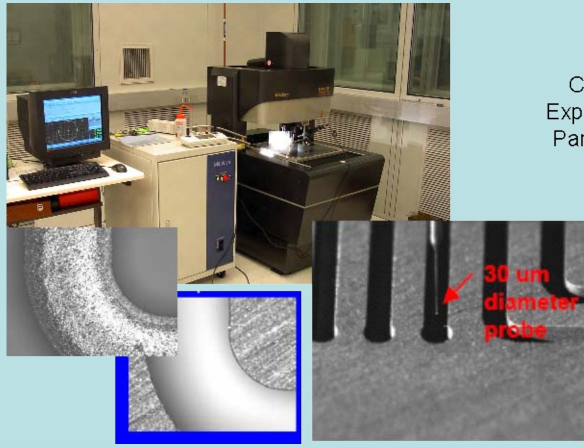
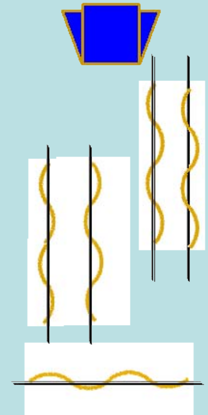
National Institute of Standards and Technology
U.S. Department of Commerce

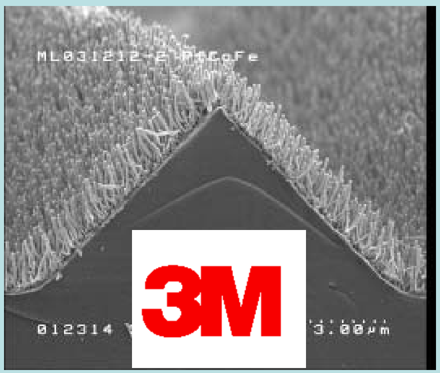


Channel Experimental Parameters

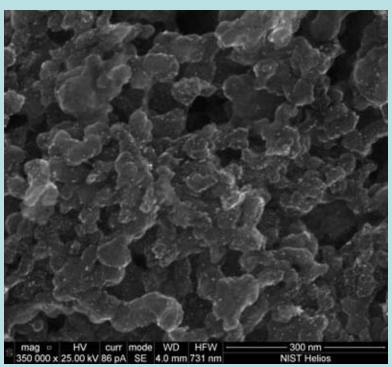


DOE Hydrogen Program

Metrology for Fuel Cell Manufacturing



DOE Hydrogen and Fuel Cells Program Review Project ID# MN006
May 12, 2011



Eric Stanfield (NIST)
Mike Stocker (NIST)

Overview

Timeline

August 1, 2009
September 30, 2012

Barriers

B. Lack of High-Speed Bipolar Plate Manufacturing Processes
F. Low Levels of Quality Control and Inflexible Processes

Partners

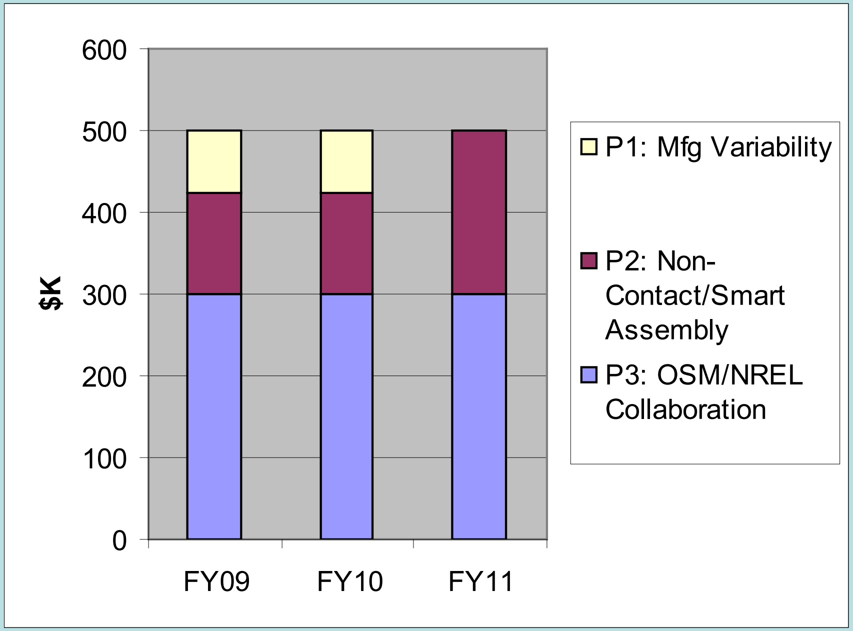
Subproject #1 Only (Funded $100K): Los Alamos (sub) – Tommy Rockward

Other Interactions & Collaborations addressed in each subproject section.

Overall Budget

- DOE Share - $1,500,000
- FY10 - DOE $400K
- FY11 - DOE $400K (Anticipated)

*** Cost share not required but NIST contribution to effort estimated at ~ 40% to 50% matching ***



Subproject Overview

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

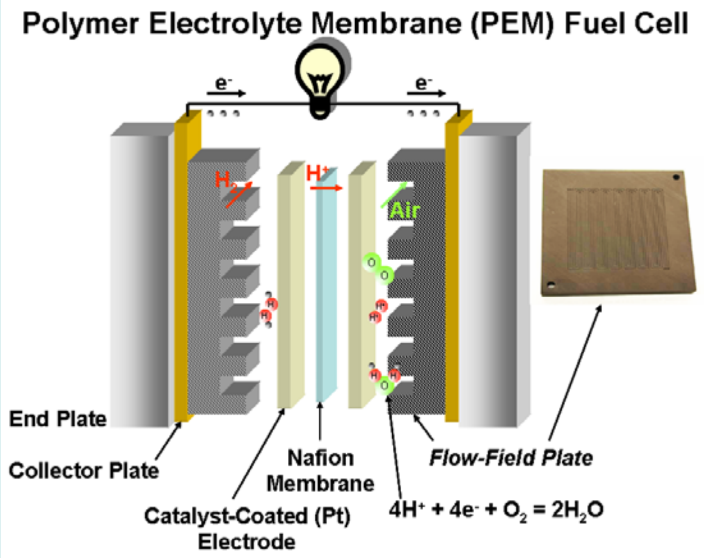
Objective: Develop a pre-competitive knowledge base of engineering data relating performance variation to manufacturing process dimensional variability.

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 robust protocol, quantify the performance effects, 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 tolerance decisions to enable reduction of fabrication costs.

NIST

- Dimensional Metrology
- Manufacturing Metrology
- Statistical Engineering



LANL

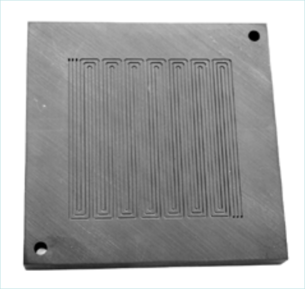
- Operational Knowledge
- Advanced Testing Facilities

Overview

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

Cell Specifications

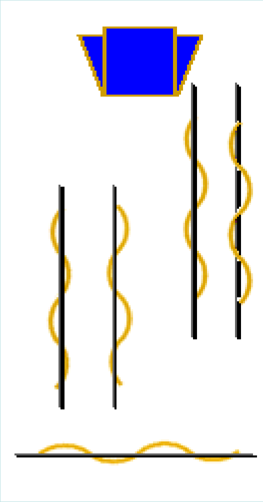
- 50 cm^2 Hardware (Teledyne CH-50)
- Gas Diffusion Media: SGL 25 BC
- Commercially Available CCM 0.1 / 0.2 mg/cm^2…Anode and Cathode
- Hydrogen Electrolysis-Grade) and Air (oilless-compressor)
- NIST Fabricated Reference Anode Plate and [10] Cathode Experimental Plates (POCO AXF-5QCF), Triple Channel Serpentine Design



Experimental Parameters and Level of Variability

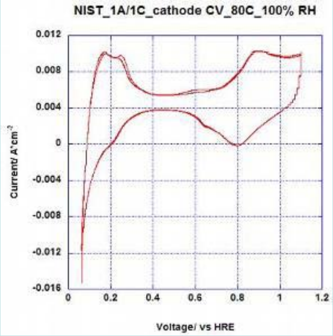
Full Factorial Design of Experiments $2^{(4-1)}$

- Sidewall Taper 0º to 10º
- Bottom Straightness 0 to 50 µm
- Sidewall Straightness 0 to 50 µm
- Variation-in-Width 0 to 100 µm



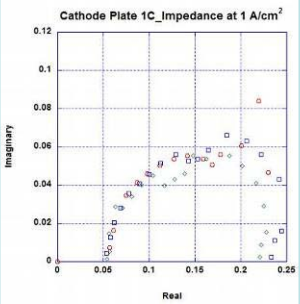
Beginning-of-Test (BOT) and End-of-Test (EOT) Diagnostics – MEA Q.C.

- Electrochemical H_2 Crossover
- Cathode Side Active Area Measurements



Performance Testing (Gas Access and H_2O Mgmt Impacts)

- Polarization curves in air measured in both directions
- AC-Impedance measurements



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

Technical Accomplishments and Progress

Performance Testing of Experimental Plates (Partnered with Tommy Rockward at LANL (2009/2010)

Current Testing Protocol***

- VI Curves @ 80ºC and 60ºC, 50% and 100% RH
- VI Curve data collected in constant current mode with 2 minute settling times
- Utilization Rates 83.3% H_2 and 71% Air
- Outlet Pressure: ambient to 25 psig both anode and cathode

****Initial testing conditions chosen to highlight performance differences between plates, then as more realistic testing conditions become available retest a select subset. All conditions fall within specifications found in DOE Cell Component Accelerated Stress Test Protocols for PEM Fuel Cells (Mar 2007). Conditions are controversial but most OEM durability protocols are considered intellectual property however SAE J2722 is a draft standard attempting to establish a baseline drive durability cycle.*

Major Challenge
Isolate Variations to Only Experimental Cathode Plates!

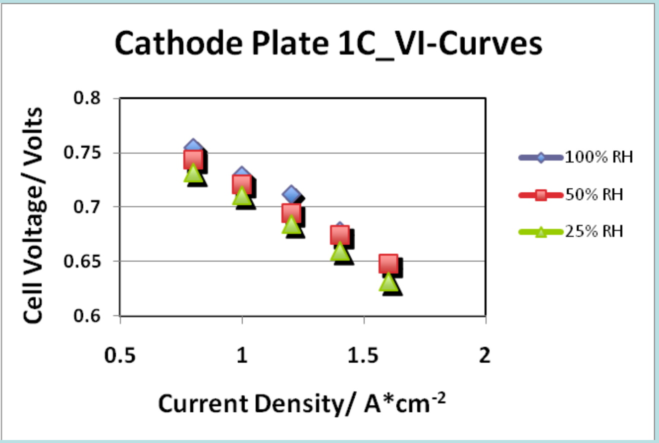
- Testing Equipment
- Assembly/Disassembly
- Performance Testing Procedure
- CCM-to-CCM Variation – Reuse Not Possible

Protocol Revisions

- Same CCM could not be reused…new CCM with each assembly from same supplier lot
- Shut down / start up must be avoided once testing begins
- Anode and cathode loadings reduced from 0.2 / 0.4 mg/cm^2 to 0.1 / 0.2 mg/cm^2 and utilization on cathode side increased from 50% to 71% to better highlight mass transport differences

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

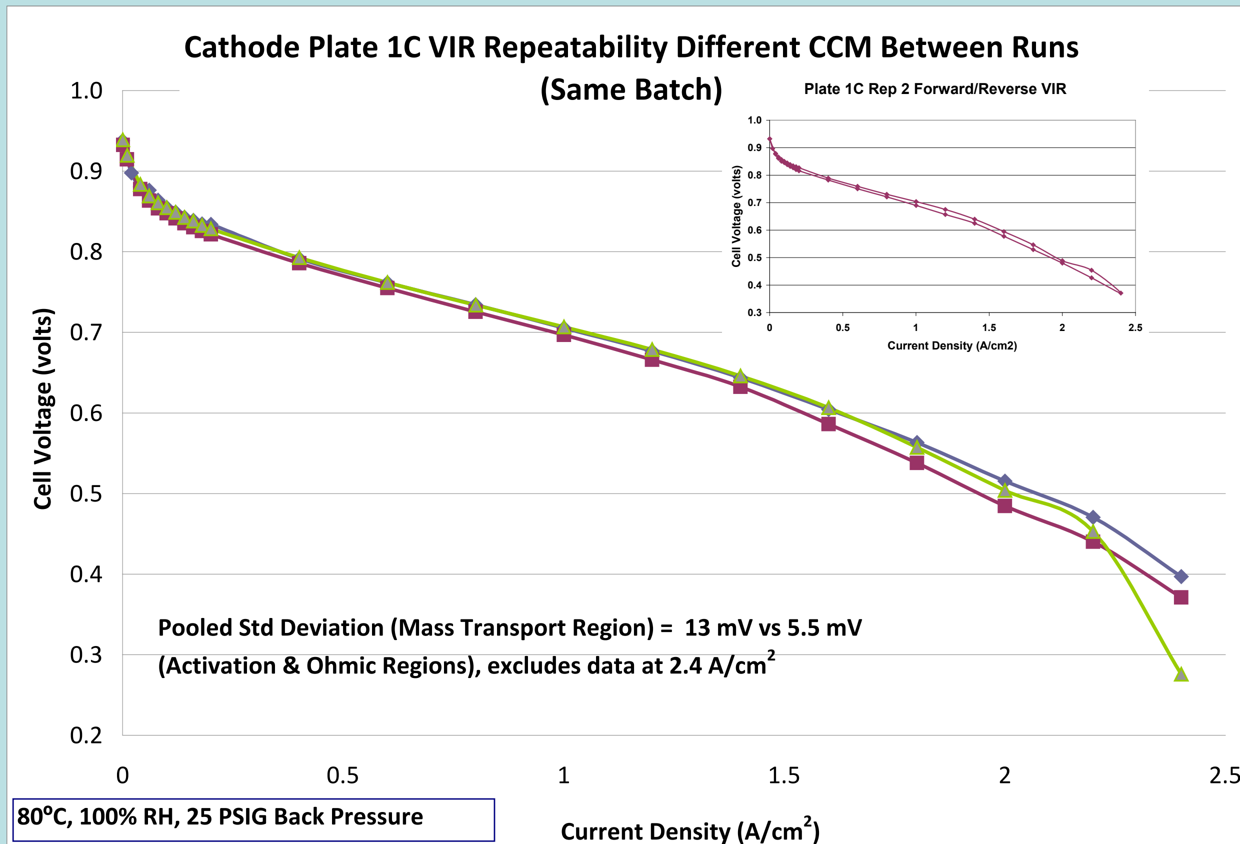
Technical Accomplishments and Progress



Performance Testing Results Reference Plate Performance & Repeatability

Reference* Cathode #1C and Anode #1A, 80ºC, 100% RH, 25 PSIG Back Pressure

Results show good repeatability using three different MEAs, GDLs, and gaskets with the reference plate.

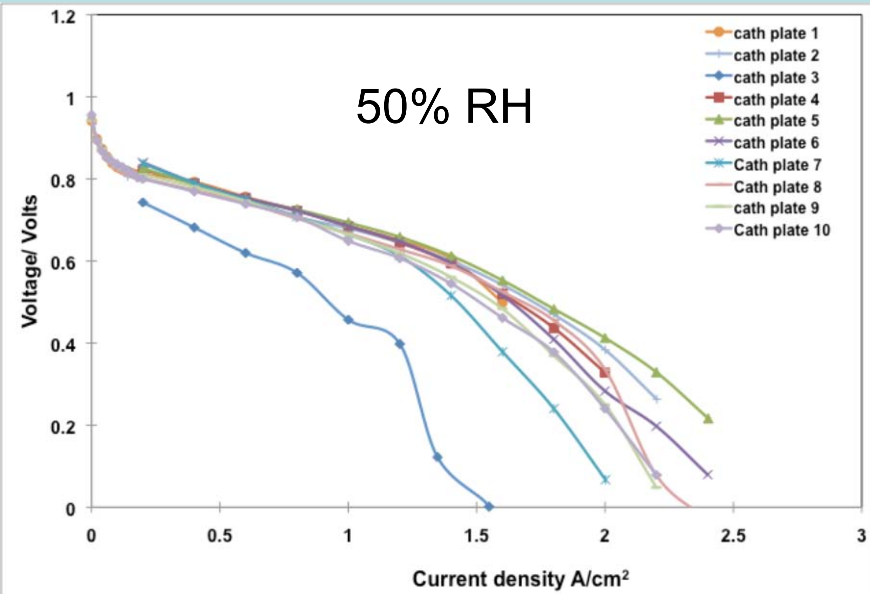


* Reference means plate channels with nominal rectangular cross-section and minimal dimensional variation.

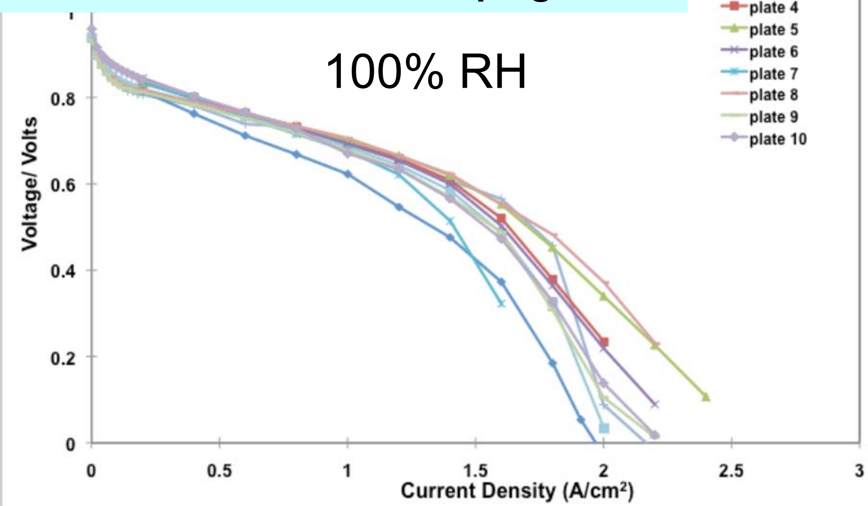
P1 **Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance**

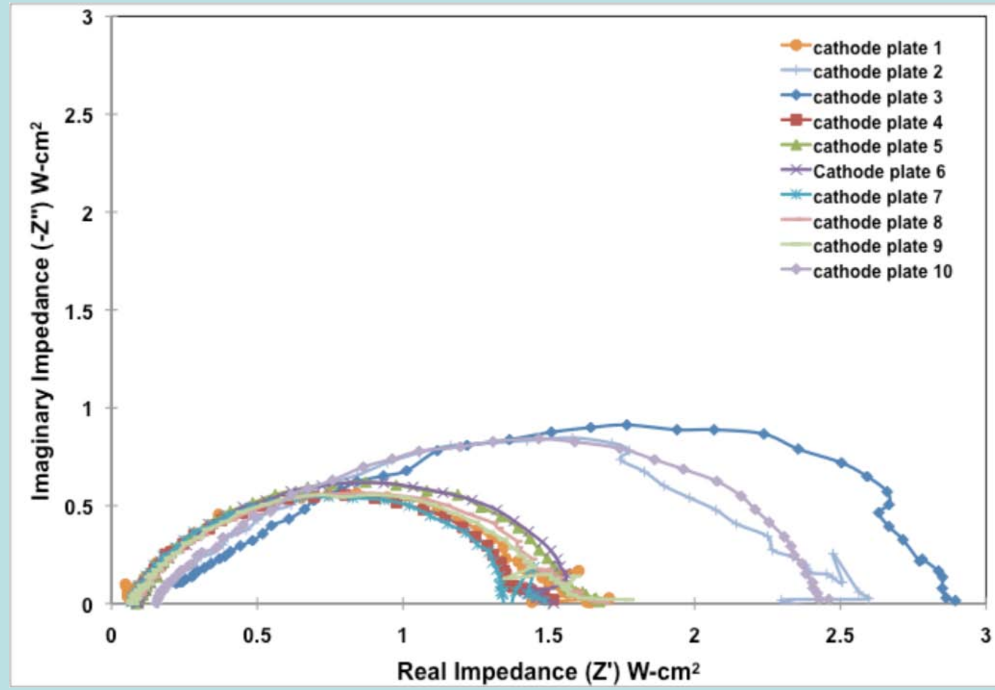
Technical Accomplishments and Progress

Performance Testing Results



Initial VIR curves for all plates @ 80ºC, 100% & 50% RH, 25 psig



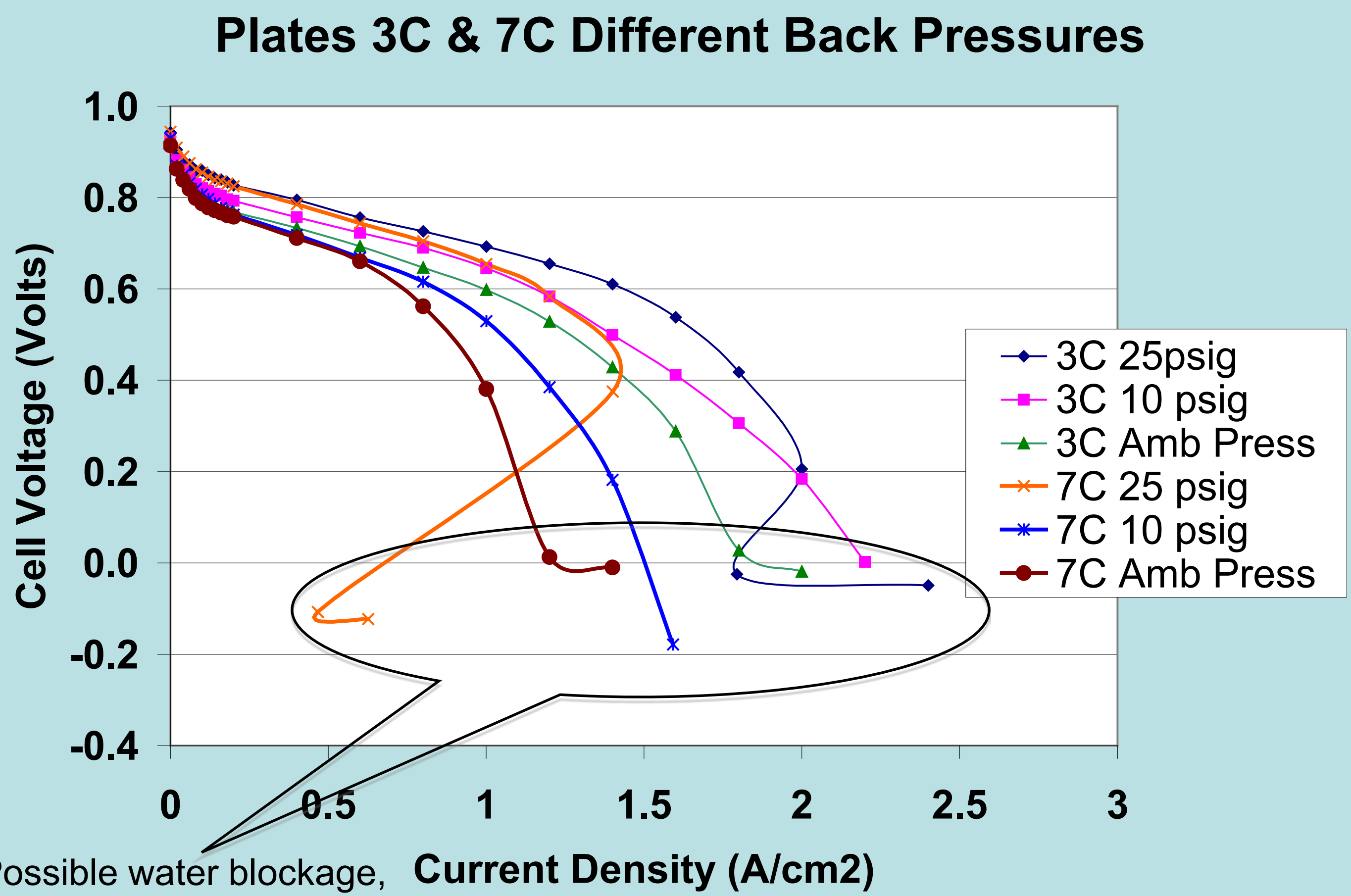


Cathode Plate Impedances at 1 A and 50%RH

Performance differences are observed using single-serpentine channels with the various intentional ‘inaccuracies’. **Similar tests with different flow-field geometries to be incorporated**.

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

Technical Accomplishments and Progress



Possible water blockage, Neutron Imaging needed to verify.

Both plates exhibit decreasing performance with diminishing back pressure (as expected), but between plate performance differences maintain order (3C>7C)

Performance vs. Back Pressure addresses 2010 AMR Reviewer's comments ---- Worst Performers

******Plate 5C best performer subject to same tests but plate broke during assembly******

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

Summary

- Intentional flow field dimensional perturbations, at the levels chosen, DO affect performance as can be seen from the VI curves and ac impedance tests
- Repeatability with replacement of CCMs combined with disassembly / assembly operations (to accommodate the change-out) between runs appears to be similar to past single cell intercomparison repeatability without these changes / operations between runs.
- Other plate data must be repeated to confirm repeatable results.

Future Work

- NIST to fabricate and verify replacement experimental cathode plate #5 (original broken during assembly in middle of repeatability testing)
- LANL will complete performance repeatability testing for plate #5
- LANL to provide NIST with quantitative VI data for experimental plates
- Analysis of experimental data by NIST's Statistical Engineering Division
- NIST & LANL to publish results

(Contingent Upon Future Industry Interest & DOE Funding Support)

- Integrate other variations such as surface finish and land height variation (variation in pressure profile) to support recently published modeling results
- Test other channel designs (i.e., parallel) – FreedomCAR Fuel Cell Tech Team Recommendation
- Vary operating conditions (e.g., ambient outlet pressure)
- Implement Neutron imaging to verify hypotheses regarding differences in performance.
- Development/refine/validate two-phase flow model using experimental data.

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

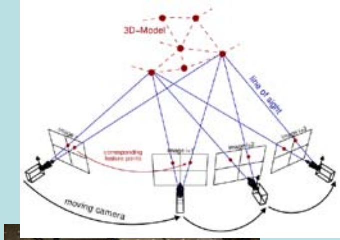
Overview

Objectives:

1) Identify and evaluate the capability and uncertainty of commercially available non-contact, high-speed scanning technologies for applicability to bipolar plate manufacturing process control.
2) Using capabilities identified in (1) demonstrate a “smart assembly” concept whereby plate parallelism data for each plate can be used to assemble the stack so that the cumulative parallelism of stack can be controlled tightly while simultaneously permitting greater parallelism variability for each individual plate.

Approach:

- Evaluate suitability based on typical plate materials and methods of fabrication
- Evaluate the ability to measure dimensional parameters of interest
- For GO technologies
  - Uncertainty determination
  - Development of measurement protocols
    - sensor evaluation
    - plate evaluation
  - Accuracy evaluation as a function of scan rate
  - Approaches to achieving contractual traceability requirements (calibration artifact vs. procedures)
  - Demonstrate “Smart Assembly” concept



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

Overview

Benefits (Relevance):

- Provide bipolar plate manufacturers with a high-speed fully automated approach for process control dimensional inspection
- **Enable rapid commercialization by indentifying, demonstrating, and characterizing QC inspection technologies suitable for high-volume bipolar plate production.**

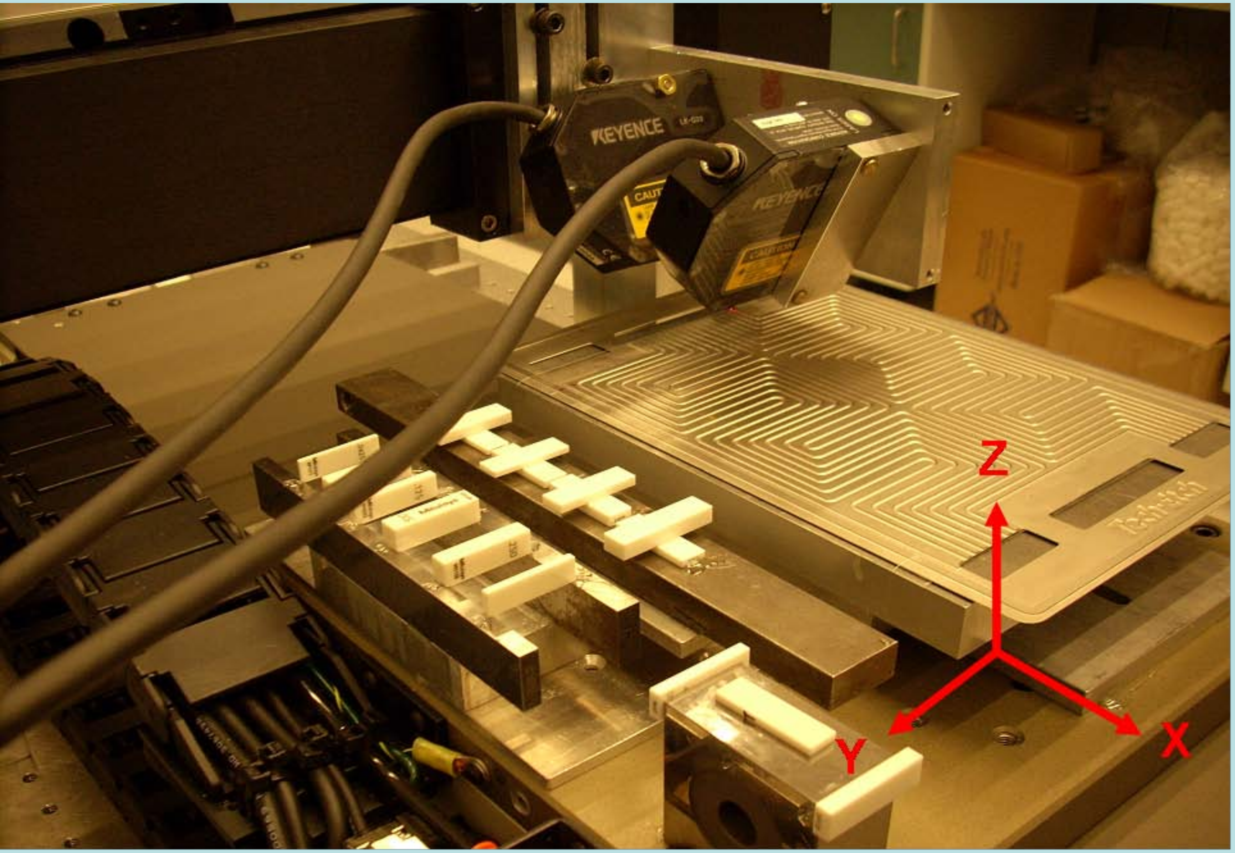
Background:

- Plate Dimensional Tolerances 50 μm → 12 μm
- Target Measurement Solution Uncertainty ~ 5 μm.
- Current State of the Industry
    - **Video Based Optical Inspection Predominantly Used** Semi-Automated, Very Subjective, Measurement Uncertainty versus higher end requirements questionable.
    - **Measurement Technology: No Packaged Systems Commercially Available**….numerous structured light systems for depth and flatness measurements but feature (lateral) size and location questionable at required accuracy.
- Industry (Plate Mfgr) Perception: A solution is needed but the priority is low as they are nowhere near high-volume production rates.

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

Accomplishments

The Solution NIST designed measurement system using commercially available precision translation stages coupled with dual non-contact laser spot triangulation probes configured in a unique manner to enable **sub-10 µm accuracy of vertical and lateral measurements** for feature size and location.



System Cost Est. Provided to Directed Technologies 2010 ~$50K (as shown) + $14K per set of additional dual sensors [See Supplement Slides for Details]

Acknowledgements:

Bala Muralikrishnan
Dennis Everett
Wei Ren
Ted Doiron
Patrick Egan

Keyence LK-G32 Probe and LK-GD500 Controller, Aerotech ALS 50060 Precision Stage (X-Axis) and Aerotech PRO 115 (Y-Axis)

Click for Video ->

Accomplishments

Design Progression (3rd "Current" Design Iteration)

Duel Probe Configuration using Position Synchronized Output (PSO) of the Stage - GO!

- Vertical and lateral measurements through combination of stage and probe reading.
- Requires precise calibration of two orientation angles for both probes (Fig 2-1)
- Scale and linearity dependent on material and incident angle (Fig 2-2)
- Must match part and reference master material and finish when calibrating scale and linearity of probes (absolute must for these type of non-contact probes regardless of application) (Fig 2-3)

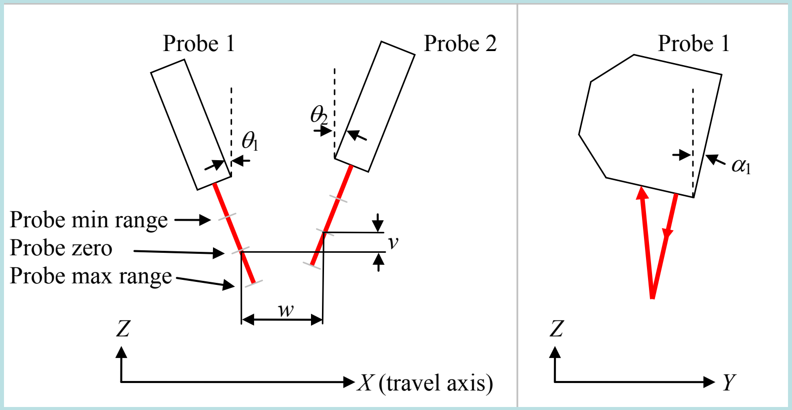


Fig 2-1 Probe Cal Angles

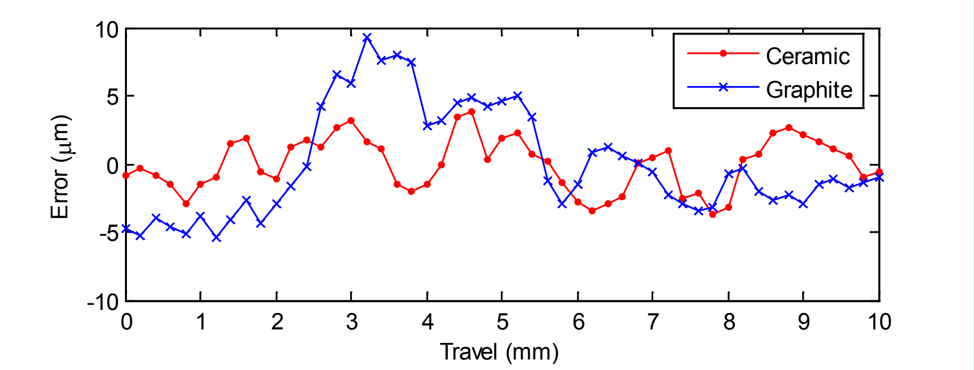
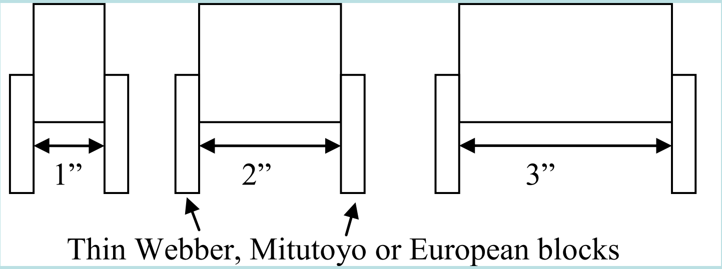


Fig 2-2 Linearity Material Dependence (Normal Incidence)



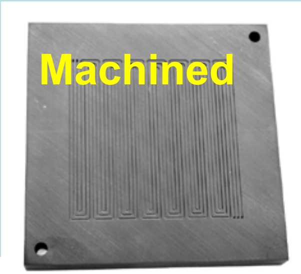
Nominal Block Size (inches)	1	2	3
	Error (micrometers)		
Manufacturer			
European	0	0.4	1.1
Mitutoyo	-4.5	-4.6	-5.1
Webber	-7.1	-5.6	-6.1

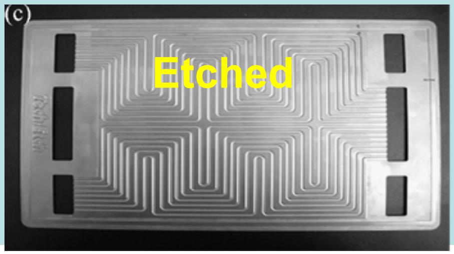
Fig 2-3 Material Dependence using Difference Ceramic Gage Blocks

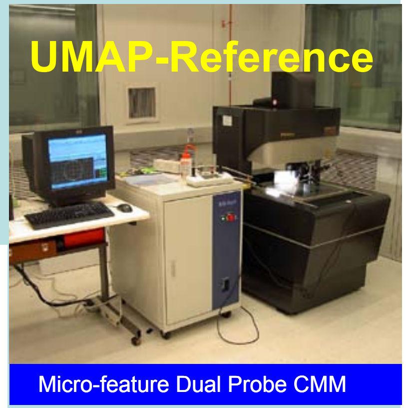
P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control and Smart Assembly of Fuel Cell Stacks.

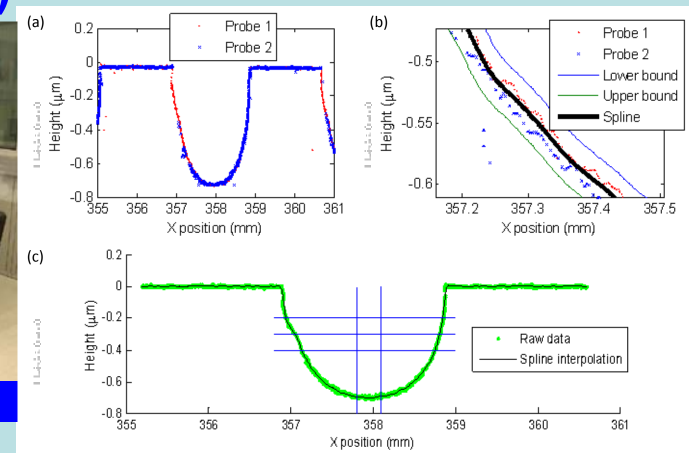
Accomplishments

NIST Measurement System Validation Data Using Machined and Chemically Etched Flow Field Plates – Non-Contact System vs. Reference Measurements (Micro-Feature Dual-Probe CMM)









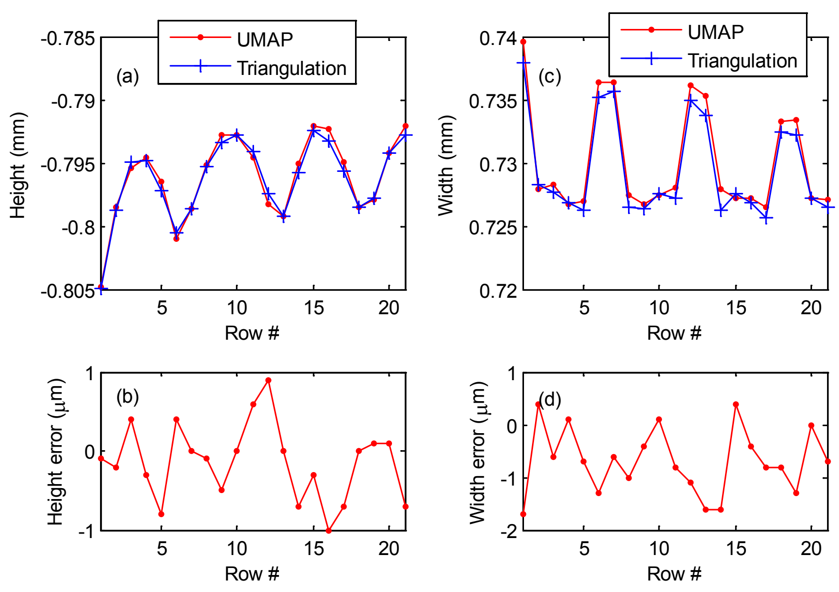
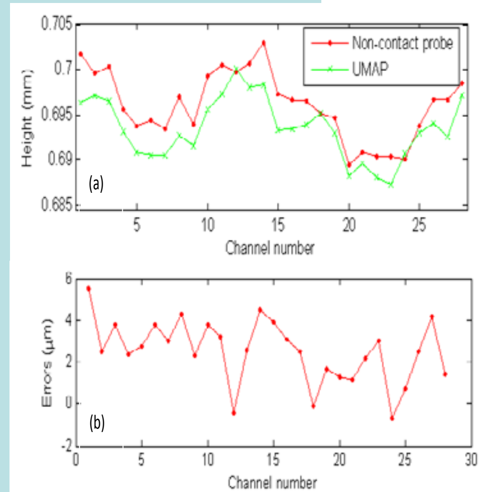


Fig 2-4 Comparison results for width and height measurements using a **machined** carbon composite sample plate (straight sidewalls - no taper).



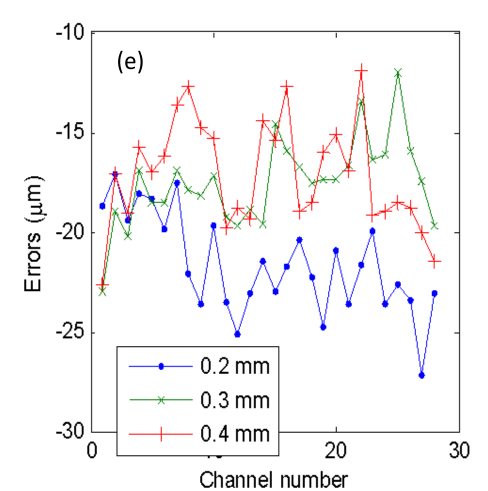


Fig 2-5 Comparison results for width and height using a **etched** metallic sample plate.

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

Accomplishments

NIST Measurement System Uncertainty

Machined Carbon (Optimal Quality Surfaces)

Expanded Uncertainty (k=2) Width = ± 6.0 µm Height = ± 3.8 µm

Etched Metallic Plate (Worst Case Surface Quality)

(Estimated) Expanded Uncertainty (k=2) Width = ± 15 µm, Height = ± 10 µm

Dominant Error Sources (Optimal Quality)

#4 Probe linearity
#6 Z straightness of X axis (error could be mapped)

(Non-Optimal Quality)

#9 and #10

	Source	Uncertainty in height (µm)	Uncertainty in width (µm)
System parameters			
1	Uncertainty in the estimation of tilt angle θ	0.2	0.5
2	Uncertainty in the estimation of α	0.1	0
3	Uncertainty in the estimation of vertical offset between probes	0	
Probe			
4	Probe linearity	1.5	1.4
5	Probe repeatability	0.1	0.1
Stage			
6	Z straightness of the stage	0.9	
7	Pitch motion of the stage		1.0
8	Uncertainty in encoder readings		0.1
Part			
9	Part surface texture and form	0.1	0.1
10	Repeatability from parallel profiles on part	0.5	1
Alignment			
11	Non-zero α and Y displacement	0.4	
12	Part misalignment		0.1
13	Offset between probes along Y axis		0.5
Other			
14	Realizing the definition of the measurand	0.2	0.2
15	Uncertainty in the estimation of horizontal offset between probes (root sum square of above described terms)		2.1
	Combined standard uncertainty ($k = 1$)	1.9	3

Uncertainty Budget Optimal Plate - Machined

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

Collaborations/Interactions

Informal Collaborations

- **Faraday Technologies**
  - SBIR to investigate application of their patented pulsing electric field technique to optimization of chemical etching SS plates
    - dimensional evaluation of plates
    - validation data for this project
    - 1st samples submitted and assessment report provided

Capability Demonstrations/Feedback:

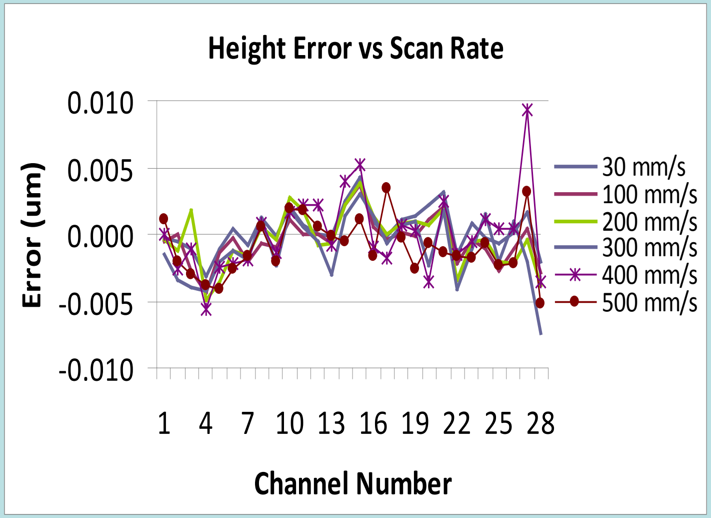
- **Tech-Etch**
  - supplied measurement evaluation report of sample plate.
- **Dana Corporation**
  - carbon composite (U.S.), compression molding most concerned about variation-in-thickness
  - publication and discussion information being forwarded to facility in Germany (metallic plates)
- **GM**
  - using stamped metallic plates
  - forwarded demonstration information and publication to them for evaluation

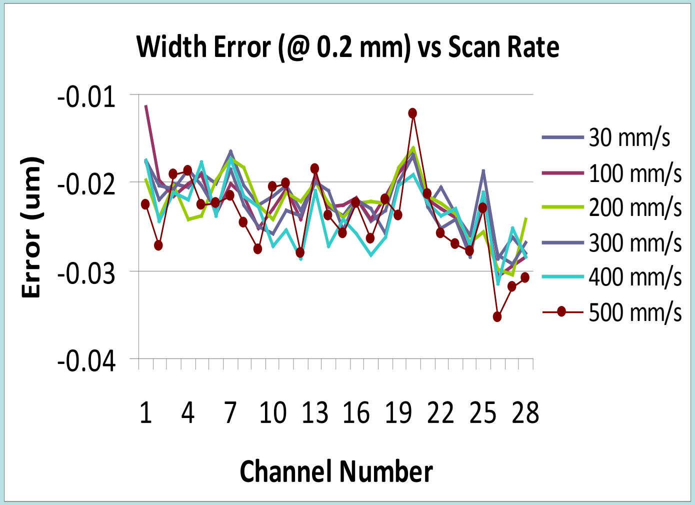
Other Interactions: Aerotek, Keyence, MetroMold, GrafTech, FotoBab, Borit, Morphic, Coran Precision, SGL Carbon, Porvair, GenCell

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

Accomplishments

Error as a function of scan rate using etched plate…….. 30 mm/s to 500 mm/s…very little accuracy degradation!





Future Work

- Configure Measurement System for Plate Parallelism and Variation-in-Thickness (VIT)
  - Demonstrate Capability
  - Uncertainty Determination
  - Scan Rate Sensitivity Testing
- Plate ID and Tracking Strategy
- Stack Assembly Algorithm (using plate parallelism data due to variation-in-thickness)

Publications

B. Muralikrishnan, W. Ren, D. Everett, E. Stanfield, T. Doiron, Performance evaluation experiments on a laser spot triangulation probe, to be submitted to Measurement: Journal of the IMEKO

B. Muralikrishnan, W. Ren, D. Everett, E. Stanfield, T. Doiron, Application of surface profile filters for non-contact dimensional measurements of an etched fuel cell plate, currently under review at Measurement Science and Technology.

Abstract for Fuel Cell Seminar 2011 will be submitted.

Subproject Overview

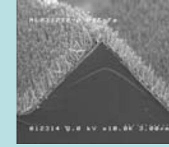
P3 Optical Scatterfield Metrology for Online Catalyst Coating Inspection of PEM (Fuel Cell) Soft Goods

Objective: Using catalyst coated samples provided by manufacturers with variations in critical parameters (i.e. Pt and Pt alloy catalyst loading) and inclusion of various types of defects characterized using standard 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/transfer 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.

P3 Accomplishments 2010→2011 Experimental

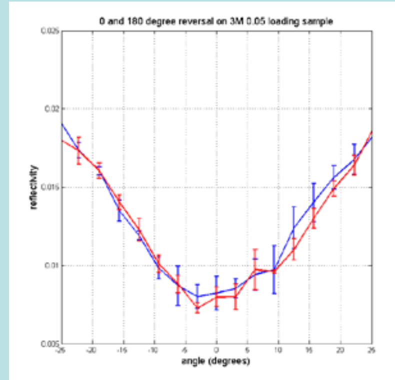


1st Sample Package: PtCoMn NSTF CCM

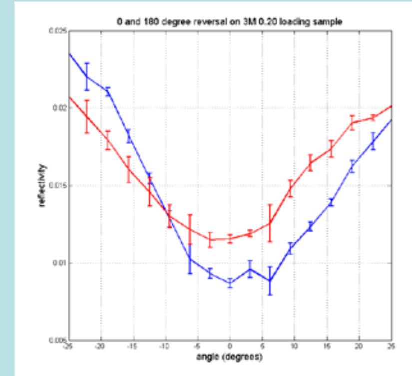
- 0.10, 0.15, and 0.20 mg/cm² samples
- 2010 AMR demonstrated successful application of approach to measurement of catalyst loading (**sensitivity ~ 0.01 mg/cm²**)
- **GO** decision for continued work

2nd Sample Package: Pure Pt NSTF CCM (0.05, 0.10, 0.15, 0.20 mg/cm² samples)

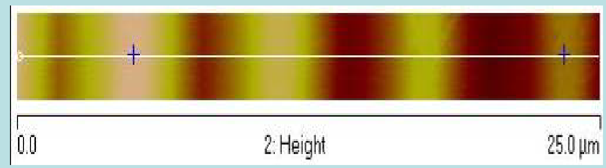
- 2010 AMR demonstrated sensitivity
  - Sensitivity level undetermined, encountered asymmetry problems with 0.20 mg/cm² sample
- **2011 Asymmetry investigation**
  - A **"reversal"** is performed on the 0.20 mg Pt/cm² sample – **conclusion** is that there is an issue with this sample
  - AFM showed that the angles of the micro-triangular substructure for the 0.20 mg/cm² sample were measurably different.
  - Eliminated this sample from our experiments.
- **First data** taken on the 3M pure Pt NSTF samples
  - There is sensitivity but, not at the same level as the 3M PtCoMn samples.
  - **Important to note:** the pure Pt NSTF samples are 2nd generation samples, the dimensions of the underlying micro-triangular substructure are different, possible cause of sensitivity difference.

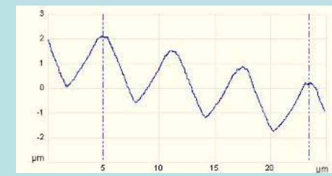


Sample Reversal on 0.05 sample (0.10 and 0.15 looked the same)

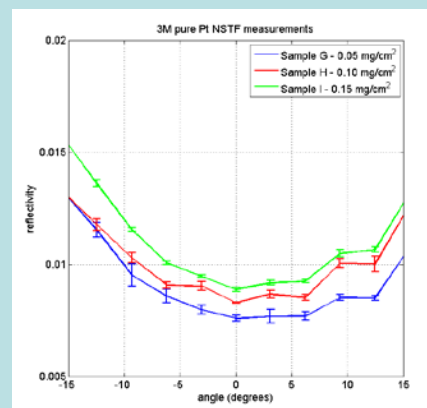


Sample Reversal on 0.20 sample





AFM investigation into geometrical cause of asymmetry

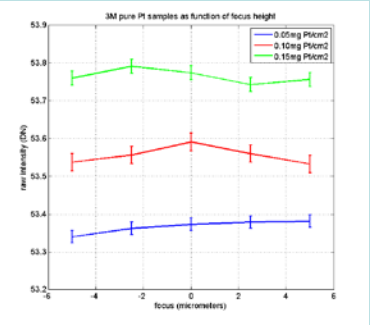


First pure Pt data set in 2011 (3 random locations, 3 repeats)

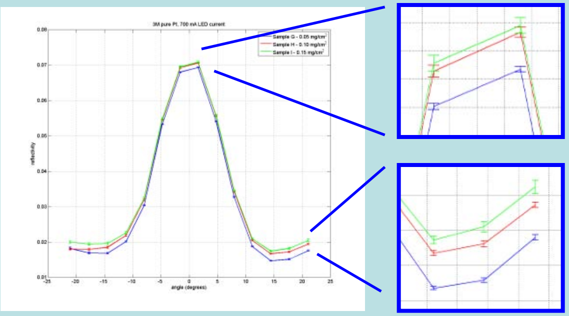
P3 Accomplishments 2010→2011 Experimental

2nd Sample Package: Pure Pt NSTF CCM (0.05, 0.10, 0.15, 0.20 mg/cm² samples)

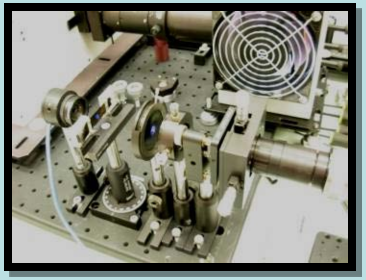
- Began efforts to **improve the sensitivity** to 0.01 level and to **understand sources of uncertainty**
- Performed a **focus vs sensitivity** study, maintained sensitivity over a **10 µm focal range**.
- More than doubled the illumination intensity (LED current set to 700 mA) of the Scatterfield Tool to improve signal to noise ratio. **Sensitivity is on the order of 0.03 – 0.05 mg Pt/cm²** at higher angles. The reflectivity curves as a function of angle open downward, opposite of first set. Became suspicious that samples were not opaque.
- Measured transmission properties of 3M pure Pt NSTF samples. **Not opaque!**
- Began investigating effects of how the pure Pt NSTF samples were mounted. Went through several iterations of mounting jigs. The current one suspends a strip of the CCM approximately ½ inch from a light absorbing material to ensure no light reflects back from mount. Curves open upward again. Data not so nice. **Work in progress.**



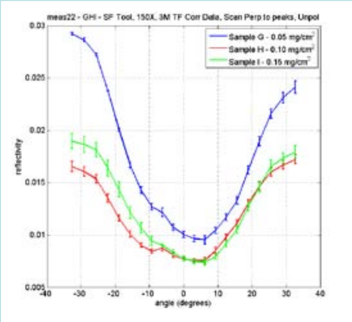
Focus vs sensitivity study



Effort to improve s/n ratio (3 locations, 3 different focus postions)



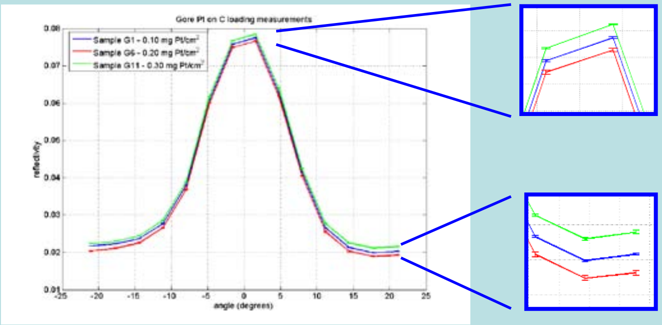
Transmission measurements of pure Pt samples



3M samples suspended above light abosrbing material (9 random locations)

3rd Sample Package: Gore Pt on Carbon CCM (A510/M710.18/C510)

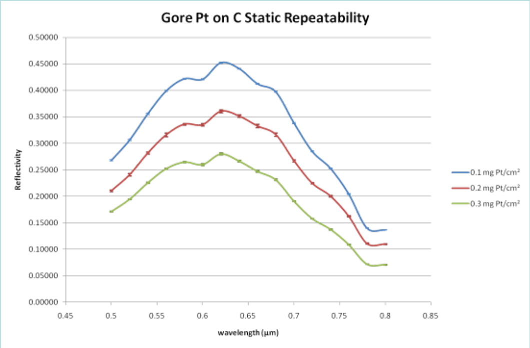
- (Sample Loadings 0.10, 0.20, 0.30, and 0.40 mg/cm²)
- These measurements were performed on the Scatterfield Tool (only three of the loadings were measured). The data demonstates a **sensitivity on the order of 0.03 – 0.04 mg Pt/cm².** (Sensitivity appears better at high angles.)



First set of loading measurements on **Gore** CCM samples (9 random locations)

P3 Accomplishments 2011 – Reflectance measurements with Ellipsometer

Spectroscopic Ellipsometer
Sopra GES5 from Semi-labs
Arrived in February 2011, enables **n & k** measurements as well as **reflectance** and **transmission** measurements.

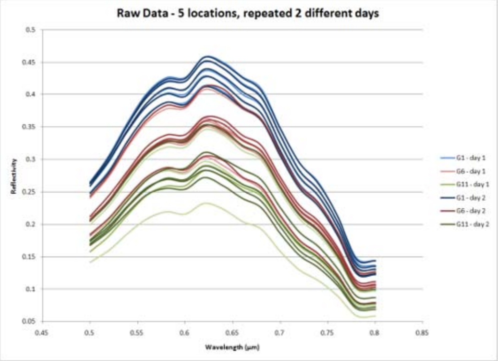


Excellent Repeatability!!!
(3 locations, 5 times each)

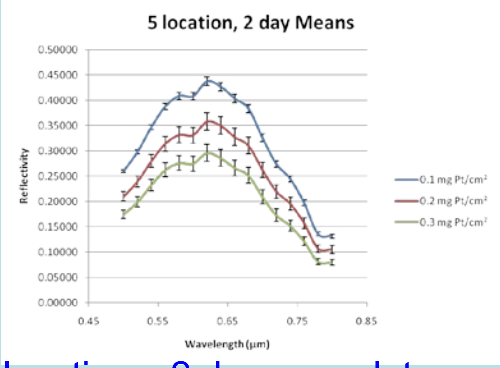
3rd Sample Package: W.L. Gore Pt on Carbon CCM (A510/M710.18/C510)

(Sample Loadings 0.10, 0.20, 0.30, 0.40 mg/cm^2)

- Reflectivity measurements were done on the new ellipsometer as a function of **wavelength**
- Static repeatability test produced excellent data.
- The next test looked at reproducibility, 5 locations (several mm apart) on two different days. Data shows definite **local variations in loading** across the sample. The 5 dark blue curves are the 5 locations measured on one loading and the 5 light blue curves are the same exact locations measured on a different day. **The curves for each location track exceptionally well!**
- The middle plot is the raw data from the reproducibility experiment just described. The bottom plot is the same data with the variations represented in the error bars.
  - Further experimental work and model simulation needed to achieve 0.01 mg/cm^2



5 locations, reproducibility over 2 days (data show localized loading variations)

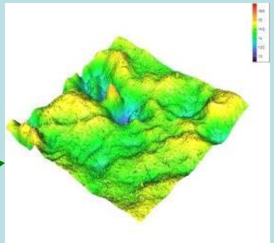


5 locations, 2 days raw data, means with error bars equal to the pooled std. error.

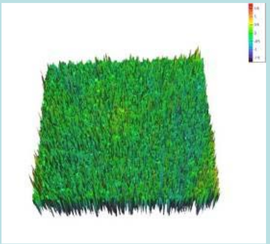
P3 Accomplishments 2011 – n & k measurements with Ellipsometer

3rd Sample Package: W.L. Gore Pt on Carbon CCM (A510/M710.18/C510)
(Sample Loadings 0.10, 0.20, 0.30, 0.40 mg/cm^2)

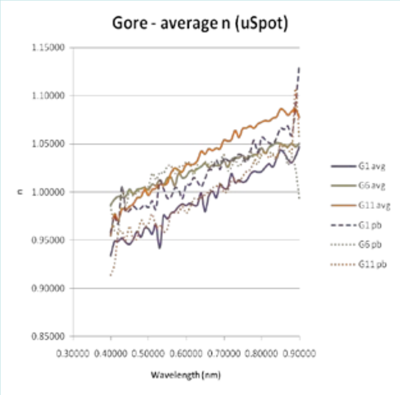
- **Began n&k measurements of consituent materials** of the samples. **Surface roughness** presented a problem.
- Performed spectroscopic ellipsometry on 3 of the 4 Gore Pt on C samples, treated the samples as effective medium, measuring 1 n&k for the sample, plug this into the model to develop self-consistency
- The experimental and theoretical data are **trending correctly** with respect to angle. The behaviour as a function of wavelength is under investigation.

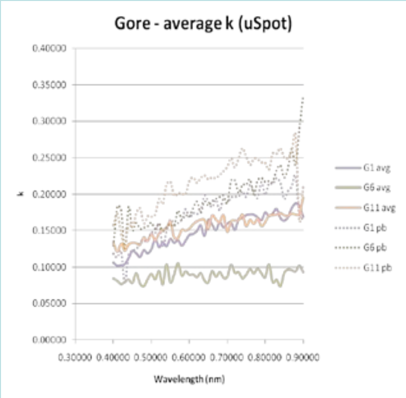


3M PEM film surface finish measurement



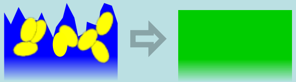
Gore PEM film surface finish measurement

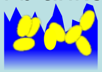
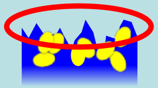


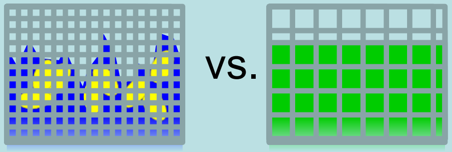


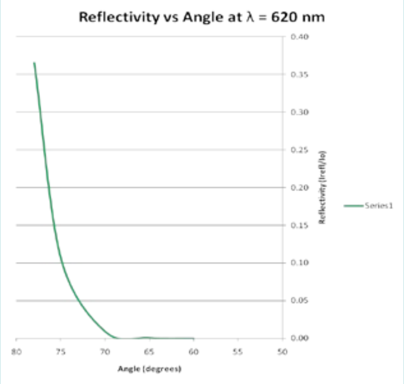
Measured 3 Gore samples as **effective medium** n & k values

Motivations for using an Effective Medium Approximation

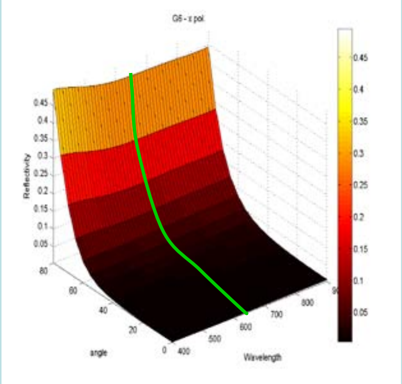


- We have a lack of blanket constituent films.
  - To model  requires knowledge of [blue] and [yellow]
- Surface roughness drives need for EMA. 
  - Rigorous modeling of roughness requires 3-D models
- Computational requirements necessitate approximation.
  - Memory constraints
  - CPU constraints





Experimental - Ellipsometer angle scan



Theoretical - Reflectivity vs angle and wavelength

P3 Accomplishments 2011 – Simulations

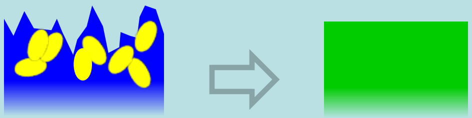
Modeling Solutions for Maxwell's Equations

A significant amount of time was invested in testing our 3D electromagnetic scattering models with respect to modeling complicated fuel cell structures. We now have a much better understanding of some of the limitations and some of the strengths and weaknesses of the 3D models as applied to fuel cell structures. The various model-specific input parameters affecting convergence were studied. The next slide "**Accomplishments 2011 – Simulation Convergence Testing**" shows the results from some of the convergence testing done on our in-house FDTD code.

- **2-D** Modeling

RCWA (EMA)

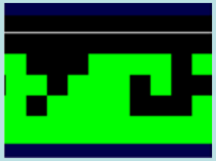
Rigorous coupled-wave analysis /
Effective Medium Approximation



- **3-D** Modeling

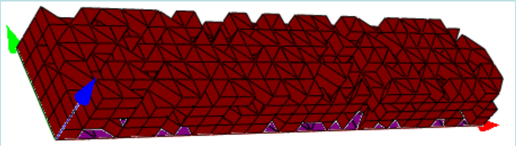
FDTD

Finite-difference time-domain



FEM

Finite element method



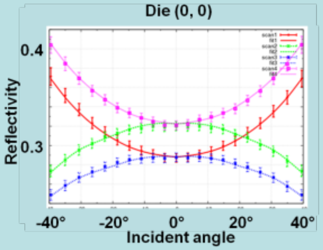
<u>Convergence Issues</u>

# of orders

Domain Size
Grid Size
Grain Size

***Goal**: to develop and validate a rigorous EMA for fuel cell*

P3 Accomplishments 2011 – Simulation Convergence Testing

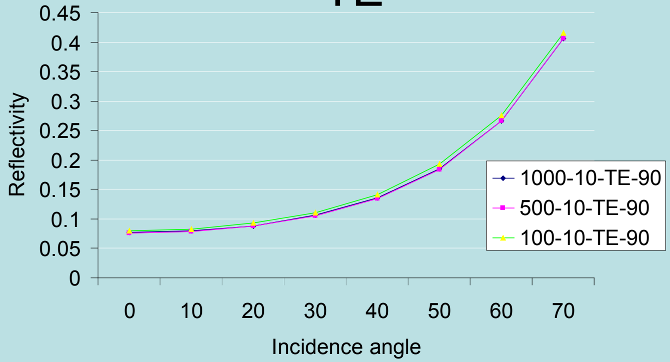


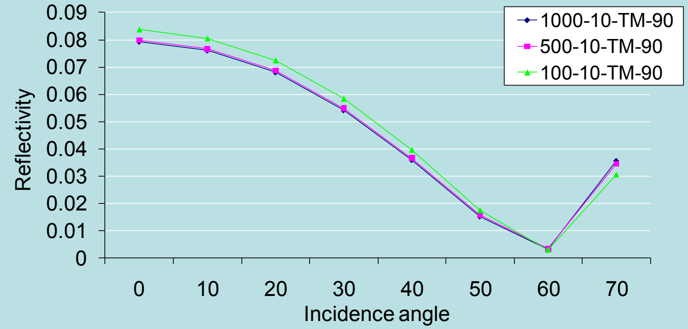
FDTD 3D simulation

TE

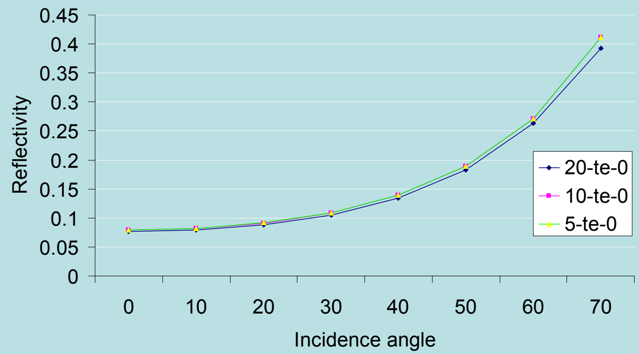
TM

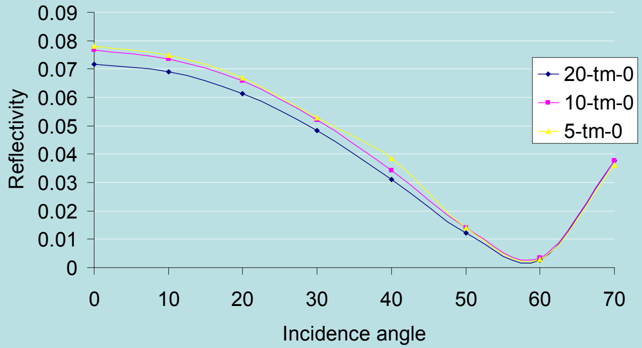
Domain Size:



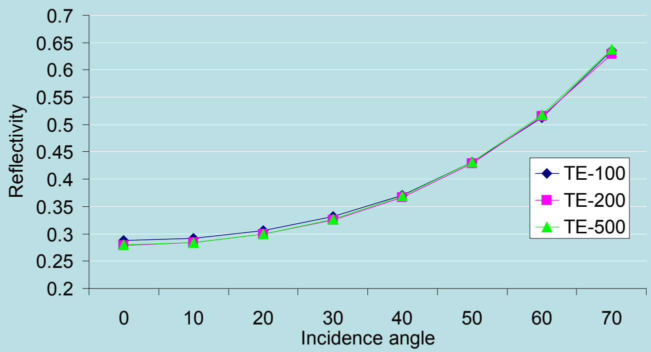


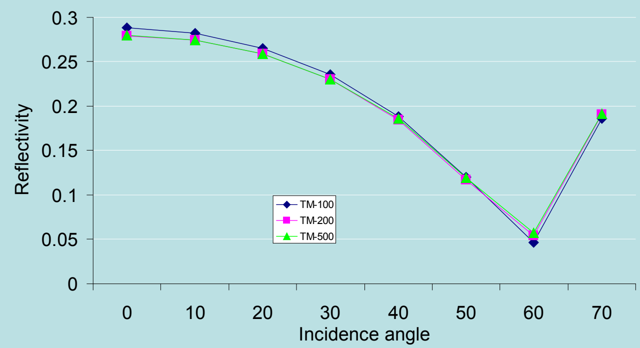
Grain Size:





Grid Size:





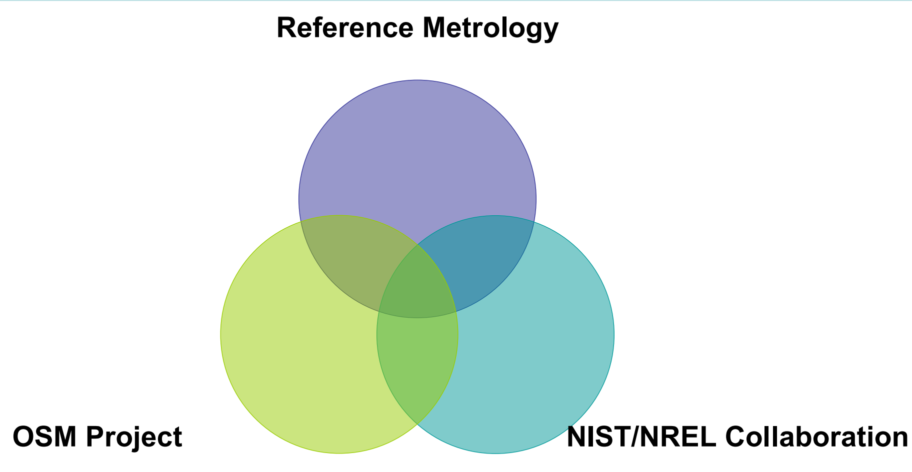
P3 Collaborations/Acknowledgements/Cost Estimate/NIST-NREL Collaboration

NIST/NREL Collaboration in Support of Fuel Cell Manufacturing:

NIST/NREL
Three-Way NDA Process

- **3M ✓**
- **Gore ✓**
- **Arkema ✓**
- **Ballard ✓**
- **Johnson-Matthey ✓**
- **BASF**

OSM Project & NIST/NREL Collaboration Relationship



Acknowlegments:

NIST

Optics Project Team

Dr. Richard Silver - Group leader / Physicist
Mike Stocker – Metrologist
Dr. Bryan Barnes – Physicist
Dr. Hui Zhou – Chemical Physicist
Dr. Yeung-Joon Sohn – Physicist
Dr. Egon Marx – Theoretical Physicist
Francois Goasmat – Computer Scientist
Dr. Jing Qin – Physicist

Microscopy/Reference Metrology

Andras Vladar
Brad Damazo
Bin Ming

Fuel Cell Project Special Acknowledgements

Mark Debe (3M)
Judy Rudolf (Gore)
Guido Bender (NREL formerly HNEI)
Mike Ulsh (NREL)

System Cost Est. Provided to Directed Technologies 2010 $50K to $100K for either of two different approaches [See Supplement Slides for Details]

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

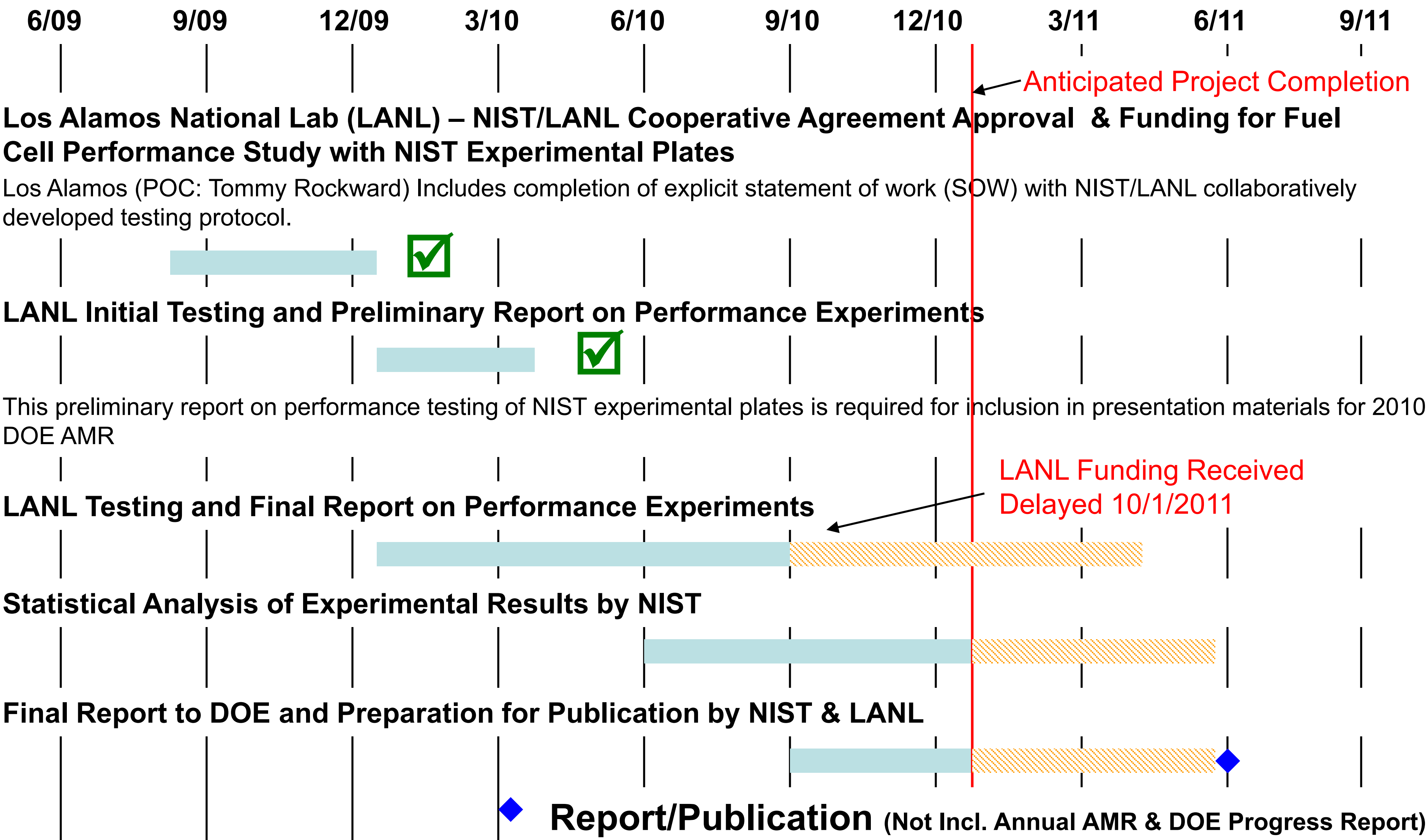
Future Work

- Perform measurements on 3M NSTF and Gore samples using the 193 nm tool, expanding our spectroscopic experimental investigation of optimal sensitivity into the UV range.
- Continue modeling work based on Effective Medium Theory. Successful application of this approximation would significantly decrease simulation times. Models must be developed for both the 3M NSTF CMM and conventional Pt on C CCM.
- Aggressively acquire index of refraction data (n and k) for each constituent material for each CCM supplier samples we have tested experimentally using the new generation ellipsometer recently delivered.
- Expand OSM loading sensitivity applicability study to included CCMs produced by other manfuacturers and GDEs with different substrates/morphologies …carbon paper and cloth.
- Work closely with NREL and industry collaborators to identify other material parameters and defects most appropriate for the OSM approach (i.e., defects that don't require entire material inspection). Then study these parameters and/or defects through experimentation and modeling.
- Develop an OSM conceptual design suitable for a manufacturing web line once theory (simulation) to experimental agreement is demonstrated. Then pursue company to commercialize product.
- Provide assistance to NREL by way of micro structural characterization and defect fabrication and quantification (reference metrology).
- Continue reference metrology support for the OSM Project by further refining/developing our microanalysis capabilities to provide further insight for the OSM experimental and model simulation efforts.

Technical Back-Up Slides

Deliverables 3rd Quarter FY2009 – End of FY2011

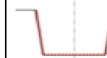
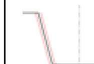
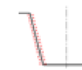
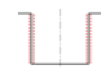
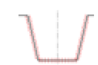
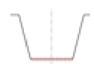
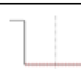
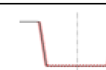
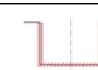
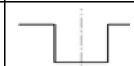
P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance



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

Technical Accomplishments and Progress

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

	2^{4-1} Fractional Factorial Design with replicated center point (k=4,n=10)							
	Sidewall Straightness	Sidewall Straightness	Bottom Straightness	Sidewall Taper				
	Amplitude	Phase	Amplitude			Sequence		Drawing
Part	*X1*	*X2*	*X3*	*X4*	*Machining*	*Measuring*	*Perf. Testing*	*Cross-Section*
9	0(25µm)	0(90)	0(25µm)	0(5)	1	1	1	
3	-1(0)	+1(180)	-1(0)	+1(10)	2	2	2	
2	+1(50µm)	-1(0)	-1(0)	+1(10)	3	3	3	
4	+1(50µm)	+1(180)	-1(0)	-1(0)	4	4	4	
8	+1(50µm)	+1(180)	+1(50µm)	+1(10)	5	5	5	
5	-1(0)	-1(0)	+1(50µm)	+1(10)	6	6	6	
7	-1(0)	+1(180)	+1(50µm)	-1(0)	7	7	7	
10	0(25µm)	0(90)	0(25µm)	0(5)	8	8	8	
6	+1(50µm)	-1(0)	+1(50µm)	-1(0)	9	9	9	
1	-1(0)	-1(0)	-1(0)	-1(0)	10	10	10	

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

Technical Accomplishments and Progress

Design of Experiments – Rational for Parameters (2006-2007)

*****Any parameter that might disrupt or unintentionally improve mass transport*****

Bottom Channel Straightness

Thin compression molded carbon composite bipolar plate design with orthogonal parallel channels: visual bleed-through of channels on one side to the bottom of the channels on the other, measured to be ~ 50 μm sinusoidal amplitude.

Side-Wall Taper

Prototype research done with machined rectangular channels…the reality…channels produced by high-speed manufacturing processes will have tapered sidewalls

Does the water management performance advantages of triangular channel cross-sections over rectangular channels extend to trapezoidal shaped (tapered channels) as noted in the following paper: *J.P. Owejan, T.A. Trabold, D.L. Jacobson, M. Arif, S.G. Kandlikar, "Effects of flowfield and diffusion layer properties on water accumulation in a PEM fuel cell," International Journal of Hydrogen Energy 32 (2007) 4489 – 4502*

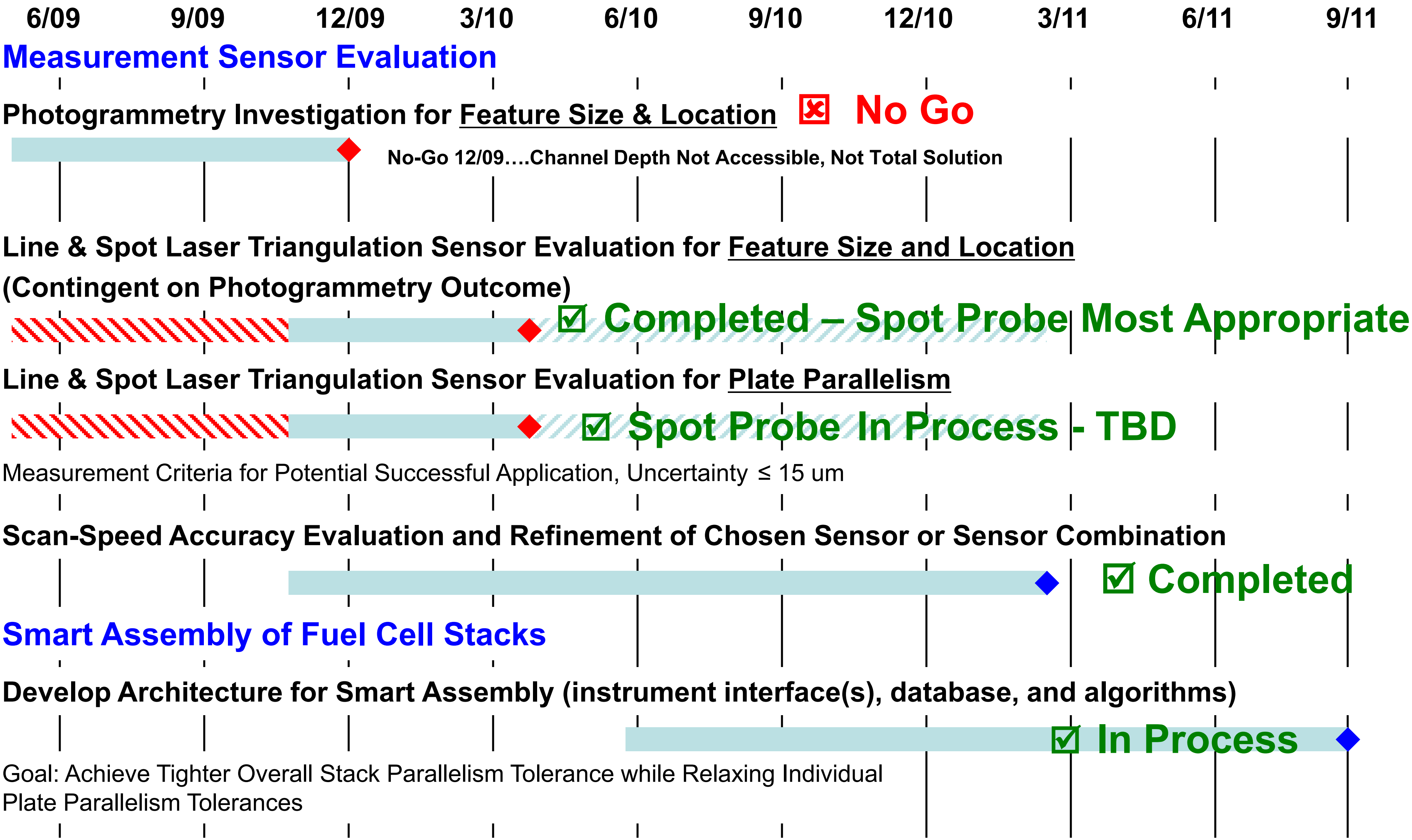
Side-Wall Straightness and Variation-in-Width

Seemed logical and width variation (100 μm) easily fabricated as extension of 50 μm side-wall sinusoidal straightness error by varying straightness phase between sides.

Surface Finish Considered important but could not accommodate at time of DoE development

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

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

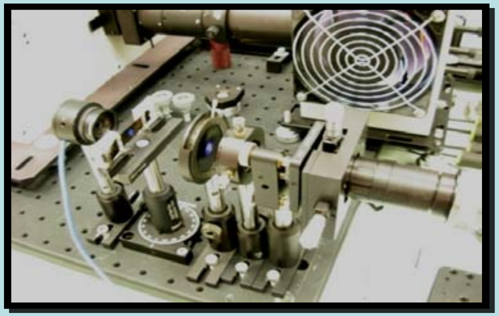


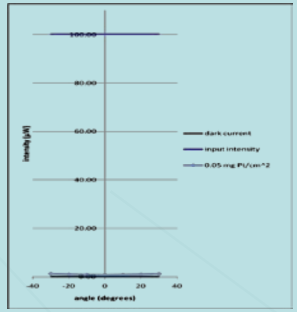
◆ Go/No-Go – Proceed? ◆ Report/Publication (Not Incl. Annual AMR & DOE Progress Report)

P3 Accomplishments 2011

2nd Sample Package: Pure Pt NSTF CCM (0.05, 0.10, 0.15, 0.20 mg/cm2 samples)

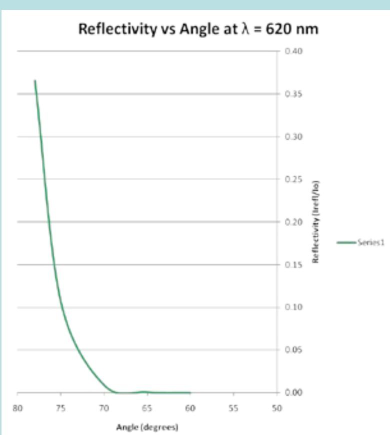
- Loading uncertainty still quite large relative to Pt alloy CCM data.
  - Pure Pt samples - not completely opaque.
  - Investigation of transmission properties of sample on loading data



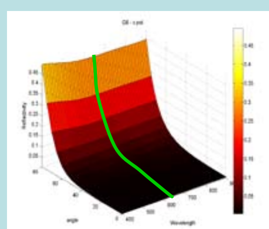


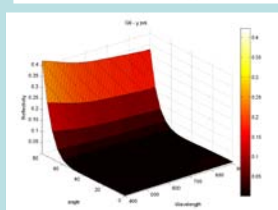
Experimental vs Theoretical

Experimental Ellipsometer, p-pol

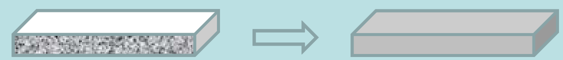


Theoretical (RCWA data, EMA)





Effective Medium Approximation



The optical properties of composite materials can often be approximated by a uniform effective medium when the length scales associated with the local variations in permittivity are small compared to the wavelength of the light in the media.
We will use a generalized anisotropic Bruggeman EMA to model the effective medium layers. This model can be derived from the generalized form of Maxwell Garnett's EMA using the expression for the polarizability of ellipsoidal particles.

$$\frac{\varepsilon_{\text{eff}} - \varepsilon_0}{\varepsilon_0 + L(\varepsilon_{\text{eff}} - \varepsilon_0)} = \sum_{i=1}^{M} f_i \frac{\varepsilon_i - \varepsilon_0}{\varepsilon_0 + L(\varepsilon_i - \varepsilon_0)},$$

where ε_{eff} is the effective medium permittivity, ε_0 is the permittivity of the host medium, and ε_i and f_i are the permittivities and volume fractions of each of the *M* materials.

(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 ☑ Ongoing

NDA Establishment with Industry Mfgrs & 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 ☑ NSTF Pt Alloy & Pt Completed

Validate Sensitivity OSM sensitivity using other conventional catalyst coated components (other CCMs, GDEs (cloth and paper). Ongoing ☑ Pt/C CCM → GDE

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)