

# NIST Fuel Cell Manufacturing Research Project

## Metrology for Fuel Cell Manufacturing

Eric Stanfield

NIST

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Project ID #

**MF-7**

# Overview

## Timeline

- October 1, 2006
- September 30, 2009
- Percent complete (40%)

## Budget

- Total project funding
  - DOE \$900K
  - NIST \$800K
- Funding received in FY07
  - DOE \$0K
  - NIST \$200K
- Funding for FY08
  - DOE \$300K
  - NIST \$300K

## Barriers

- A. Lack of High-Volume Membrane Electrode Assembly (MEA) Process
- B. Lack of High-Speed Bipolar Plate Manufacturing Processes
- F. Low Levels of Quality Control and Inflexible Processes

## Interactions

- LANL
- HNEI
- Porvair
- 3M
- W.L. Gore
- SFCC
- GM
- UTC
- SGL Carbon
- Tech-Etch

# Overview Cont'd

The interagency agreement covering this work was drafted in FY07, not approved and funded until October 2007 (FY08). The work being done under this project was subdivided into three phases, each corresponding to a fiscal year (FY07-FY09). Progress reported can be referenced against the FY07 and FY08 expectations as defined in the interagency agreement.

# Objectives

## **Metrology for Fuel Cell Manufacturing** Subprojects.

- **P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance** (FY08 DOE Funding, Completion FY08)

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

- **P2 Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control.** (FY08-FY09 DOE Funding)

**Objective:** 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.

- **P3 Optical Scatterfield Metrology for Online Catalyst Coating Inspection of PEM (Fuel Cell) Soft Goods.** (FY08 NIST Funded, FY09 DOE Funded)

**Objective:** Evaluate the suitability of Optical Scatterfield Metrology as a viable measurement tool for in-situ process control of catalyst coatings

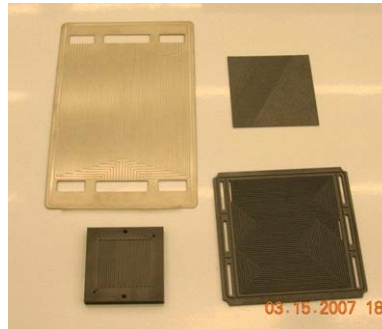
# Approach / Milestones

## (P1) Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance

- Using a reference single cell (PEM) fuel cell, fabricate as series of replacement cathode flow field plates with dimensional perturbations that simulate potential geometric variability attributed to the fabrication techniques currently used in industry and evaluate the impact of these variabilities by performance testing (polarization curve measurements).
  - Select single cell reference design (2005)
    - Teledyne CH50 and soft goods as specified in the USFCC Single Cell Testing Protocol, 2004*
  - Determine dimensional parameters to vary (2005)

### Evaluate Samples from Common Industry Mfg Methods, 2004

- Injection molded (carbon)
- Compression molded (carbon and metal)
- Machining (carbon)
- Chemical Etching (metal)



### Dimensional Parameters Chosen, 2005

- Vertical Channel Sidewall Taper
- Horizontal Channel Sidewall Straightness
- Horizontal Channel Variation-in-Width
- Horizontal Channel Bottom Straightness

- Design  $2^{4-1}$  factorial experiment with replica center point (2006)

See Slide #9

# Approach / Milestones Cont'd

## (P1) Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance

- **Develop single cell testing capability and validate**
  - *2006-2008*
- **Fabricate replica cathode plate and validate performance versus reference**
  - *FY07-FY08*
- **Fabricate experimental plates, dimensionally quantify perturbations, conduct performance test**
  - *FY08*
- **Publish results**
  - *FY08*

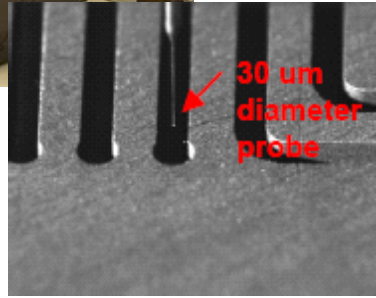
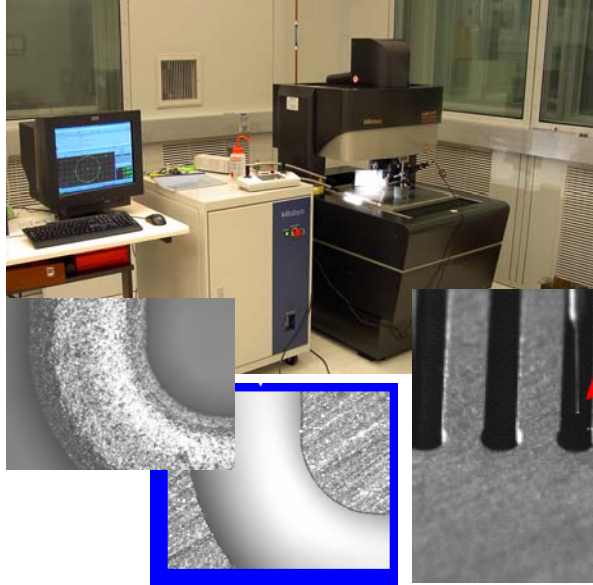
# Accomplishments

(P1) Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance

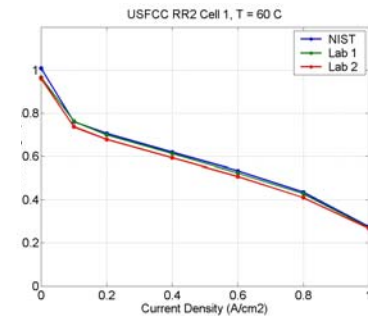
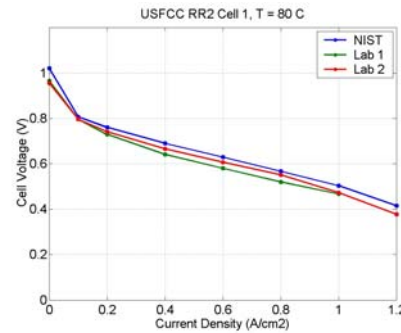
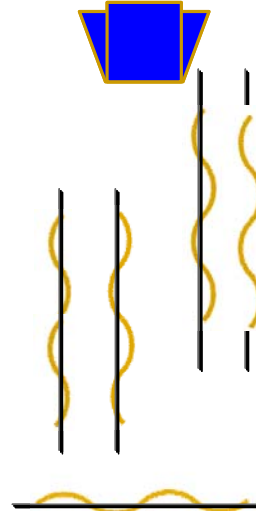
- **Single Cell Testing Capability - VALIDATED**
  - USFCC Single Cell Testing Task Force RR#2 – **COMPLETED**
  - USFCC Joint Hydrogen Quality Task Force (JHQTF) LANL Single Cell Design, Single Cell RR – **IN PROCESS**
- **NIST Facsimile Cathode Flow Field Plate**
  - Plate fabrication and performance verification – **SUCCESSFUL**
- **NIST Experimental Flow Field Plates**
  - Fabrication - **COMPLETED**

# Accomplishments Cont'd

## (P1) Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance



Channel Experimental Parameters





# Accomplishments Cont'd

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

2 <sup>4-1</sup> Fractional Factorial Design with replicated center point (k=4,n=10) (donmez.xls)								
	Sidewall Stra	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	

# Future Work

## (P1) Cause-and-Effect: Flow Field Plate Manufacturing Variability and it's Impact on Performance

- **Complete 2<sup>nd</sup> Validation JHQTF RR (6/08)**
- **Demonstrate Success Soft Good Substitution (6/08)**
  - **Go/No-Go**: Proceed with USFCC soft good recipe while working out soft good substitution issues
- **Dimensionally Verify all Experimental Plates (6/08)**
- **Complete Performance Testing with Experimental Plates (9/08)**
- **Publish Results (10/08)**

# Approach / Milestones

## (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control

Identify, procure, and evaluate potential commercially available non contact sensors capable of measuring topography changes of surfaces ranging from reflective (metallic) to non-reflective (carbon composite).

### Phase 1

- Conduct market research to identify 2 to 3 of the most promising technologies (2007)
- Procure and interface sensors (2007)

### Phase 2

- Test sensitivity to test surface reflectivity and incident angle (FY08)
- Test linearity of sensor as a function of surface reflectivity and incident angle (FY08)

### Phase 3

- Test probes using three-axis machine tool as test-bed to evaluate scanning capability in two-dimensional operation (FY08-09)
  - Evaluate scan direction feature dimensional accuracy (i.e. channel width) as a function of scan speed and sensor sampling rate.
  - Develop software algorithm to combine data into a quasi 3-D surface map
  - Evaluate overall feature measurement uncertainty versus reference metrology measurements.

# Accomplishments

## (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control

- **Market Research and Procurement** – Completed

- **Spot Laser Triangulation Probe**

- **Procured**

- Tech Note: 30 um spot, 2 ms/pt scan rate

- **Conoscopic (Holographic) Probe**

- **Procured**

- Tech Note: 30 um Spot size, 0.3 ms/pt scan rate

- **Line Laser Triangulation Probe**

- Recent consideration

- 30 um line width and 25 mm line length

- **Sensor Interfacing** – **Completed**



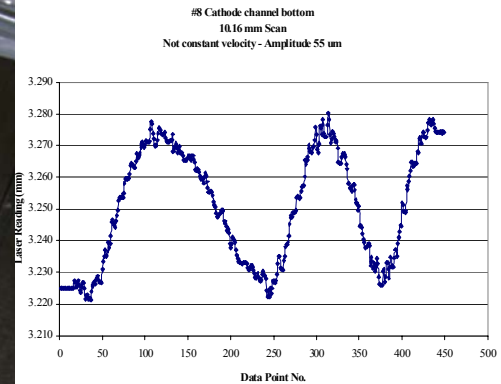
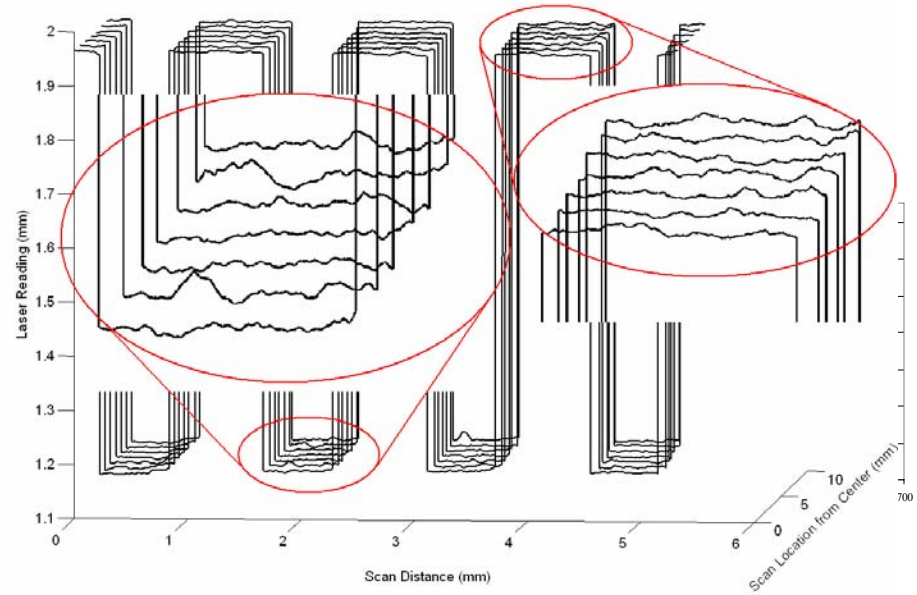
## Accomplishments Cont'd

### (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control

- **Probe material sensitivity and linearity testing (Phase 2)**
  - **Spot Laser Triangulation Probe**
    - Phase 2 testing - **Completed**
    - Tech Note: Automatic gain adjustment feature provided excellent results on shiny and dull surfaces, incident angles up to 60° with virtually no change in linearity.
  - **Conoscopic Probe**
    - Phase 2 Preliminary Testing – **Partially Completed**
    - Tech Note: Issue with very reflective surfaces but possibly will still work for chemically etched metallic plates (matte surface)
- **Phase 3 – Test bed Testing**
  - **Spot Laser Triangulation Probe**
    - Designed and fabricated articulating mechanism for mounting probe to machine tool – **Completed**
    - Probe evaluation on test bed - **In Process**
      - Tech Note: Very promising preliminary results

# Accomplishments Cont'd

## (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control



# Future Work

## (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control

- **Complete Phase 2 Preliminary Testing of Conoscopic Probe (10/08)**
  - **Go/No-Go:** If it measures our sample metallic plate then we will proceed to performance evaluation on test bed.
- **Spot Laser Triangulation Probe Test bed Evaluation (FY09)**
  - Evaluate feature amplitude uncertainty
  - Evaluate scan direction dimensional accuracy (at machine scan rate of  $\sim 1$  mm/s)
  - Develop variable speed scan direction system to allow testing of sensor's accuracy (orthogonal and co-linear) versus scan rate.
  - Develop algorithm to combine data into quasi-3D surface map (combining of two direction scan data)
- **Conoscopic Probe Test bed Evaluation (Phase 2 Conditional FY09)**
  - Same details as above.

# Future Work Cont'd

## (P2) Non-Contact Sensor Evaluation for Bi-polar Plate Manufacturing Process Control

- **Develop a Calibration Target (FY09)**
  - More durable material with similar optical properties and features of flow field plates being inspected (calibration check standard)
  - Needed to establish traceability (common contractual requirement)
- **Other Alternative Technologies (FY09+?)**
  - Line Scanning Probe (faster)
  - Close Range Photogrammetry (fastest)



# Approach / Milestones

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

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

### **Phase 1 (FY08)**

- Obtain commercially available catalyst coated samples on various substrates (GDE cloth, GDE paper, CCM) for evaluation using the SEM to identify potential morphology differences that could cause variability in the OSM measurements.
- Interview and solicit manufacturers to identify critical parameters and to obtain sample packages that vary these parameters for a detail sensitivity study using OSM.

### **Phase 2 (FY08-09)**

- Conduct sensitivity studies using sample packages and OSM
  - Develop models to optimize sensitivity.

### **Phase 3 (FY08-09) Go/No-Go Point**

- Accurately characterize sample package parameters to calibrate sensitivity of OSM measurements.

### **Phase 4 (FY09+)**

- Conduct inline measure studies (accuracy sensitivity versus scanning speed) and develop statistical sampling strategies.

# Accomplishments

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

- **Sample Morphology Study**

- **Sample Types**

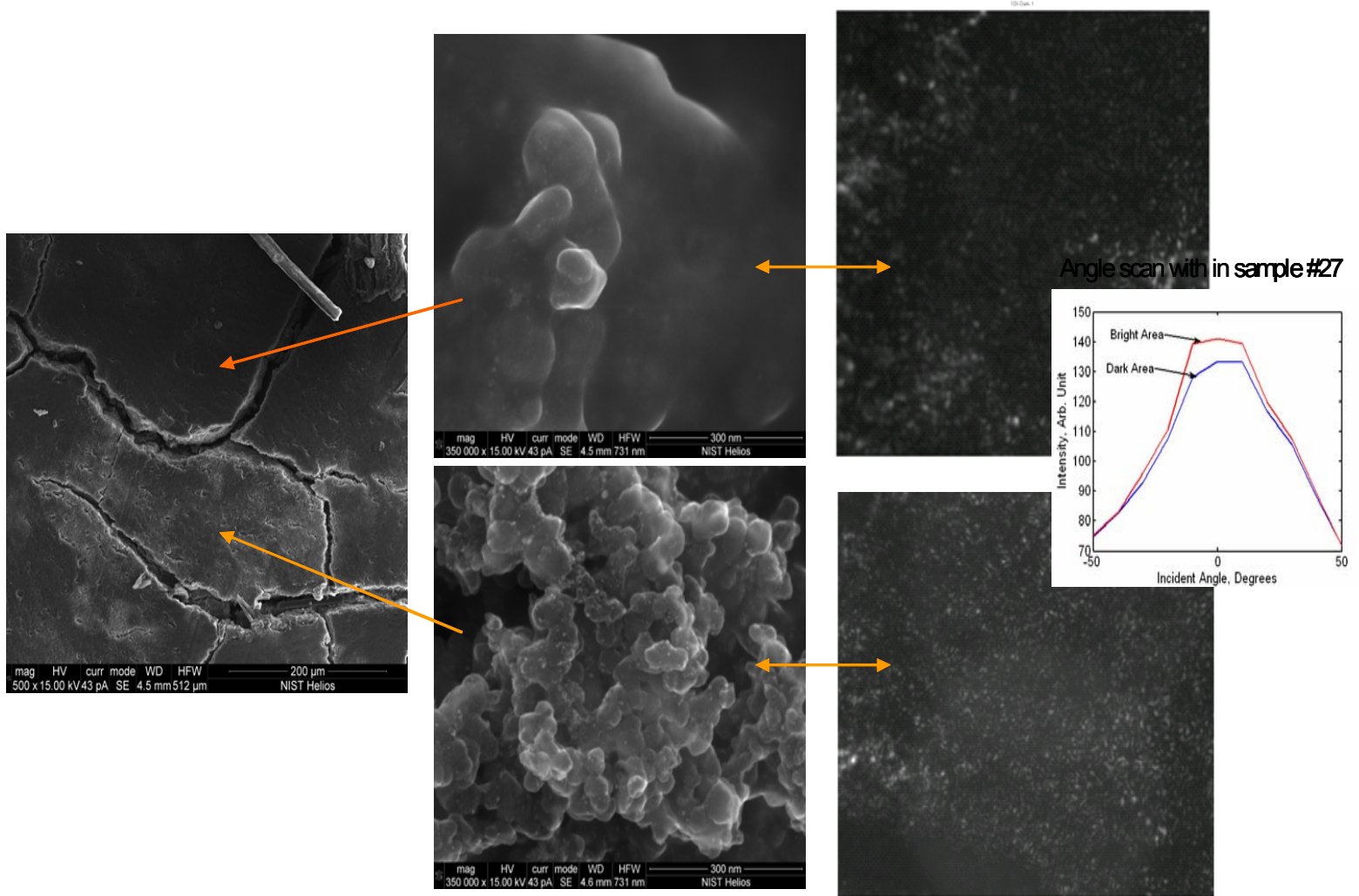
- Woven Cloth GDE – Very Poor
- CCM – Much better
- Carbon Paper GDE – Not obtained yet

- **Conclusions Derived**

- OSM method parameter sensitivity testing to be done with CCM's
- Manufacturer input – no one sample type dominates
- If OSM proves useful
  - develop models and re-engineer illumination field to deal with variability of less than optimal samples (cloth GDE)
  - suggest “design for metrology” versus “design for manufacture” to promote CCM as preference

# Accomplishments Cont'd

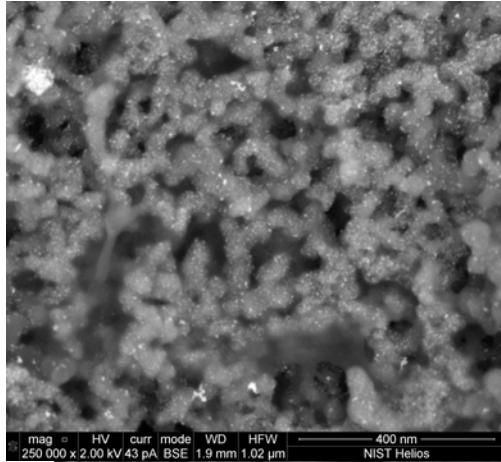
## (P3) Optical Scatterfield Metrology (OSM) for Online Catalyst Coating Inspection of PEM (Fuel Cell) Soft Goods



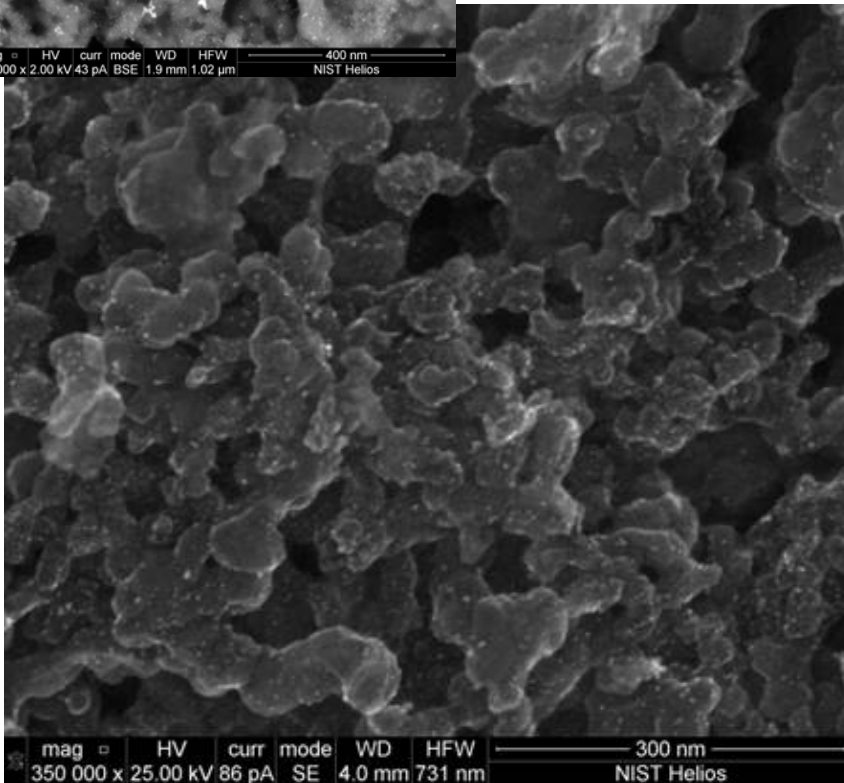
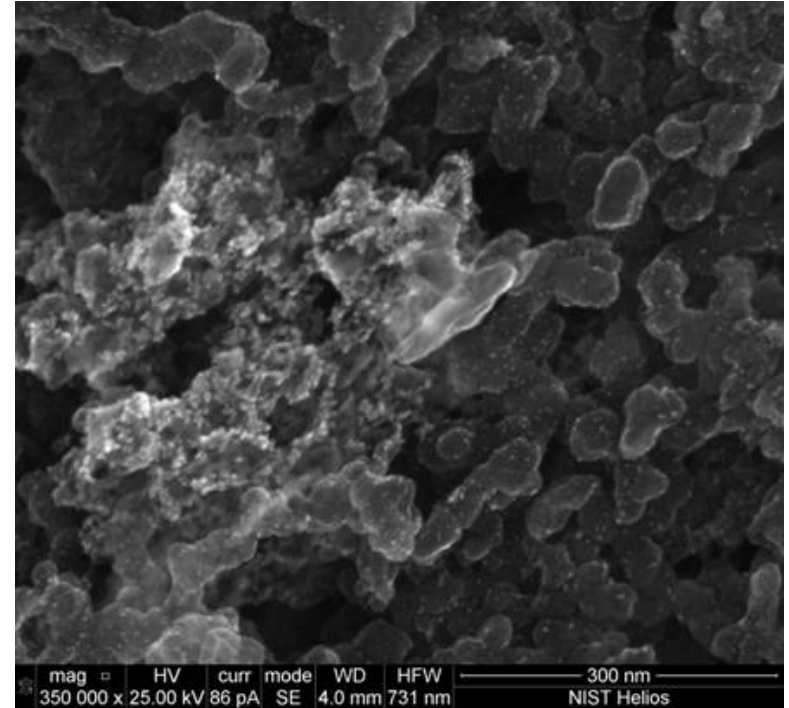
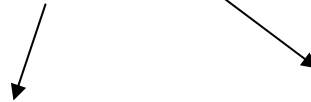
First three pictures (from left) are SEM images showing variability of cloth GDE.....last two pictures are OSM images and simple intensity measurement

# Accomplishments Cont'd

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



Standard  
secondary electron  
images



All SEM images of CCM sample...bottom left - typical homogeneous sample image found over most of the sample area...top right - unusual platinum conglomeration...top left - backscattered electron image enhances platinum particle contrast

# Future Work

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

- **Resolve NDA terms with manufacturers (5/08)**
- **Complete Sample Morphology Study (6/08)**
  - Procure and image carbon paper based GDE
- **Work with manufacturers on sample package composition (6/08)**
  - Different parameters than HNEI
- **Receive and Evaluate Sample Package from HNEI (Guido Bender)**

Sample Package Includes:

  - 5 Samples, 50% Pt/C, 5 Different Loadings ( $\sim 0.05 \text{ mg/cm}^2 - 0.50 \text{ mg/cm}^2$ )
  - 3 Samples, 20 % Pt/C, 3 Different Loadings ( $\sim 0.10 \text{ mg/cm}^2 - 0.50 \text{ mg/cm}^2$ )
  - 1 Carbon Black Sample, Nafion with no Pt
  - **Go/No Go:** Use samples to determine is OSM shows sensitivity to platinum loading and if the weight percent of Pt/C has any influence.(9/08)
- **Evaluate OSM sensitivity to test sample package parameters supplied by manufacturers (9/08)**
  - **Go/No Go:** Based on all results of sample packages and review of these results with suppliers, do we continue R&D of the technique or stop (FY09).