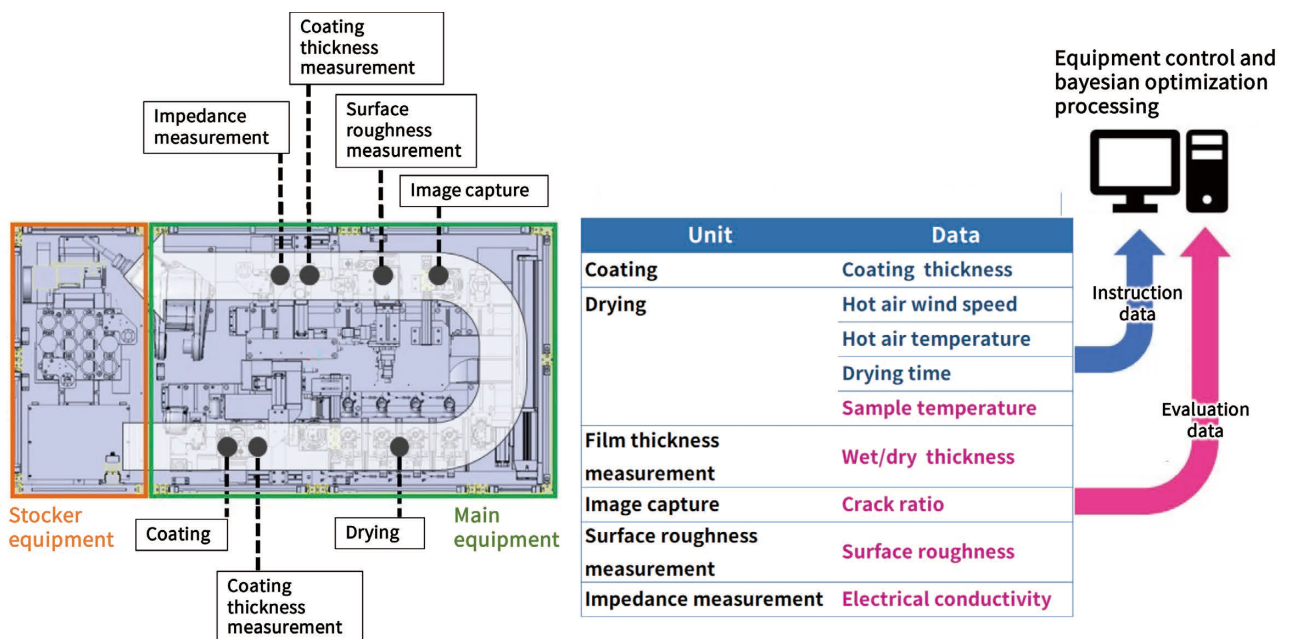


Figure 3 Top view of the equipment and overview of data transfer

the same principle as production processes. The coating thickness can be specified arbitrarily within the range of 50 μm to 200 μm for each test. To address the issue of ink drying in the slit die between tests, a small amount of ink is dispensed onto a waste sheet, enabling continuous testing within a single batch (Figure 4).

Film Thickness Measurement

The quality of the coating process is monitored by measuring the film thickness before and after drying, as well as by shape measurement via image capture. For film thickness measurement, both the WET thickness immediately after coating and the DRY thickness immediately after drying can be measured. A confocal optical displacement sensor is used, offering a height resolution of 1 μm , suitable for the sample characteristics. The sensor is positioned immediately after the slit die to synchronize with the coating operation and enable measurement of the film thickness directly after coating.



Figure 4 Coating test sample.

Drying

To simulate the production line and allow for independent setting of drying conditions, four zones of hot air drying furnaces are provided (Figure 5). Each furnace allows for arbitrary settings of airflow speed (0–10 m/sec), temperature (ambient to 180°C), and drying time. The hot air flow is precisely controlled using HORIBA STEC's mass flow controller SEC-E50. The central surface temperature of the sample during drying is measured using HORIBA's IT-480F radiation thermometer, which has been custom-designed for measurement distance and spot size for this apparatus, contributing to its compactness. Additionally, multiple points within the furnace are monitored with sheath thermocouples, enabling comparison of temperature conditions when scaling up to production facilities.

Image Capture and Surface Roughness Measurement

Depending on coating and drying conditions, cracks and differences in surface roughness may occur in the dried samples. To accurately assess the crack rate within the coated area, images are captured before and after drying using a telecentric optical system and transmitted light. Image processing is then used to measure the crack rate within the coated region. A high-resolution camera enables measurement of areas larger than 10 mm square with a pixel resolution of 6.9 $\mu\text{m}/\text{pixel}$.

Surface roughness is measured after drying using a white-light interferometer, allowing for height resolution of 1 μm within a 1 mm square area. By selecting the cutoff frequency, both fine roughness below the cutoff and coarse roughness above the cutoff can be measured, providing indicators of sample surface properties.

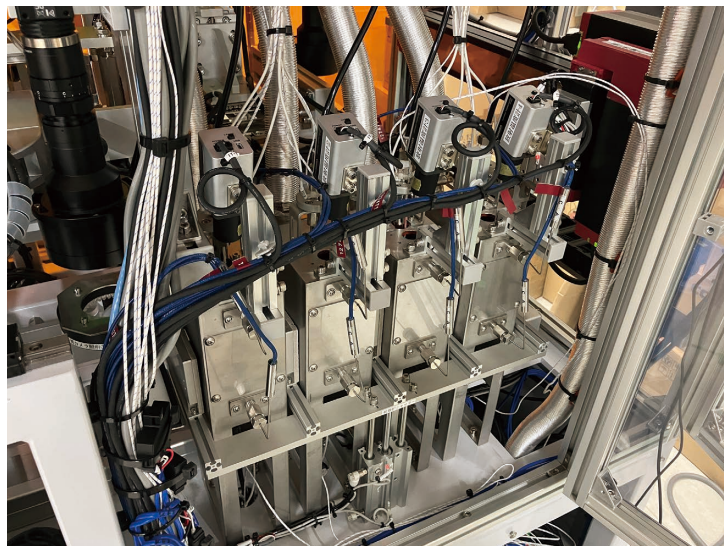


Figure 5 Drying unit

Impedance Measurement

Finally, contact-type impedance measurement is performed for evaluation. A compact sample probe is used to measure small samples, with the probe fixed to ensure consistent contact pressure for reproducible measurements. After passing through each unit described above, the sample holder is returned to the stocker. Bayesian optimization is then performed based on the obtained data to determine the next fabrication parameters, and the sample fabrication process begins anew. Throughout this process, no human intervention is required, enabling fully automated experiments to derive the optimal parameter values.

Conclusion

This paper has introduced an automated experimental apparatus developed by HORIBA, Ltd. that simulates the coating and drying production processes for fuel cells. The apparatus enables integrated data control of both production and evaluation parameters for fuel cells, while maintaining a compact form factor. Although coating and drying are only a part of the overall fuel cell production process, once data-driven optimization of production conditions is achieved using data obtained from this apparatus, it will become possible to implement production equipment in society that is labor-saving, space-efficient, and rapid, utilizing process informatics. Furthermore, by applying various analytical instruments as shown in Figure 1, new evaluation items—such as management of ink materials prior to coating or degradation evaluation of battery packs—can be implemented, enabling acquisition of diverse data and the introduction of new indicators. As such, the apparatus can play an important role as a DX solution throughout the entire lifecycle of fuel cells. As a manufacturer of analytical instruments that handle critically important data for research, we are committed to further promoting the use of process informatics and proposing DX solutions that meet the needs of developers and researchers.

Acknowledgment

We would like to express our sincere gratitude to all members of NEDO for their overall management and invaluable support throughout this project. We also extend our deep appreciation to the members of All-Star FC and FC-Cubic for sharing their profound knowledge and providing various insightful advice regarding production equipment for coating and drying. Furthermore, we wish to convey our

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* Editorial note: This content is based on HORIBA's investigation at the year of issue unless otherwise stated.

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