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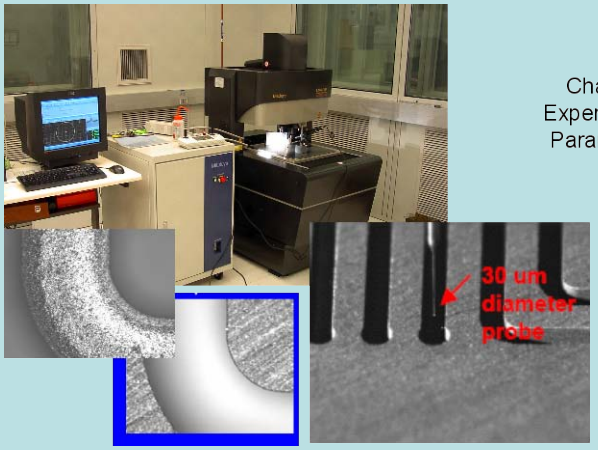
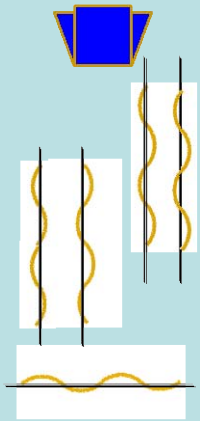
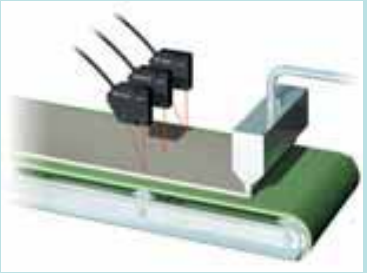
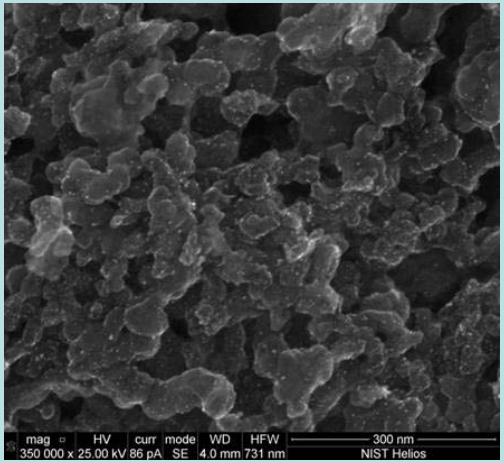






Metrology for Fuel Cell Manufacturing

2010 DOE Annual Merit Review
Project ID# MN006



Eric Stanfield
NIST

June 12, 2010

This presentation does not contain any proprietary, confidential, or otherwise restricted information.

Overview

Timeline

October 1, 2009
September 30, 2011

Barriers

B. Lack of High-Speed Bipolar Plate Manufacturing Processes

F. Low Levels of Quality Control and Inflexible Processes

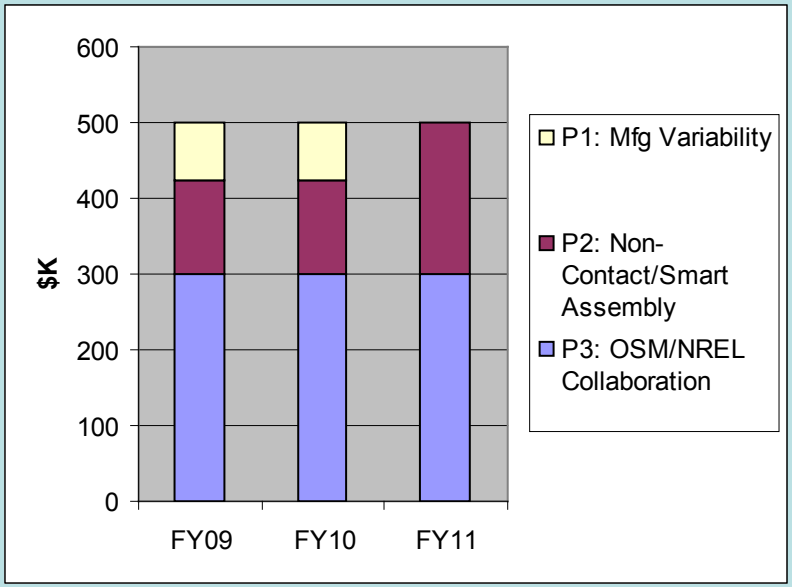
Partners

Subproject #1 Only (Funded $100K):
Los Alamos (sub) – Tommy Rockward

Other Interactions & Collaborations addressed in each subproject section.

Overall Budget

- FY09
  - DOE $500K (9/09)
  - NIST Est. $100K+
- FY10 & FY11 (Anticipated)
  - DOE $1M
  - NIST $100K and counting



Past Funding: FY07 DOE $0K, NIST $200K / FY08 DOE $300K NIST $300K

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Subproject Overview

Los Alamos NATIONAL LABORATORY EST. 1943

P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance

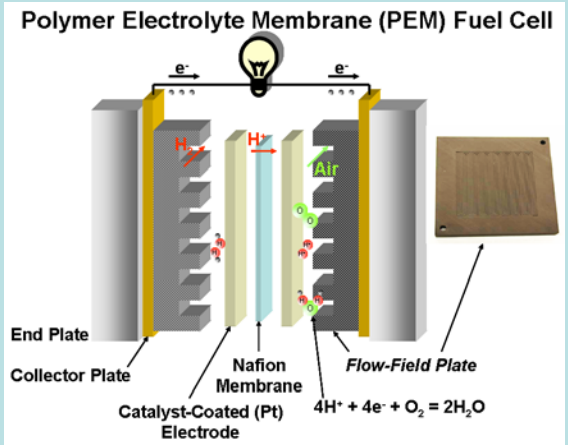
Objective: Develop a pre-competitive knowledge base of engineering data relating performance variation to manufacturing process parameters and variability.

Approach: Using a statistically based design-of-experiments, fabricate experimental "cathode" side flow field plates with various well defined combinations of flow field channel dimensional variations; then through single cell fuel cell performance testing using a well defined protocol, quantify the performance affects, if any, and correlate these results into required dimensional fabrication tolerance levels.

Benefits (Relevance): Provide bipolar plate manufacturers and designers with the data necessary to make informed decisions associated with reducing fabrication costs (i.e., process control inspection intervals and fabrication rates).

NIST

- Dimensional Metrology
- Manufacturing Metrology
- Statistical Engineering



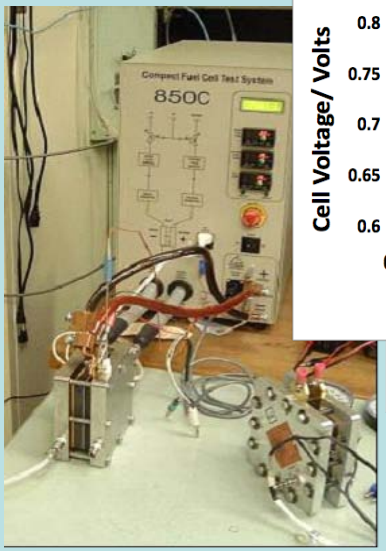
LANL

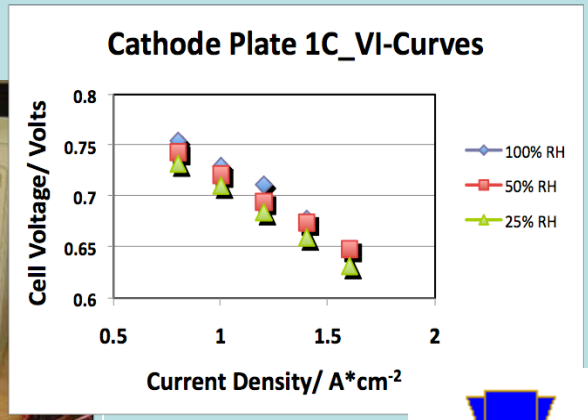
- Operational Knowledge
- Advanced Testing Facilities

Technical Accomplishments and Progress

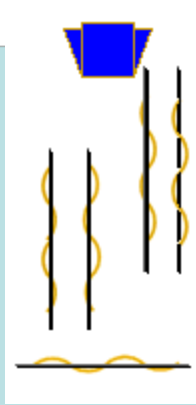
Past Accomplishments (2008 DOE AMR)

- Reverse Engineering Reference Fuel Cell Design (with numerous interactions).
- Developed Single Cell Testing Capability.
- Fabricated "Gold" Plates (Dimensionally Ideal = Geometric Variations < 5 um).
- Fabricated Experimental Plates per the Design of Experiments
- Validated Single Cell Testing Capability through participation in two intercomparisons.
- Dimensionally Quantified all Experimental Plates.





Experimental Parameters



Latest Progress

(maps directly with deliverable chart in additional slides)

- Developed Experimental Plate Fuel Cell Testing Protocol with Numerous Iterations. Latest document in the LANL/NIST SOW.
- Experimental Plate Testing In-Progress at LANL and Preliminary Testing Report Received

Challenges

- Minimizing Variation to Only Dimensional Parameters of Interest (vs. test system components and cell materials).
- New Institutional Safety Requirements: FC performance testing NIST → LANL.

Design of Experiment: 2^{4-1} Full Factorial (4 dimensional parameters, 2 levels each, with center point replication).....Total of 10 Experimental Cathode Plates

Technical Accomplishments and Progress

LANL Experimental Plate Testing Plan

Q. ***What information are we trying to obtain for the experimental results?***

A. Does dimensional variation of the cathode flow field impact gas access and water management (mass transport).

Testing Basics

Beginning-of-Test (BOT) and End-of-Test (EOT) Diagnostics – MEA Q.C.

- Electrochemical H2 Crossover
- Active surface area measurements on cathode side

Performance Testing (Gas Access and H2O Mgmt Impacts):

- Polarization curves in air and oxygen measured in both directions (Gas Access and H_2O mgmt impacts).
- AC-Impedance measurements

Identical Hardware and Components

- 50cm^2 Hardware (Teledyne CH-50)
- Gas Diffusion Media: SGL 25 BC
- Commercially Available MEA
- Hydrogen Electrolysis-Grade) and Air (oiless-compressor)

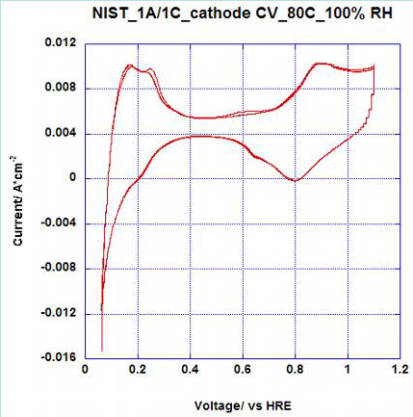
Fuel Cell Testing Conditions

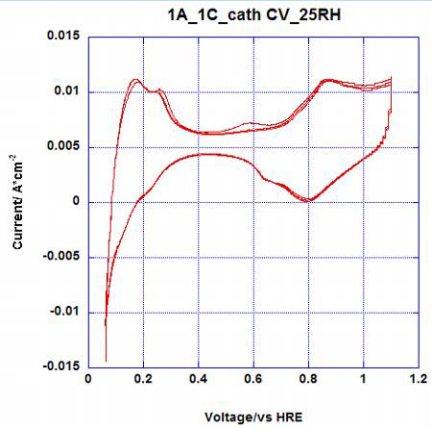
- 80ºC, 25 psig
- Varying RH: 100%, 50% , and 25%H2/air and oxygen (83.3% H2 and 50% utilization for air
  - CVs at each RH on the cathode
  - AC Impedance at 1 A/cm2

Baseline polarization curve between each experimental flow field configuration using a single reference MEA

Technical Accomplishments and Progress

Preliminary Results - Electrochemical H2 crossover/Surface Area

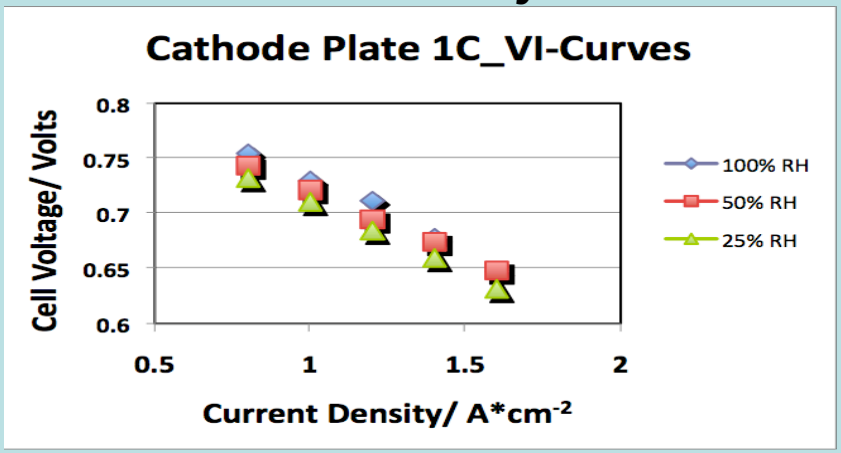


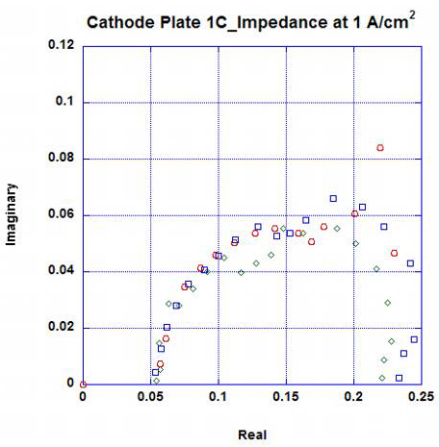


H_2/N_2 @ 500 sccm
80°C, 25 psig
Scan Rate: 20 mV/s
0.1 – 1.1 V, 3 cycles

- CVs are used for quality control as well as to confirm hydrogen cross-over measurements throughout the experiments.
- We will be able to compare CVs at the different RHs for each cathode and to ensure the MEA is still usable for other experiments – **(Protocol Revision) MEA can not be reused run-to-run.**

Preliminary Results - VI Curves / AC Impendance





- VI curves reflect high current density (increase H_20/establish mass transport) AC Impedance was run at 1 A/cm^2 for each RH – **(Protocol Revision) no clear differences observed – initial limits not high enough thus AC Impendence doesn't show what was expected, not in mass transport region!**

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Summary

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Testing alternatives based on preliminary testing:

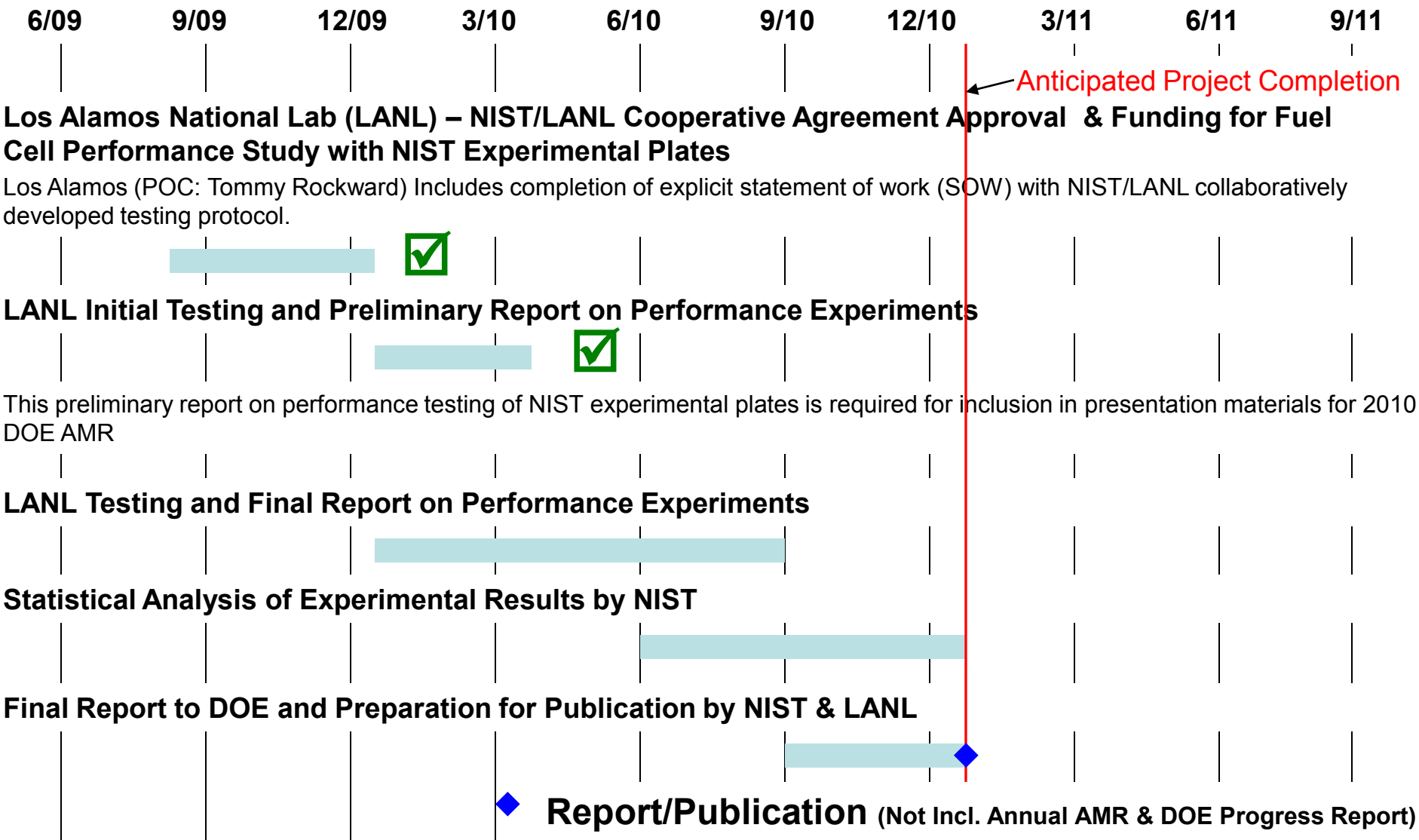
- Fresh identical MEAs will be used for each flow field
- VI curves will be run until mass transport region is clearly observed
- AC impedance will be run in both the kinetic region (i.e ~ 0.9V) and at the onset of mass transport

Future Work

- Run VI curves to higher current densities until mass transport observed and investigate AC Impedance results for differences at various RH conditions.
- Repeated reference cell (cathode 1C) testing with new MEA installed between each run to verify acceptable consistency with currently commercially available MEA.
  - Go – Continue with experimental plate testing
  - No-Go – Review other potentially more consistent / durable commercially available MEA suppliers.
- Final LANL performance testing and report (Expected: October 2010)
- Final NIST Statistical Engineering Division data evaluation and formal report on conclusions (January 2011).

(Revised 11/25/2009) Deliverables 3rd Quarter FY2009 – End of FY2011

Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance



Subproject Overview

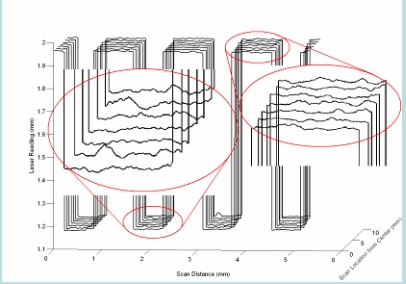
P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks.

Objective: (1) Identify and evaluate the capability and uncertainty of commercially available non-contact, high-speed scanning technologies for applicability to bi-polar plate manufacturing process control. (2) Using capabilities identified in (1) demonstrate smart assembly concept.

Approach: The development, integration, and evaluation of high-speed non-contact sensors or system of sensors for application in process control of bipolar plates.

The evaluation will include:

- Suitability based on typical plate materials and methods of fabrication
- Dimensional parameters of interest
- Development of measurement protocols
    - sensor evaluation
    - plate evaluation
- Accuracy evaluation as a function of scan rate
- Approaches to achieving contractual traceability requirements
- Demonstrate “Smart Assembly” concept



Benefits (Relevance): Provide bipolar plate manufacturers with a high-speed fully automated approach for process control inspection, while also providing the information needed to allow for more rapid inspection with decreased tolerance requirements (accuracy evaluation as a function of scan rate). And provide calibration/evaluation methods to ensure contractual traceability….**Enable Cost Reduction and Rapid Commercialization**

Subproject Overview

P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks.

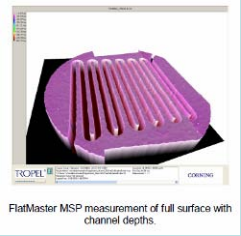
Current / Past Interactions (Plate Manufacturers):

Currently No Formal Collaborations

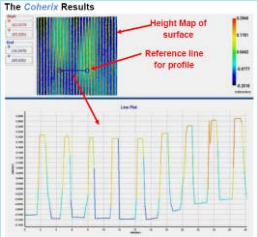
- **MetroMold* – machining and molding**
- **GrafTech* – molding**
- **FotoFab* –chemical etching**
- **Borit – molding**
- **Morphic – kinetic stamping**
- **Coran Precision – conventional stamping**
- **SGL Carbon – injection molding**
- **TechEtch – chemical etching**
- **Porvair – compression molded plates**
- **GenCell – stamping & etching**

Current / Past Interactions (Measurement Instruments & Precision Motion Control):

- **Aerotech – precision stage**
- **Keyence – measurement sensor**
- **Corning Tropel – measurement system**
- **Konica Minolta - measurement sensor**
- **Coherix – measurement system**



FlatMaster MSP measurement of full surface with channel depths.



Relevant Past Progress (2008 DOE AMR)

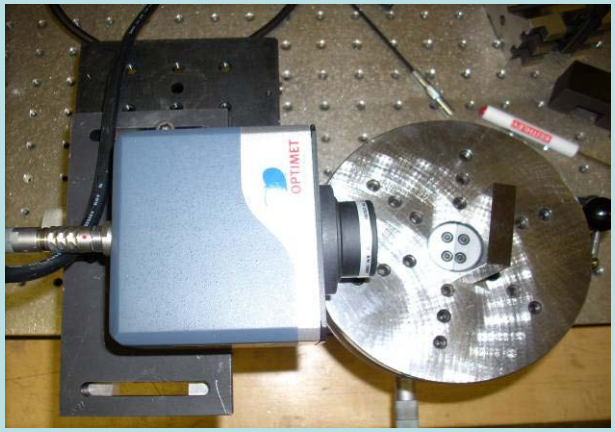
P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks.

Market Research: assessed needs versus measurement technology available:

- Dimensional Tolerances 50 um → 12 um
- Target Sensor Uncertainty ~ 5 um.
- Current State of the Industry (under constant observation)
  - **Video Based Optical Inspection Predominantly Used** Semi-Automated, Very Subjective, Measurement Uncertainty versus higher end requirements questionable.
  - **Measurement Technology: No Real All-In-One Systems Commercially Available**….numerous structured light systems for depth and flatness measurements but feature (lateral) size and location questionable at required accuracy.

Procured and initially evaluated two different non-contact triangulation probes.

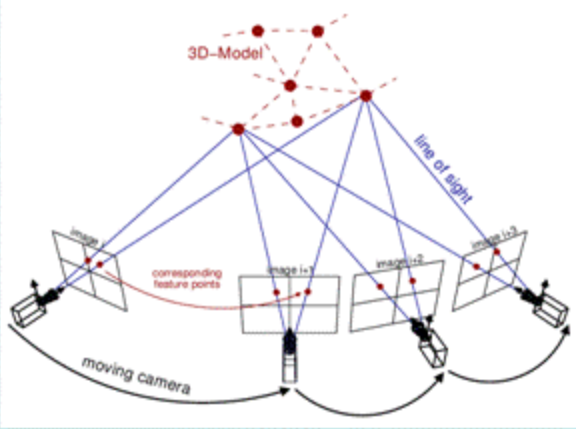
- Evaluated sensitivity to surface finish, material, and angle of incidence.
  - One excellent the other had limitations but still remained a potential solution



Optimet Inc. – Smart Conoprobe (Conoscopic Holography)

Keyence – LK-G (CCD Laser Displacement Sensor)

Recent Accomplishments



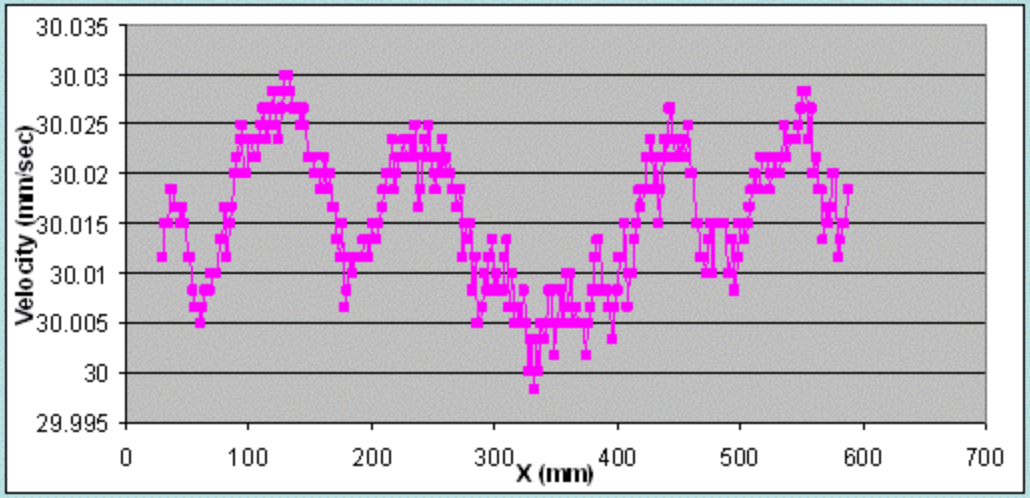
- **Evaluated Photogrammetry Potential – No Go!**
  - Even with incredible resolution of digital SLR cameras, aspect ratio of channel depth to width restricted this application.
  - Regardless accuracy seems limited to ~ 50 um.
- **Continued Performance Testing of Spot Triangulation Probe (Keyence LK-G)**

Approach (1): Data Acquisition based on known scan velocity

Evaluated in-house instrumentation for potential as a probe testbed using laser displacement interferometer.

- Requirements: variable velocity, minimum capability of 100 mm/s(300 mm/s desired), and velocity stability ≤ 0.03 mm/s.
- Several failed but one stood out to be the best immediately available option.

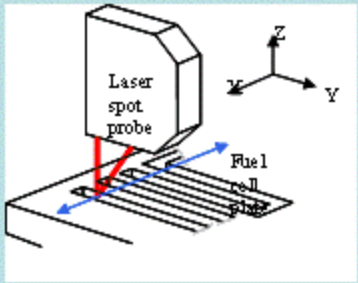
Coordinate Measuring Machine (CMM) velocity profile as a function of position.

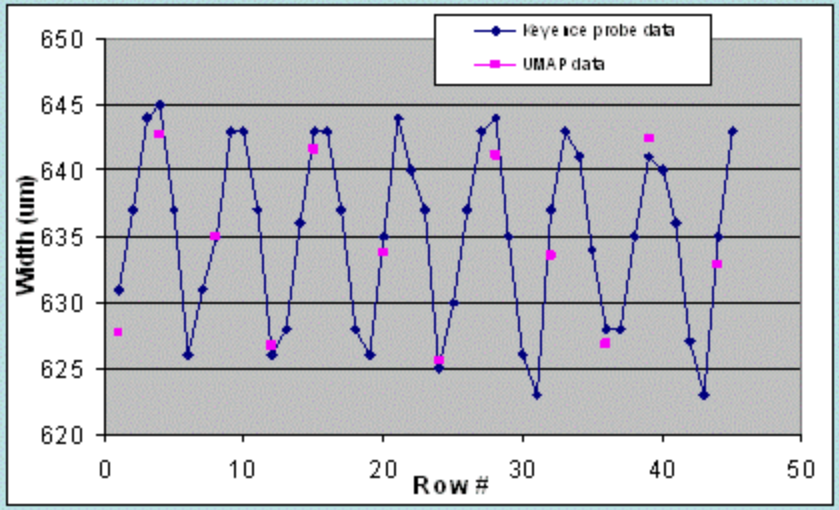


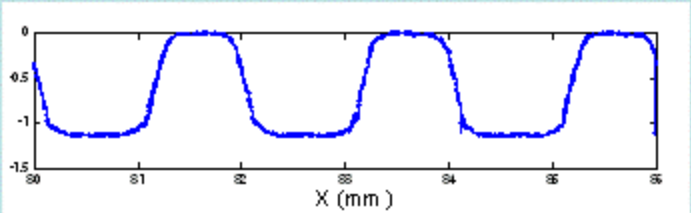
Recent Accomplishments

Performance Testing of Spot Triangulation Probe…..Continued

- **Absolute accuracy testing using "gage blocks" as reference standards (image on right).**
  - Height Errors ~ 1 – 7 um
  - Width Errors ~ 10 um
  - Identified Error Sources: laser spot diameter, sampling rate, probe test bed velocity stability, probe behavior near sharp edge, side wall reflections, probing system's software settings (how it handles outliers)
  - Less than desirable results lead to formulation of Approach (2): Simultaneous synchronization of probe data acquisition with precise position.
- **Repeatability and Correlation Evaluation** using a carbon-composite flow field plate.
  - 1.5 µm (one sigma) repeatability of channel width (5 traces, sharp edges)
  - Demonstrated agreement on channel width to a few micrometers versus reference values (bottom right figure).
  - Demonstrated plate-parallelism computation capability on a fuel cell plate
  - Successfully obtained profile data from a plate with tapered edges (bottom left)







Future Work

Non-Contact Sensor Metrology Workstation/Testbed:

- Assemble procured XY stages and ancillary equipment and integrate sensor output with stage output to facilitate **Approach (2) - Simultaneous synchronization of probe data acquisition with precise position.**
  - Pre-procurement sensor and stage signal integration testing successful.
- Identify error sources that contribute to nearly 10 µm errors in gage blocks
- Identify and study error sources in fuel cell plate measurements
- Develop error budget for parameters such as channel width, depth and parallelism
- Evaluate the performance of non-contact laser conoscopic probe and the laser line probe (procured and received).
- Measure and characterize plates with side wall tapers
- Characterize measurement errors as a function of scan rate
  - Stages being procured can move up to 2 m/sec versus 70 mm/sec using CMM.
- Smart Assembly research (FY2011)

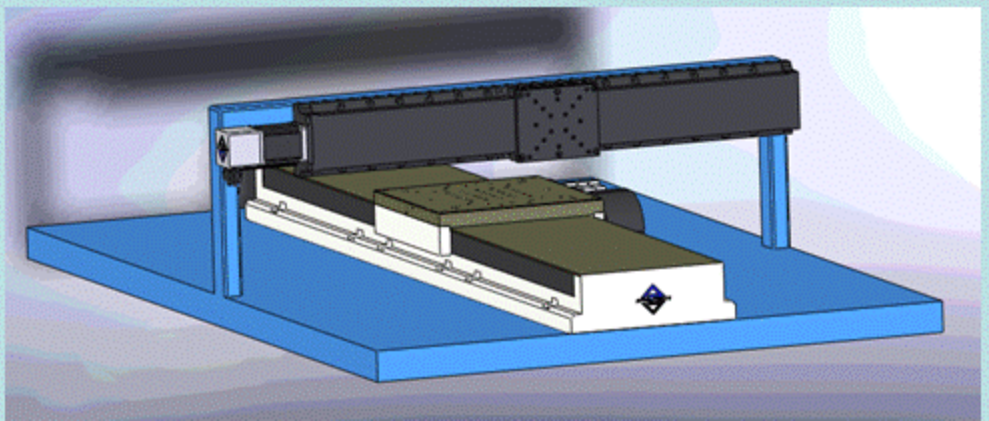
Parallel Activities:

- Work with other manufacturers of probing systems: Coherix and Corning-Tropel have expressed interest in measuring fuel cell plates.
- Present Paper Titled "**Performance Evaluation of Laser Triangulation Probes**" at the American Society of Precision Engineering (ASPE) Annual Meeting November 2010 [Abstract Submitted]

Summary

- Approach (1) based on data acquisition relative to a known scan velocity has significant limitations thus Approach (2) simultaneous synchronization of probe data acquisition with precise position is expected to be far superior.
- Efforts using Approach (1) were not in vain they revealed:
  - We have at least one probing technology with the potential repeatability and accuracy needed for this application.
- Procured and nearing assembly, the sensor workstation/testbed provides:
  - Dedicated platform for this work
  - Component specifications that will support either approach
  - Provide the capability to evaluate accuracy over the entire scan velocity range of interest.
- Lateral dimensions (channel width) of fuel cell plates with sharp edges are simple but chamfered edges create difficulties that have yet to be overcome; yet chamfered edges are the reality of mass produced bipolar plates regardless of material.
- Parallel efforts with manufacturers of other potential instrumentation ensures an optimal solution. This will be done by supplying these manufacturers with characterized plates for comparison.

Future NIST Sensor Testbed



(Revised 11/25/2009) Deliverables 3rd Quarter FY2009 – End of FY2011

Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks

6/09 9/09 12/09 3/10 6/10 9/10 12/10 3/11 6/11 9/11

Measurement Sensor Evaluation

Photogrammetry Investigation for Feature Size & Location ☒ No Go

No-Go 12/09….Channel Depth Not Accessible, Not Total Solution

Line & Spot Laser Triangulation Sensor Evaluation for Feature Size and Location (Contingent on Photogrammetry Outcome) ☑ In Process

Line & Spot Laser Triangulation Sensor Evaluation for Plate Parallelism ☑ In Process

Delayed start due to FY2010 funding uncertainty and subproject prioritization.

Measurement Criteria for Potential Successful Application, Uncertainty ≤ 15 um

Scan-Speed Accuracy Evaluation and Refinement of Chosen Sensor or Sensor Combination

Equipment Delay

Smart Assembly of Fuel Cell Stacks

Develop Architecture for Smart Assembly (instrument interface(s), database, and algorithms)

Goal: Achieve Tighter Overall Stack Parallelism Tolerance while Relaxing Individual Plate Parallelism Tolerances

◆ Go/No-Go – Proceed? ◆ Report/Publication (Not Incl. Annual AMR & DOE Progress Report)

Subproject Overview

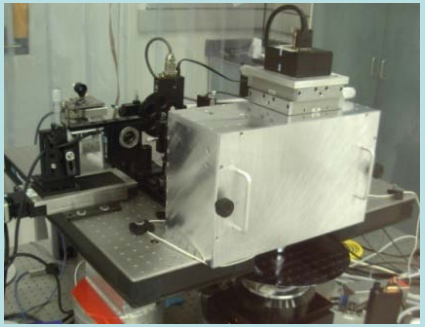
P3 Optical Scatterfield Metrology for Online Catalyst Coating Inspection of PEM (Fuel Cell) Soft Goods

Objective: Using catalyst coated samples provided by manufacturers with variations in critical parameters (i.e. Pt and Pt alloy catalyst loading, porosity, particle size, defects) characterized using standard industry methods (XRF, SEM), evaluate the Optical Scatterfield Metrology Tool's sensitivity to these parameters.

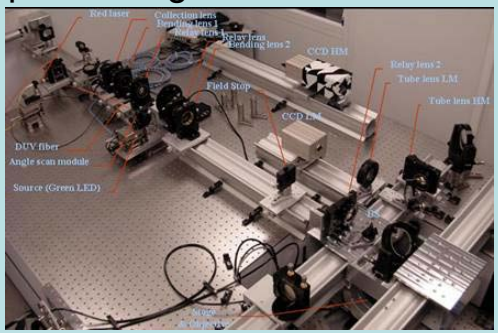
Approach: The Optical Scatterfield Microscopy technique employs both simulation and physical measurement of samples. **Simulation** is a key aspect of the approach as it allows one to develop accuracy when making optical measurements that require nanometer uncertainties. It provides a flexible and efficient platform to evaluate and optimize measurement parameters even before samples are measured. **Experimentally**, the approach involves acquiring angle and wavelength resolved data on one of three in-house custom designed and fabricated scatterfield capable microscopes (refer to figures below).

Benefits (Relevance): Provide PEM CCM and GDE manufacturers with an automated high-throughput approach for performing process control inspection of Pt loading with sensitivity equal to or better than that currently provided with XRF (and other parameters of interest simultaneously). Simulations will give insight and enable manufacturers to tune their measurement equipment to the parameters of interest. For dual side simultaneous catalyst coating operations, this method will provide the ability to concurrently perform Pt loading measurements on both sides of a CCM independently versus XRF which is "total" sample loading measurement.

The Overlay Tool
λ= 546 nm,
Arc lamp source



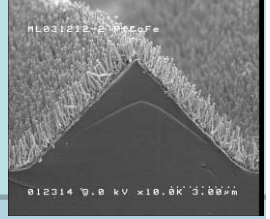
Scatterfield Tool
λ = 450 nm, until now LED
giving spectroscopic capability



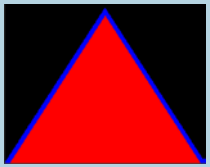
193 nm Tool
λ = 193 nm, higher resolution
imaging, Excimer laser source

Recent Accomplishments - Simulation

Initial Sample Package: (PtCoMn NSTF CCM)

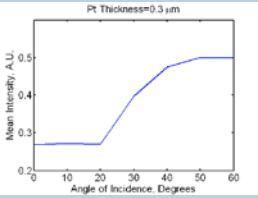


Simulating Pt layer on C triangular substrate

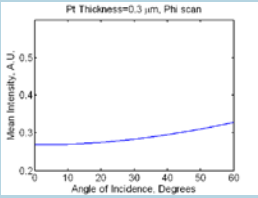


Red: Carbon
Blue: Pt (0.2 μm - 0.4 μm)
Period: 12 micrometer
Height: 6 micrometer
Wavelength: 546 nm

Theta angle scan

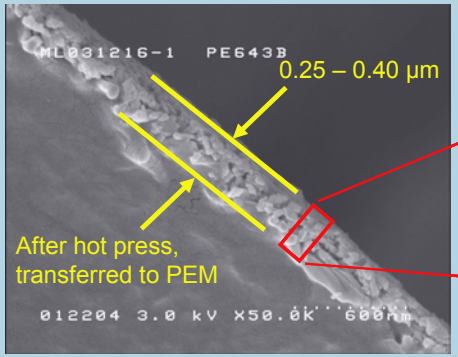


Phi angle scan

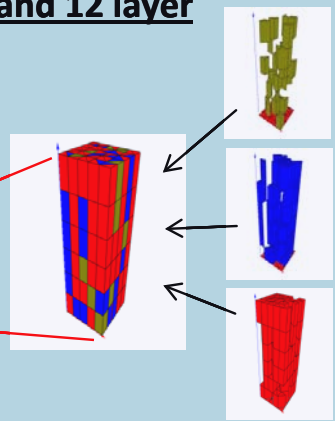


- Initial simulations performed (above) to give insight into light scattering properties of Pt and C for the given geometrical characteristics
- A more complete and realistic 3D model of the catalyst layer (on right) was constructed and simulated.
- A 6 layer and a more comprehensive and accurate 12 layer model (each layer equal to 25 nm) was constructed and run.
- In both the initial and more complicated simulations, we observed the type of Pt loading sensitivities that were anticipated.

3D simulation of CCM layer: 6 and 12 layer



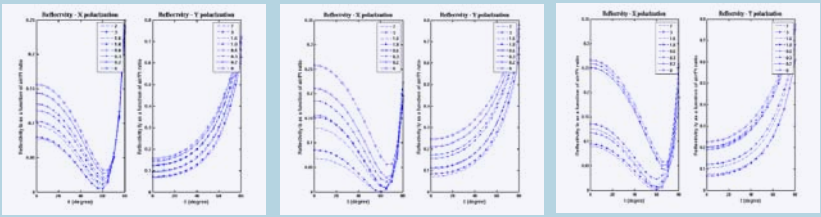
3M DOE AMR SEM Image



6 layer model

λ = 193 nm λ = 450 nm λ = 800 nm

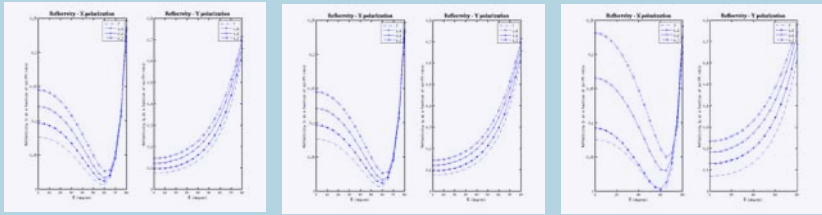
Reflectivity



Angle of Illumination

12 layer model

Reflectivity



Angle of Illumination

Recent Accomplishments – Experimental

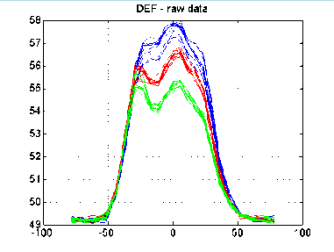
Overlay Microscope Measurements

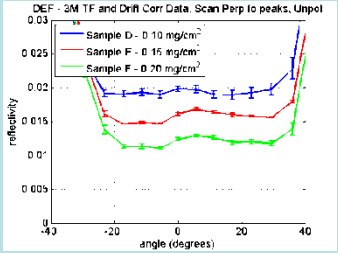
3M PtCoMn NSTF Samples D,E,F

- Studied effects of magnification and polarization on Pt loading sensitivity
- Successfully applied angle resolved scatterfield microscopy to Pt loading measurements on Overlay Tool
- Demonstrated repeatability and sensitivity to Pt loading values ranging from 0.1 mg Pt / cm^2 to 0.2 mg Pt / cm^2
- These data show a comparison of magnification from top to middle and a comparison of polarization from middle to bottom. From 50X to 10X, there doesn't appear to be much of a change in sensitivity. There is a substantial difference in sensitivity from the unpolarized to polarized measurements.
- Modified our in-house developed auto-focus and auto-centering routines to work with CCM samples
- Developed custom holders for CCM samples.
- A sample image taken at 50X can be seen on the right.

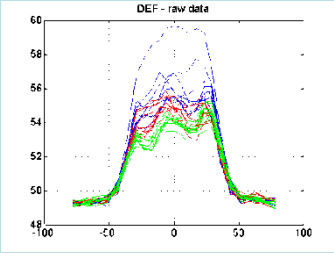
Raw data | Final Reflectivity data

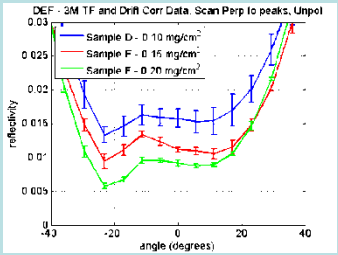
50X, Unpolarized



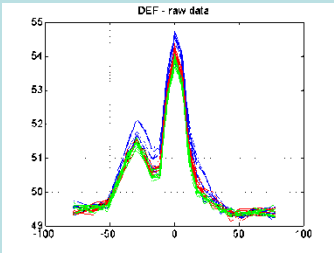


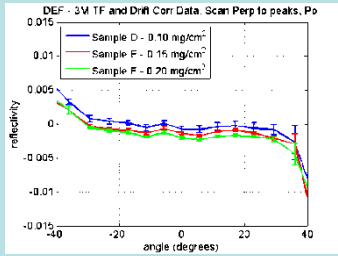
10X, Unpolarized

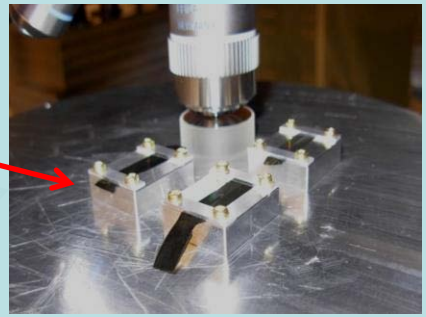




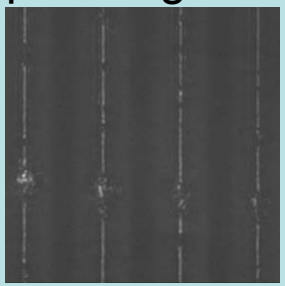
10X, polarized







Sample image at 50X



Recent Accomplishments - Experimental

Scatterfield Microscope Measurements

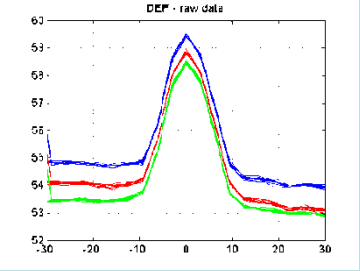
3M PtCoMn NSTF Samples D,E,F

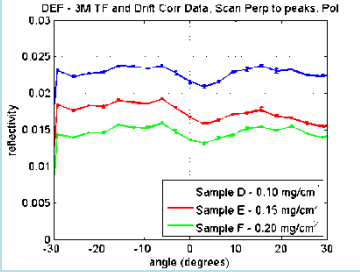
- Successfully applied angle-resolved scatterfield microscopy to Pt loading measurements on the Scatterfield Tool, demonstrating repeatability and sensitivity to Pt loading values ranging from 0.1 mg Pt /cm^2 - 0.2 mg Pt /cm^2. The data indicates that OSM could provide sensitivity to Pt loading on the order of 0.01 mg Pt / cm^2 or better. A more rigorous sensitivity study will be performed in the future.
- Angle resolved measurements on the Scatterfield Tool were performed at 150X magnification. Data is smooth and symmetric.
- Run 1 looked at the same location 9 times on the 3 different loading samples (base repeatability). Run 2 looked at 3 random locations on each of the 3 different loading samples, beginning to look at Pt loading variation across the samples.
- Successfully applied wavelength-resolved scatterfield microscopy to Pt loading measurements on the Scatterfield Tool, demonstrating repeatability and sensitivity to Pt loading values ranging from 0.1 mg Pt /cm^2 - 0.2 mg Pt /cm^2. Measurement 1 and 2 were performed on separate days.

Angle-resolved measurements

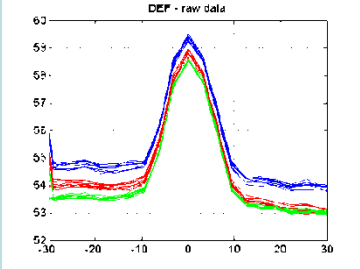
Raw data | Final Reflectivity data

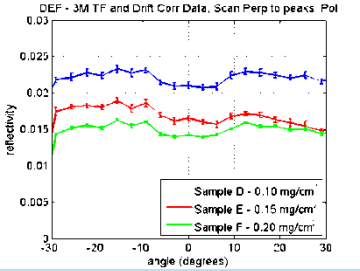
Run 1





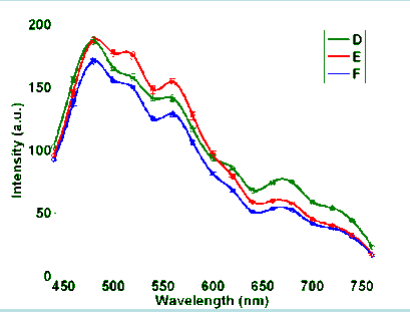
Run 2

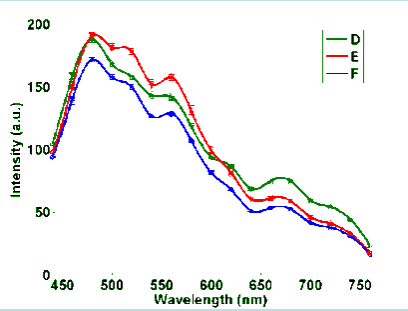




Wavelength-resolved measurements

Measurement 1 | Measurement 2

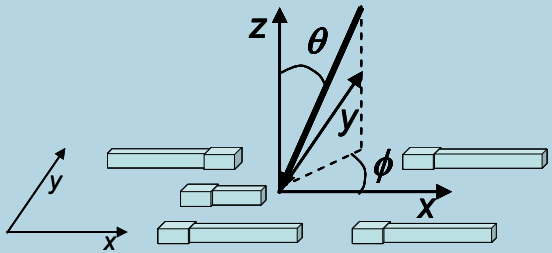


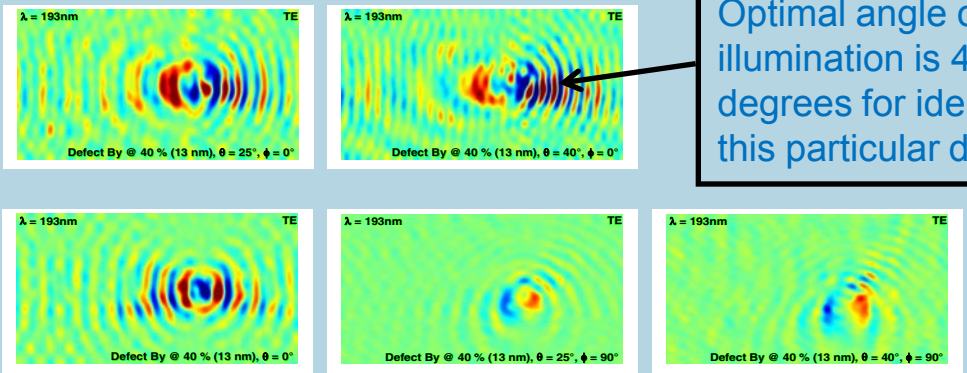


OSM Future Work

- Model more accurately the actual materials and structure of the CCM samples
  - Need to develop accurate *n* and *k* data using a new generation ellipsometer
- Look at different materials composition of CCMs and GDEs from different suppliers to understand the breadth of the technique. Study and understand environmental factors (temperature, pressure, humidity, etc.) on measurements.
- Make CCM measurements on the NIST 193 nm Scatterfield Tool.
- Extend and evaluate the application of Scatterfield microscopy defect related work to fuel cell defect measurements.
- Comprehensively separate and quantify materials effects from geometrical effects
  - Optimize hardware (spectroscopic, angle, polarization, etc.) and software algorithms
- Develop uncertainty budget for Scatterfield microscopy CCM measurements
- Perform a rigorous sensitivity analysis for angle-resolved and wavelength-resolved CCM measurements. Initial data indicate that Scatterfield microscopy is sensitive to Pt loading at the level of 0.01 mg Pt / cm^2 or better.
- Design a compact Scatterfield microscope that could be packaged into a useable form for inline process control of Pt loading.

Scatterfield Microscopy defect inspection in Semicondutor Manufacturing



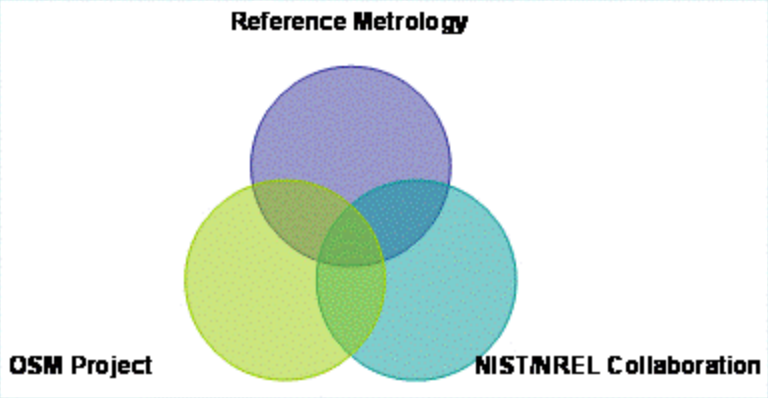


Optimal angle of illumination is 40 degrees for identifying this particular defect.

NIST/NREL Collaboration in Support of Fuel Cell Manufacturing:

- Effort summary developed and distributed to potential Industry partners (right).
- NIST/NREL Three-Way NDA – Drafted and Approved by both NIST & NREL Legal…..distributed to potential partners.
  - 3M, Gore, Arkema, BASF, Ballard, Johnson-Matthey

OSM Project & NIST/NREL Collaboration Relationship



U.S. DEPARTMENT OF ENERGY | NREL | NIST National Institute of Standards and Technology U.S. Department of Commerce

Proposed NREL-NIST collaboration on fuel cell MEA manufacturing R&D

Recognizing the need to develop manufacturing methods concurrent with technology at this stage in the maturation of the fuel cells market, the DOE Fuel Cell Technologies Program's Multi-year Program Plan documents the risks and barriers to the transition to high volume manufacturing. One of the barriers identified is in-line quality control for MEAs and components. NREL and NIST have been supporting DOE and industry with efforts to address this need. NREL is evaluating and developing in-line diagnostics for MEA component materials, with current focus areas of 2D membrane thickness measurement and defect identification and catalyst uniformity. NREL is also studying the effects that as-manufactured defects in component materials have on the performance and durability of MEAs. NIST is studying bipolar plate flow field channel dimensional manufacturing variability and the associated effects on PEM fuel cell performance while concurrently evaluating the accuracy and establishing traceability methodology for potential in-line high-speed high-density dimensional inspection technologies. NIST is also evaluating the suitability of a novel optical scatterfield microscopy tool as an in-line diagnostic for catalyst coated membrane (CCM) inspection (currently focused on the measurement of platinum loading). The capabilities of NREL and NIST to support the manufacturing initiative are very complementary, including reference metrology, analytical characterization, materials fabrication, *ex-situ* and *in-situ* fuel cell testing, and continuous processing of web materials for in-line device validation (soon).

Given this synergy, and to most effectively support our industry partners, NREL and NIST propose – with DOE support – to initiate a more formal collaboration. Our industry partners and DOE will benefit by closer communication and integration of project activities between the two organizations, taking advantage of the complementary capabilities, and maximizing the effectiveness of DOE support. As an example, we envision the following work breakdown:

Phase	Task	Lead	Asst.
Definition Phase			
	Verify/update prioritized defect list	NIST/NREL	
	Select defect for work	NIST/NREL	
	Select development lead (DL)	NIST/NREL	
	Select reference metrology	NIST	NREL
	Determine collaborators (industry, academia)	NIST/NREL	
Initial Development Phase			
	Sensor/detector development/evaluation	DL	
	Prep required MEA samples	NIST/NREL	
	Perform reference metrology on samples	NIST	
	Develop effects of defects test methodology	NREL	
	Review with collaborators	NIST/NREL	
System Development and Validation Phase			
	System development/evaluation	DL	
	Determine system specs (from defects testing)	NREL	NIST
	Perform End of Test/Life reference metrology	NIST	
	Perform appropriate modeling	NREL (LBNL)	
	Technology transfer plan	DL	
	In-line validation	NREL	
	Review with collaborators	NIST/NREL	

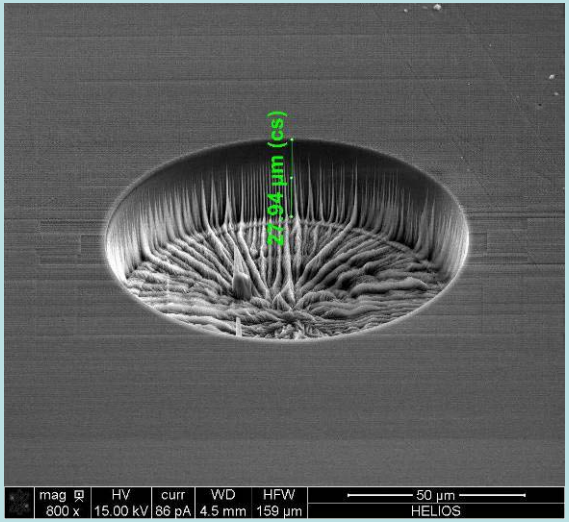
NREL and NIST will continue to have separate non-disclosure agreements with our industry partners. However, to enable this collaboration, we ask that our partners accept separate agreements with *both* organizations. We are developing a section of common language for these agreements so that, though separate, each agreement describes the intents of the collaboration, in terms of information and materials sharing between NREL and NIST.

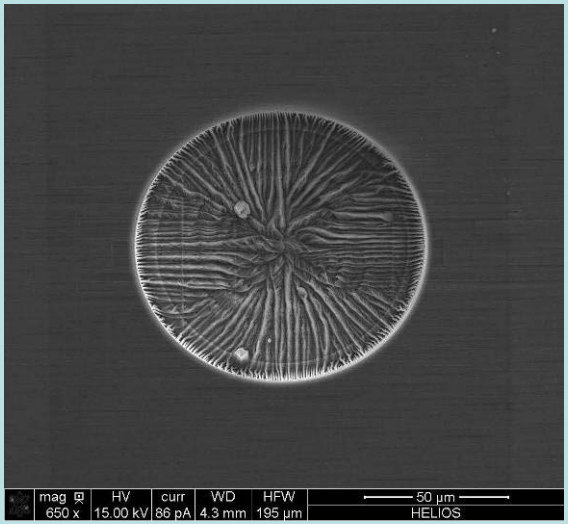
NIST/NREL Collaboration

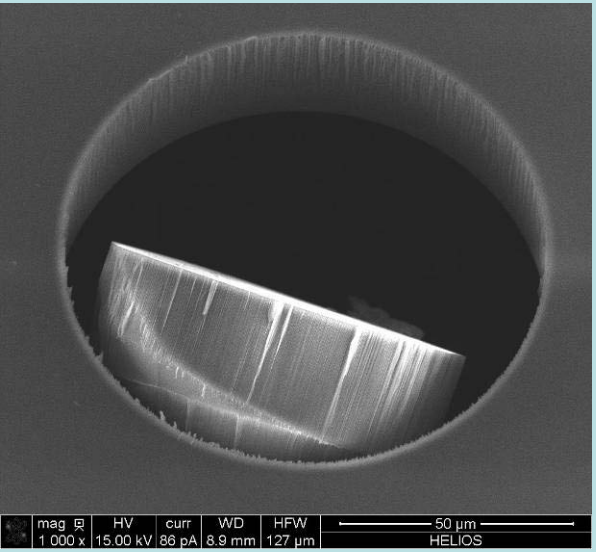
Technical Accomplishments and Progress

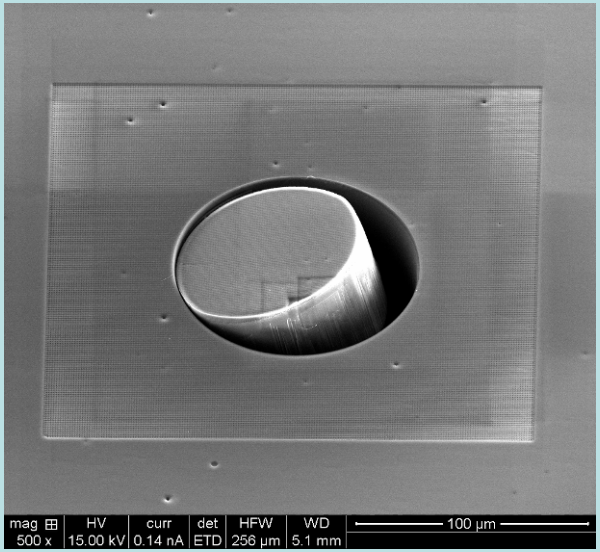
Membrane defect generation in support of NREL's defect study using Dual Beam SEM/Gallium FIB:

- 100 um diameter cavity / blind hole ½ membrane thickness.
- 100 diameter through hole









(Left Images) Gallium Contamination Study [Does Ga ion contamination affect fuel cell performance and/or diagnositics?]

- (100 um Hole #1) Ga milled hole with minimum contamination
- (100 um Hole #2 Ga milled then surface rastered with Ga to maximize contamination.

(Revised 11/25/2009) Deliverables 3rd Quarter FY2009 – End of FY2011

Optical Scatterfield Microscopy (OSM) for Online Catalyst Coating Inspection of Proton Exchange Membrane (PEM) Fuel Cell Soft Goods

6/09 | 9/09 | 12/09 | 3/10 | 6/10 | 9/10 | 12/10 | 3/11 | 6/11 | 9/11

NIST/NREL Cooperative Effort

Develop NIST/NREL Cooperative Plan ☑ NDA process slow but work still proceeds

Reconcile NDAs — **Execution of Deliverables**

Define scope of support with deliverables, cross-link manufacturer agreements, information sharing

NIST OSM Catalyst Coating Sensitivity Study

Preliminary Catalyst Loading Measurements of Novel CCM Samples ☑ Completed

Validate Sensitivity OSM sensitivity using other conventional catalyst coated components (other CCMs, GDEs (cloth and paper). ☑ In Process (NDA – Slows Progress)

2nd Package consists of samples with various Pt loadings from various Pt/C weight percentage mixtures

OSM Parameter Investigation for Catalyst Loading Measurements ☑ GO! Success

(1) Investigate Obvious Technique Modifications to Enhance Sensitivity to Loading [Instrument Illumination: angle, wavelength, polarization, and spatial filters…..Instrument Collection: spatial (wavelength) filters…..Algorithm Studies: intensity, thresholding, spatial (pixel size) filters, pixel histograms]

(2) Investigate New Approaches to Enhancing and Understanding OSM's Potential Sensitivity to Pt Loading: (a) incorporation of time-dependent illumination and CO tagging into current OSM technique and (b) perform finite domain time difference (FDTD) parametric modeling of one or both types of CCMs.

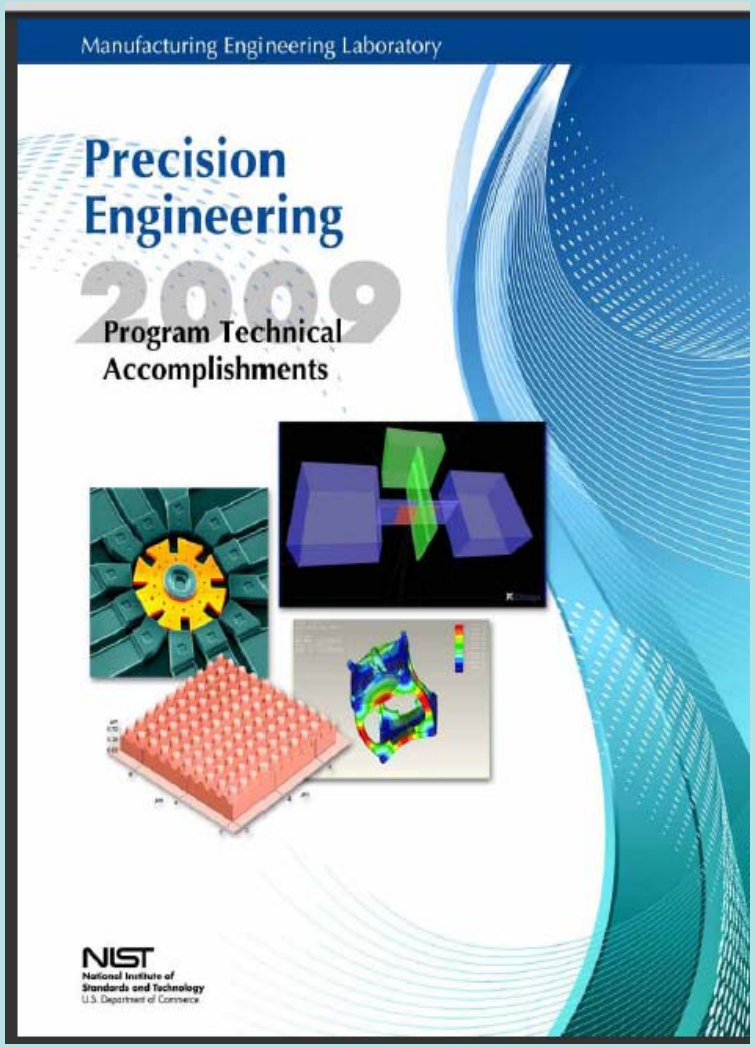
OSM Parameter Investigation for Other Critical Catalyst Coating Parameters

Specific parameters to be identified as a result of NIST/NREL cooperative plan

◆ **Go/No-Go – Go to Proceed!** ◆ **Report/Publication** (Not Incl. Annual AMR & DOE Progress Report)

Supplemental Slides

Publication Covering All Our Work



Precision Engineering 2009 Program Technical Accomplishments, pp 42-45, 2010
http://www.nist.gov/mel/ped/index.cfm

P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance

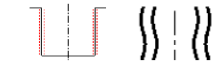
Accomplishments associated with project effort but not apart of project objective:

- Contributing Member of USFCC Materials Testing Working Group – Single Cell Testing Task Force.
  - USFCC Single Cell Testing Protocol 05-014B-2
  - IEC TC 105/241 International Single Cell Testing Protocol 62282-7-1
  - Single Cell Round Robin
  - USFCC Joint Hydrogen Quality Task Force – DOE North American Hydrogen Quality Team, H2 Quality SAE J2719/ISO 14687
  - Single Cell Round Robin (LANL Reference Lab - Publication Pending)
  - Liaison between NIST (Gas Metrology Experts) and this group (representing ASTM E4, SAE, ISO)

P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance

Design of Experiment Full Factorial 2^{4-1} (4 dimensional parameters, 2 levels each with center replica point)

Units are um

	2^{4-1} Fractional Factorial Design with replicated center point (k=4,n=10) (donmez.xls)							
	Sidewall Strai	Sidewall Stra	Bottom Straight	Sidewall Taper				
	Amplitude	Phase	Amplitude			Sequence		Drawing
Part(index)	*X1*	*X2*	*X3*	*X4*	*Machining(Brian)*	*Measuring(Eric)*	*Perf. Testing(Dave)*	*Cross-Section* *Top*
9	0(25–m)	0(90)	0(25–m)	0(5)	1	1	1	
3	-1(0)	+1(180)	-1(0)	+1(10)	2	2	2	
2	+1(50–m)	-1(0)	-1(0)	+1(10)	3	3	3	
4	+1(50–m)	+1(180)	-1(0)	-1(0)	4	4	4	
8	+1(50–m)	+1(180)	+1(50–m)	+1(10)	5	5	5	
5	-1(0)	-1(0)	+1(50–m)	+1(10)	6	6	6	
7	-1(0)	+1(180)	+1(50–m)	-1(0)	7	7	7	
10	0(25–m)	0(90)	0(25–m)	0(5)	8	8	8	
6	+1(50–m)	-1(0)	+1(50–m)	-1(0)	9	9	9	
1	-1(0)	-1(0)	-1(0)	-1(0)	10	10	10	

NIST/NREL Collaboration Future Work

- Depth of field imaging using helium ion microscope
- Develop methods to FIB mill sub 100 nm cross sections for imaging and quantifying membrane coating thickness
- Develop methods to image and quantify defects “as supplied” membrane defects (3D defect reconstruction).
- TBD on an “as needed” basis for OSM and NREL support.