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# Reduction in Fabrication Costs of Gas Diffusion Layers

**Jason Morgan**

**Ballard Material Products**
**2 Industrial Avenue, Lowell, MA**

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Project ID # MN002

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# Project Overview

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## Timeline

- Start date: Sept. 1, 2008
- End date: Aug. 31, 2011
- **85 % complete**

## Barriers

- A.) Lack of high-volume membrane electrode assembly (MEA) processes
- F.) Low levels of quality control and inflexible processes

## Budget

- Total project funding:
  - DOE share: $3,000,000
  - Contractor share: $1,617,949
- Fed. funding received in FY10: $250,000
- Fed. funding for FY11: $743,926

## Partners

- The Pennsylvania State University – Dr. Mike Hickner
- Ballard Power Systems
- Ballard Material Products - Prime

# Project Relevance (1)

- **This project addresses the following 2 DOE Technical Barriers:**
  - **A) Lack of high-volume membrane electrode assembly (MEA) processes:**
    - The process modifications introduced in this project, coupled with additional Ballard investments, have increased GDL production volumes nearly 4 fold from the beginning of the program to date
  - **F) Low levels of quality control and inflexible processes:**
    - Ballard has introduced new quality control technologies such as mass flow meters to control MPL loadings, providing more uniform properties and reducing the amount of ex-situ testing required
    - Ballard has added an in-line visual inspection station as a final quality tool improving processing efficiency and accuracy by eliminating slow manual inspection and operator determination of a defect
    - This project reduces the manufacturing costs associated with state-of-the-art GDLs by eliminating process steps, improving production yields, reducing scrap and increasing production efficiency, while also creating a modular production process that can be easily modified for different production designs and scaled up to meet future production needs

# Project Relevance (2)

- **Objectives of Program are to reduce GDL fabrication costs by:**
  - Utilization of continuous mixing equipment for GDL fabrication process
    - Address technical barriers associated with continuous mixing
    - Manufacture sample inks within production specifications
    - Coat in-line inks on standard material
  - Successfully use (Many-At-A-Time) MAAT coating process to manufacture GDLs
    - Address technical barriers associated with MAAT coating
    - Coat standard GDL material, with standard inks, using MAAT coating equipment
  - Integrate continuous mixing and MAAT coating processes
  - Introduce on-line process control tools to improve product quality and reduce ex-situ testing requirements
  - Reduce GDL fabrication costs by improving manufacturing efficiency, improving process yields and reducing scrap rates
  - Determine relationships between process parameters and key GDL properties

# Project Approach (1)

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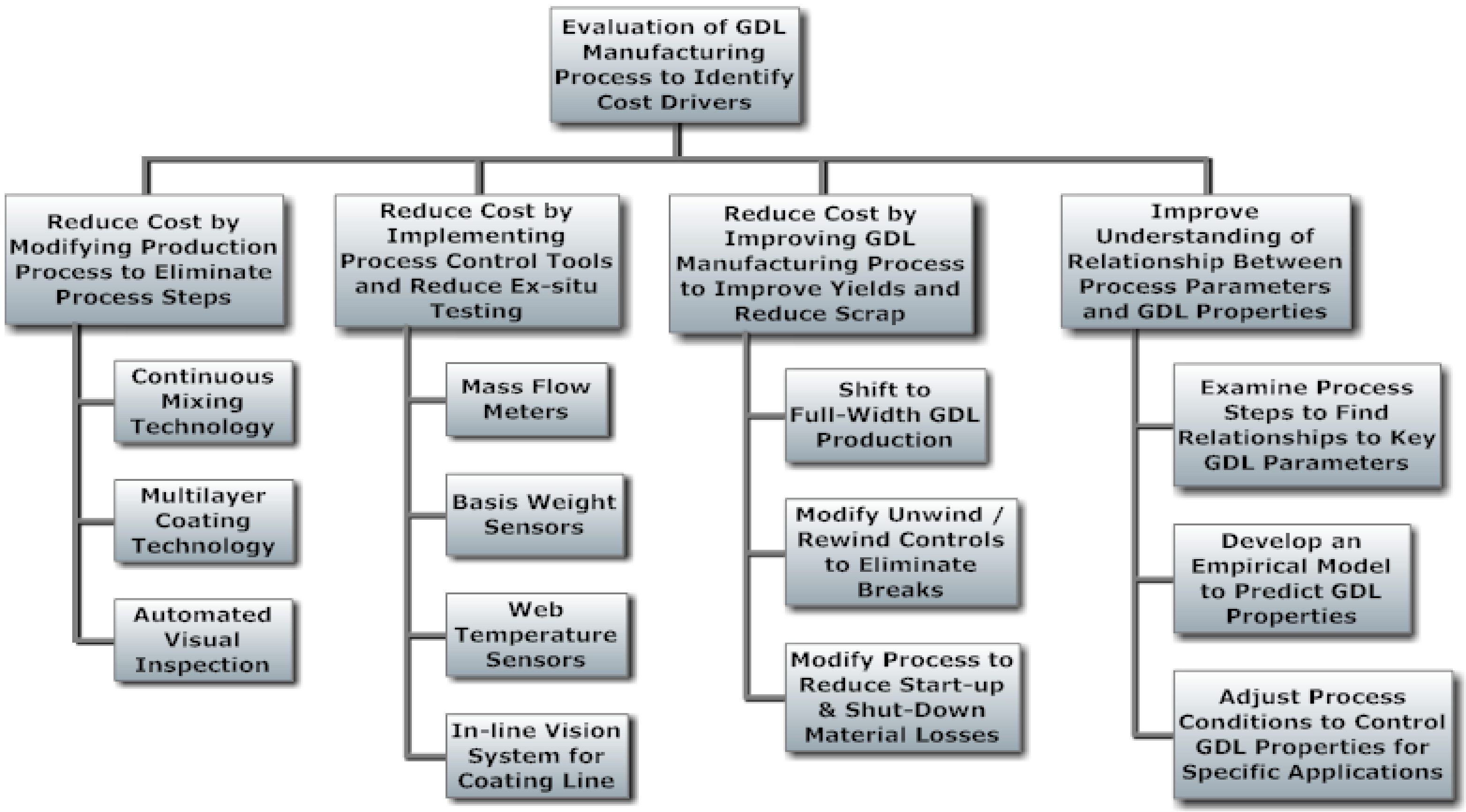


**Evaluating state-of-the-art GDL manufacturing processes allows for process improvements to reduce manufacturing cost at current production volumes and design next generation equipment to reach DOE target costs at higher volumes**

# Project Approach (2)

| Date Completed | Milestone / Deliverable | Measure of Success |
|---|---|---|
| Mar-10 | Installation of In-Line Mixing Equipment Complete | Inks manufactured to production specifications, initial problems associated with mixing were addressed |
| Mar-10 | Installation of MAAT Coating Equipment Complete | MPLs were successfully MAAT coated, problems such as micro-cracking were eliminated |
| Jun-10 | Evaluation of GDL made with MAAT/In-Line Mixing Technologies (Non-Integrated) Completed | Measurement of ex-situ properties of GDLs manufactured with MAAT or In-Line Mixing technologies was completed. Single Cell testing was also completed and compared to baseline GDL material for a variety of operating conditions |
| Nov-10 | MAAT and In-Line Mixing Technologies Fully Integrated | Successful demonstration of MAAT coating using inks produced with In-Line mixing technologies, some issues noted to be addressed |
| Mar-11 | Ex-Situ Evaluation of GDL made with MAAT/In-Line Mixing Technologies Completed | 4 Rolls of GDL material (2 anode/2 cathode) were manufactured using the integrated new technologies. Ex-situ testing was completed and showed that the GDL properties were within production specification limits |
| Projected: Jun-11 | In-Situ Short Stack Evaluation of GDL made with MAAT/In-Line Mixing Technologies Completed | In-situ testing will be completed (both single cell and short stack testing) and compared with results from baseline GDL |
| Projected: Sept-11 | Design of Greenfield Facility Complete | Design of a facility capable of producing GDLs at volumes & cost consistent with DOE 2015 targets |
| Projected: Sept-11 | In-Situ Full Stack Evaluation of GDL made with Integrated Technologies Completed | In-situ testing will be completed (full stack) and compared with results from baseline GDL |

# Project Approach (3)

- **What GDL(s) should be used for the baseline in this project?**
  - Anode & Cathode designs were selected from a real near-term fuel cell application (materials handling)
  - Separate designs for anode and cathode allow for verification of continuous mixing, MAAT coating and on-line tools with different components (GDL substrates, ink designs, loadings, etc.)
  - High volume design that is currently in production
    - Large amount of baseline data to capture true manufacturing variability
    - Real production design makes product specifications representative of true manufacturing needs
    - Designs in this program have been proven with more than 5,000 $m^2$ of each GDL design sold last year

**Sample of Production Cathode Roll**

**Sample of Typical Production Rolls**

# Technical Accomplishments and Progress (1)

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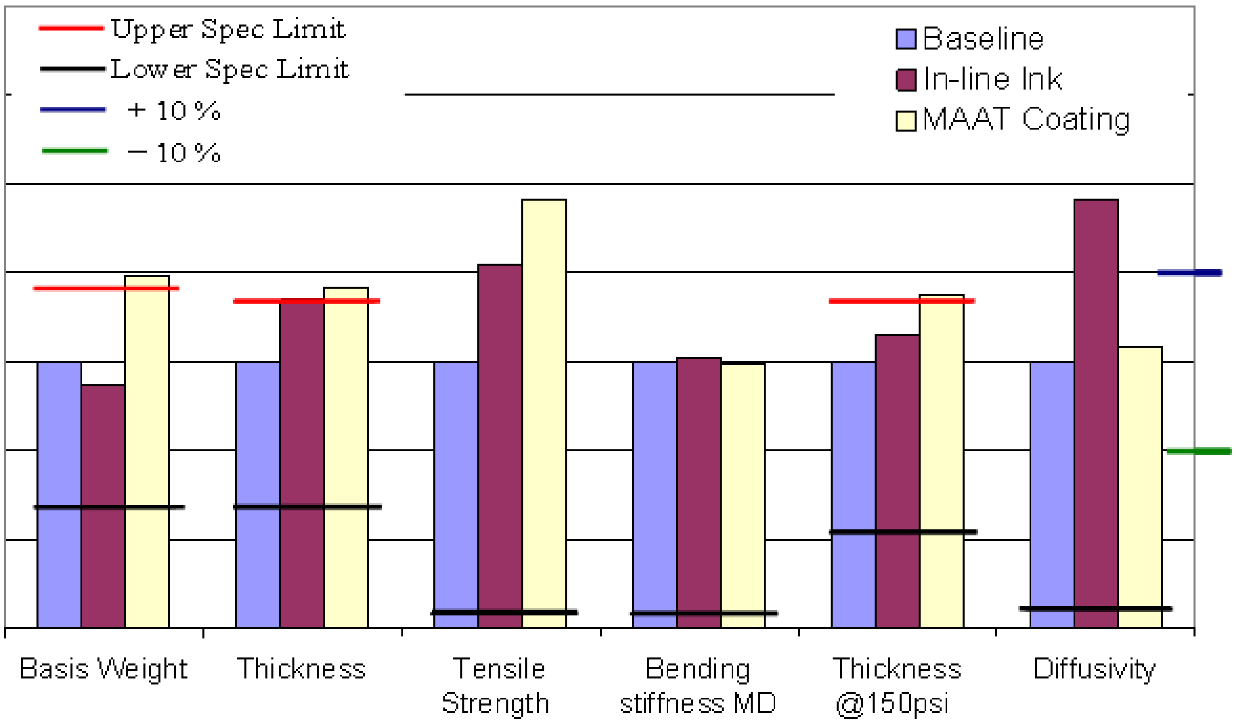
- **Validation of continuous mixing and MAAT coating processes**
  - Results for baseline GDL are from averages of many lots of commercial GDL
  - Results for In-Line Mixing & MAAT coating are from 40m rolls with samples every ~2m
  - **Note that all property values have been "normalized" to the baseline value**

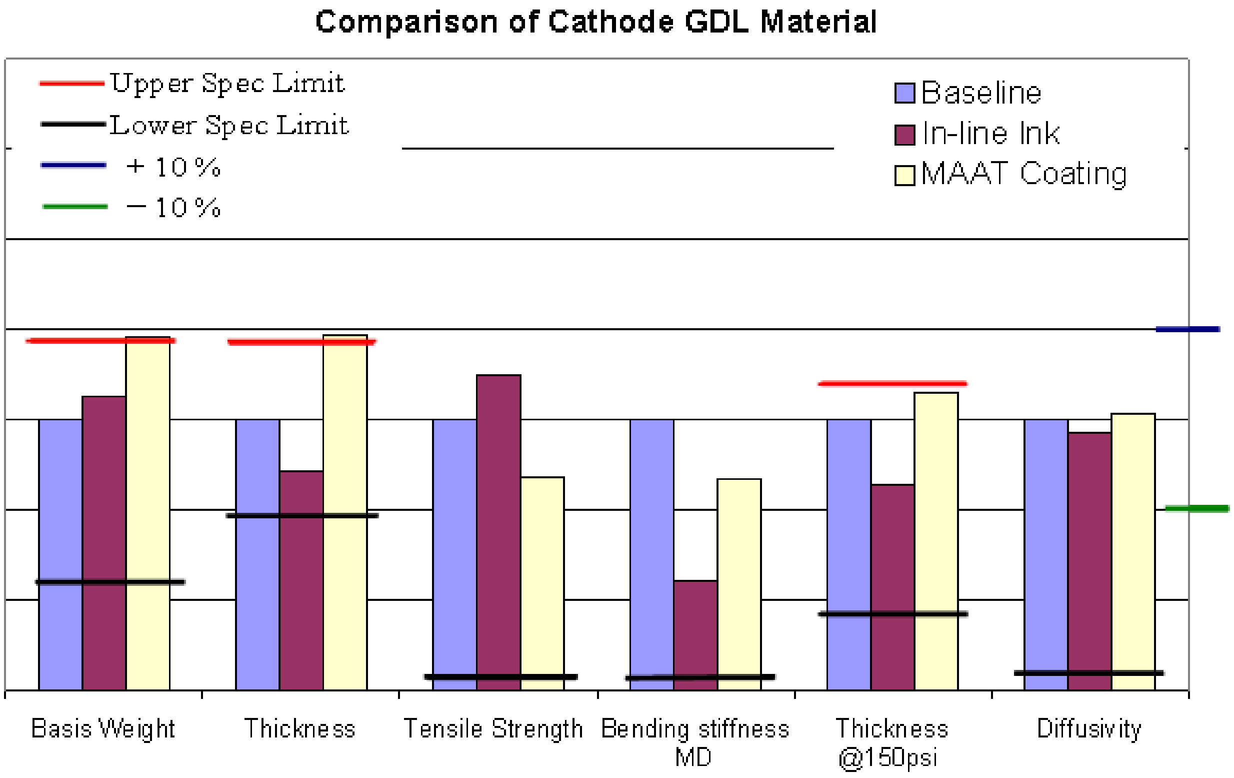
**Comparison of Anode GDL Material**




- **Properties for the anode roll are mostly within production specifications as shown**
  - Thickness and BW of MAAT anode are high due to inaccurate feed rates
  - Tensile strength, bending stiffness and diffusivity are all well-above the required values
  - Thickness at 150 psi is above the specification limit for the MAAT anode, but this is attributed to the higher MPL coatings due to inaccurate feed rates

# Technical Accomplishments and Progress (2)

- **Validation of continuous mixing and MAAT coating processes**
  - Results for baseline GDL are from averages of many lots of commercial GDL
  - Results for In-Line Mixing & MAAT coating are from 40m rolls with samples every ~2m
  - **Note that all property values have been "normalized" to the baseline value**




- **Properties for the cathode roll are mostly within production specifications as shown**
  - Thickness and BW of MAAT cathode are high due to inaccurate feed rates
  - Bending stiffness of the In-Line Ink is above the required specification, but is below the baseline value. This is not believed to be related to the inks.
  - Tensile strength and diffusivity are well above required values

# Technical Accomplishments and Progress (3)

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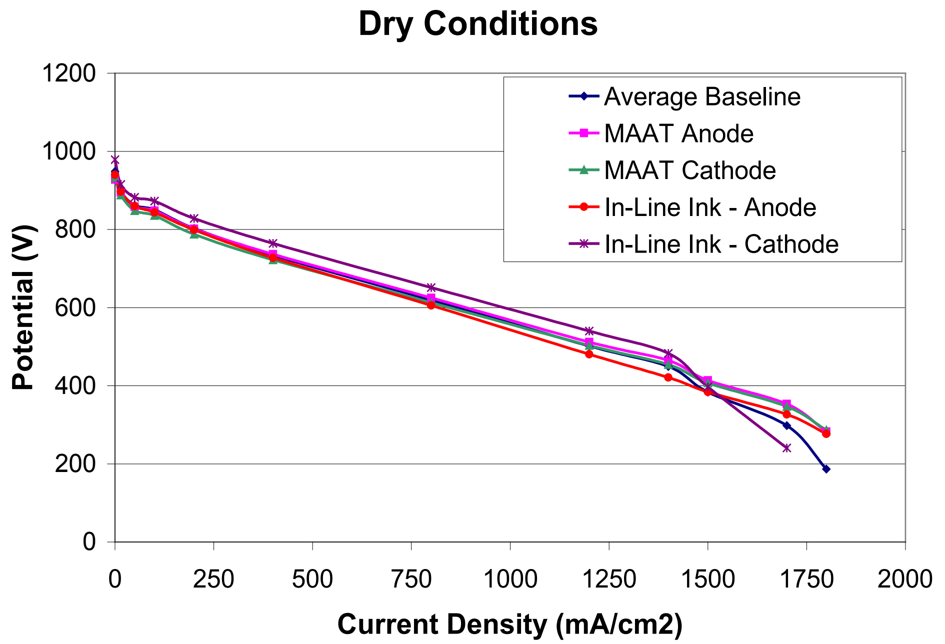
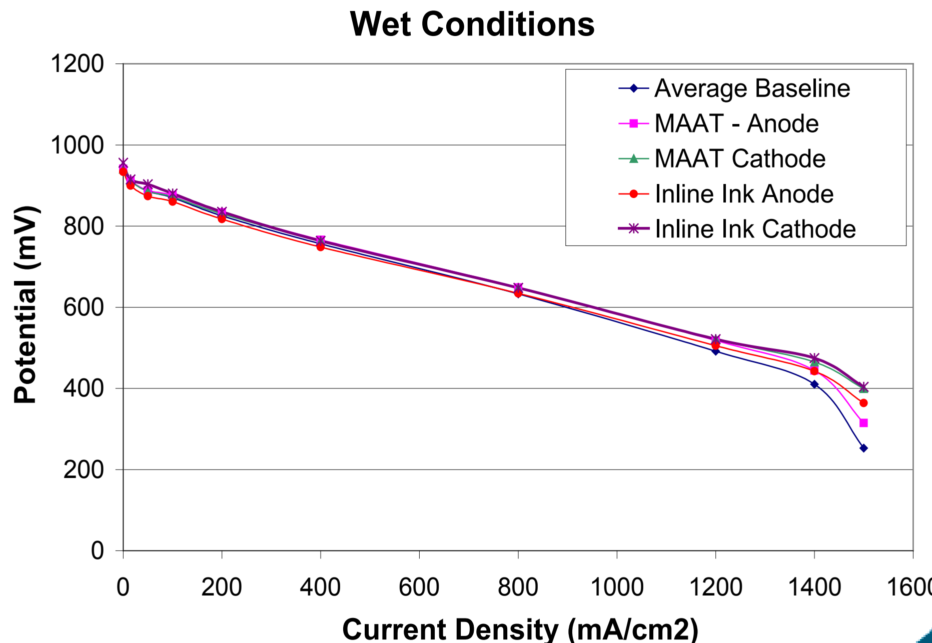
- **Validation of continuous mixing & MAAT coating processes in a single cell under dry conditions**
  - Results are averaged for at least 3 separate tests for each curve (both up and down polarizations)
  - Results showed minimal voltage difference (<10 mV at 1 A/cm$^2$) for these conditions




- **Validation of continuous mixing & MAAT coating processes in a single cell under wet conditions**
  - Results are averaged for at least 3 separate tests for each curve (both up and down polarizations)
  - Results show no adverse affect on GDL performance due to new technologies

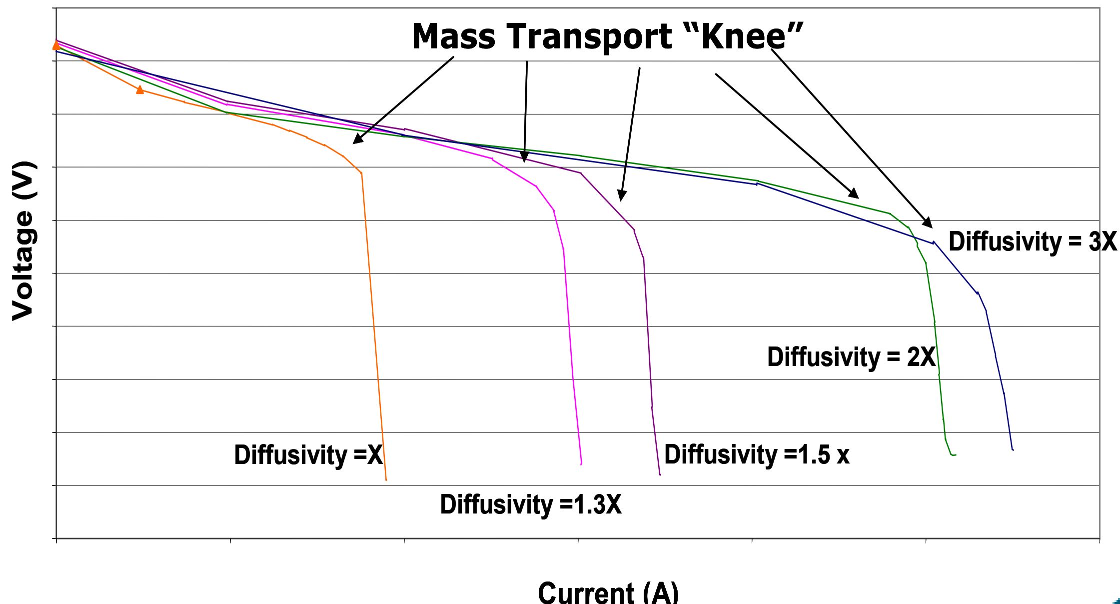


# Technical Accomplishments and Progress (4)

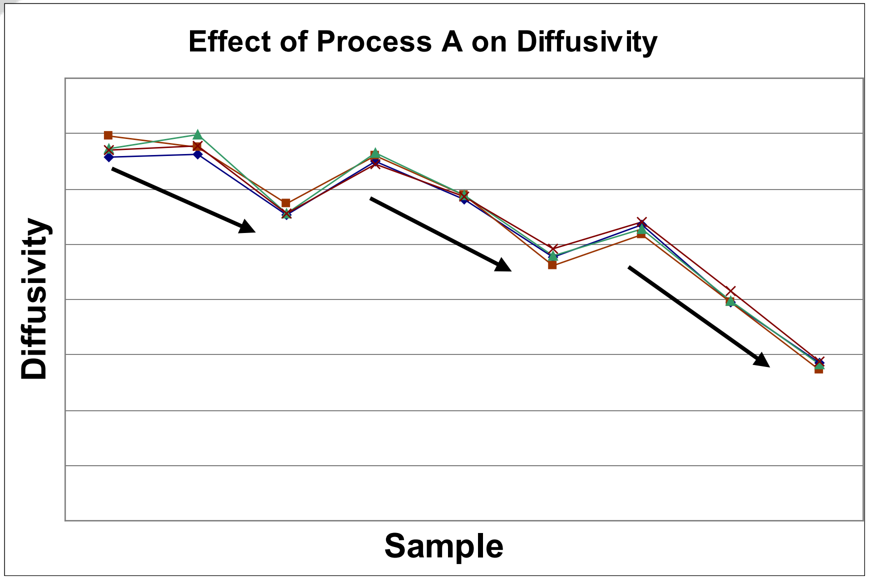
| Property | Impact |
|---|---|
| Diffusivity | Critical for controlling water content in the GDL |
| Thickness | Required for proper sealing in the MEA |
| Surface Roughness | Could influence membrane durability |
| MPL Loading | Controls the pore structure at the CCM/CL interface |
| Permeability | Relates to water management in a stack |
| Basis Weight | Impacts GDL durability, thickness and cell performance |
| Tensile Strength | Minimum value is required for continuous processing |
| Flexural Stiffness | Indicates the potential of GDL deflection into the channel |

- **Critical GDL Properties**
  - These properties can be influenced during the GDL manufacturing process
  - Significant effort has been made to relate these properties to specific process steps and operation parameters

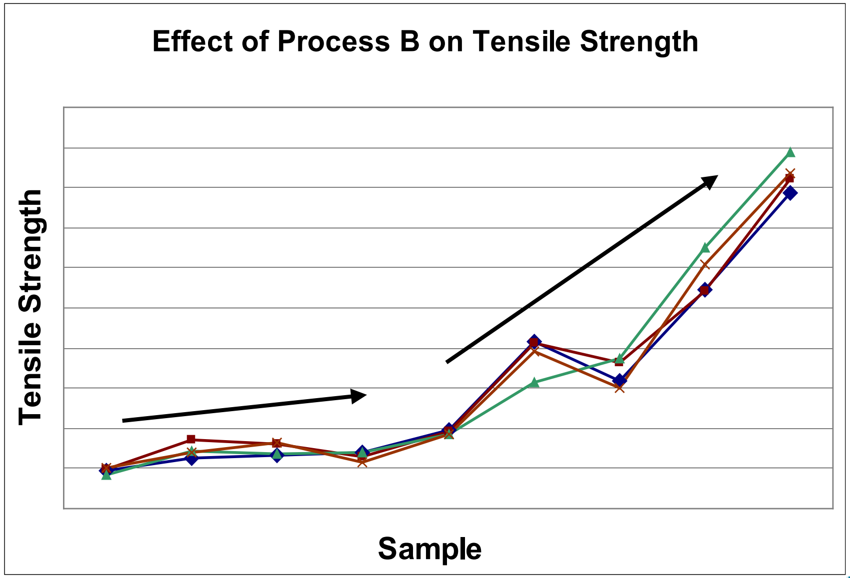
- **Cell Performance as a Function of GDL Diffusivity**
  - Diffusivity is measured using a proprietary Ballard measurement tool
  - The GDL can have a significant impact on the performance of a fuel cell
  - The optimal GDL diffusivity is determined by the operating conditions, hardware and other components used in the system



# Technical Accomplishments and Progress (5)



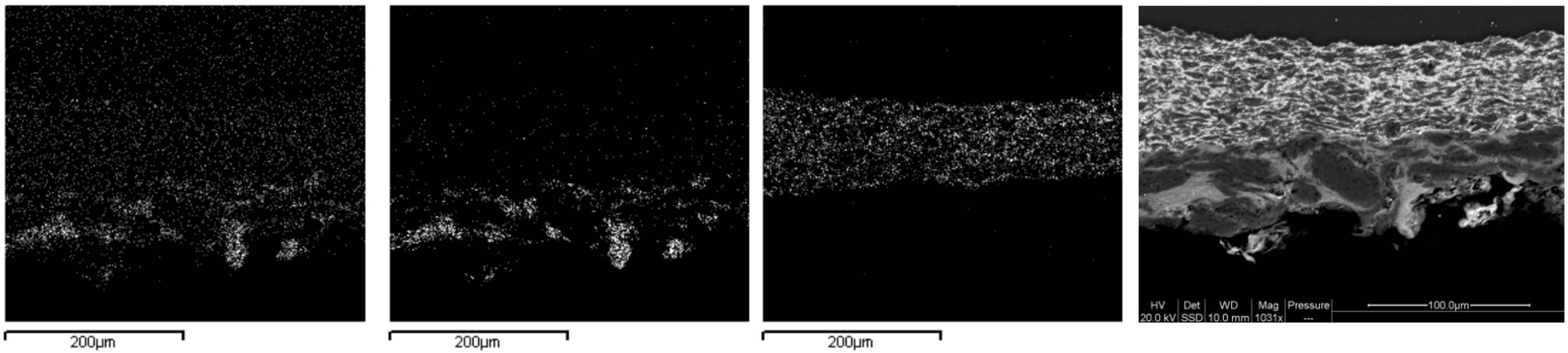

- **Process A was identified as the main lever for controlling GDL diffusivity**
- **From these data, an empirical model was developed to predict diffusivity, allowing BMP to manufacture GDLs engineered for optimum performance in specific applications**



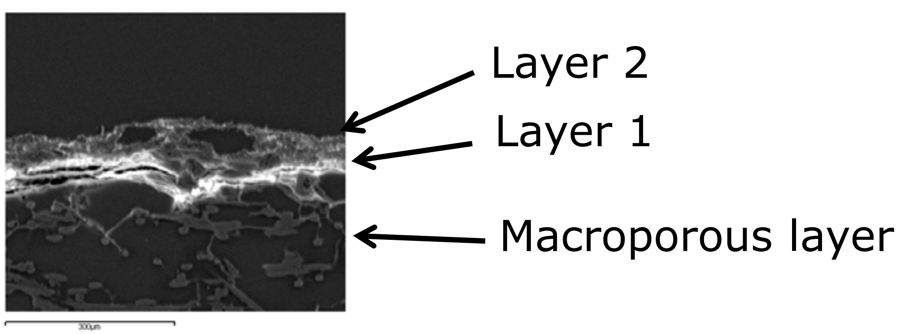

- **Process B was identified as a strong influence on GDL Tensile Strength**
- **Analysis was done to understand the trade-offs of modifying the GDL design (i.e. higher diffusivity = lower tensile strength, etc.)**
- **Additional correlations were found for: Electrical Resistivity, Thermal Conductivity, Basis Weight, Thickness, and Permeability**

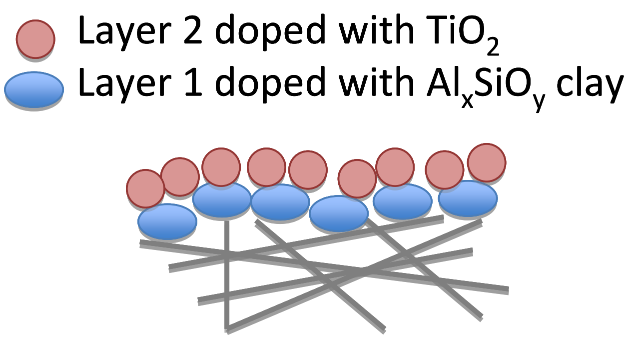
# Technical Accomplishments and Progress (6)

High contrast doping of multilayered samples provides key information on interlayer mixing during coating processes.




Al EDS | Si EDS | Ti EDS | SEM



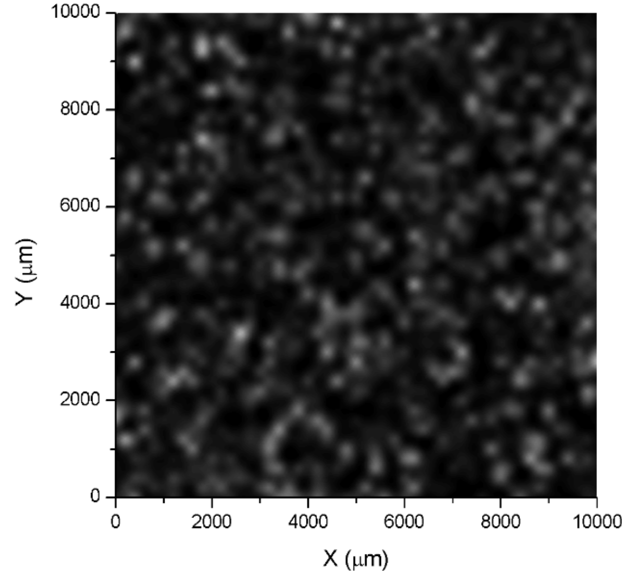



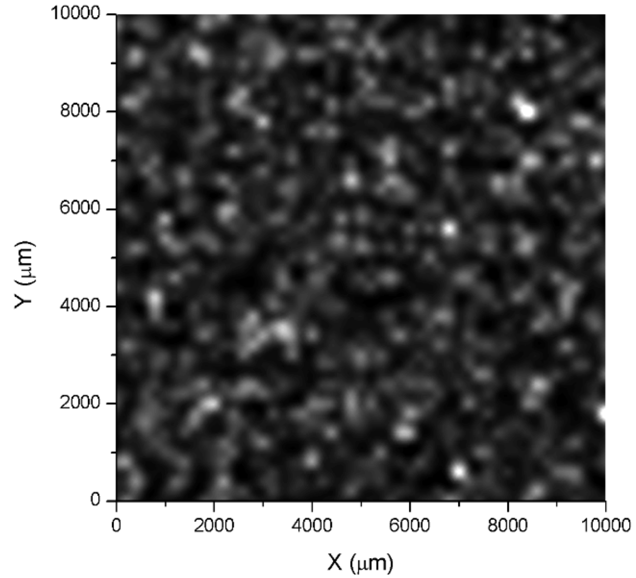
# Technical Accomplishments and Progress (7)

## Raman scanning for 2D resolution of PTFE and carbon distribution

Mendoza, Hickner, Morgan, Rutter, Legzdins *Fuel Cells* **2011**.

10 mm x 10 mm Raman maps collected to measure size and distribution of PTFE clusters. PTFE clusters shown in lighter colors.




| | S1 - left | S2 - right |
|---|---|---|
| Average size (mm) | 0.040 | 0.100 |
| Area fraction of PTFE | 0.63 | 0.70 |
| # islands | 95 | 108 |

Areal scanning shows that sample on the right has more, higher intensity PTFE regions on the surface of the sample. We've correlated these changes to specific processing conditions.

# Technical Accomplishments and Progress (8)

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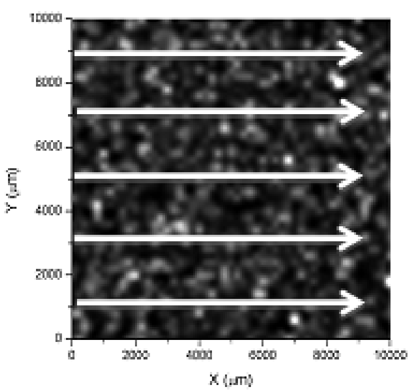
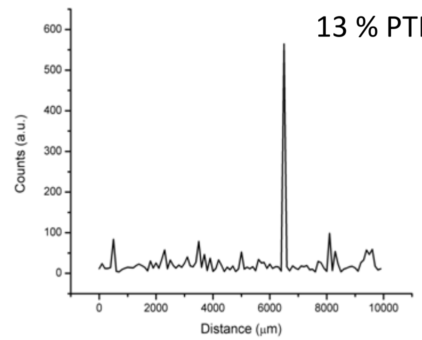
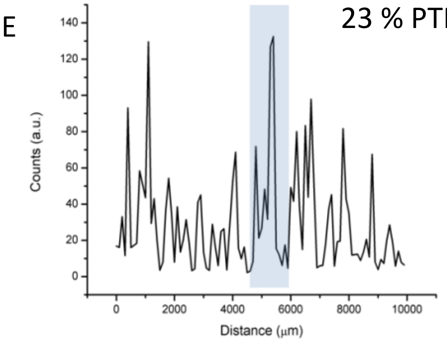
## Analysis of line-scanning data shows potential of Raman as an on-line technique

Line scans gathered to record Raman spectra vs distance.

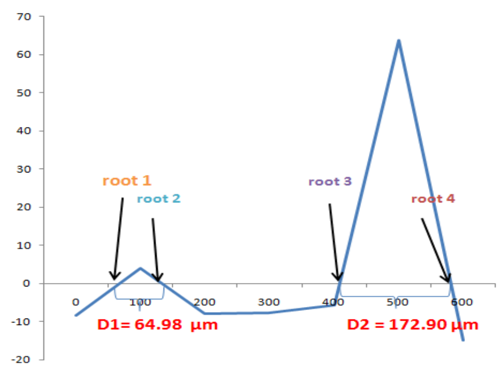



Raman peaks at 734 $cm^{-1}$ (PTFE) used for intensity vs distance analysis to determine characteristic PTFE sizes and frequency in a linear scan (similar to down-web scanning).




Peak widths and intensities are extracted from the line scanning data to represent the characteristic sizes and intensities of the PTFE features.




Average peak area extracted from line scans gives similar information to the 2D PTFE area analysis. Demonstrates potential that 2D data (slow, not-online technique) and 1D data (faster, possible application to downweb scanning) can be correlated.

| | S1 - left | S2 - right |
|---|---|---|
| Average 734 $cm^{-1}$ peak area | 3093 | 3971 |
| Area fraction | 0.63 | 0.70 |

# Technical Accomplishments and Progress (9)

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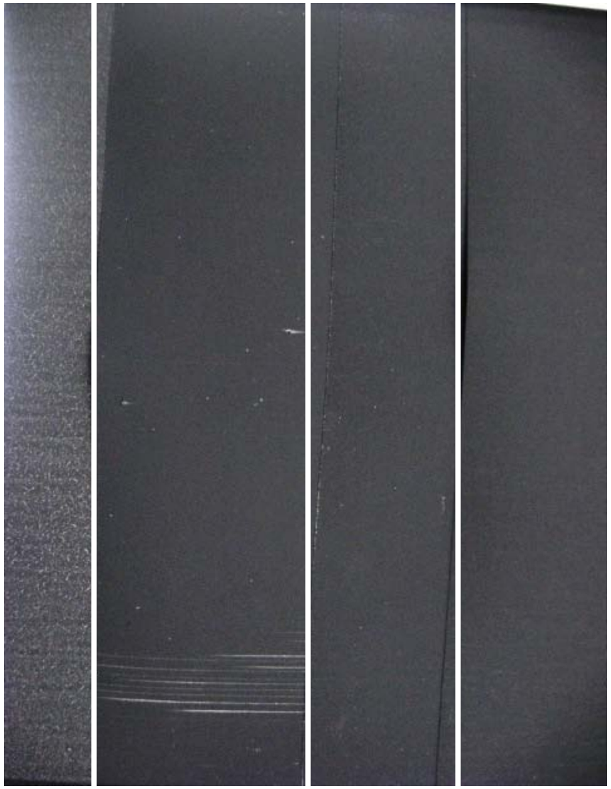
| In-line Mixing | | |
|---|---|---|
| **Tool** | **Type** | **Benefit** |
| **Mass Flow Meters** | | |
| PTFE & other liquids | Rheotherm | Accurate control of feed liquids to the mixer leading to a more uniform mix |
| DI water | Flocat - Diff. Press. | Accurate control of DI water flow into the mixer improving mix uniformity |
| **Gravimetric Feeders** | Schenck Accurate | Accurate feed of dry materials to in-line mixer to produce a more uniform mix with accurate solids % |
| **Rheometer** | Brookfield | Measures viscoelastic parameters of fluid - can give insight into causes of shelf-life issues and formation of agglomerations |
| **Surface Tensiometer** | Sensydyne LV500 | Measures surface tension of high viscosity fluids. For coating multilayer coatings - top fluid must have lowest surface tension, dynamic surface tension curve gives idea of potential drying issues |

| Many-At-A-Time Coating | | |
|---|---|---|
| **Tool** | **Type** | **Benefit** |
| **Mass Flow Meters** | Micromotion Coriolis | More accurately meters solution to coating head - improves basis weight and thickness uniformity (as seen with the ex-situ analysis of trial rolls) |
| **Basis Weight Tool** | NDC | Continous measurement of coated and substrate basis weight |
| **In-line Viscometers** | Cambridge Viscosity | Continuous reading of in-line viscosity, changes could affect coat weights |
| **Non-contact IR Thermometer** | Exergen | Continuously measure web temperature throughout oven to maintain consistent drying profile. With air cooling can operate in temperatures up to 150°C |
| **High Temp Non-Contact IR Thermometer** | Raytek-FA | Continuously measure web temperature throughout oven to maintain consistent heat treatment profile. With high temperature optics and fiber optic cable can accurately read temperatures above 250°C without added cooling |
| **Chilled Mirror - Dewpoint Measurement** | Kahn Optidew | Measure dewpoint in each zone of the oven, |
| **In-line Vision System** | NI/Labview | Alert operators to coating defects that can be fixed in real-time |

# Technical Accomplishments and Progress (10)




## MAAT coating with In-line mixed inks

### Enhancements to coating line:

- Modified solutions (e.g. increase solids to reduce wet-load)
- Optimized dryer profile utilizing dew point sensors to prevent premature drying of top layer
- Due to different solution formulations – slot heights were modified to produce defect-free coating and improve cross-web basis weight uniformity
- Successfully produced physically defect free Anode and Cathode material with multilayer coating head
- Improved repeatability of basis weights by installing Micromotion flow meters for each solution

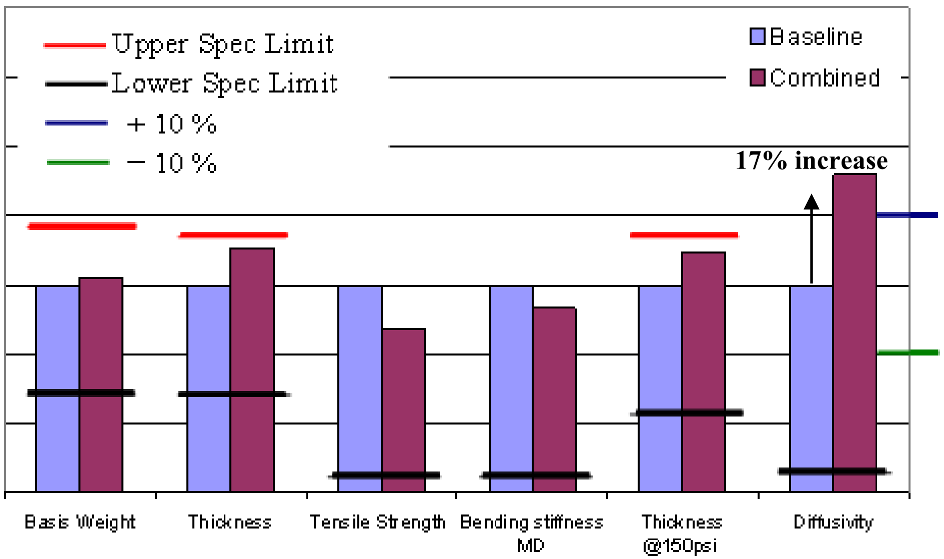
### Problems left to address:

- Small agglomerates formed in in-line ink due to solution modifications
  - Trade-off between modified solutions and mix quality
- High amount of entrained air present with in-line ink
  - Examining methods to improve de-gas technique to remove air more efficiently

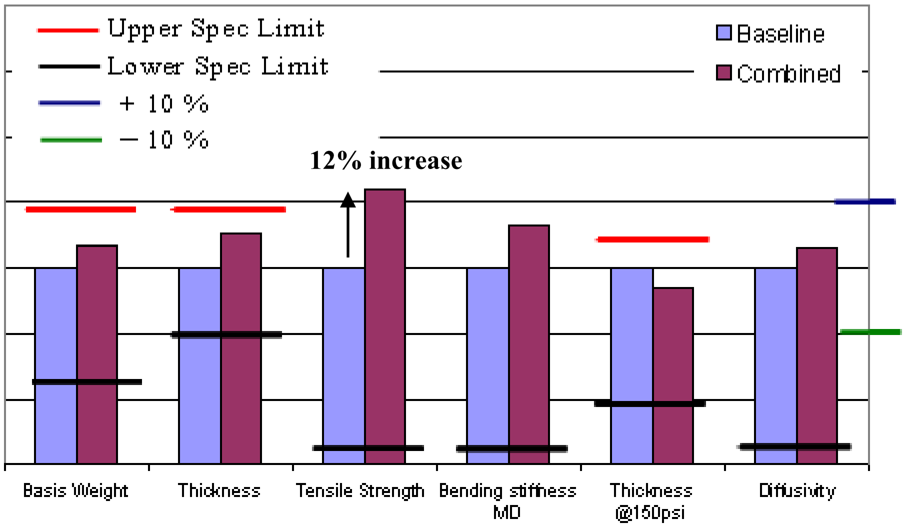
# Technical Accomplishments and Progress (11)

- **Key GDL properties were within production specification requirements and all measurements were within 10% of the baseline values, except where values were improved (Anode Diffusivity & Cathode TS)**

Comparison of Anode GDL Material



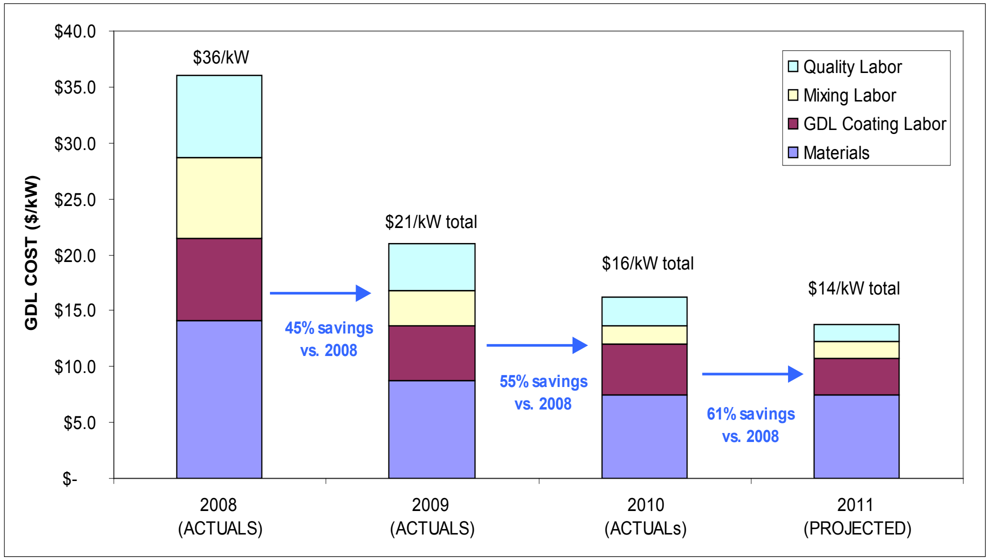

Comparison of Cathode GDL Material



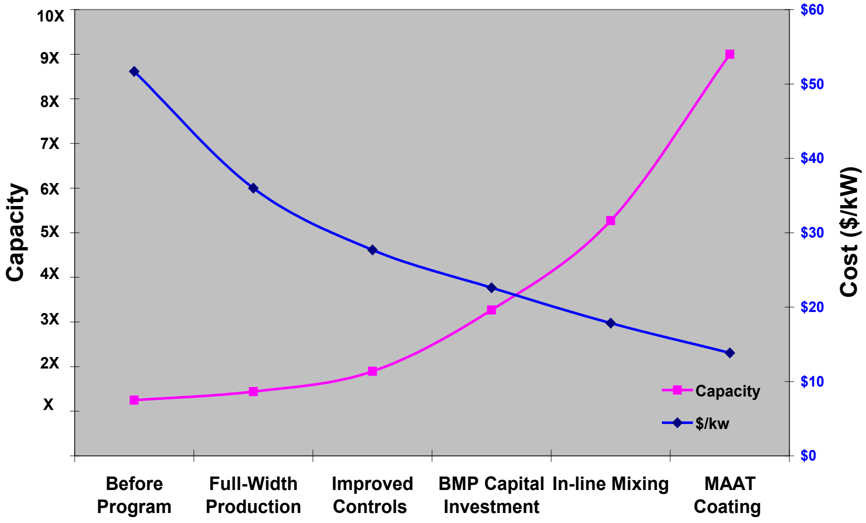

- **Improved thickness and basis weight uniformity are attributed to the mass flow meters added to the MAAT coating equipment**
- **Diffusivity was improved for both Anode & Cathode and can be attributed to improved process controls**

# Technical Accomplishments and Progress (12)

- **GDL costs have been reduced over 50% since the start of the program**
- **Demonstration has focused on specific anode & cathode designs, but are applicable to all BMP paper-based GDL designs**
- **Manufacturing capacity has been increased nearly 4 fold since the program began, with the total increase expected to reach 9 fold once continuous mixing and MAAT coating are qualified and fully implemented**




BMP's Paper Based GDL Capacity and Costs



# Collaborations (1)

- **Ballard Material Products – Prime**
- **The Pennsylvania State University – Partner**
  - Dr. Michael Hickner is leading the effort to:
    - Establish an in-line method for measuring the chemical homogeneity of GDLs
    - Develop a method to dope the carbon particles in the MPLs to better quantify the degree of intermixing
- **Ballard Power Systems – Partner**
  - The GDL R&D team is the leading the effort to:
    - Perform additional ex-situ analysis of new low-cost GDL designs and compare values to baseline material
    - Perform in-situ short stack (10 cell) testing examining:
      - Up and Down Polarization curves
      - Fuel/Air Stoich. Sensitivities
      - Water Crossover
- **NREL – Collaborator**
  - Supporting Dr. Mike Ulsh's work with materials and information about our GDL manufacturing process

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# Proposed Future Work (1)

- **Further Testing of Initial Anode/Cathode Rolls Manufactured with Integrated Technologies (In-Line Ink & MAAT Coating)**
  - In-situ testing
    - 10 cell stacks, fuel/air stoich. Sensitivities, water crossover, etc.
  - Ex-situ testing
    - MIP, TPP, IPP, Surface Roughness, etc.
- **Improvements to In-Line Mixing / MAAT based on initial results**
  - Adjustments to ink mixing to eliminate particulates in coating inks
  - Modify manufacturing process, if necessary, based on in-situ test results (e.g. increase diffusivity if flooding is seen)
- **Production of full-scale rolls of both Anode/Cathode**
  - Increased run time to screen for potential issues
  - In-situ/ex-situ testing including full stack testing
- **Design of Greenfield Facility to Reach DOE Cost Target**

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# Summary (2)

- **BMP has examined the GDL manufacturing process and improved manufacturing cost by:**
  - Eliminating process steps
    - Introduction of continuous mixing and MAAT coating
  - Implementation of On-Line Tools to Improve Quality & Reduce Ex-situ Testing
    - Use of mass flow meters, basis weight tools, web temperature sensors, etc. to improve product uniformity and eliminate ex-situ testing between process steps
  - Improve Manufacturing Efficiency to Increase Product Yields and Reduce Scrap
    - Improved GDL properties and material handling equipment to move to full-width production (850 mm wide) and full-length (900m) manufacturing to improve yields and reduce scrap
  - Understand Relationship Between Process Parameters and Key GDL Properties to Improve Performance for Specific Applications
    - Developed correlations between key GDL properties and process variables at multiple process steps
    - Performed design trade-off analysis to determine how to optimize GDLs for specific properties
- **The result of this work has been a 55% drop in the manufacturing cost of the GDL as well as a 4x increase in manufacturing capacity**
- **This work makes it possible to reach to DOE 2015 target cost of GDL with the design of a new Greenfield facility and appropriate capital investment**

# Technical Back-Up Slides - Supplemental

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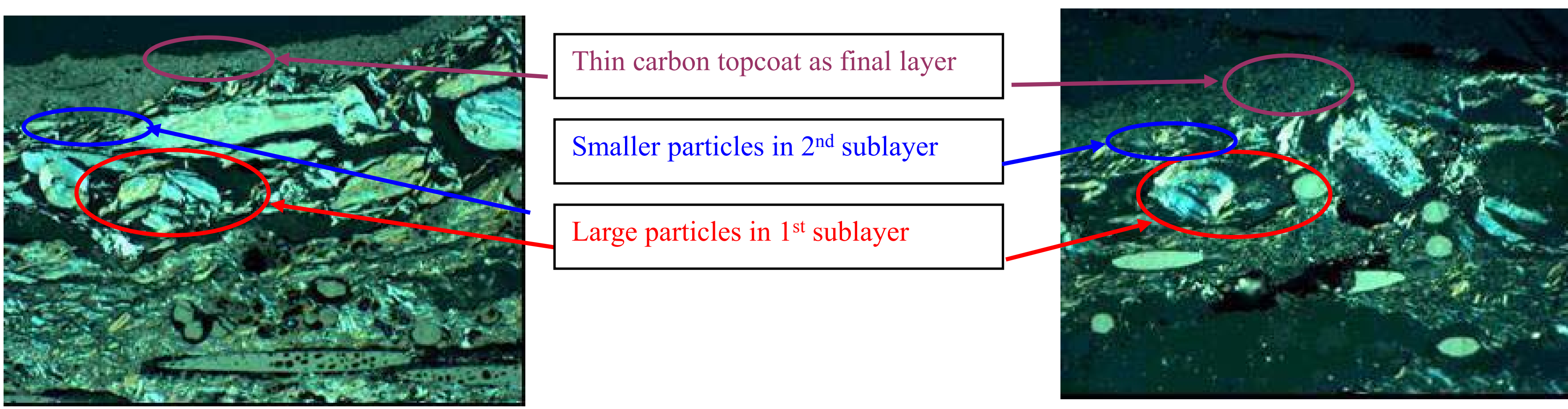
Technical Back-Up Slides

# Technical Back-up Slides (1)

## Challenges

- **How can we tell if the "Many At A Time" (MAAT) coating structure and the "One At A Time" (OAAT) coating structure are similar?**
  - We are looking at cross-section SEM images of multilayer coated samples
    - This allows us to approximate the thickness of each layer
    - This provides us with some insight into how the layers are laying on top of each other (amount of penetration into the substrate)
  - Work was performed on both standard and multilayer samples and initial results appear promising

**Multilayer** **Standard**



# Technical Back-up Slides (2)

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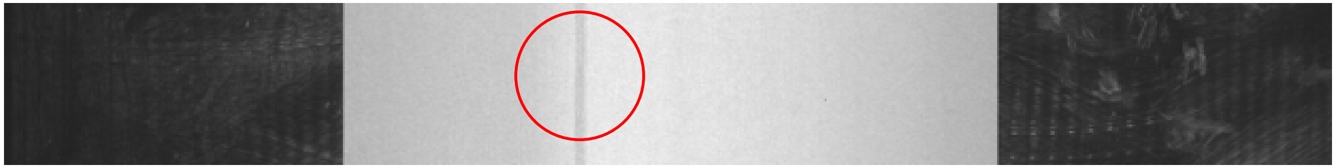
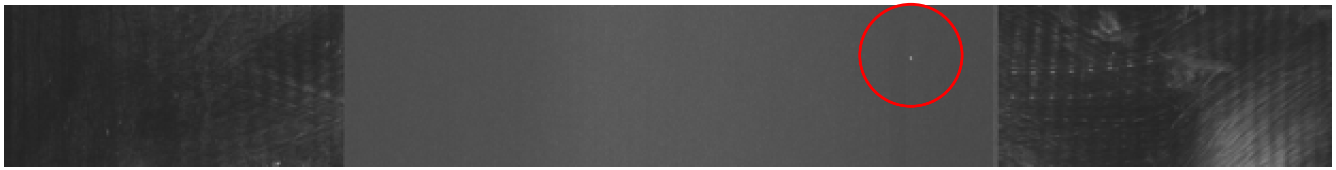
## Examples of defects as seen in the coating line vision system

- System can accurately detect start and final length of a coating streak
- Real-time defect map can be used to view images of defects
- Alarm system will notify operators of high levels of defects or streaks, so that adjustments can be made
- System is being programmed to detect "repeat" defects that may otherwise be overlooked



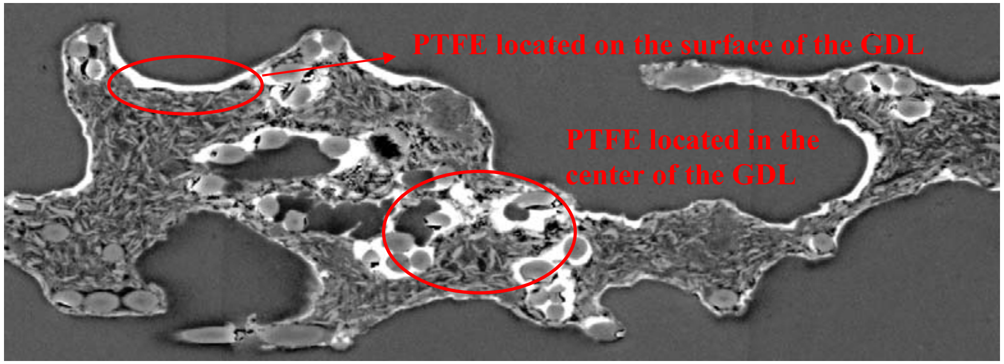
Example of a coating streak as seen in the on-line vision system



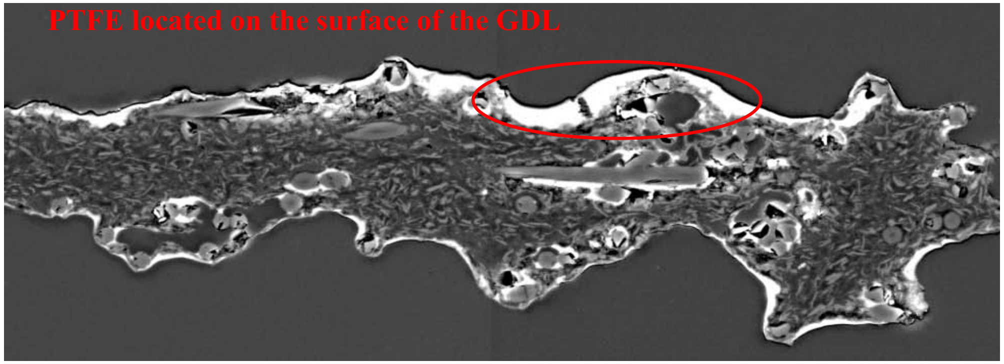
Example of a coating blemish as seen in the on-line vision system

# Technical Back-up Slides (3)

- Controlling PTFE distribution in the GDL
  - **PTFE distribution in the GDL can be altered by the drying process**
  - **Penn. State is working on developing a relationship between Raman line scans and PTFE distribution**
  - **Investigations into the effect of PTFE distribution on GDL properties and cell performance will be continued during this project**



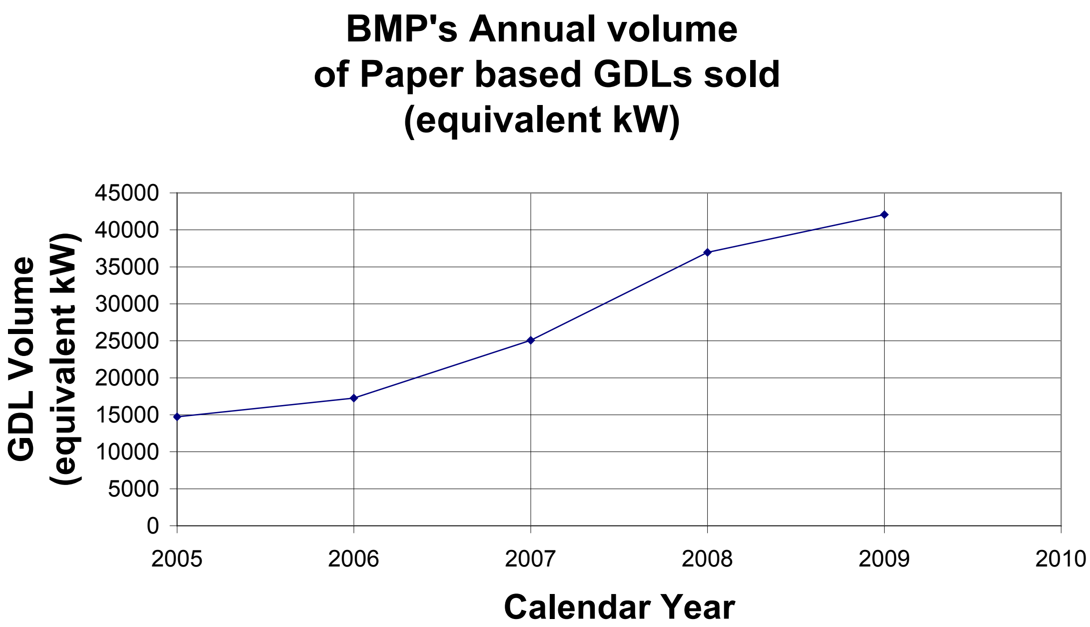

**PTFE distribution throughout the GDL that was air-dried, showing high concentration in the center of the GDL and less on the surface**

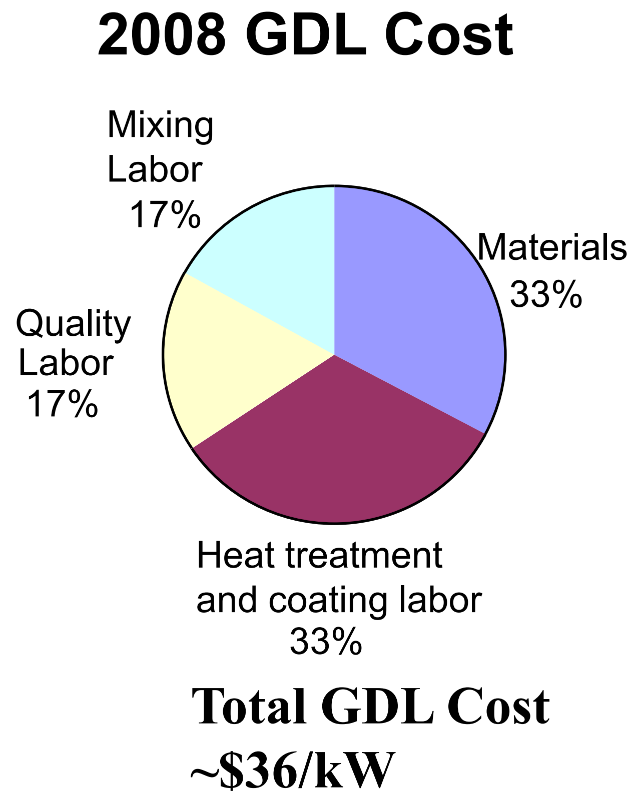



**PTFE distribution throughout the GDL that was oven-dried, showing high concentration on the surface and minimal concentration in the center**

# Technical Back-up Slides (4)

- **BMP's GDL Production Rates**
  - Considers only paper based GDLs
  - Sales volumes indicate a need to continue increasing production capacity, improving GDL quality and reducing manufacturing costs




- **Breakdown of GDL costs at the end of 2008**