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# Reduction in Fabrication Costs of Gas Diffusion Layers

**Colleen Legzdins**

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

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Project ID #
mf_02_legzdins

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# Table of Contents

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# Project Overview

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## Timeline

- Start date: Oct. 1, 2008
- End date: Sep. 30, 2011
- **15 % complete**

## Budget

- Total project funding:
  - DOE share: $3,000,000
  - Contractor share: $1,697,949
- Funding received in FY09: $999,994
- Funding for FY10: $1,006,072

## Barriers

- High material & manufacturing costs
- Lack of high-volume membrane electrode assembly (GDL) processes
- Low levels of quality control and inflexible processes

## Partners

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

# Project Relevance

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## Project Objectives

- Reduce the fabrication costs of Gas Diffusion Layer (GDL) products and demonstrate the means of achieving six sigma quality standards at high volume manufacturing
  - Development, implementation, and demonstration of novel manufacturing technology
    - Reduce the number of process steps
    - Enable a growth in product volumes and yields
    - Enable innovative product development of low cost GDL roll goods
  - Development, implementation, and verification of real-time, on-line measurement tools
    - Reduce/eliminate costly ex-situ characterization sampling and testing
    - demonstrate continuous high volume, low-cost GDLs to six sigma quality standards
  - Development, implementation, and application of real-time, on-line process control tools
    - Maximize production yields
  - Sustained development of an understanding of the relationships between process variables, GDL product properties, and fuel cell performance to direct process specifications that maximize production yields of high performance, low cost GDLs
- Produce high performance GDLs at lower cost in the near term
- Verify the design of a new production facility incorporating new GDL process technologies to meet automotive volume requirements at the DOE 2015 cost target of $30/kW for the fuel cell system (GDL cost target = $4/kW)

# Project Approach

- **Develop new process technology**
  - Conducted pre-design technical and cost analysis of state of the art production technologies feasible for high volume GDL manufacturing
  - Selection criteria for process technologies also included environmental impact, health and safety, customer quality requirements, and future product development needs
  - Technologies selected to be integrated into current manufacturing process
  - Multilayer coating technology
    - Reduce process steps – increase production volume
    - Reduce probability of coating defects – increase yields
    - Open low cost GDL design window
  - In-line mixing and delivery technologies
    - Reduce waste of raw materials
    - Reduce processing time & energy costs
    - Reduce/eliminate coating liquid variability
    - Enable longer campaigns – increase production volume
    - Open low cost GDL design window

# Project Approach

- **Develop new on-line process control and measurement tools**
  - Process control tools required for new process technologies
    - Ensure process variables and product properties on target
    - Enable higher line speeds
  - Tools required for improved process specifications to achieve six sigma or better quality standards
    - Provide full roll length characterization vs. beginning & end of roll sampling
  - Tools for new control strategies arising from continued development of our understanding of the relationships between process variables, GDL properties, and fuel cell performance
  - Replace/eliminate costly ex-situ materials testing

- **Develop on-line quality inspection system**
  - Eliminate human error – cost of inspectors missing defects
  - Permit high line speeds
  - Monitor line start-up, changeover and real time process upsets
  - Allow real-time and long-term process improvements

- **Establish process variables – GDL properties – fuel cell performance relationships**
  - Develop new process specifications
  - Establish critical-to-quality and critical-to-performance characteristics

# Project Approach

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## Milestones & Go/No-Go Decisions

| Date | Milestone |
|---|---|
| March 31st, 2009 | Design feasibility and scalability analysis completed. |
| September 30th, 2009 | All in-line mixing & delivery and multilayer coating process equipment and process control & on-line measurement tools (designed, specified, fabricated) purchased and received. |
| March 31st, 2010 | Installation and determination of initial process capability metrics of new process equipment, process control & measurement tools complete. |
| September 30th, 2010 | Assessment of process parameters and their effect on GDL properties complete – inline ink mixing & delivery and multilayer coating processes separated. **GO/NO-GO Decision** |
| September 30th, 2010 | Demonstrate that single cell fuel cell performance of lower cost GDL produced with the new process technologies is equivalent to or improves the baseline GDL fuel cell performance - inline ink mixing & delivery and multilayer coating processes separated. **GO/NO-GO Decision** |
| March 31st, 2011 | Demonstrate that the variability of GDL material properties within low cost, high volume GDL roll good is equivalent to the higher cost, baseline GDL roll good, with no degradation in fuel cell stack performance (inline ink mixing & delivery and multilayer processes now combined). |

# Technical Accomplishments and Progress

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| Task # | Project Task | Planned Completion Date | Percent Complete |
|---|---|---|---|
| **PHASE 1. PROCESS DESIGN AND IMPLEMENTATION** | | **9/30/2010** | |
| **1** | **Design Feasibility and Scalability Analysis of the New Process Technologies** | 3/31/2009 | 90% |
| **2** | **System Design, Specification, Fabrication and Receipt** | 9/30/2009 | |
| 2.1 | Multi-layer coating equipment | 9/30/2009 | 45% |
| 2.2 | Multilayer coating process control and measurement tools | 9/30/2009 | 50% |
| 2.3 | Solids and liquid delivery equipment | 9/30/2009 | 45% |
| 2.4 | Ink mixing and delivery equipment | 9/30/2009 | 50% |
| 2.5 | In-line mixing & delivery process control and measurement tools | 9/30/2009 | 45% |
| 2.6 | Substrate paper process control and measurement tools | 9/30/2009 | 65% |
| 2.7 | On-line substrate paper and coated GDL product quality tool | 9/30/2009 | 50% |
| **3** | **Commissioning of process technology and on-line measurement tools** | 9/30/2010 | |
| 3.1 | Commissioning of process technology | 3/31/2010 | Not started |
| 3.2 | Short roll uniformity studies | 9/30/2010 | Not started |
| 3.3 | Evaluate process control and on-line measurement systems capability | 9/30/2010 | Not started |
| **4** | **Establish Process, Product and Performance Relationships** | 9/30/2010 | |
| 4.1 | Build processing parameters-product properties relationships | 9/30/2010 | 5% |
| 4.2 | Single cell fuel cell testing | 9/30/2010 | Not started |
| 4.3 | Establish critical-to-quality and critical-to-performance characteristics | 9/30/2010 | 5% |

# Technical Accomplishments and Progress

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| Task # | Project Task | Planned Completion Date | Percent Complete |
|---|---|---|---|
| **PHASE 2 - PRODUCT/PROCESS DEMONSTRATION & VALIDATION** | | **9/30/2011** | |
| **5** | **Synchronize and Validate New Technologies** | 9/30/2011 | |
| 5.1 | Synchronize and validate new technologies | 9/30/2011 | Not started |
| 5.2 | Establish process specifications and validate process control systems | 9/30/2011 | Not started |
| 5.3 | Short roll and production run trials | 9/30/2011 | Not started |
| 5.4 | New facility design | 9/30/2011 | Not started |
| **6** | **Product Performance Validation** | 3/31/2011 | Not started |
| **7** | **Statistical Process Control (SPC) and ISO9001 & TS16949 Procedures** | 9/30/2011 | Not started |
| **8** | **Project Management** | 9/30/2011 | |
| 8.1 | Environmental health and safety, hazard assessment and SOPs | 9/30/2011 | 10% |
| 8.2 | Training of operators and maintenance staff | 9/30/2011 | 5% |
| 8.3 | Project reporting | 9/30/2011 | 15% |

# Technical Accomplishments and Progress

## PHASE 1 – PROCESS DESIGN AND IMPLEMENTATION

- **Task 1. Design feasibility and scalability analysis of the new process technologies.**
  - On-track to completion
  - Analysis benchmarks established that include: performance, cost, quality, timing/delivery, flexibility, equipment requirements, maintenance requirements, automation techniques, spatial layout, and waste minimization strategies
- **Task 2. System design, specification, fabrication & receipt.**
  - Subtask 2.1 Multi-layer coating equipment
    - Characterization of coating solutions' rheological and physical properties complete
    - Modifications to coating fluid formulations for multi-layer coating compatibility identified
    - Lab-scale multi-layer coating trials in-progress to direct key design parameters of system and establish preliminary coating window
  - Subtask 2.2 Multi-layer coating process control and measurement tools
    - Down-selected on-line GDL coating weight measurement tool
      - Setup & calibration in-progress, with first run web scanning data collected
    - Prospective on-line moisture content and thickness tools under assessment
    - Review and assessment of measurement techniques feasible for characterization of GDL chemical homogeneity in-progress

# Technical Accomplishments and Progress

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- On-line surface topology sensor identified
  - Selection criteria established and assessment of non-contact surface profilometry techniques/instruments completed
  - Representative substrate and coated GDL samples sent to various manufacturers of appropriate on-line sensors for demonstration of capability
  - Evaluation criteria of surface topography characterization defined
    - ISO defined 2D profiles and 3D surface topography
    - 2D & 3D parameters in accordance with international standards using advanced ISO 16610 filtering techniques
    - Long scan 2D profiles at representative scanning speed
  - Identified requirement for accurate web velocity measurement
    - 2 approaches under review: accurate process control of web velocity or on-line surface velocimeter measurement input real-time to instrument

## Subtask 2.3 Solids and liquid delivery equipment

- Physical and rheological properties of liquid feed materials characterized
- Continuous solids feeding systems under evaluation (e.g. volumetric and gravimetric screw, vibrating, rotary valve, belt, agitator, and disc feeders)

# Technical Accomplishments and Progress

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- Subtask 2.4 Ink mixing and delivery equipment
  - Dynamic and static in-line mixers evaluated
    - Criteria for the mixer design include: incorporation and wetting out of powders, ink holdup, mixing shear rate range, bubble generation, and ink flow pulsation
  - Pressurized and pump delivery systems evaluated
    - Criteria for the delivery equipment include: interface with the mixer and deliver a non-pulsating, bubble-free, agglomerate-free, precision (metered) flow to the coater
  - In-line mixer pilot trials in-progress

- Subtask 2.5 In-line mixing & delivery process control and measurement tools
  - On-line viscometers under evaluation
    - Criteria for evaluation include: simplicity of use, ease of maintenance & calibration, accuracy, repeatability, ability to accommodate different ink rheologies & particle sizes, and measurement temperature & shear rates
  - Assessment of bubble content and surface tension measurement techniques in-progress
  - Identified additional tools for assessment
    - Solids content, pH, and particle size distribution

# Technical Accomplishments and Progress

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- Subtask 2.6  Substrate paper process control & measurement tools
  - Initiated substrate carbonization process study
    - Combined experimental thermogravimetric analysis, lab-scale process experiments, and production plant trials
    - Process parameters affecting final substrate morphology identified
      - Parameters include: soak temperatures and periods; heating rates; gas composition, flow rate and pressure; concentration of volatiles & their subsequent reactions; precursor composition, porosity, pore size & additives
    - Identified process parameters requiring tighter control to reduce product variability
    - Identified high resolution 3D imaging technique to provide information on the topology of the pore space and its changes during processing
  - Review and assessment of potential measurement techniques to characterize substrate chemical homogeneity in-progress
    - Initial results demonstrate the potential of Raman and IR spectroscopy to map the distribution of surface Teflon® during on-line processing
  - On-line surface topology sensor identified

# Technical Accomplishments and Progress

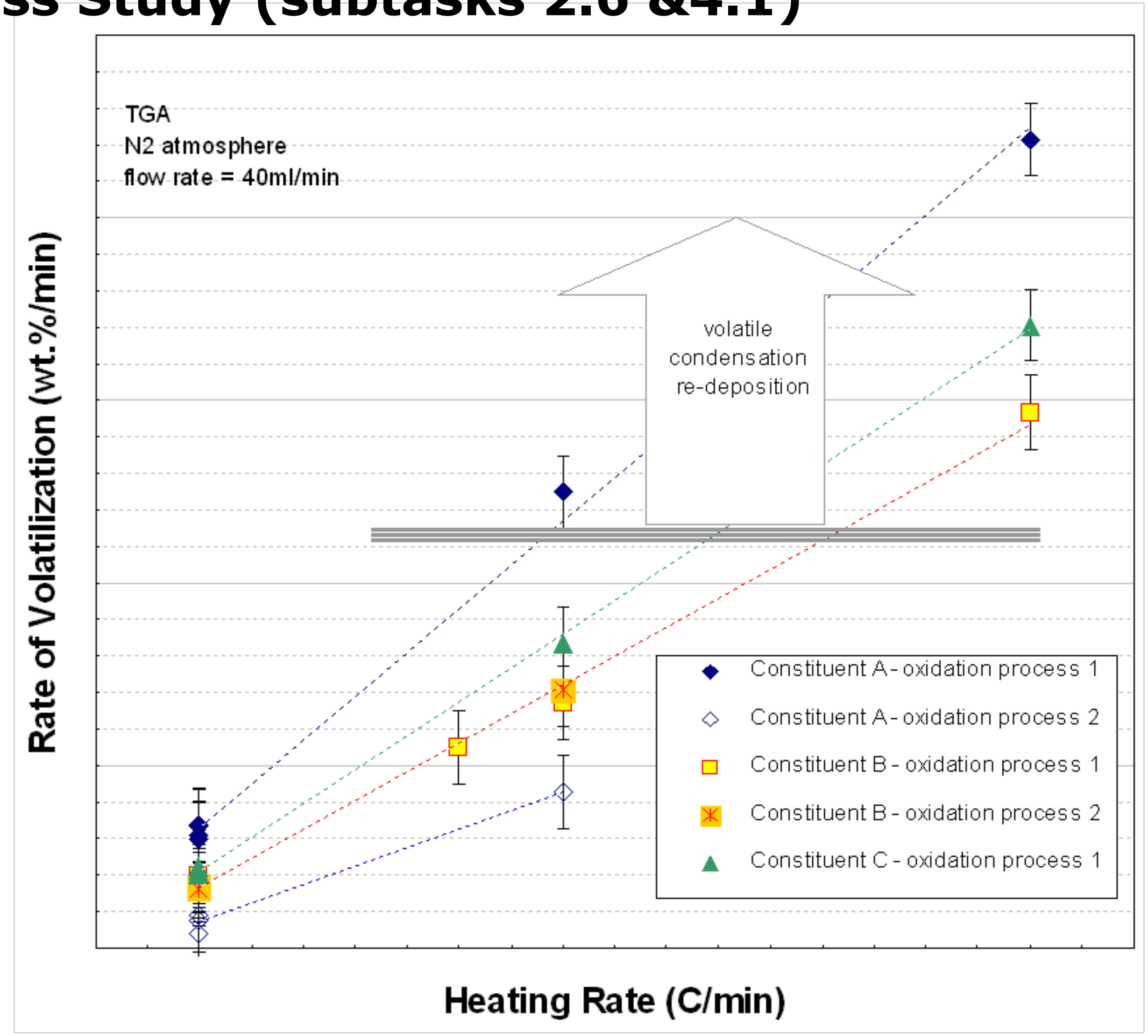
- Subtask 2.7  On-line substrate paper and coated GDL product quality tool
  - Review of surface quality & coating defect specifications for substrate and GDL products on-going
    - Defects identified and their classification on-going
    - Allowable frequencies of each defect type and size class on-going
  - Evaluation of machine vision inspection systems in-progress
    - Evaluation criteria include: camera type, capability (minimum defect size detected at maximum coating/line speed), data capture rate, image processing rate, robustness, versatility, stability, ease of use, working distance, magnification, range (field of view), resolution, and accuracy

# Technical Accomplishments and Progress

## Carbonization Process Study (subtasks 2.6 &4.1)

- Higher carbonization heating rates can be utilized with optimized oxidation process

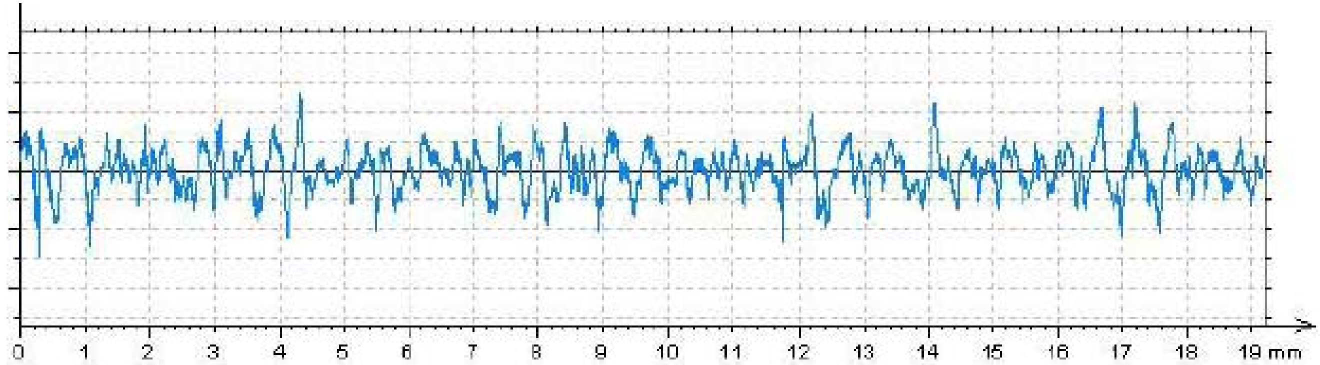
- Translation of experimental conditions to local furnace process conditions to determine maximum heating rate at onset of volatile condensation & re-deposition



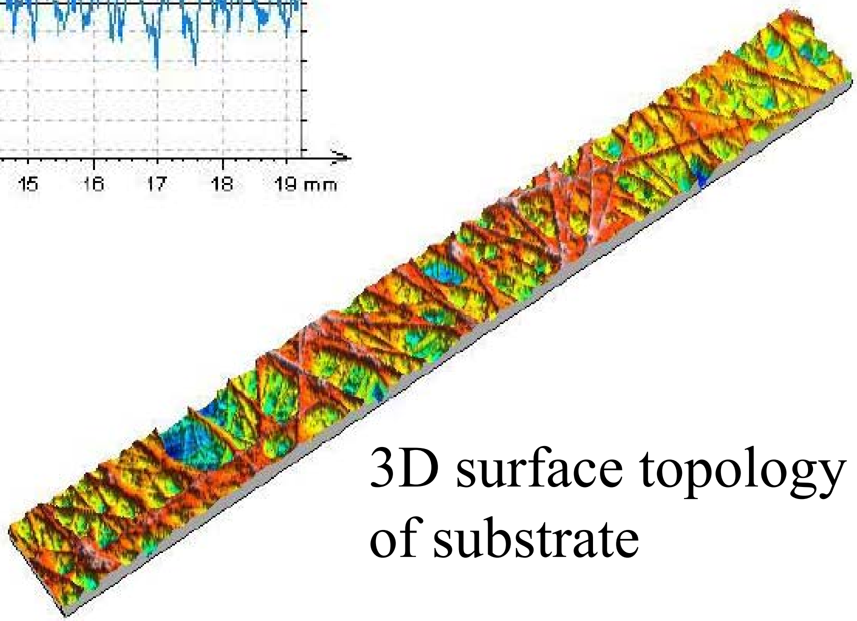
# Technical Accomplishments and Progress

## On-line Surface Topology Tool (subtask 2.2)

2D Surface profile of coated GDL obtained at representative scanning rate (web speed) - Length of scan = 19mm




- Sensor capable of 2D long run scanning
- Instrument capable of resolving rough surfaces



3D surface topology of substrate

# Technical Accomplishments and Progress

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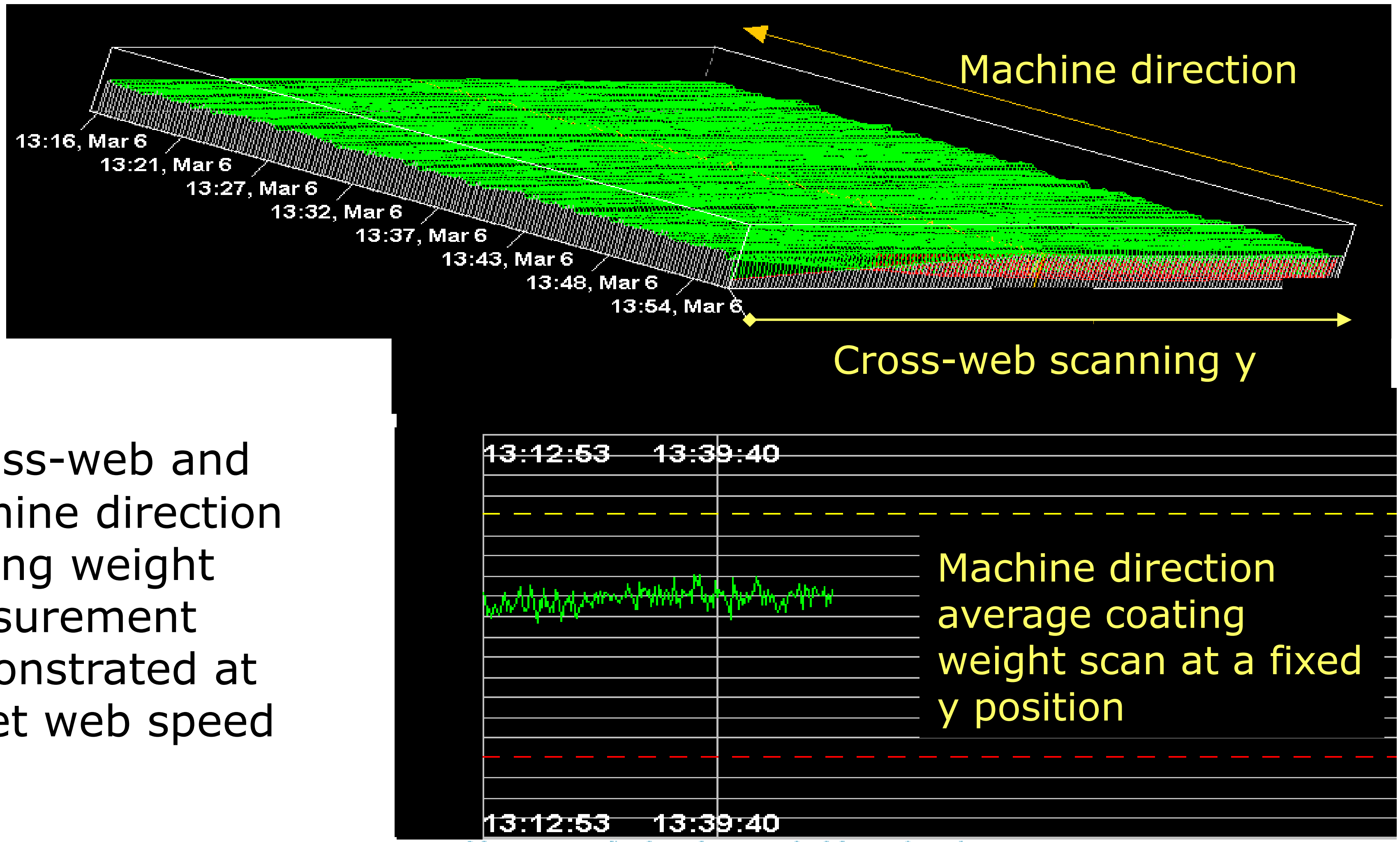
## On-line Coating Weight Tool (subtask 2.2)




- Cross-web and machine direction coating weight measurement demonstrated at target web speed

# Technical Accomplishments and Progress

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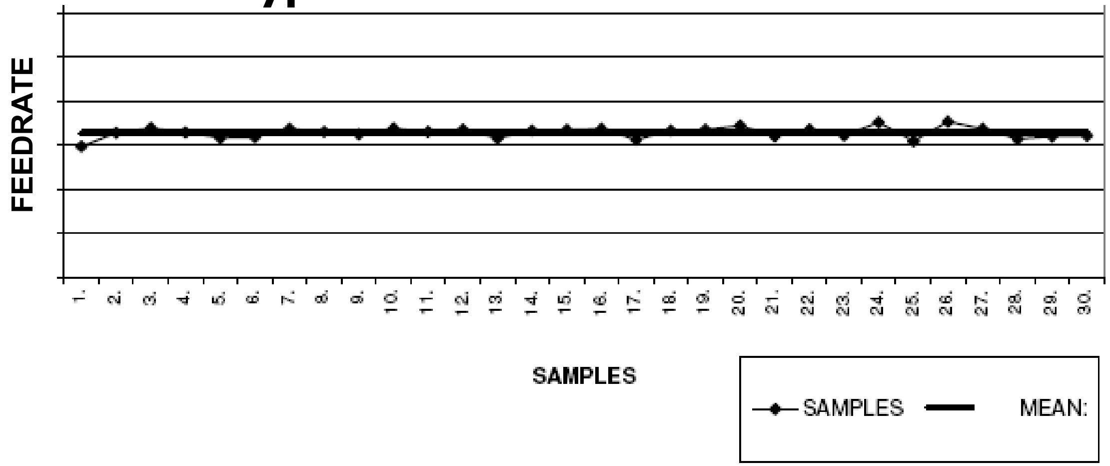
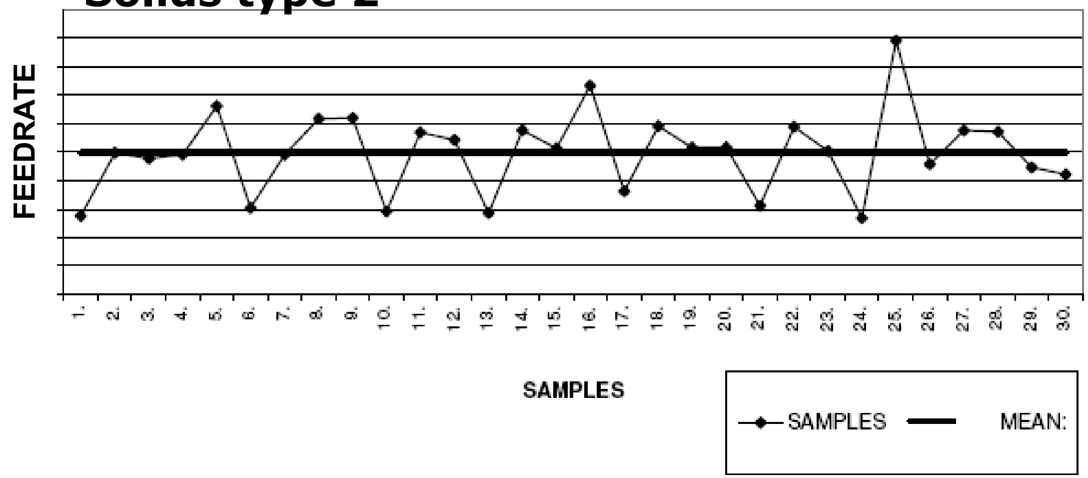
## Continuous solids delivery equipment (subtask 2.3)

- Representative laboratory test data utilized in feeder evaluations
- Feedrate accuracy best for solids type 1 versus solids type 2 with this feeder technology
- Feeder design driven by characteristics of each raw material

**Solids type 1**



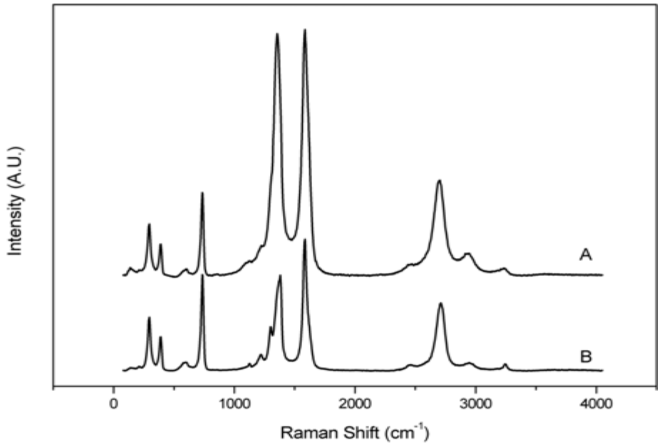

**Solids type 2**

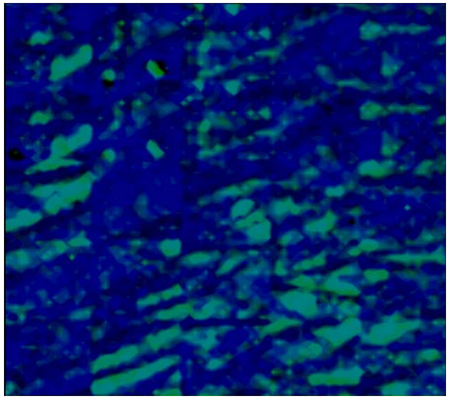


# Technical Accomplishments and Progress

## Vibrational spectroscopy imaging to determine surface PTFE distribution of GDLs on-line during processing (subtasks 2.2 &2.6)

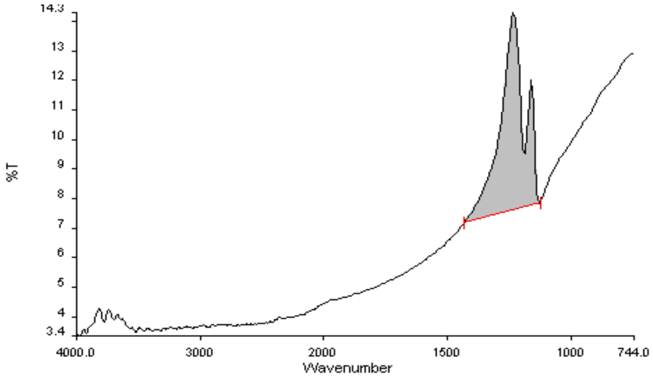
*Raman spectra and intensity map for PTFE peak at 734 $cm^{-1}$*

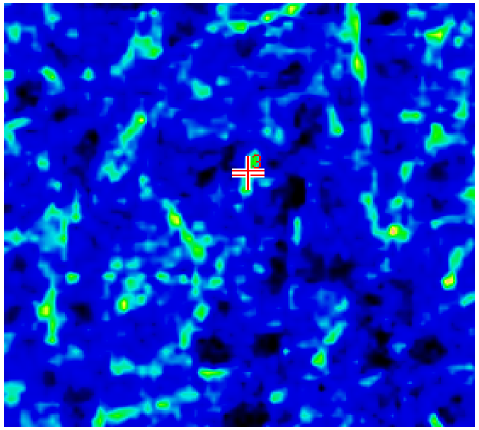


750 x 750 μm

*FTIR spectrum and intensity map for PTFE peaks between 1450 and 1100 $cm^{-1}$*



987 x 987 μm

Lighter areas show higher concentrations of PTFE

# Technical Accomplishments and Progress

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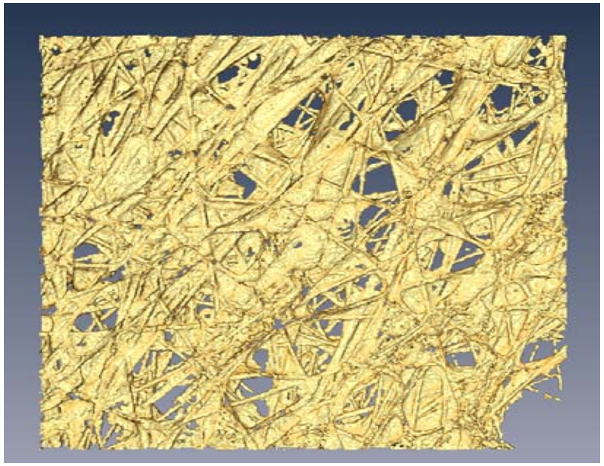
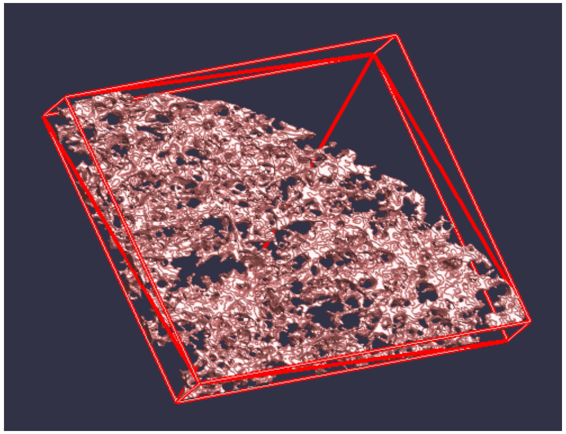
## High resolution x-ray CT scanning of GDL pore structure (Subtasks 2.2, 2.6 & 4.1)

High-resolution 3D imaging can provide information on the topology of the pore space and its changes during processing in post-analyses.

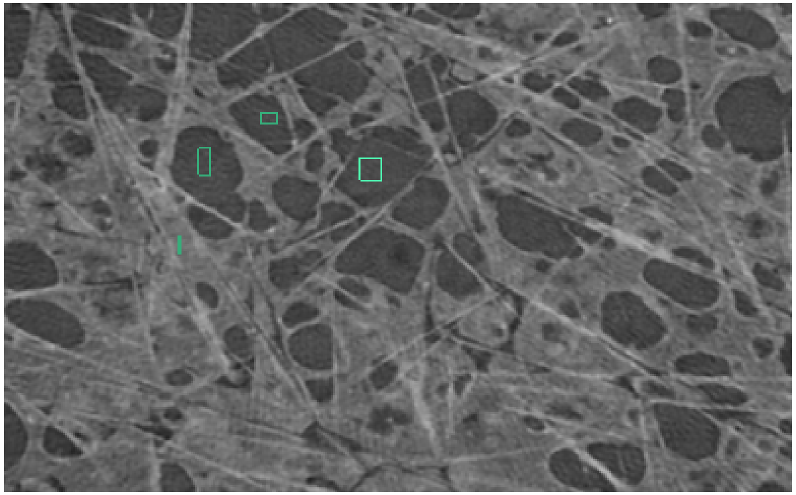
*3D structure of a PTFE-laden GDL*



P75T – 2.45 μm per voxel side



*Raw data slide showing solid and pore space*



PENNSTATE

DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING
COLLEGE OF EARTH AND MINERAL SCIENCES

# Collaborations

- Prime - Ballard Material Products, Industry, within DOE $H_2$ Program
- Partner – Dr. Michael Hickner, The Pennsylvania State University, Academia, within DOE $H_2$ Program
- Partner – Ballard Power Systems, Industry, within DOE $H_2$ Program
- Collaborator – Dr. Michael Ulsh, National Renewable Energy Lab, Federal Laboratory, within DOE $H_2$ Program
- Collaborators – GDL customers across all fuel cell market segments, outside of DOE $H_2$ Program

# Proposed Future Work

- **FY09: Complete Tasks 2.1 – 2.7**
  - 2.1 Multi-layer coating equipment
    - Analyze pilot trial data; establish equipment design parameters; specify, fabricate and purchase equipment
  - 2.2 Multi-layer coating process control and measurement tools
    - Complete assessment of moisture content and thickness tools; evaluate potential sensors; demonstrate capability and purchase if meets technical criteria
    - Complete assessment of GDL chemical homogeneity characterization techniques; select viable technique(s) for further development
    - Complete surface topology and coating weight sensors capability studies
  - 2.3 Solids and liquid delivery equipment
    - Complete evaluation of continuous solids feeding systems and liquid delivery equipment; specify design with cascaded requirements from ink mixing equipment, and purchase
  - 2.4 In-line ink mixing and delivery equipment
    - Analyze pilot trial data; establish equipment design parameters with cascaded requirements from multi-layer coater design; specify, fabricate and purchase equipment

# Proposed Future Work

- 2.5 In-line mixing & delivery process control and measurement tools
  - Complete evaluation of in-line viscometers; specify and purchase
  - Complete assessment of in-line bubble content, surface tension, solids content, pH, and particle size distribution techniques; evaluate potential tools; demonstrate capability and purchase if meets technical criteria
- 2.6 Substrate paper process control & measurement tools
  - Complete substrate carbonization process study; introduce new process control procedures
  - Complete assessment of GDL chemical homogeneity characterization techniques; select viable technique(s) for further development
  - Complete assessment of thickness tools; evaluate potential sensors; demonstrate capability and purchase if meets technical criteria
  - Complete surface topology sensor capability study
- 2.7 On-line substrate paper and coated GDL product quality tool
  - Complete evaluation of inspection systems; demonstrate capability and purchase if meets technical requirements

# Proposed Future Work

- **FY10: Complete phase 1, Process Design and Implementation, Tasks 3 & 4**
  - 3.1-3.3 Commissioning of process technology and on-line measurement tools
  - 4.1-4.3 Establish process, product and performance relationships
  - Demonstrate that single cell fuel cell performance of lower cost GDLs produced with the new process technologies is equivalent to or improves the baseline GDL fuel cell performance

# Summary

- **Relevance:**
  - Addresses technical barriers of high material & manufacturing costs, lack of high-volume membrane electrode assembly (GDL) processes, and low levels of quality control and inflexible processes
  - Enables the production of lower cost GDLs in the near term, and verification of the design of a new production facility incorporating these process technologies to meet automotive volume requirements at the DOE 2015 cost target

- **Approach:**
  - Reduce the fabrication costs of Gas Diffusion Layer (GDL) products and demonstrate the means of achieving six sigma quality standards at high volume manufacturing
  - Develop new process technologies that include multi-layer coating and in-line mixing
  - Develop on-line process control and measurement tools
  - Establish process variables – GDL properties – fuel cell performance relationships to direct process specifications that maximize production yields of high performance, low cost GDLs

- **Technical Accomplishments and Progress**
  - Carbonization process study has identified process parameters requiring tighter control and established key process parameters for bulk of pyrolysis reaction
  - Pilot and lab trials of in-line ink mixing and multi-layer coating have been initiated
  - Coating weight tool capability has been demonstrated on-line
  - On-line surface topology sensor identified and capability study in-progress
  - Assessment and evaluation of on-line tools and inspection system well underway
  - Promising new techniques for characterization of GDL chemical & physical homogeneity have been identified

# Summary

- **Technology Transfer/Collaborations:**
  - Active partnerships with Dr. Michael Hickner of the Pennsylvania State University and Ballard Power Systems
  - Collaboration initiated with Dr. Michael Ulsh of NREL
  - On-going collaborations with GDL customers within the fuel cell industry
- **Proposed Future Research**
  - FY09: complete Task 2, System design, specification, fabrication and receipt of process equipment and on-line tools
  - FY10: complete Task 3 & 4, Commissioning of process technology and on-line measurement tools; Establish process, product and performance relationships