



Rapid detection of defects in fuel-cell electrodes using infrared reactive-flow-through technique



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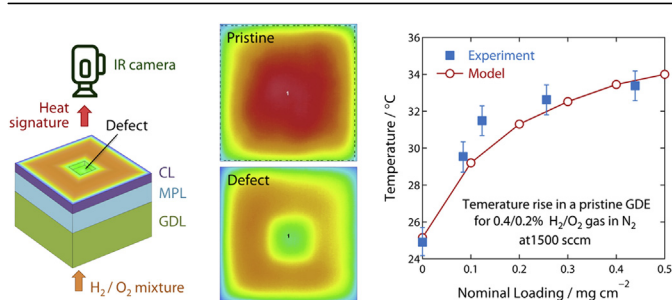
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HIGHLIGHTS

- A nondestructive, noncontact QA/QC technique for fuel-cell electrodes is presented.
- The technique uses IR thermography and dilute H₂ to detect electrode's defects.
- Both modeling and experiments are presented to demonstrate the technique.
- The technique can detect a 200 μm electrode crack.

GRAPHICAL ABSTRACT



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ABSTRACT

As fuel cells become more prominent, new manufacturing and production methods will need to be developed to deal efficiently and effectively with increased demand. One necessary component of this industrial growth is the accurate measurement of the variability in the manufacturing process. In this study, we present a diagnostic system that combines infrared thermography with a reactive-flow-through technique to detect catalyst-loading defects in fuel-cell gas-diffusion electrodes accurately with high spatial and temporal resolutions. Experimental results are compared with model predictions of thermal response with good agreement. Data analysis, operating-condition impacts, and detection limits are explored using both experiments and simulation. Overall, the results demonstrate the potential of this technique to measure defects on the millimeter length scale with temporal resolutions appropriate for use on a web-line. Thus we present the first development stage of a next-generation non-destructive diagnostic tool, which may be amenable to eventual use on roll-to-roll manufacturing lines.

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1. Introduction

Proton-exchange-membrane fuel cells (PEMFCs) are considered a potential key zero-emission energy-conversion technology of the 21st century [1]. PEMFC systems are currently being deployed in

some early markets, and as this commercialization expands, there must be a transition from low-volume, largely manual manufacturing methods, to high-volume, continuous and automated processes. In parallel with this transition to high-volume-manufacturing methods, there must be an effort to evaluate and develop quality-control techniques that are rapid, noncontact, and non-destructive, and which can be implemented in continuous or automated production lines. Scalable quality-control methods are especially needed for membrane-electrode-assembly (MEA)

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components [2], including membranes, electrodes, and gas-diffusion media (GDM), which are in many cases inspected visually before use in a stack. Of particular importance, given the high cost of platinum and other noble-metal constituents, is the detection of variations in catalyst layers (CLs). CL thickness variations, such as a spot with excessive or limited loading, can yield performance losses beyond those expected from the platinum (Pt) variation or the composition of the CL alone, especially at the local area near the defect [3–8]. This could be due to uneven compression of the GDM at the location of the defect, causing an increase in serial resistance or impedance (e.g., contact losses) and leading to water-management (e.g., flooding at the interface) and/or other issues, such as cell-performance reduction [3,5,9]. In addition, such variations could lead to initiation sites for membrane and other cell degradation and failure [10].

MEAs for PEMFCs are typically fabricated using roll-to-roll processes, wherein a sheet of material that is continuously formed by coating, casting, or other methods, is conveyed through required process steps by a roller system, with the final product wound into a continuous roll. Therefore, maintaining the quality of the final product is challenging. For in-line inspection, the fuel-cell industry is dependent on the optical inspection of visible defects or point measurements of coating thickness. While these measurements can be extremely useful, they will ultimately not be the best solution for continuous production of PEMFC MEA components. First, not all defects are visible, particularly relative to the typically black, non-reflecting electrode coatings and GDM. Second, point measurements only provide representative statistical data – they cannot generally locate discrete defects, even if scanned across the sheet of material being fabricated, as is often done in the industry. Given that discrete defects can act as failure-initiation points for MEAs in operation [11], it is critical to develop an inspection technique that measures every location on the sheet material such that defects in MEA components can be removed prior to assembly into complete cells with minimal scrap. This 100% inspection requirement is necessary to avoid premature cell failures, which often can cascade and cause complete cell and stack failure; therefore necessitating expensive replacements. In addition, the time required for the detection of defects is a key metric for any technique that is required to be on-line in a manufacturing process [2].

For CL defects, X-ray Fluorescence (XRF) spectroscopy has recently been applied for the in-line measurement of catalyst loading [12,13]. Unfortunately, this technique cannot at this time provide 100% inspection for CL defects. Acquisition times provided by commercially available systems that provide quantitative analysis of Pt loading are such that, for a roll width of 18 in. and a web-speed of 30 ft min⁻¹, data is only gathered on less than 1% of the sample area. Therefore, this technique can only provide a statistical metric of Pt loading for a given production run. One way around this could be a multi-pass system through the XRF, but this would slow the web speed and add complexity. Another option is to use multiple XRF setups, but this is cost prohibitive. In summary, XRF spectroscopy is an established diagnostic technique, but it has limitations relative to use as a diagnostic tool for continuous production of PEMFC electrodes.

Infrared (IR) thermography may allow for complete, rapid (with response times on the order of 1 s for large areas), non-contact, and non-destructive detection of defects. Several patents mention using IR thermography for detection of various PEMFC parameters, including MEA and membrane defects [14–16]. However, little public information is available on the ability of specific excitation techniques that allow for the detection of specific (size and type) defects in PEMFC materials. Recently, we have demonstrated an IR thermography and direct-current (DC) electronic-excitation

method to detect variations in CL thicknesses on decals or membranes, i.e., electrically insulating substrates, with high spatial and temporal resolution [17]. Although IR thermography techniques may not be element specific (as is XRF), our work shows that the IR-DC technique can be used to identify CL-thickness variations rapidly, at a relatively low cost, while providing 100% inspection.

In this paper, we examine an IR thermography-reactive flow through (RFT) technique that is applicable to gas-diffusion electrodes (GDEs), i.e., a CL coated on the microporous layer (MPL) of an electrically conductive gas diffusion layer (GDL). IR-RFT detects variations in Pt loadings, which, in this study, is identical to variations in CL thickness. As an initial study in the use of IR detection with reactive flow-through for CL defects, this work focuses on detecting square spots of different thickness in a surrounding layer of nominal thickness. Mathematical modeling is used to explore the effects of various process and material parameters.

The outline of this paper is as follows. First, the experiment, theory, and model for the technique are described. Second, the results of IR measurements with defined defects are presented and used for model validation. Third, both experiments and model are used to examine the impact of different variables of the technique including gas-mixture concentration, flowrate, defect size, and aspect ratio. Finally, the extension and applicability of this technique on a web-line, including simulating a slot flow rather than a uniform excitation, is discussed.

2. Experimental

IR-RFT was employed to detect variations of catalyst (i.e., Pt) loading in GDEs. The RFT technique involves the flow of a dilute non-flammable H₂/O₂ gas-mixture through the GDE where reaction occurs at the Pt catalytic sites in the CL. For the experiments, a flow of 0.4/0.2% H₂/O₂ gas in N₂ balance was introduced into an inlet manifold of a bench-top hardware, then passed through the gas-diffusion electrode, and finally exited through an outlet manifold as shown in Fig. 1(a). The heat signature of the reaction at the electrode was captured through an IR transmitting material (POLY IR[®], Fresnel Technologies) using an IR camera (Jenoptik Vario-Cam HiRes camera) with a 640 × 480 pixel detector with an accuracy of under ±1.5 K. A uniform emissivity of 0.95 was used for the GDE. This value was determined empirically by comparing a piece of 3 M[™] brand masking tape, which has a known emissivity of 0.95 [18], at the same ambient conditions as the CL.

CL samples with and without defects were prepared in-house from a catalyst ink containing 20 wt% Nafion solution with 1100 EW (Ion Power, DE2020), n-propanol, water, and 46 wt% Pt/carbon catalyst (TKK, TEC10E50E). The ionomer to carbon mass ratio was adjusted to 0.8:1 and mixing was performed overnight using a vial, zirconium oxide beads, and a ball mill. The resulting catalyst ink was then sprayed onto sheets of the commercially available GDL material BC25 from SGL, using a home-built ultrasonic spray system with a Sono-Tek Impact spray head. The spray system included a syringe pump, a 120 kHz micro-bore spray head and power system, and a programmable x–y stage. The flowrate of the catalyst ink during spraying and the speed of the sample holder were adjusted to achieve a platinum loading of 0.025 mg cm⁻² in a single coat. During spraying, the temperature of the sample holder was controlled to 80 °C. No additional time for drying was needed between coats.

Defects with 50% and 100% loading reduction were intentionally introduced to select samples by placing stainless steel masks of either 0.25 or 2 cm² on the GDL material for part of the spray process. The resulting loading reduction of the defect was controllable by the number of spray coats the mask was kept in place. For a loading reduction of 50%, for example, the mask was

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