



燃料電池材料の構造・組成を見える化！開発・製造に活かす評価技術

2025年9月5日

株式会社リガク グローバルプロダクトマーケティング部

松田 渉



燃料電池車市場の成長

- 世界の FCEV 累計台数

2023 年 ≈ 9万台 → 2030 年 160–210 万台へ¹⁾

- 商用車の開発製造が活発

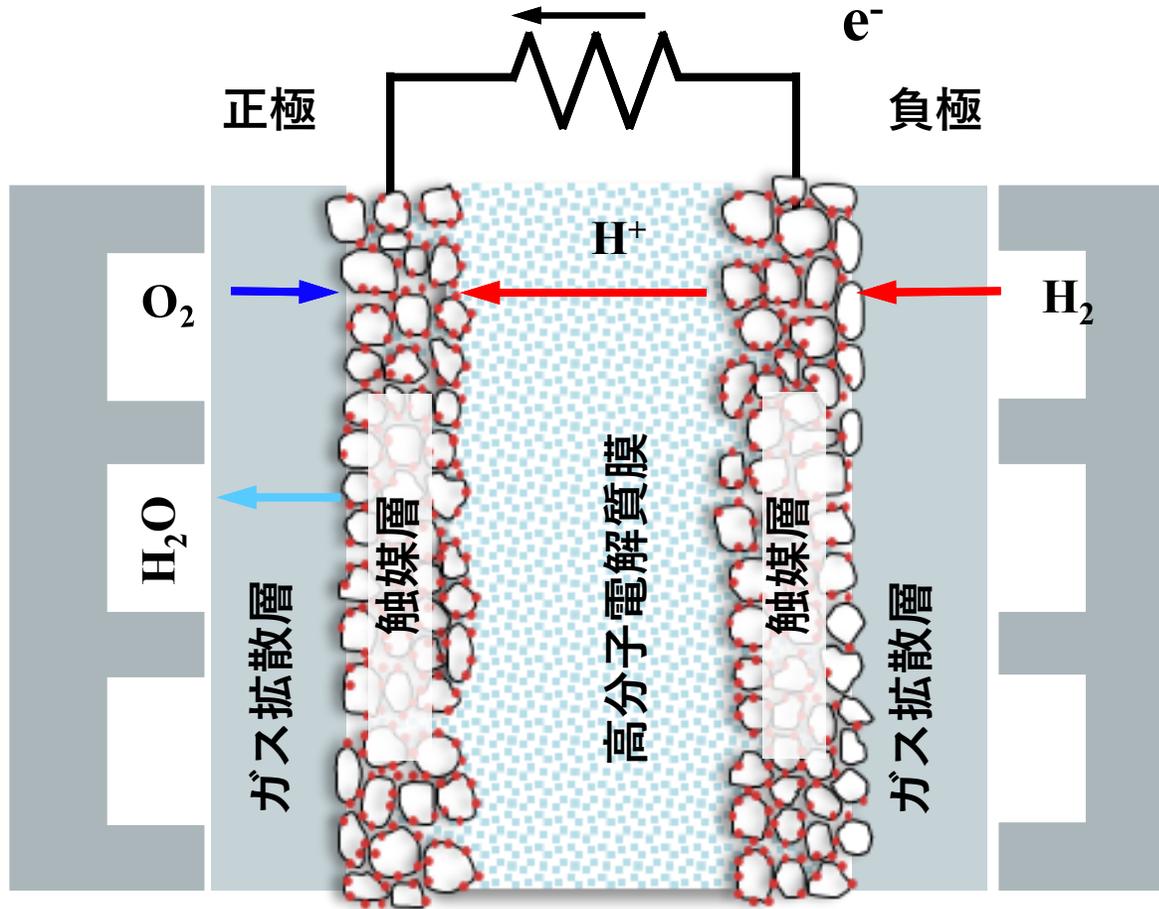
中国では 2024 年の新規 FCEV の大半がバス・トラック²⁾

- 大量生産 → 品質管理ニーズ急増

1) IEA “National FCEV targets 2030” 集計値 (2024)

2) DOE HDV Cost Analysis 2023

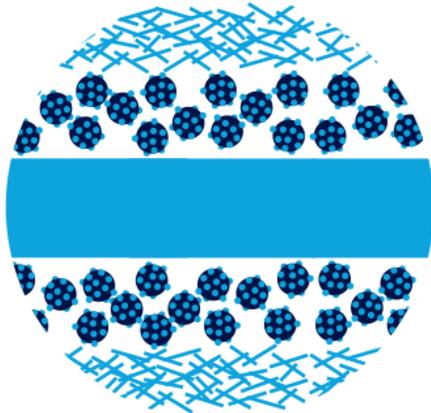
固体高分子形燃料電池 (PEFC) の仕組み



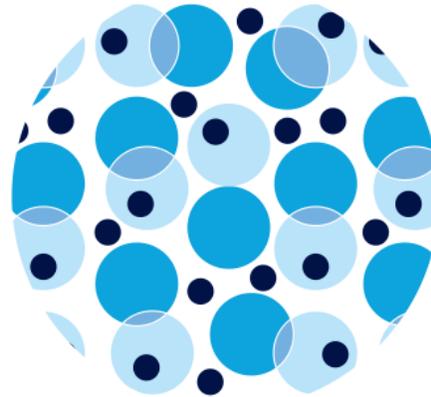
Point

- H_2 や O_2 を用いて発電するクリーンエネルギー

燃料電池の性能とコストを左右する事例



ガス拡散層の空隙閉塞
電圧の降下



Pt 粒径粗大化
電極触媒活性の低下



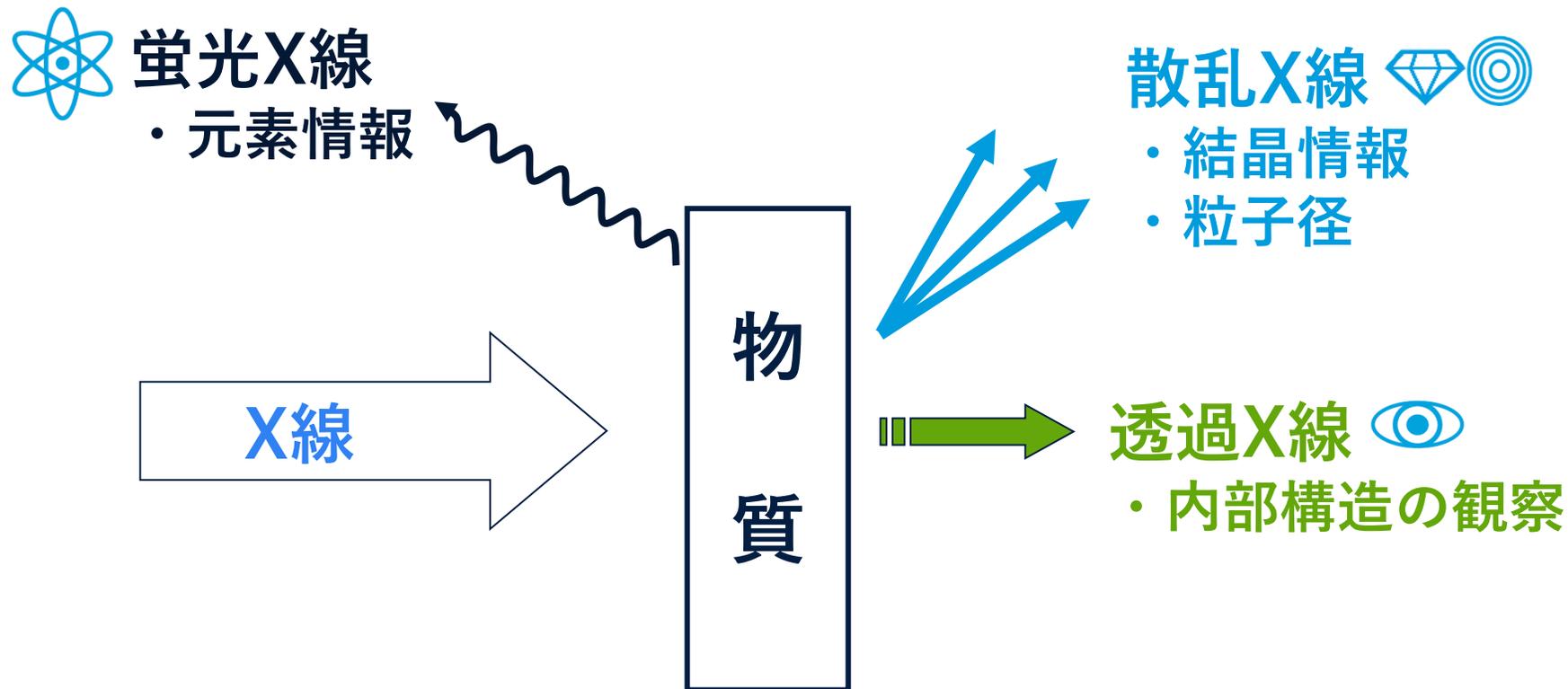
触媒の付着量のばらつき
歩留まりの悪化

★求められる技術

- 非破壊
- 定量性

→X線分析が解決します

X線と物質の相互作用



X線分析による燃料電池材料の評価



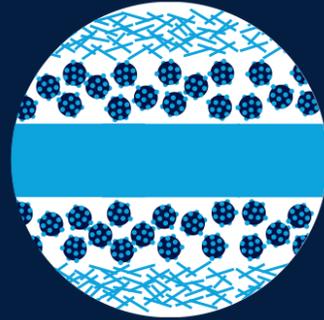
膜電極接合体の内部構造観察



触媒粒子の結晶子サイズと粒径分布の評価

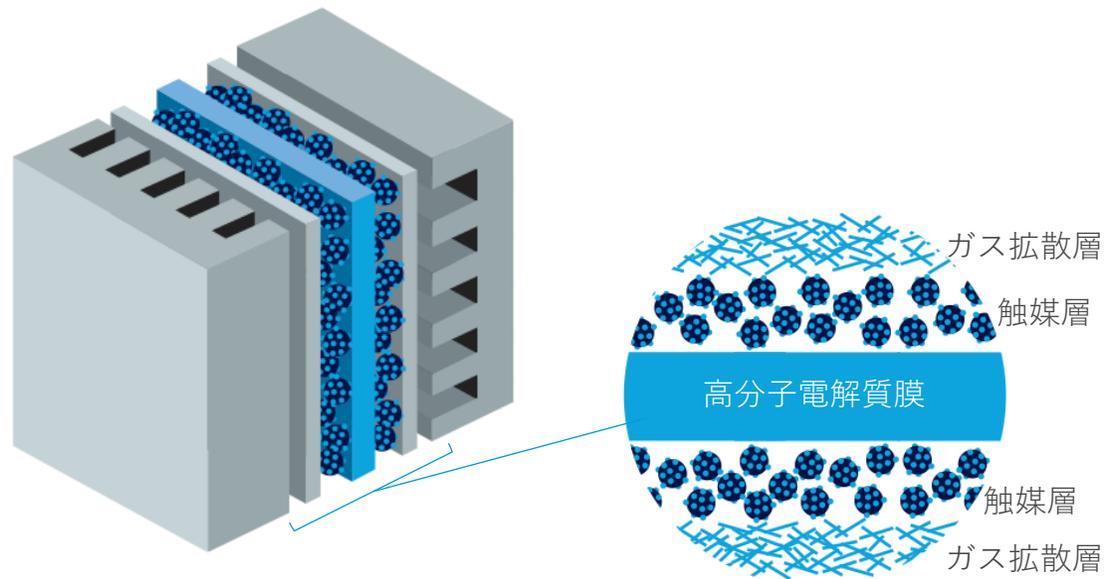


触媒シートの付着量管理



膜電極接合体の 内部構造観察

MEA(膜電極接合体) 内部構造が性能を左右する



MEA：（膜電極接合体Membrane Electrode Assembly）
高分子電解質膜+触媒層+ガス拡散層を一体化した発電部

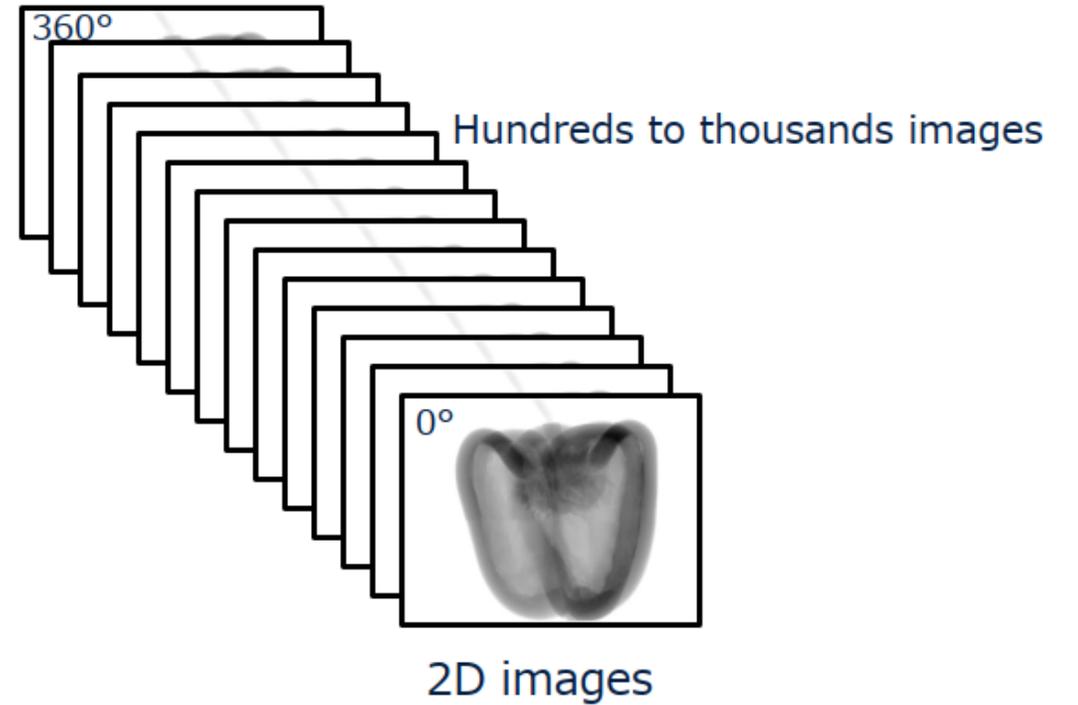
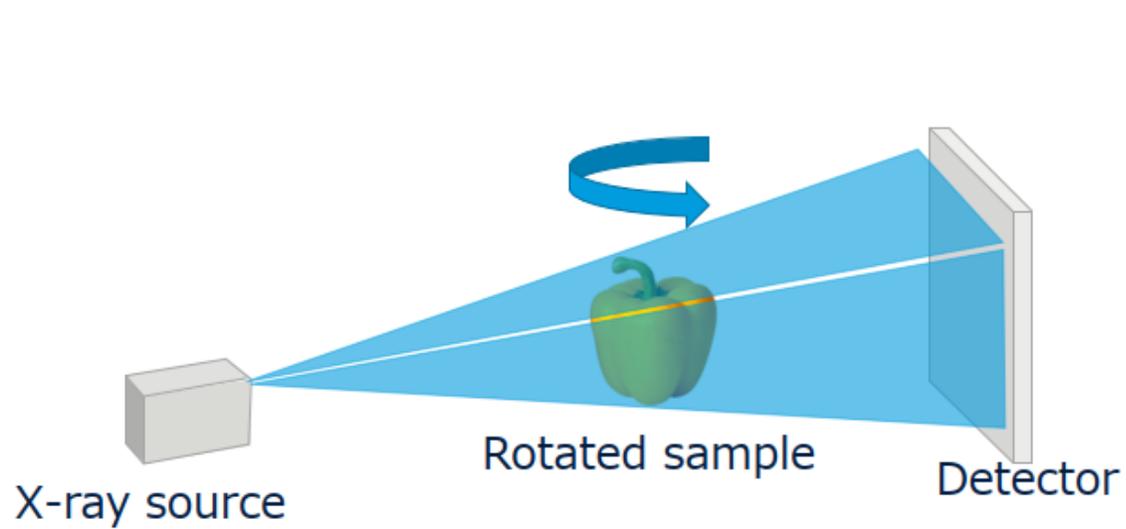
★ガス拡散層や触媒層の空隙構造の役割
水素・酸素の供給経路、水の排出経路

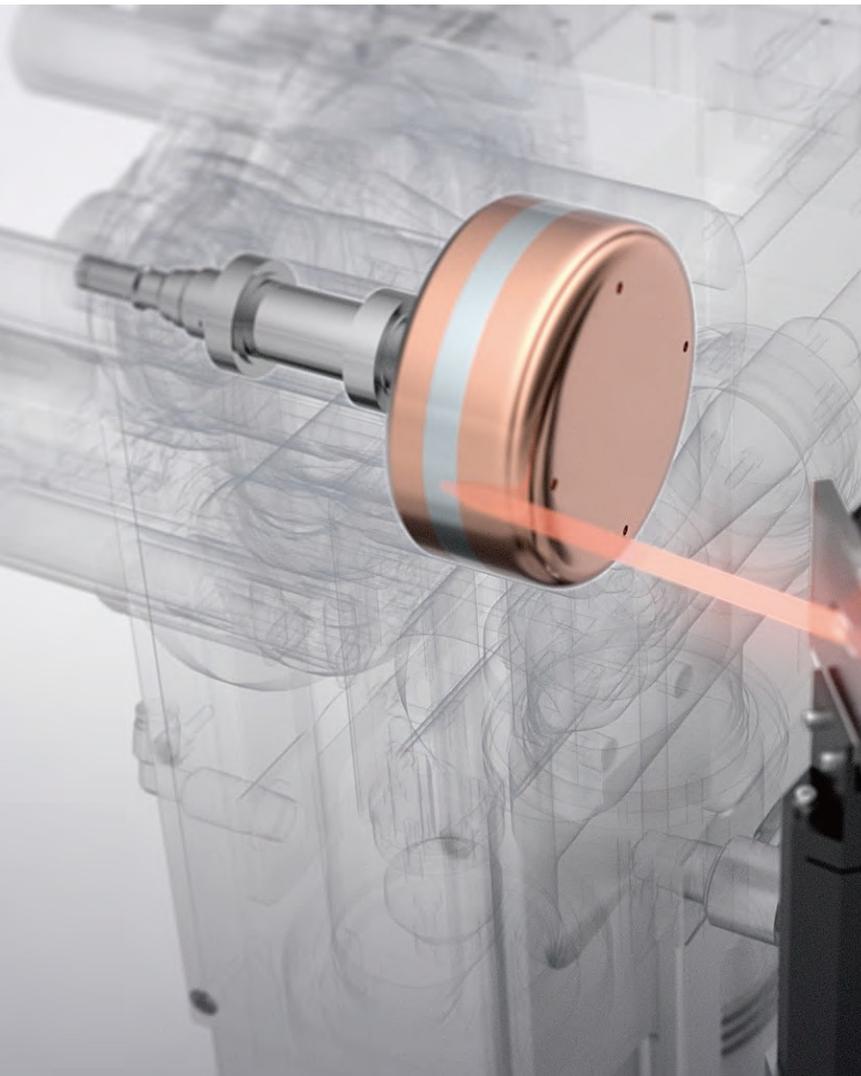
空隙のサイズ・連結性が不適切だと

- ・ガス供給不足 → 発電性能の低下
- ・水滞留（フラッディング）→ 電圧の低減
- ・ナフィオンの乾燥 → 導電率の低下

内部構造を、そのまま観察する技術として
X線CTを提案

X線CTによる内部構造観察



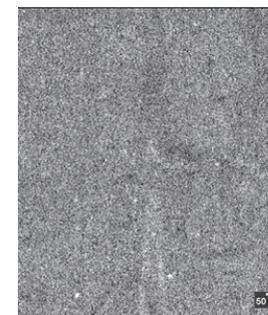


高分解能3DX線顕微鏡 nano3DX

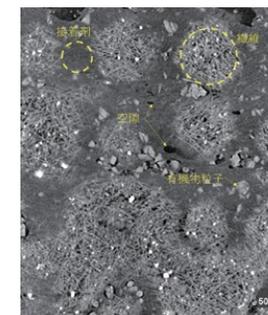


- 高輝度X線源 1200W
- 特性X線の活用

有機物試料の観察事例



W線源

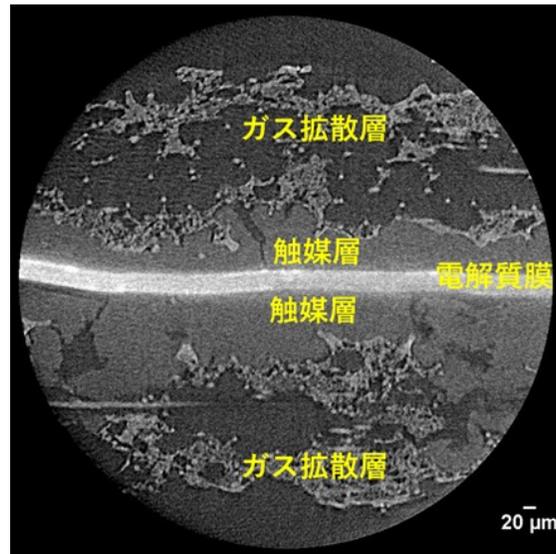


Cu線源

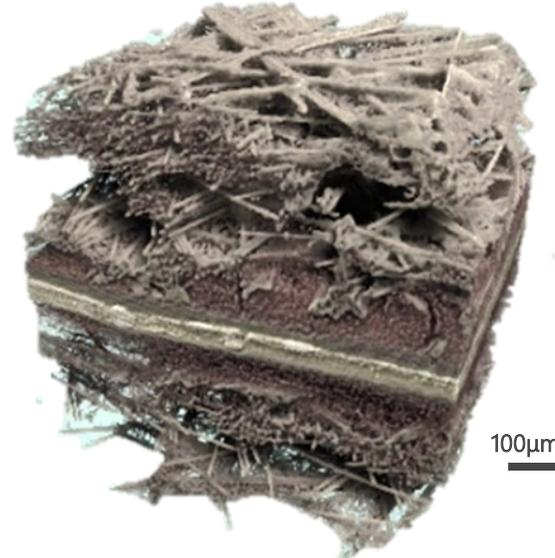
短時間でキレイな画像を取得できます

MEA内部の空隙や材料の積層状態を非破壊で観察

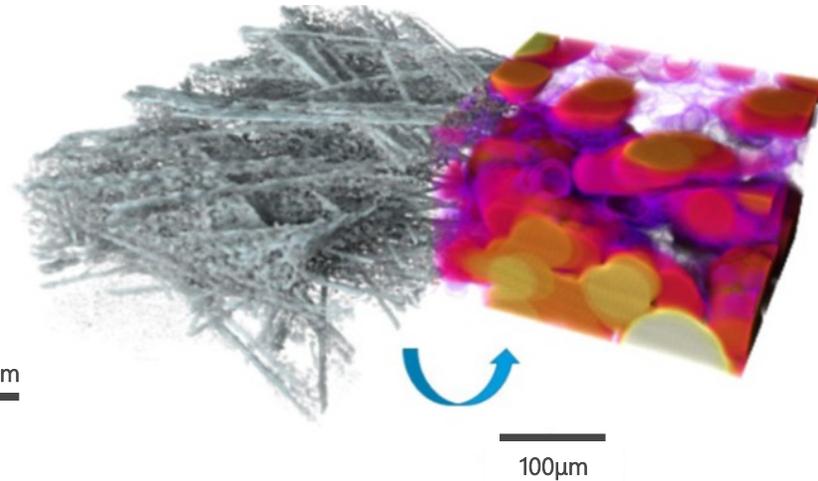
断面画像



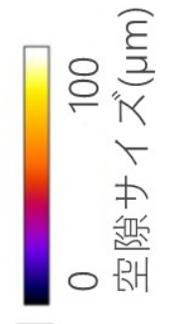
三次元画像



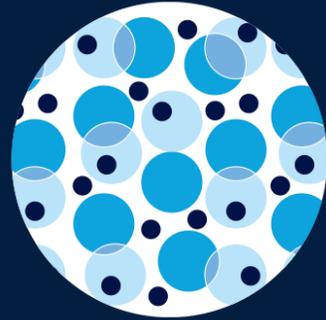
ガス拡散層



抽出した空隙の
三次元有効サイズマップ

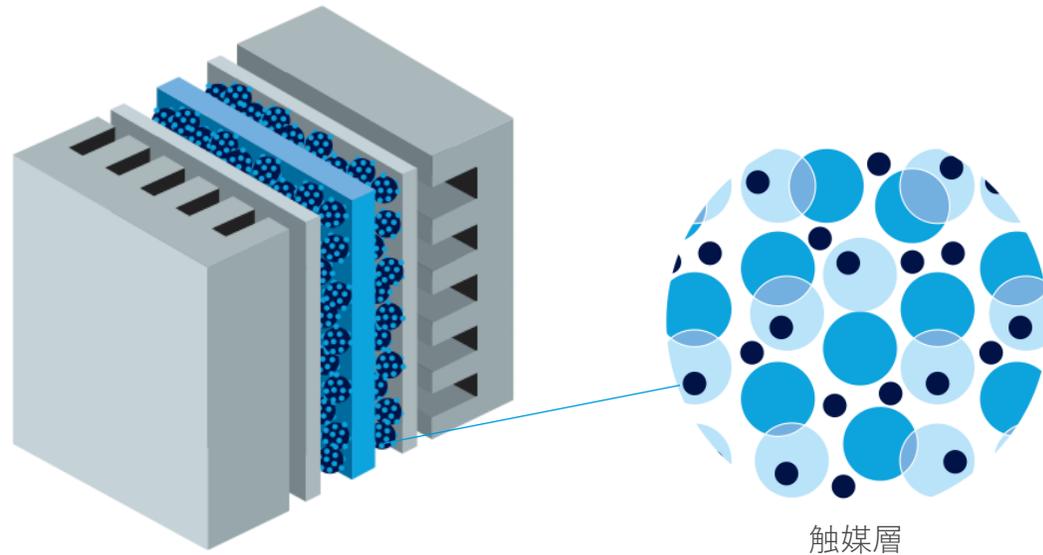


非破壊で、内部構造を観察できるだけでなく、空隙サイズの定量も可能です



触媒粒子の結晶子サイズ と粒径分布の評価

触媒ナノ粒子の“粒径と結晶子サイズ”が性能を左右する



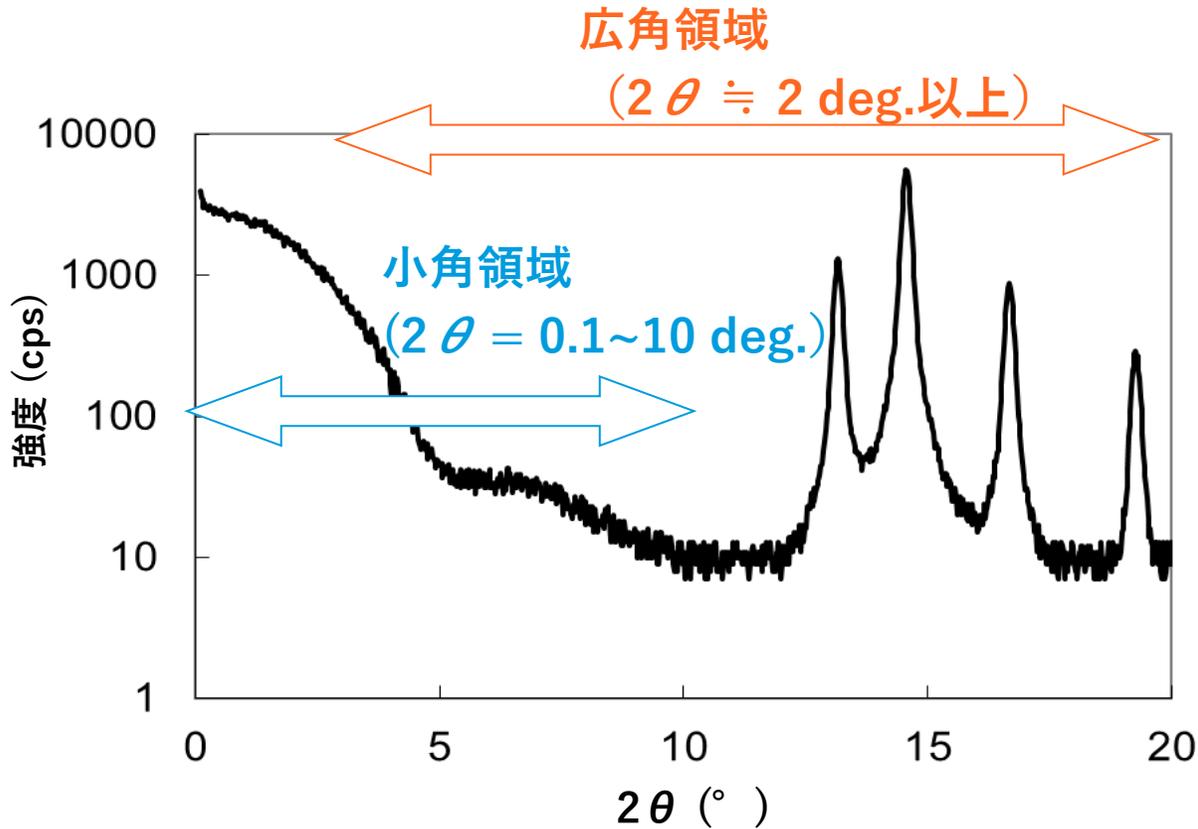
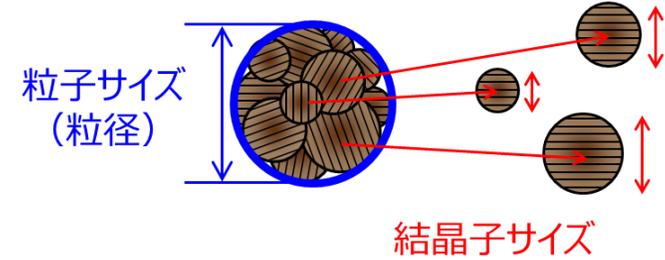
触媒ナノ粒子の役割：

酸素還元反応/水素酸化反応の活性中心

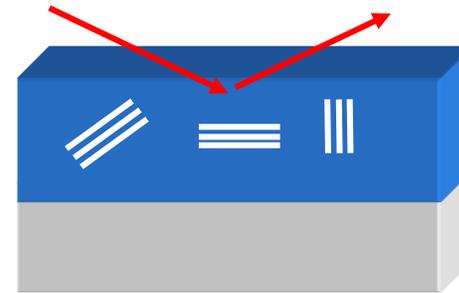
- 粒径の増加 → 活性の低下
- 結晶子の粗大化 → 電圧劣化を引き起こす
- 粒径分布大 → Pt使用量増加

粒径1~100nmと結晶子サイズを
非破壊で測定する技術としてXRDとSAXSを提案

X線回折法と小角X線散乱法

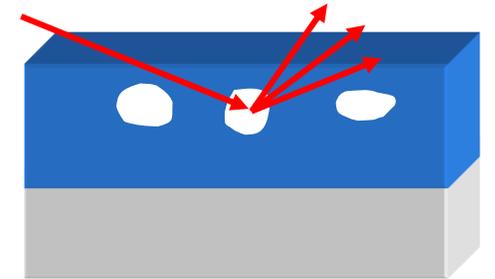


X線回折法 XRD



- 定性、定量分析
- 結晶構造解析
- 結晶性評価
(結晶子サイズ、歪み)

小角X線散乱法 SAXS



- 粒子径・
空孔径分布解析

全自動多目的X線回折装置 SmartLab



- 測定目的に対応した光学系
- あらゆる実験環境を再現

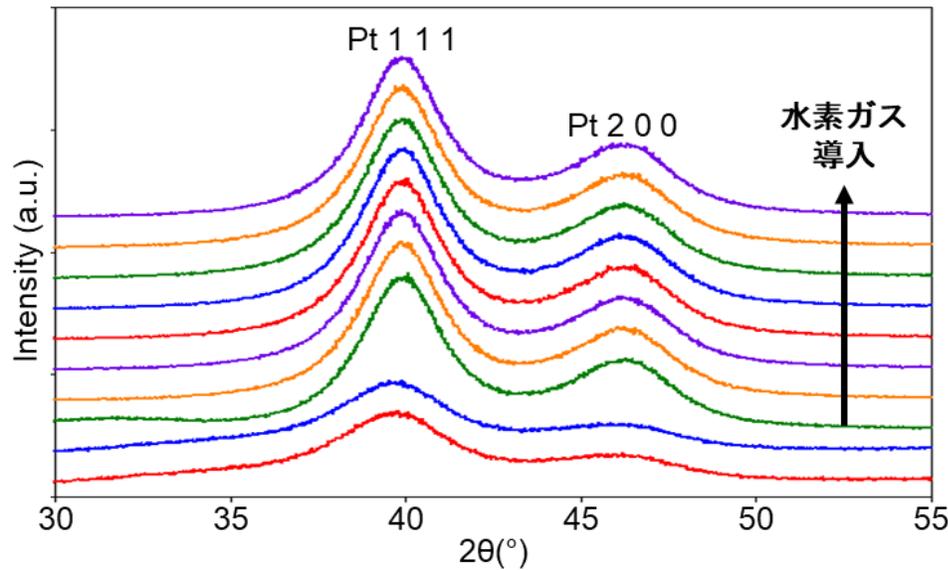


水素ガス雰囲気下で
*In-situ*測定が可能

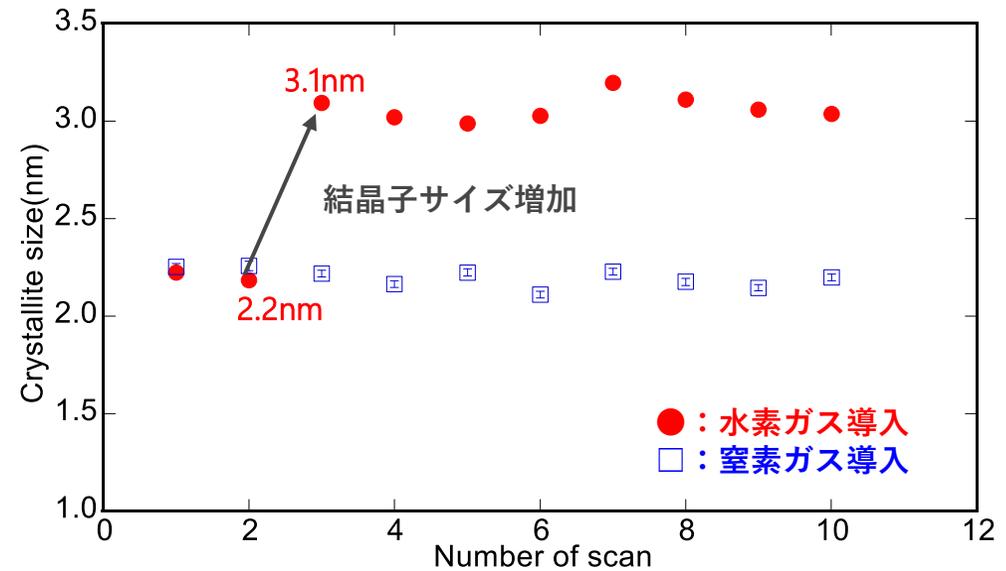
非破壊で結晶子サイズや粒径を測定できます

水素ガス雰囲気下 *in-situ* XRD測定結果

試料：Ptナノ粒子（粒径2~3 nm）



水素ガス導入によるXRDパターン

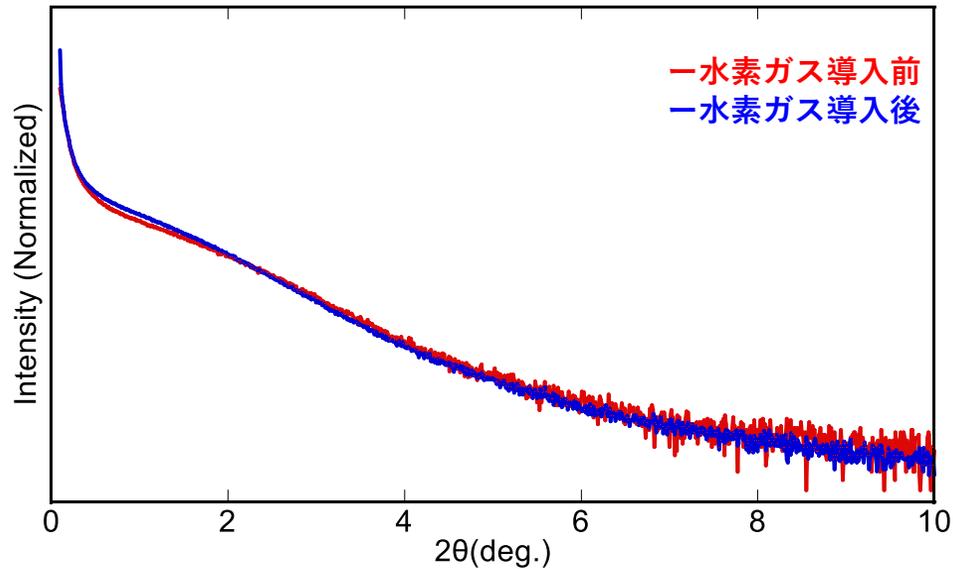


水素ガス導入によるPtナノ粒子の結晶子サイズ変化

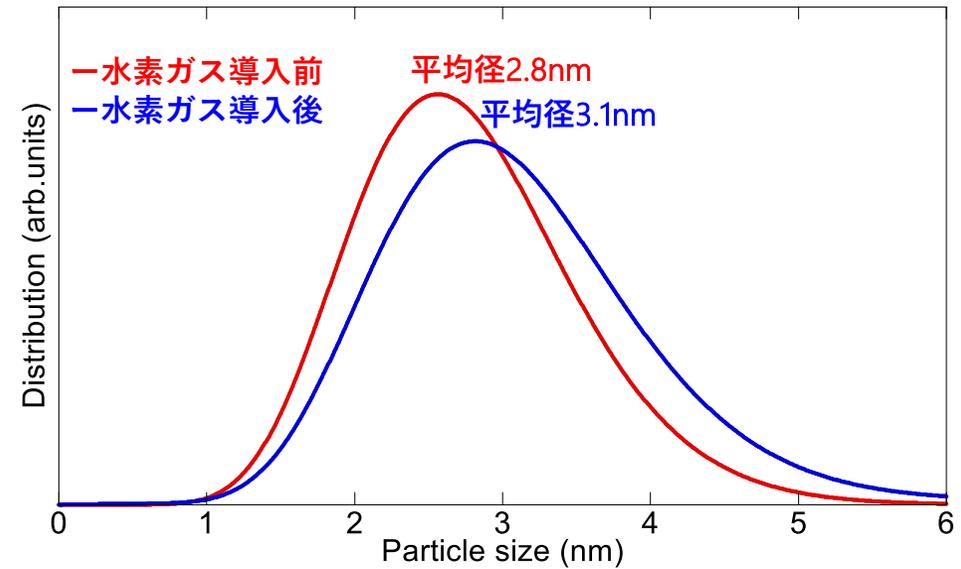
ガス導入により回折プロファイル変化をその場で観測が可能です

水素導入前後のSAXS測定結果

試料：Ptナノ粒子（粒径2~3 nm）



水素ガス導入前後における小角散乱パターン



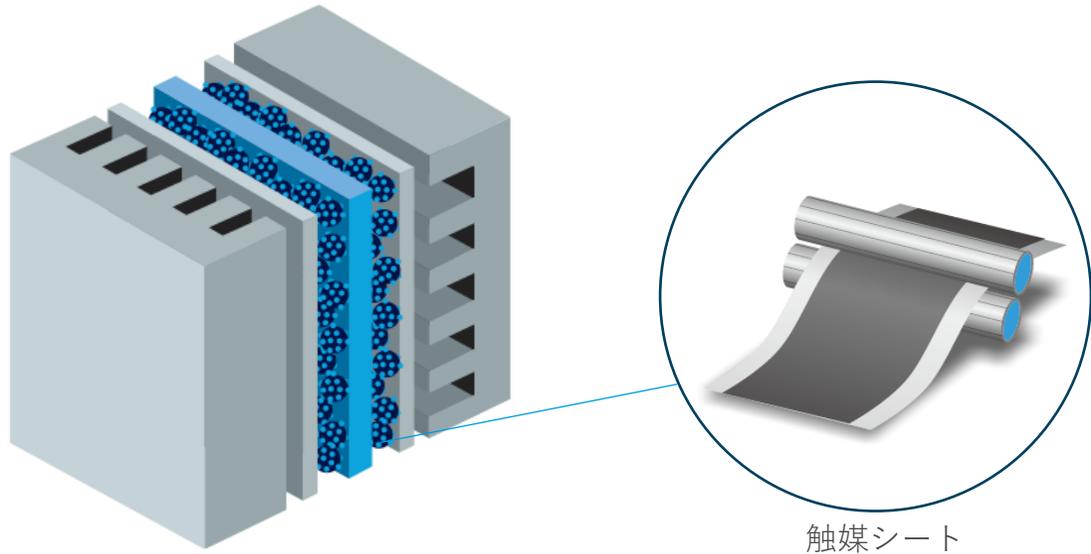
水素ガス導入前後におけるPtナノ粒子の粒径分布解析結果

水素導入に伴い酸化被膜が金属Ptに還元され、結晶子サイズとともに平均粒径が増加しました



触媒シート の 付着量管理

触媒塗布量の“過多⇔不足”がコストと歩留まりを左右する



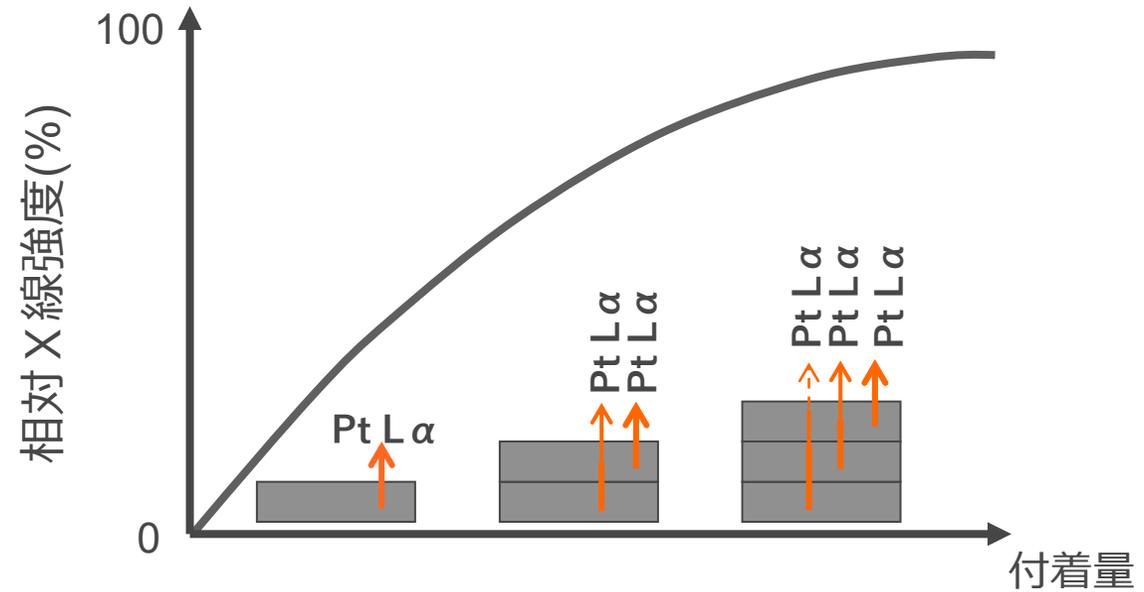
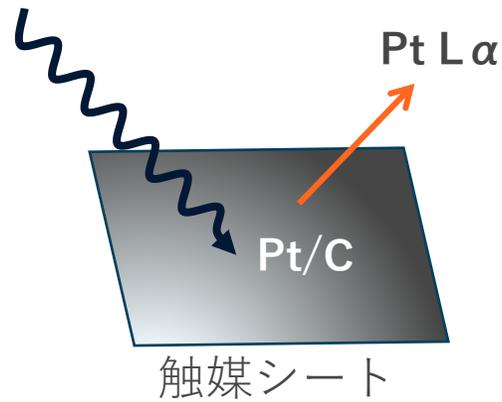
燃料電池材料コストうち、約 30 %*がPt

- ・ 過剰な塗布 →材料費増、ガス拡散阻害
- ・ 不足な塗布 →出力低下、耐久劣化
- ・ 面内ばらつき →歩留まりの悪化

非破壊で高速に面内の触媒の塗布量（付着量）を
高速で測定する技術→XRFが最適

※DOE Fuel Cell Cost Analysis 2023 の HDV/LDV ブレークダウン値を四捨五入

Ptの付着量の評価方法



元素情報（蛍光X線）から付着量を定量可能です

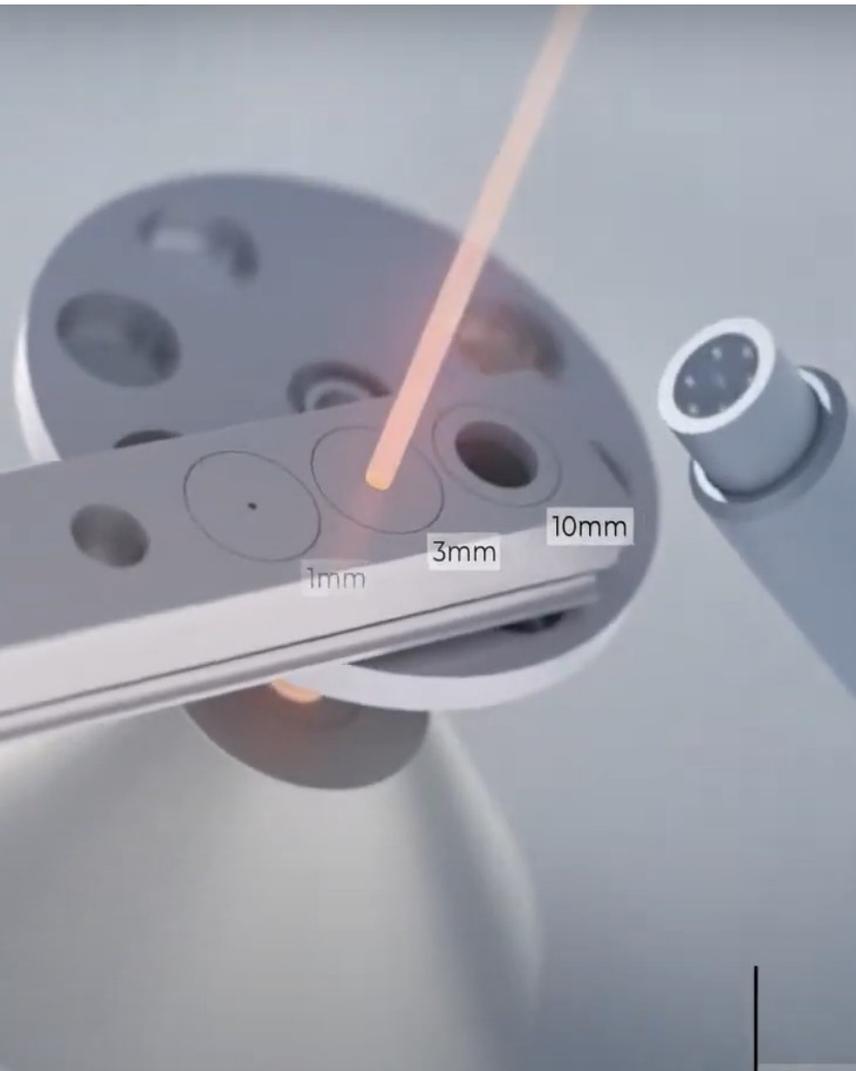
蛍光X線分析装置 NEX DE

- 30 × 30 × 10cmの試料室
- 1, 3, 10mm径の測定機能

有機物試料の観察事例

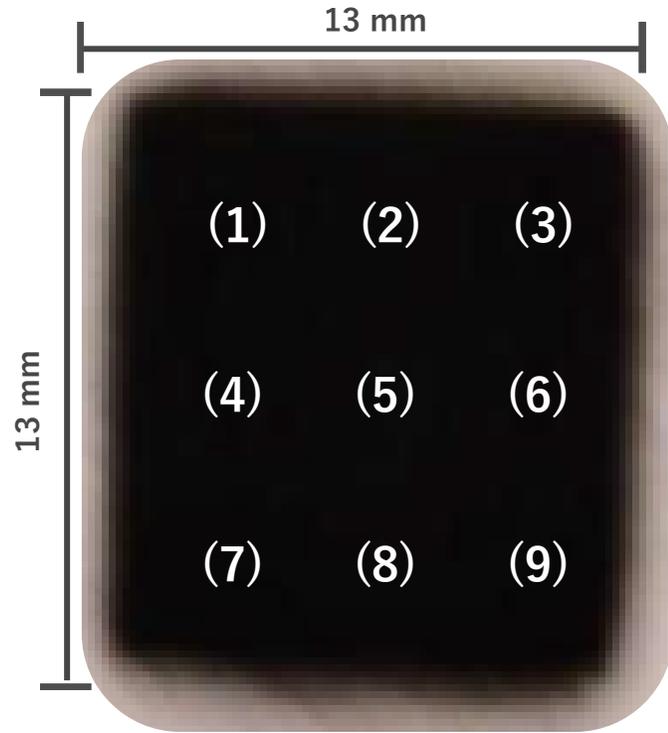


装置に置くだけ，内蔵カメラでポイント指定



非破壊で触媒シートの付着量を分析できます

触媒シートへのPt付着量分析



触媒シートおよび測定座標

XRFによる各座標の分析結果[mg/cm²]

(1) 0.191	(2) 0.184	(3) 0.171
(4) 0.171	(5) 0.167	(6) 0.163
(7) 0.163	(8) 0.142	(9) 0.139

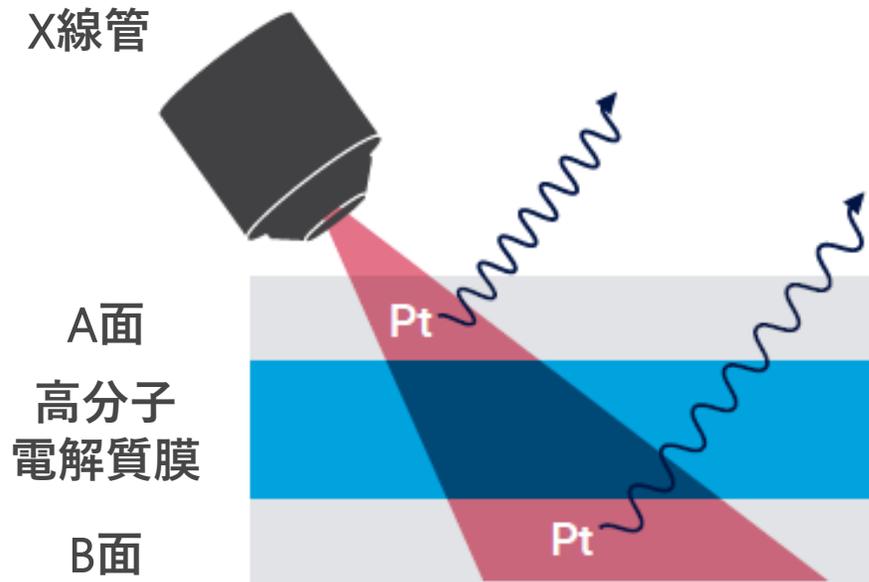
MAX. 0.20

MIN. 0.10

() : 座標番号

(1), (2), (8), (9)は±0.020 mg/cm²以上のムラがあることが分かりました

触媒シートへのPt付着量分析



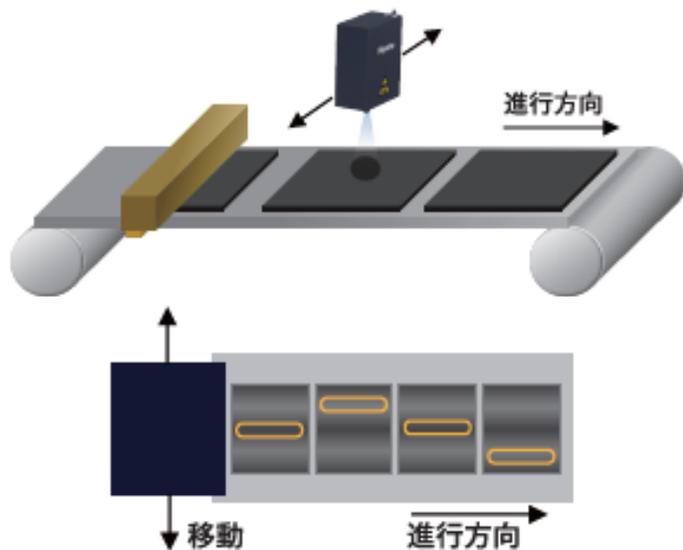
XRFによる各面の分析結果[mg/cm²]

測定面	A面	B面
分析値	0.201	0.420
設計値	0.203	0.405
相対誤差(%)	1.0	3.7

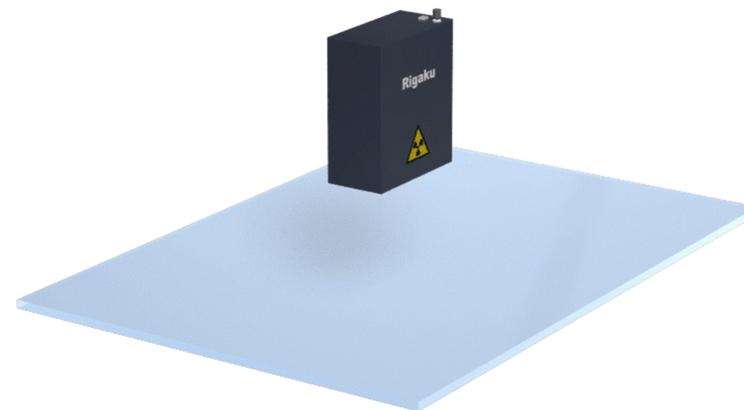
表面だけでなく、裏面の付着量を同時に分析可能です

触媒シート インライン・オンライン対応ヘッド

ロールtoロール



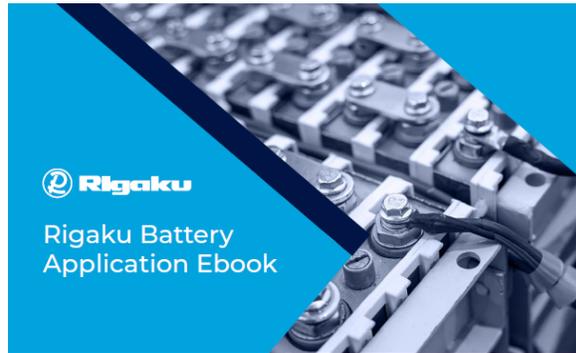
枚葉



相対標準偏差 $\pm 1\%$ ¹⁾の精度で管理可能

¹⁾膜種や測定時間により変わります

燃料電池のアプリケーションを揃えたe-book



Rigaku
Rigaku Battery
Application Ebook

About Rigaku



Who We Are

Since its inception in 1951, Rigaku has been at the forefront of analytical and industrial instrumentation technology. Today, with hundreds of major innovations to their credit, the Rigaku group of companies are world leaders in the fields of general X-ray diffraction, thin film analysis, X-ray fluorescence spectrometry, small angle X-ray scattering, protein and small molecule

X-ray crystallography, Raman spectroscopy, X-ray optics, semiconductor metrology, X-ray sources, computed tomography, nondestructive testing and thermal analysis.

Corporate Mission

To contribute to the enhancement of humanity through scientific and technological development.

RIGAKU BATTERY APPLICATION EBOOK

Fuel Cell

Rigaku's analysis technologies support R&D for safer and more efficient cells.

Fuel Cell

Separator
Gas diffusion layer
Electrolyte membrane

Analyze: Electrolyte membrane
For: Evaluation of thermal stability
Thermal analysis

Catalyst

Analyze: Catalyst
For: Optimizing electrochemical performance and quality assurance
Particle size and structure analysis
Elemental analysis

MEA, Membrane Electrode Assembly (Gas diffusion layer, catalyst layer and electrolyte membrane)

Analyze: MEA, Membrane Electrode Assembly
For: Optimizing electrochemical performance
Non-destructive imaging

RIGAKU BATTERY APPLICATION EBOOK

Analysis of Adhesion within Both Sides of a Fuel Cell (PEFC) Electrode

Analyze: Fuel cell
Use: Quality assurance
Elemental Analysis

Analyzed materials:
Pt as catalyst

In a polymer electrolyte fuel cell (PEFC: Polymer Electrolyte Fuel Cell), a cathode layer and an anode layer are placed on both sides of the electrolyte membrane. Since the amount of platinum (Pt) catalyst on each electrode layer is different, each layer must be managed separately. Conventional control methods require each surface to be measured separately, resulting in a large workload and time cost. X-ray fluorescence analysis is an effective method for efficiently solving these problems because it has high penetrating power, is nondestructive, and can simultaneously evaluate the amount of Pt adhered on both surfaces in a single measurement.

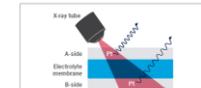


Figure 1: Schematic diagram of measurement

Measurement surface	A-side	B-side
Analysis value	0.001	0.42
Design value	0.003	0.405
Relative error (%)	1.0	3.7

Table 1: Analysis results for each aspect

Conclusion: Both sides (A-side and B-side) of the electrolyte membrane sheet coated with platinum-loaded carbon electrode were measured for 60 seconds using X-ray fluorescence of platinum and quantitatively analyzed by the thin film FP method. As shown in Table 1, the analysis results for each measurement point agreed with the design values for each surface within 15% relative error, confirming that the platinum loading in this film formation process was properly controlled.

RIGAKU BATTERY APPLICATION EBOOK

Evaluation of Water Absorption and Swelling in Polymer Electrolyte Membranes

Analyze: Fuel cell
Use: Evaluation of thermal stability
TA: Thermal Analysis

Analyzed materials:
Electrolyte membrane

In the fuel cell operating environment, changes in temperature and humidity affect the expansion and contraction of materials. Dimensional changes due to water absorption in polymer electrolyte membranes can have a significant impact on cell sealing and adhesive durability. There are limited methods to evaluate these properties in a humid environment, and STA and TMA with controlled humidity are effective approaches in material design and selection.

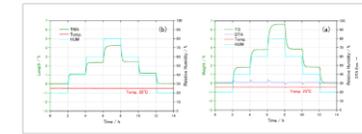


Figure 1: (a) weight change and (b) dimensional change of polymer electrolyte membranes as a function of relative humidity.

Conclusion: STA measurements under humidity-controlled conditions allow quantitative evaluation of water absorption by measuring the weight change of a sample under each relative humidity condition. TMA can also be used to determine the swelling behavior under each humidity condition.

RIGAKU BATTERY APPLICATION EBOOK

Internal Structure Evaluation of Fuel Cell Membrane Electrode Assembly (MEA)

Analyze: Fuel cell
Use: Optimizing electrochemical performance
Non-destructive analysis

Analyzed materials:
MEA, Membrane Electrode Assembly

For improving the performance and quality of fuel cells, it is important to evaluate the internal structure of the gas diffusion layer (GDL) in the membrane electrode assembly (MEA), a multifunctional base material responsible for gas diffusion, electron collection, and discharge of produced water. GDLs are composed of low-density materials such as carbon fiber, and conventional X-ray CT imaging using high-energy X-rays does not provide contrast because X-rays are not absorbed by GDLs, making it difficult to analyze pore size and diffusion pathways. The nanoCT, which is capable of low-energy X-ray CT imaging, makes it possible to visualize and evaluate the structure of GDLs, which is expected.



Figure 1: X-ray CT image of a membrane electrode assembly (MEA) and analysis of gas diffusion layer (GDL) pores

Conclusion: CT imaging using low-energy characteristics: X-rays of the NanoCT enabled high-contrast and clear observation of GDLs even in the presence of electrolyte membranes with high-density platinum catalyst attached. By visualizing the internal structures of all the substrates comprising the prototype MEA in three dimensions and quantitatively evaluating structural indices such as the GDL pore size, it will be possible to study the optimization of the MEA preparation conditions.

RIGAKU BATTERY APPLICATION EBOOK

SAXS-RMC Method for Estimating 3D Structure of Pt/GDC Supported Nanoparticles

Analyze: Fuel cell
Use: Optimizing electrochemical performance
Particle size and structure analysis

Analyzed materials:
Pt as catalyst

Catalysts consisting of platinum nanoparticles supported on gadolinium-doped ceria (Gd-doped ceria: GDC) (Pt/GDC catalysts) have been reported to exhibit better four electron oxygen reduction reaction activity than commercially available Pt/C catalysts. It is important to ensure that the reactions at the catalyst surface in the catalyst bed are facilitated, e.g., by the oxygen and hydrogen gas supply pathways. To understand these properties, it is useful to evaluate the three-dimensional structure of the entire catalyst layer, which is a complex aggregation of nanoparticles. However, with conventional observation methods, it is difficult to nondestructively determine how the nanoparticles are aggregated in three dimensions. By combining small angle X-ray scattering (SAXS) and reverse Monte Carlo (RMC) methods, it is possible to estimate the three-dimensional aggregation structure of nanoparticles non-destructively and statistically.

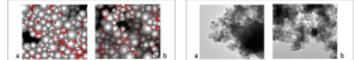


Figure 1: The resultant structural models obtained by RMC to match the observed SAXS patterns. Red and gray spheres for Pt/GDC particles, respectively.

Figure 2: A typical TEM image of Pt/GDC.

Conclusion: The structural model constructed by the combination of SAXS and RMC methods was in good agreement with the TEM observation results. Furthermore, the pore size distribution calculated based on the model was in good agreement with the experimental results obtained by nitrogen gas adsorption, confirming the reproducibility of macroscopic structural indices. These results indicate that the SAXS-RMC method is a very useful 3D structural analysis technique that is superior in both visualization of local structure and statistical structure reproduction.

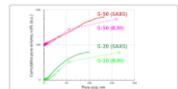


Figure 3: The SAH analysis result curves are shown in the dashed line with the measurement data points in open circles. Average curves of three RMC simulations run for SAXS patterns are shown in the solid lines.

© 2025 RIGAKU HOLDINGS CORPORATION AND ITS GLOBAL SUBSIDIARIES. ALL RIGHTS RESERVED.

まとめ：X線が様々なシーンで活躍します

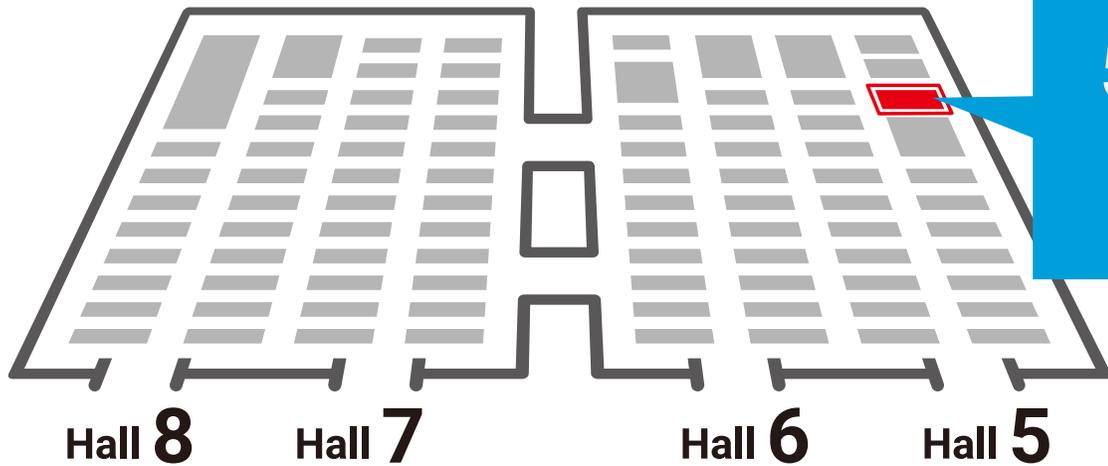
視点	主要課題	X線技術	効果
構造	空隙詰まり	X線CT	発電安定
ナノ粒子	粗大化	XRD + SAXS	活性維持/劣化予測
付着量	過多・不足・面内ムラ	XRF	歩留まりの向上



リガクウェブサイトの右上のボタン 「**専門家に相談する**」より お問い合わせください



ぜひ、リガクブースへ
お立ち寄りください！



 Rigaku

5ホール 入って直進！

5A-801



