Feature Article

特集論文

Materials Analysis of SOFC/SOEC Stack's Components using HORIBA's Scientific Instruments

HORIBAの科学機器によるSOFC/SOECスタック構成部材の材料分析

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This article summarises measurements done on Solid Oxide Fuel Cell (SOFC)/Solid Oxide Electrolysis Cell (SOEC) in HORIBA application lab using inhouse scientific instruments. It also gives an analysis of SOFC and SOEC market trend in testing, and summarises insights from research literature. The focus of this review was introducing materials analysis in SOFC/SOEC sector and explaining application of HORIBA's scientific instruments in this sector. It was highlighted that SOFC/SOEC markets not only needs materials analysis at the R&D step, but also along the production line. This includes measurements from the preparation of raw materials to in-line quality control and post-mortem analysis. HORIBA can provide scientific instruments to make a large number of these measurements. These measurements will help to improve SOFC/SOEC cells, stacks and systems performance and durability, which can be determined with HORIBA test stations and Balance of Plants.

HORIBAの科学機器を使用したアプリケーションラボでの測定結果，文献，固体酸化物形燃料電池(SOFC)/固体酸化物形電解槽(SOEC)の市場動向に関するHORIBAの分析結果をまとめて紹介する。このレビューの焦点はSOFC/SOEC分野における材料分析の紹介と，HORIBAの科学機器の応用について説明する。SOFC/SOEC市場では，研究開発段階での材料分析だけでなく，生産ラインでの材料分析も必要であることが強調された。これには原材料の準備からインラインの品質管理，事後分析までの測定が含まれる。HORIBAはこれらの測定を数多く行うための科学機器を提供することができ，これらの測定はSOFCセル，スタック，システムの性能と耐久性の向上に役立ち，HORIBAのテストステーションやBalance of Plantsで判定することが可能である。

Introduction

Due to climate change, Alternative Energy Solutions (AES) are under investigation to decarbonize societies. Among these solutions, fuel cells and electrolysis cells are considered as promising solutions to participate in the energy transition. Fuel cells (Solid Oxide Fuel Cell, Proton Exchange Membrane Fuel Cell…) convert certain fuels into electricity via an electrochemical reaction. Electrolysis cells (Solid Oxide Electrolysis Cell, Proton Exchange Membrane Electrolysis Cell, Alkaline Electrolysis Cell, Anion Exchange Membrane Electrolysis Cells…) convert electricity and certain fuels into other fuels (Figure 2, for SOFC and SOEC).

Fuel cells are promising because they can use renewable fuels such as Green Hydrogen, and their fuel to electricity efficiencies are higher than many energy systems such as gas turbine, Steam turbine and Diesel engine.[2] Many technologies of fuel cells exist, they are summarised in Table 1.

Electrolysers are promising because they can produce Hydrogen from water and electricity by emitting less CO_2 than the conventional steam-methane reforming process.[3] Also, electrolysers can produce synthetic fuels from non-fossil energy sources: CO_2 and H_2O.[4] Alkaline Electrolysis Cell (AEC), Proton Exchange Membrane Electrolysis Cell (PEMEC), Solid Oxide Electrolysis Cell (SOEC) and Anion Exchange Membrane Electrolysis Cell (AEMEC) are the four types of electrolyser technologies that are used today.

Table 1 Fuel Cell technologies.[2]

Type of fuel cell	Electrolyte	Operating Temperature	Fuel	Oxidant	Electrical Efficiency	Application
PEM (Proton Exchange Membrane)	Polymer: proton exchange membrane	50-80°C	Pure Hydrogen produced from hydrocarbons, melthanol or electrolysis	O_2/Air	40-50%	Transportation, H2 production.
AFC (Alkaline Fuel Cell)	Potassium hydroxide (KOH)	50-200°C	Pure Hydrogen or hydrazine liquid or methanol	O_2/Air	50-55%	Stationary (Apollo).
AEM (Anion Exchange Membrane)	Polymer	25-70°C	Hydrogen, methanol, maybe other alcohols and N-fuels (research)	O_2/Air	60%	Transportation, H2 production.
PAFC (Phosphoric Acid Fuel Cell)	Phosphoric Acid	160-210°C	Hydrogen produced from hydrocarbons or methanol	O_2/Air	40-50%	Stationary 100-400kW, Heavy duty, Air-independent propulsion (submarine)
MCFC (Molten Carbonate Fuel Cell)	Molten salts such as NO_3, SO_4, and CO_3	630-650°C	Hydrogen, CO, natural gas, propane, marine diesel	CO_2/O_2/Air	50-60%	Stationary
DMFC (Direct Methanol Fuel Cell)	Polymer	60-200°C	Liquid methanol	O_2/Air	40-55%	Power for man-portable tactical equipment, Battery chargers, and Autonomous power for test and training instrumentation
SOFC (Solid Oxide Fuel Cell)	Ceramic such as YSZ or CGO	600-1000°C	Hydrogen, natural gas or propane	O_2/Air	45-70%	Stationary, H2 production, CHP, Integrated coal gasification sysems, Integrated steam turbines, Electric vehicles (APU)

Components of a SOFC/SOEC Stack

Fuel Cells and Electrolysis Cells technologies differ in the materials used to make them. These technologies (Table 1) are not necessarily in direct competition; they can have different applications. This article focuses on the Solid Oxide Fuel Cell (SOFC) and Solid Oxide Electrolysis Cell (SOEC) technologies which are publicly known as High temperature Fuel Cell and High temperature Electrolysis Cell. When these technologies are used, they are part of a system made of a stack and auxiliaries. A stack is made of cells (fuel cells or electrolysis cells), stacked together with interconnectors, glass sealings, end plates and compression rods (not shown) (Figure 1). Interconnectors conduct gases through the stack and connect electrically cells together. Glass sealings seal gases compartment (H_2/air, H_2/atmosphere, and O_2/atmosphere compartments). End plates participate in the compression of the stack, provide electrical insulation between adjacent cells, and enable the stack to be connected to gases and electricity. Compression rods maintain the stack compressed. Depending on the kind of Solid Oxide Cells used (High temperature, Intermediate temperature, Low temperature), SOFC and SOEC stacks use different materials for the cathode, electrolyte, anode, and interconnectors. The most common materials used in SOFC stacks are listed in Table 2.

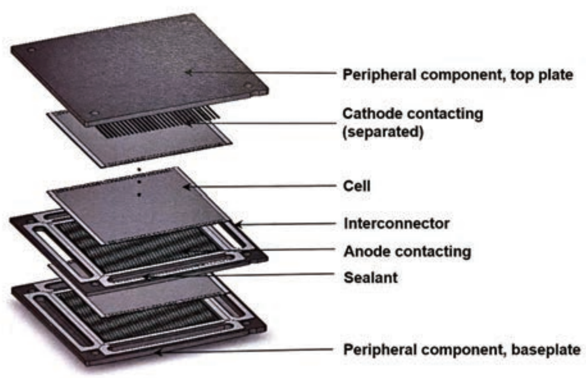


Figure 1 Exploded view of an assembled SOFC/SOEC planar cell,[5] where four rods and nuts at each corner crimp these planar cells together.

SOFCs and SOECs

SOFCs and SOECs are made of solid oxides, especially electroceramics. Some SOFC are reversible, so they can be used as SOEC.[7] The SOFC technology is well-known for its fuel tolerance and flexibility, high electrical efficiency (45-70%)[2] and high operating temperature (600-1000°C).[2] Three families of SOFC exist:

- High temperature SOFC, operating between 900°C and 1000°C;
- Intermediate temperature SOFC, operating between 700°C and 900°C;
- Low temperature SOFC, operating between 500°C and 700°C.

High temperature SOFCs are mostly used in Integrated

Table 2 Main materials used in SOFC.[6]

	Cathode	Electrolyte	Anode	Interconnect
High temperature SOFC (900-1000°C)	$La(Sr)MnO_3$	$Zr(Y)O_{2-x}$	$Ni-Zr(Y)O_2$	$LaCr(Mg)O_3$
Intermediate temperature SOFC (700-900°C)	$La(Sr)MnO_3$, $La(Sr)Co(Fe)O_{3-x}$	$Zr(Y)O_{2-x}$, $Ce(Gd)O_{2-x}$, $La(Sr)Ga(Mn)O_{3-x}$	$Ni-Zr(Y)O_2$	$Cr-Fe(Y_2O_3)$, $Inconel-Al_2O_3$
Low temperature SOFC (500-700°C)	$La(Sr)Co(Fe)O_{3-x}$	$Ce(Gd)O_{2-x}$, $La(Sr)Ga(Mn)O_{3-x}$	$Ni-Zr(Y)O_2$, $Ni-Ce(Gd)O_{2-x}$	Stainless steel

coal gasification systems and grid power distribution.[4] Intermediate temperature SOFCs are mainly used in Combined Heat and Power (CHP) systems,[8] integrated gas turbines[9] and vehicles (Auxiliar Power Unit).[10],[11] Low temperature SOFCs are principally used in Combined Heat and Power (CHP) systems, Auxiliary Power Units (APU) of electric vehicles.

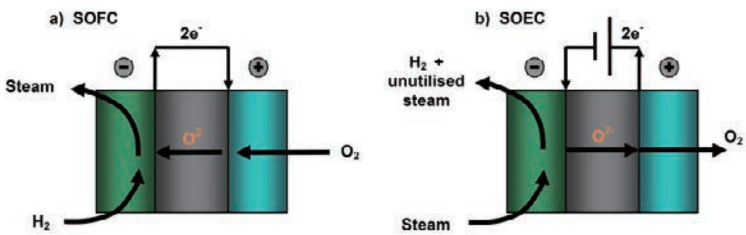
SOFCs can use hydrogen as fuel, this will produce electricity and water (Figure 2): at the cathode $O_2(g)$ is reduced to O^{2-}; in the electrolyte O^{2-} ions are transported from the cathode to the anode and at the anode $H_2(g)$ is oxidised to $H_2O(g)$. Hydrogen can be produced by Solid Oxide Electrolysis Cell (SOEC) (Figure 2): at the cathode $H_2O(g)$ is reduced to $H_2(g)$ and O^{2-} ions; in the electrolyte O^{2-} ions are transported from the cathode to the anode and at the anode O^{2-} ions are oxidised to $O_2(g)$.

SOFC/SOEC Materials Analysis

Along the development cycle of a SOFC/SOEC stack, all constituting materials (interconnector, glass sealing, end plate, compression rod, cells) are analysed. In this article, a focus is made on the materials analysis of cells (SOFC/SOEC).

SOFC/SOEC Materials Properties and Performance

SOFCs/SOECs are characterised in terms of performance and durability. The performance of a SOFC/SOEC relates to its electrical efficiency, fuel utilisation, and power density at different current densities. The durability of a SOFC/SOEC has to do with the degradation of the performance over time and test cycles, in certain test conditions. The SOFC/SOEC performance and durability depend on the cell design, intrinsic properties of materials (electronic conductivity, ionic conductivity, thermal expansion coefficient, Oxygen self-diffusion coefficient, Surface exchange coefficient, Area Specific Resistance, porosity content, grains size, thickness, …)[12] and operating conditions (air and fuel compositions, temperature, gas pressure, …). To optimise SOFC/SOEC materials, the link between materials composition and microstructure, and performance and durability tests must be understood. For this, the materials composition and microstructure are analysed along the manufacturing steps, drafted in Figure 3, where firstly powders are prepared from raw materials, secondly inks are produced from powders, dispersants, solvents, and binders, thirdly inks are coated on a support and dried, fourthly cut to the right dimensions, and then fifthly coated layers are sintered.



SOFC	SOEC
Anode (-)	Anode (+)
$H_{2(g)} + O_o^x \rightarrow H_2O_{(g)} + V_o^{\bullet\bullet} + 2e'$	$O_o^x \rightarrow 2e' + \frac{1}{2}O_{2(g)} + V_o^{\bullet\bullet}$
Cathode (+)	Cathode (-)
$\frac{1}{2}O_{2(g)} + V_o^{\bullet\bullet} + 2e' \rightarrow O_o^x$	$H_2O_{(g)} + V_o^{\bullet\bullet} + 2e' \rightarrow H_{2(g)} + O_o^x$
Total	
$H_{2(g)} + \frac{1}{2}O_{2(g)} \rightarrow H_2O_{(g)}$	$H_2O_{(g)} \rightarrow H_{2(g)} + \frac{1}{2}O_{2(g)}$

Figure 2 Schematics and electrochemical reactions taking place in a SOFC and a SOEC.[1]

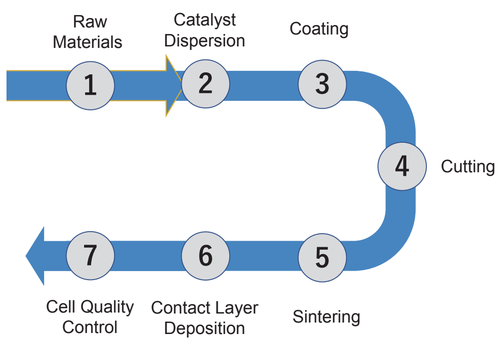


Figure 3 Schematic of the SOFC cell manufacturing.

Raw Materials Characterisation

Raw materials particles are characterised at line with instruments such as HORIBA XploRa™ PLUS confocal Raman microscope (Figure 4), HORIBA Partica LA-960V2 laser scattering particle size distribution analyzer (Figure 5), HORIBA nanoPartica SZ-100V2 Series nanoparticle analyzer (Figure 6) and HORIBA Partica CENTRIFUGE CN-300 centrifugal nanoparticle analyzer (Figure 7) to know their composition, crystallinity, size and distribution.

Ink Characterisation

Inks are characterised at line to know the particles size distribution in ink with HORIBA Partica LA-960V2 laser scattering particle size distribution analyzer (Figure 5) or with HORIBA SZ-100V2 Series nanoparticle analyzer (Figure 6) or with HORIBA Partica CENTRIFUGE CN-300 centrifugal nanoparticle analyzer (Figure 7), their rheology, viscosity, and zeta potential with HORIBA SZ-100V2 Series. Figure 8 shows results from HORIBA Partica LA-960V2 using the high concentration cell on an ink.

Figure 8 shows the results of the relationship between the scanning electron microscope (SEM) morphology of a PEFC (polymer electrolyte fuel cell), (not of SOFC here) cell coated layer and the particle size distribution of the ink analyzed by HORIBA Partica LA-960V2 using a high concentration cell. By using the high concentration cell, the particle size distribution of ink can be measured without diluting the ink as much as possible. Figures 8(a), (b), and (c) show that the particle size distribution is approximately 20 to 300 µm for Sample A, 0.2 to 1 µm for Sample B, and 0.1 µm for Sample C, respectively. Figures 8-(d), (e), and (f) show surface SEM images of the coated layers of Sample A, B, and C. Figures 8-(g), (h), and (i) show cross-sectional SEM images. The results show that the surface morphology of the coated layer becomes rougher and the variation of film thickness becomes larger when the inks with large particle size distribution are applied, that clearly confirms the effect of the catalyst dispersion solution on the microstructure of the coated layer.

Figure 4 XploRA PLUS Raman Spectrometer - Confocal Raman Microscope.

Figure 5 Partica LA-960V2 Laser Scattering Particle Size Distribution Analyzer.

Figure 6 nanoPartica SZ-100V2 Series Nanoparticle Analyzer.

Figure 7 Partica CENTRIFUGE CN-300 Centrifugal Nanoparticle Analyzer.

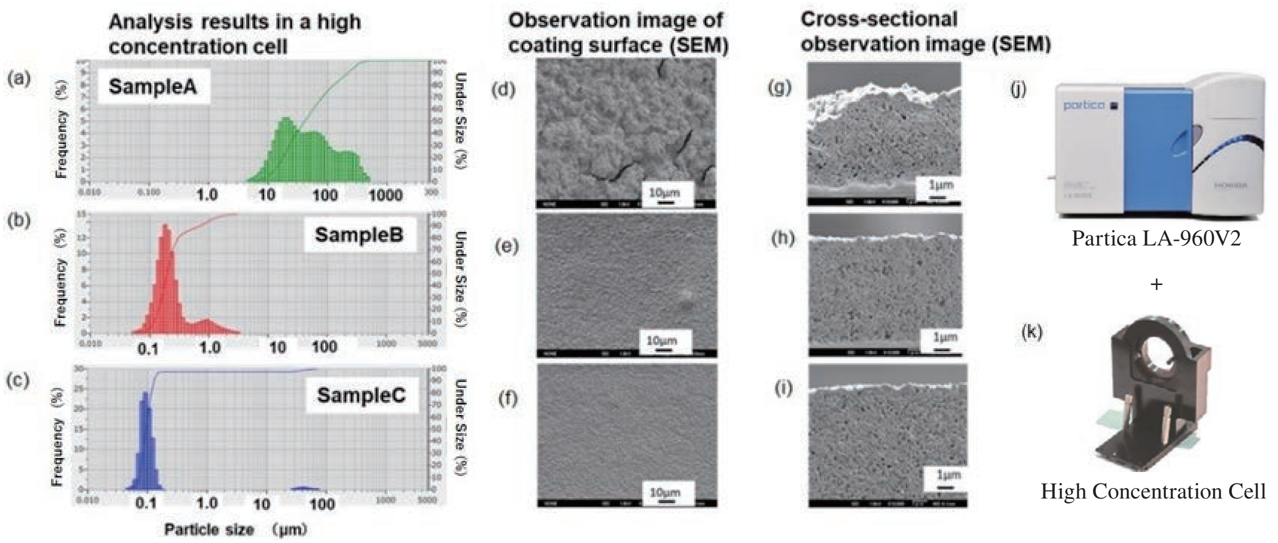


Figure 8 Particle size distributions (a-c) and SEM (d-i) data from different ink preparations. Particle size distributions were obtained with HORIBA Partica LA-960V (j) using High Concentration Cell (k) [Data provided by: Hirai & Sasabe Laboratory, Tokyo Institute of Technology, FC-Cubic].

Coating Characterisation

Nowadays, cells manufacturers want to measure the coated catalyst film thickness and catalyst loading inline, and ensure that the coated film shows a homogeneous distribution of the catalyst and the binder from the coating step to the drying step. Also, they want to detect defects (over thicknesses, impurities, pinholes, bubbles …).

HORIBA can provide an inline XRF solution to measure the amount and thickness of catalyst coated films inline. Figure 9 shows a photograph of the X-ray analysis head section (a) and an image of its X-ray analysis unit when installed in a roll-to-roll facility (b). As shown in Figure 9-(c), multiple X-ray analysis heads can be installed on the roll-to-roll coating film, or the X-ray analysis head can scan over the film while making measurements. HORIBA can also provide Raman solutions to detect defects and control the homogeneity of catalyst and binder in coated films. The example in Figure 10 shows Raman mapping (a) of the surface of a humid SOFC/SOEC air electrode using a HORIBA LabRAM Soleil Raman Spectrometer (c). The colour of each spectrum shown in Raman spectrum (b) corresponds to the colour of Raman mapping (a), where blue shows materials X, red shows materials X+C-H (polymer), and green and orange show Fe_3O_4 or Fe_2O_3 (impurity) respectively.

Sintering Characterisation

Today's market looks for inline instruments to detect defects and fast offline instruments to measure the thickness of sintered cells. HORIBA can provide inline XRF (Figure 9) or offline XRF solutions for defect detection and inline measurement of sintered layer thickness. Figure 11 shows the elemental composition analysis of dark impurity spots (b) on the SOFC anode surface using the HORIBA XGT-9000 (c), a micro X-ray fluorescence analyzer capable of elemental analysis and elemental mapping of small areas as small as φ10 µm. Figure 11-(a) shows the results of five elemental analyses, and it was found that a higher concentration of Fe_2O_3 was detected in the dark spot area than in the reference area, indicating that it was the cause of the dark spot. HORIBA can also provide a Raman solution to detect defects in the sintered layer. Figure 12 shows a microscope image and Raman mapping of a sintered SOFC anode surface by HORIBA LabRAM soleil. The colour of the Raman mapping image (a) corresponds to the colour of the Raman spectrum (b), showing red: materials X; red: ZrO2 ; blue: NiO; yellow: $BaTiO_2$ (impurity).

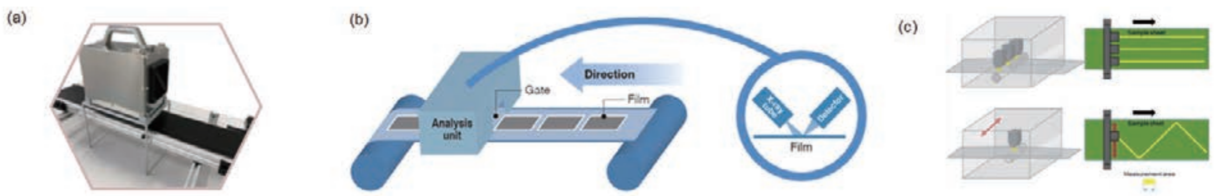


Figure 9 Photo (a) and Schematics (b and c) of inline XRF system.

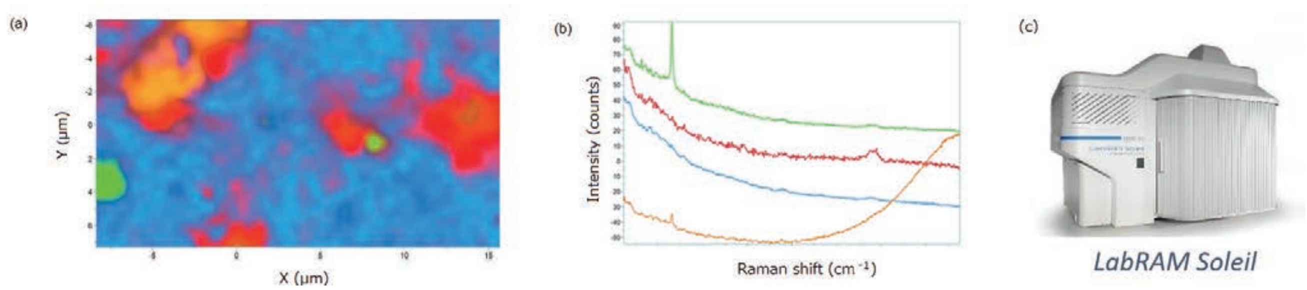


Figure 10 Raman mapping (a) and Raman spectrum (b) of a humid SOFC/SOEC air electrode measured with HORIBA LabRAM Soleil Raman Spectrometer (c). In blue: materials X; in red: materials X + C-H (polymer); in green and orange: Fe_3O_4 or Fe_2O_3 (impurity).

Post-mortem analysis

Once cells have been tested (performance and durability tests) with test stations such as the one produced by HORIBA FuelCon (Figure 13), some post-mortem analyses are done. In research, this is very common to do post-mortem analysis on cross sections with Scanning Electron Microscope (SEM)/Energy Dispersive X-ray (EDX) and Focused Ion Beam (FIB)-SEM. SEM-EDX enables to identify the chemical composition of layers (undesired phases, impurities…) and estimate both the quality of contacts and the porosity content. The FIB-SEM gives access to the 3D structure of layers (porosity content, contact surface, tortuosity…).[13] These data obtained from SEM are used to understand performance and durability tests results. For example, a loss of performance can be explained by the formation of an insulating phase at the electrode/electrolyte interface[14] or the electrode delamination[15] or the diffusion of species[14],[16] or something else. The post-mortem analysis can be completed with other characterisation technics such as Raman spectroscopy, X-ray Fluorescence spectroscopy and Glow Discharge Optical Emission Spectrometer (Figure 16), developed by HORIBA.

Raman spectroscopy was used to detect phase transformation in YSZ electrolyte enhanced by Nickel reduction.[17] It can also be used to monitor Cr poisoning behaviour of $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ (LSCF) cathodes.[18]

X-ray fluorescence spectroscopy can be used to identify, in a non-invasive way, Ni-S compound formed within a Ni-YSZ fuel electrode of a SOFC in contact with H_2S.[19] It can also be used to monitor the depletion and agglomeration of Ni in Ni-YSZ electrode[16] or a crack formation inside the YSZ electrolyte.[20]

In addition to composition and microstructural analyses, there is an interest in the industry to measure the mechanical properties of SOFC and SOEC cells.

SOFC/SOEC Stack and Hot-Box Materials Analysis

Current SOFC research is not only searching for new cathode, anode and electrolyte materials with greater performance and durability, but also for better interconnectors, glass sealings, end plates, compression rods and stack designs. That is why not only single cells are tested and characterised but also stacks and hot-box systems. Stacks and hot-box systems performance and durability can be determined with test stations such as HORIBA FuelCon test stations (Figures 14 and 15). The performance and durability test results will again be completed with measurements done from the raw materials preparation to the post-mortem analysis.

(a)

	Fe2O3	NiO	Y2O3	ZrO2	HfO2	CaO	Cr2O3	Cl	SO3
reference	0.05	28.4	7.9	61.1	2.2	0.3	0.03	0.00	0.00
Dark points									
N°1	0.11	30.2	7.8	59.3	2.2	0.3	0.02	0.02	0.00
N°2	0.18	28.7	7.9	60.6	2.2	0.3	0.03	0.03	0.00
N°3	0.09	29.9	7.8	59.9	2.2	0.2	0.02	0.00	0.00
N°4	0.24	32.3	7.5	57.4	2.2	0.2	0.05	0.05	0.00
N°5	0.21	29.6	7.8	59.9	2.1	0.2	0.04	0.05	0.00

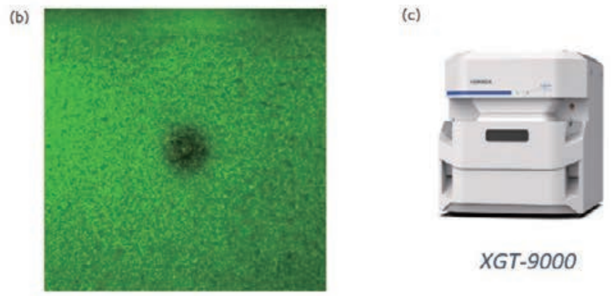


Figure 11 X-ray fluorescence signature (a) of a dark impurity spot (b) on a SOFC anode. A high concentration of Fe_2O_3 was detected with HORIBA XGT-9000 (c).

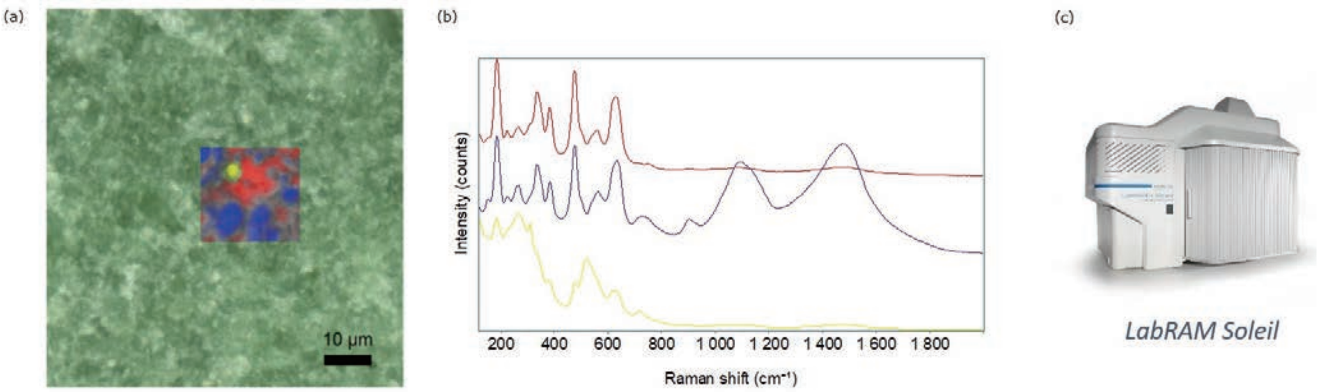


Figure 12 Microscope image and Raman mapping (a), and Raman spectrum (b) of a sintered SOFC anode measured with HORIBA LabRAM soleil (c). In red: materials X; in red: ZrO_2 ; in blue: NiO; in yellow: $BaTiO_2$ (impurity).

PEMFC/PEMEC cells, stacks, and systems Materials Analysis

Like HORIBA testing capability of SOFC/SOEC components and systems, HORIBA is capable of characterising PEMFC/PEMEC components and systems from materials to performance level. HORIBA GD-Profiler 2, coupled with HORIBA XploRa Plus Microscope are, for example, very useful to characterise the Bipolar Plates (PEM interconnectors) coating. By combining the elemental depth profile and the Raman spectrum of the Bipolar Plate coating, they helped to understand the impact of the coating morphology on the coating thickness (Figure 16).

Figure 16 shows an example of the characterization of a DLC coated film on a bipolar plate (PEM interconnector) using HORIBA GD-Profiler 2 (d) and the HORIBA XploRa Plus Microscope (e). The coated film of the bipolar plate must have a quality and thickness that satisfies high conductivity and corrosion resistance. Figure 16-(a) shows the Raman spectrum of the surface of the DLC-coated film of the bipolar plate (b). The quality of the DLC film can be evaluated by the ratio of the peak intensity of the G-band around 1580 cm-1 and the D-band around 1360 cm-1, which is derived from carbon, and the width at half maximum. Figure 16-(c) shows the elemental depth profile of a DLC-coated film on a bipolar plate. The vertical axis is indicated by the elemental emission intensity, or elemental concentration, and the horizontal axis is indicated by the sputtering time, which can be converted to the depth from the surface. The red line is the profile of carbon derived from the DLC film, and the black line is the profile of titanium, the base material of the bipolar plate. From this depth profile, the thickness of the DLC coated film can be analyzed. Thus, by combining the elemental depth profiles and Raman spectra, the influence of the DLC film quality of the bipolar plate on the thickness of the coating can also be investigated.

Conclusion

The main focus of research in SOFC/SOEC today, is the development of improved materials and designs to increase the efficiency and reduce the cost of these systems. This includes the development of new electrodes materials, electrolytes, and fuel cell components (interconnectors, glass sealings, end plates). Researchers are looking at ways to optimise the performance and durability of SOFCs/SOECs by analysing materials and their performance. The materials analysis can be made by Raman spectroscopy, X-ray Fluorescence, Dynamic light scattering, Glow Discharge Optical Spectrometer, Energy-dispersive X-ray spectroscopy and other technics. The main challenge of SOFC/SOEC industrials is the upscaling of SOFC/SOEC stacks production. To support this growth, HORIBA is offering inline monitoring solutions to control the quality of the process. Also, HORIBA is suppling end-of-line test stations to sinter stacks and validate their performance. Additionally, HORIBA provides measurement and test equipment for fuel cells and electrolysers systems development.

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

Figure 13 Evaluator C1000-HT SOFC Fuel Cell Test Station.

Figure 14 EVALUATOR S25-HT.

Figure 15 S25-HT for Hot-Box systems.

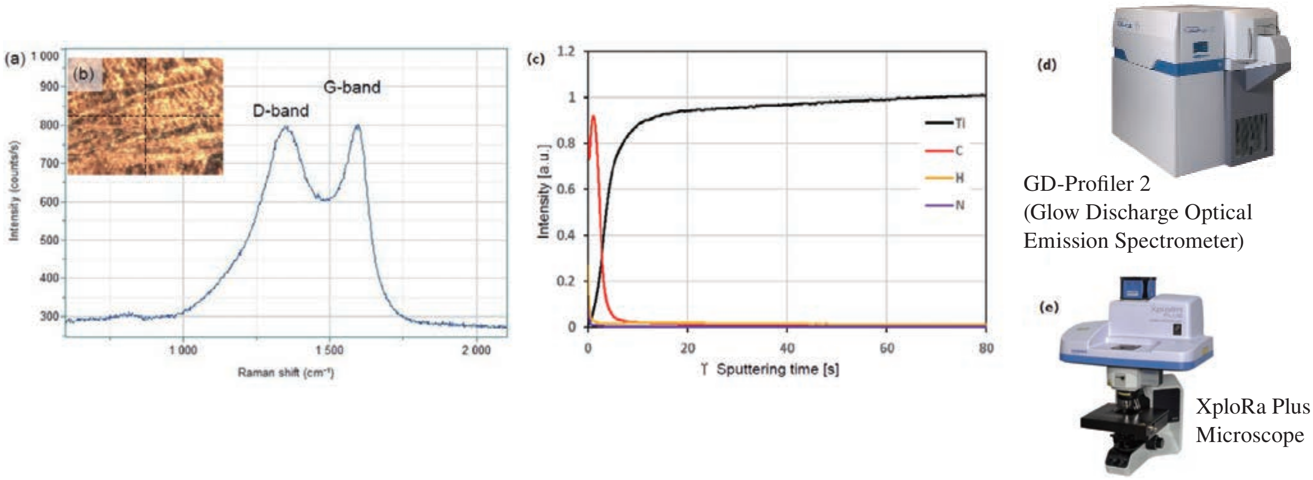


Figure 16 Raman spectrum obtained with HORIBA XploRa Plus Microscope (e) on a carbon coated surface of a bipolar plate and Optical image of the measurement area (a), and Elemental depth profile of the bipolar plate analysed by GD-Profiler 2 (d).[21]

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