VI.4 Metrology for Fuel Cell Manufacturing

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Overall Objectives

- Evaluate the scatterfield microscopy technique's sensitivity to performing catalyst loading and defect detection on industry supplied catalyst-coated membranes (CCMs) and gas diffusion electrodes (GDEs).

- Accurately measure the optical properties of the constituent materials in industry-supplied CCMs and GDEs.

- Build realistic theoretical models of CCMs and GDEs in order to generate accurate simulation data.

- Demonstrate quantitative theory-to-experiment agreement on CCM and GDE measurements.

- For dual-side catalyst coating operations, build and test a prototype optical device that will enable high-throughput simultaneous dual-side catalyst coating and defect detection measurements.

Fiscal Year (FY) 2013 Objectives

- Investigate other optics-based measurement approaches as in situ process control fuel cell manufacturing metrology solutions.

- Build and test a new scatterometry-based prototype optical measurement system for catalyst loading and defect measurements.

- Continue developing samples to allow accurate optical properties determination of CCM and GDE constituent materials.

Technical Barriers

This project addresses the following technical barriers from the Manufacturing R&D section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

(K) Low Levels of Quality Control

Contribution to Achievement of DOE Manufacturing R&D Milestones

This project will contribute to achieving the following DOE milestones from the Manufacturing R&D sections of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

- 6.1 Develop continuous in-line measurements for PEM MEA fabrication. (4Q, 2012)

- 6.2 Develop defect detection techniques in pilot scale applications for manufacturing MEAs and MEA components. (4Q, 2013)

- 6.6 Demonstrate continuous in-line measurement for MEA and MEA component fabrication. (4Q, 2015)

FY 2013 Accomplishments

- Based on the optical design work conducted in FY 2012, constructed a new large aperture projection scatterometer (LAPS) specifically for fuel cell manufacturing metrology research.

- Using the new LAPS instrument, made dynamic (as a function of velocity or web-line speed) platinum (Pt) loading measurements on Gore-provided CCM samples.

- Using the new LAPS instrument, performed diffraction pattern scans of 3M-provided nanostructured thin film (NSTF) samples as a function of Pt loading.

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INTRODUCTION

Scatterfield microscopy is an optical technique developed at the National Institute of Standards and Technology (NIST) that extends the ability to extract useful dimensional and materials related information from optical microscopy data when measuring samples and targets with features and dimensions that are beyond the resolution limit of the microscope. Applying our scatterfield microscopy techniques, we have been successful in extracting relative catalyst loading values from angle-resolved reflectivity scans of CCM fuel cell products. We have successfully

demonstrated the ability of the technique to produce industrially relevant sensitivities to changes in Pt loading. In addition, building on our previous knowledge of and work in applying scatterfield microscopy in fuel cell manufacturing metrology, we designed and built a new scatterometry-based optical metrology tool for performing fuel cell catalyst loading and defect detection measurements.

APPROACH

The scatterfield microscopy approach has been successful in achieving industrially relevant sensitivities (0.01 mg Pt/cm^2) in performing Pt loading measurements on industry provided CCMs. Progress has also been made from the inception of the project in developing electromagnetic scattering models that would allow us to generate accurate simulation data of the CCM samples we are measuring. Work has continued this year in the modeling area, but the majority of our attention focused on developing a new scatterometry-based optical instrument. The reasons for developing this tool are rooted in the experience we gained in validating the scatterfield microscopy approach in fuel cell manufacturing metrology. This new instrument was designed to resolve some open issues with the scatterfield microscopy approach (including open issues with our spectroscopic ellipsometry measurements) while providing additional functionality. First, the vertical motion of the CCM samples may introduce measurement errors if not accounted for properly. Second, the sensitivity of the scatterfield microscope and spectroscopic ellipsometer measurements to local defects and sample variations has not been fully addressed. Third, we had not previously been able to make dynamic loading measurements (as function of velocity) of CCM samples. This new instrument, called the LAPS, addresses these three issues. This new instrument is shown in Figure 1.

RESULTS

The LAPS, a completely new optical instrument designed specifically for fuel cell manufacturing metrology, was built and tested. Preliminary data were acquired from this new instrument and reported at the DOE Annual Merit Review meeting May 15, 2013. The majority of the effort on this project this year involved the design, construction, and testing of the LAPS. The primary motivations for building this tool were the following: to engineer a bigger (adjustable) spot size for large area data averaging, to facilitate better signal to noise measurements (via a more powerful source and a more sensitive detector), to add the ability to make measurements at web-line speeds, and to be able to quantify the coupled vertical motions of the membranes during measurement. Accomplishments regarding the construction of the LAPS include: completing the mechanical design, procuring necessary instrumentation, fabricating required parts, developing optical and mechanical alignment
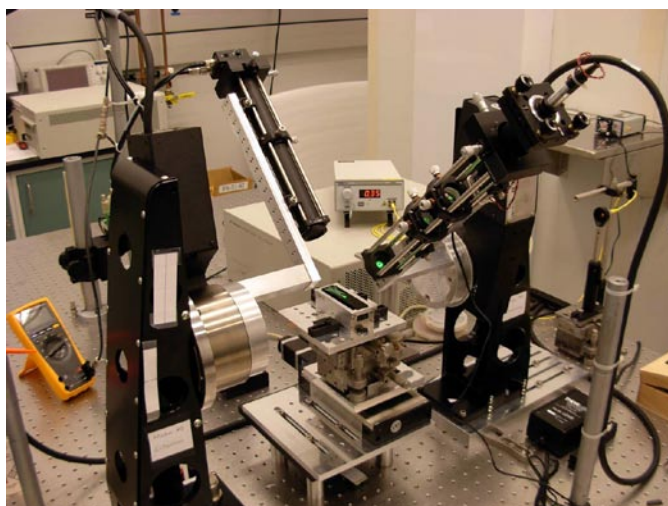

FIGURE 1. Newly Constructed LAPS Instrument

procedures, and writing LabVIEW® motion control and data acquisition code.

Preliminary dynamic CCM loading data were taken on a Gore sample set (A510/M710.18/C510) as a function of velocity, simulating web-line movement of the samples. Sample loadings in the set were 0.10, 0.20, 0.30 and 0.40 mg Pt/cm^2. The stage used in this round of data permitted sample velocities (web-line speeds) ranging from approximately 1.0 ft/min to 4.0 ft/min. These data are shown in Figure 2. The data show good sensitivity and repeatability with no apparent dependence on velocity. This tool will enable us to measure larger format samples at typical web-line speeds (30 ft/min) and we are currently in process of integrating a new stage capable of moving the samples at those speeds.

When illuminated, the 3M NSTF-type samples produce a diffraction pattern as seen in Figure 3. This diffraction presents a new measurement challenge that requires further study. The amplitude of the different peaks contains key information about the materials and surface properties of the sample. We successfully measured this diffraction pattern as a function of loading on a 3M sample set, including 0.10, 0.15, and 0.20 mg Pt/cm^2 loading values. In this particular measurement, the illumination arm is held at 80 degrees and the collection arm is scanned from 80 to 30 degrees. These diffraction pattern data, shown in Figure 4, demonstrate excellent sensitivity in certain peaks.

CONCLUSIONS AND FUTURE DIRECTIONS

In past reports, we have successfully demonstrated Pt loading measurement sensitivities at the level of competing technologies. There remains significant work to properly measure the optical properties of the CCM and GDE constituent materials to enable accurate simulations in order
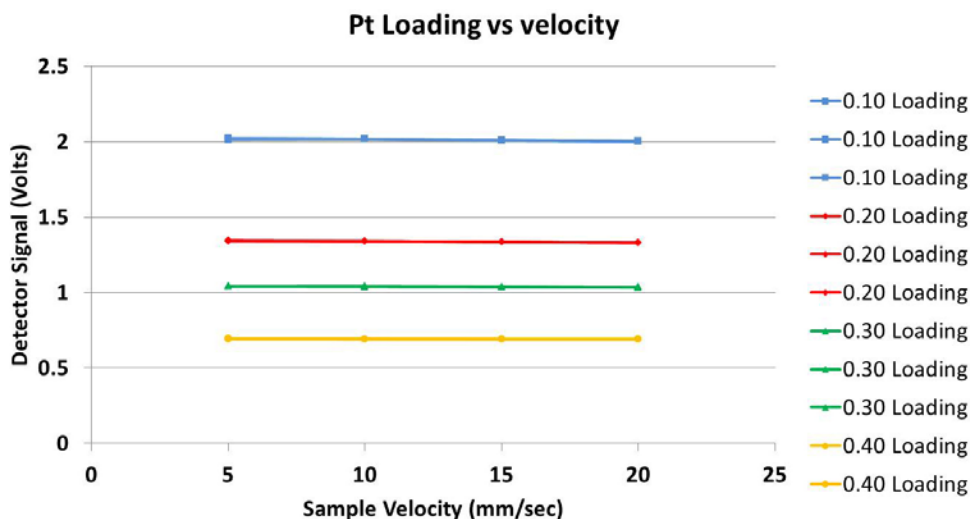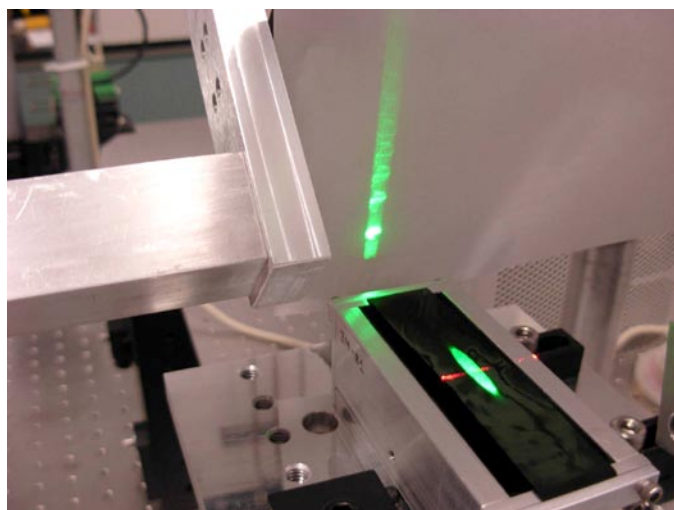
FIGURE 2. LAPS Dynamic Pt Loading Measurements

FIGURE 3. 3M NSTF Diffraction Pattern

FIGURE 4. Measured 3M NSTF Diffraction Pattern as a Function of Pt Loading

to achieve quantitative theory-to-experiment agreement. For this methodology to become a robust effective metrology solution, we need to be able to accurately model the samples to generate quantitative simulation data. To further understand the underlying measurement science and develop the necessary rigor, we designed and built a custom optical metrology tool (LAPS). The tool is now operational. We still need to comprehensively characterize and calibrate the tool. We acquired preliminary LAPS data on Gore samples (as a function of velocity) and 3M samples (diffraction pattern).

The most pressing challenge to achieving quantitative fuel cell manufacturing metrology is to develop the simulation infrastructure to enable the underlying rigor with acceptable uncertainties and sensitivities. Although we have demonstrated initial theory to experiment data fits, different
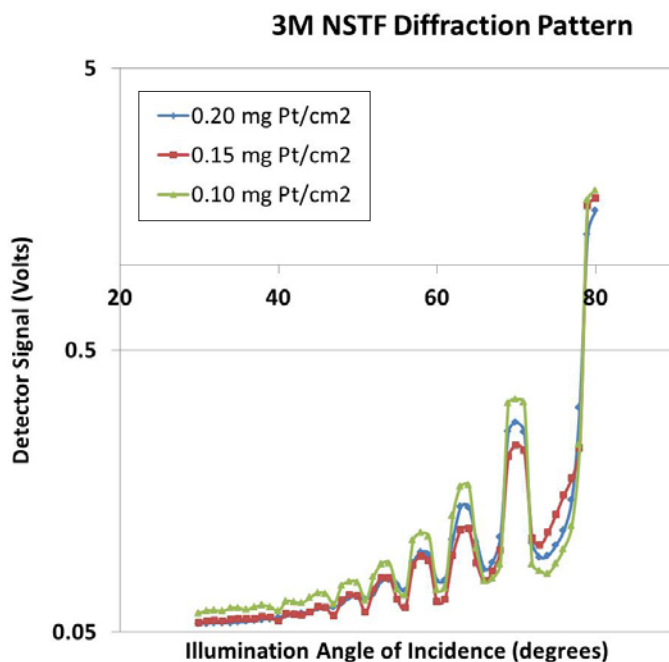
manufacturers using different methods and materials for their membranes require adaptability of the methodology. This necessitates a flexible model that allows different geometrical parameters and normalization procedures. Once a robust modeling and fitting algorithm has been established, the method is generally broadly applicable to different membrane types.

A multiple-beam and dual-side LAPS hardware architecture capable of dramatically increasing throughput will require significant further design work and research.

We will continue working with industrial collaborators to create samples that allow optical property measurements of CCM constituent materials. The next materials to characterize are the actual proton exchange membrane (Nafion® and 3M membrane) and amorphous carbon. We need to finish characterizing and calibrating the LAPS tool. We will measure larger format samples at web-line speeds. We already have another stage (Newport XMS160) to integrate that will increase travel to six inches and speeds in excess of 30 ft/min. It will be a priority to answer outstanding questions regarding the diffraction and scattering of the periodic 3M NSTF samples and optimize for Pt loading determination. We will continue to investigate the applicability of scatterfield microscopy and LAPS to fuel cell defect detection. Industry input is needed as to the types, sizes, and distributions of defects that cause real performance losses.

DISCLAIMER

Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

FY 2013 PUBLICATIONS/PRESENTATIONS

1. M. Stocker, "Metrology for Fuel Cell Manufacturing," DOE Annual Merit Review Proceedings, MN006, May 15, 2013, http://www.hydrogen.energy.gov/pdfs/review13/mn006_stocker_2013_o.pdf.