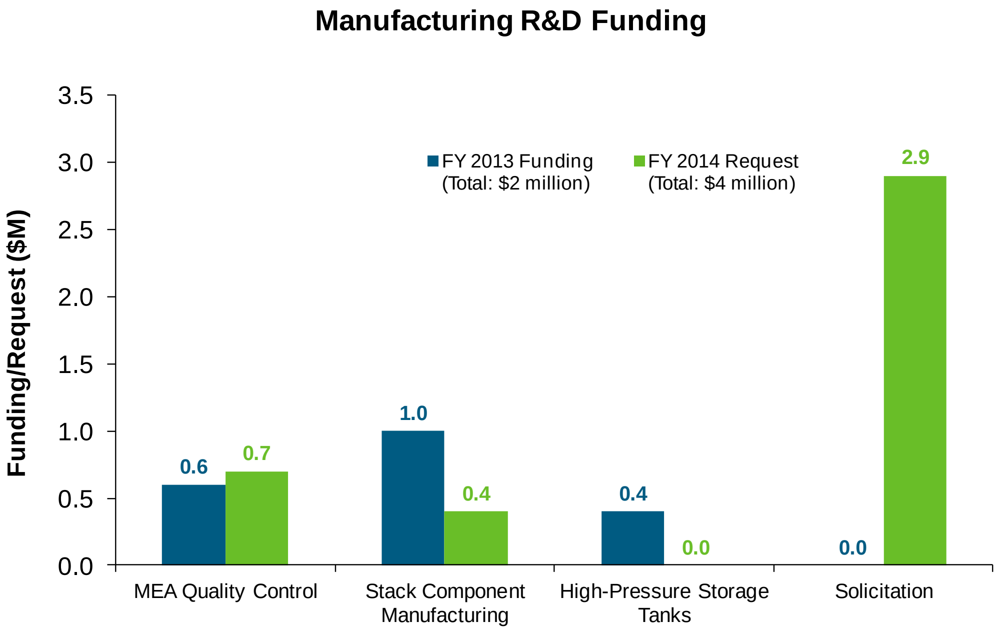
2013 — Manufacturing Research and Development (R&D)
Summary of Annual Merit Review of the Manufacturing R&D Program

Summary of Reviewer Comments on the Manufacturing R&D Program:

According to the reviewers, the objectives and progress of the Manufacturing R&D program were clearly presented and prior successes were described. The program appears to be focused, well managed, and effective in addressing the U.S. Department of Energy (DOE) Hydrogen and Fuel Cells Program needs. The reviewers suggested that DOE consider adding support for design for manufacturing and assembly relevant to polymer electrolyte membrane (PEM) and solid oxide fuel cell (SOFC) stacks as well as advanced stack assembly. In fiscal year (FY) 2013, four manufacturing projects were reviewed. These projects addressed fuel cell membrane electrode assembly (MEA) manufacturing, fabrication of catalyst-coated membranes, fuel cell stack in-line testing, and manufacturing of high-pressure vessels for hydrogen storage.

Manufacturing R&D Funding:

Funding for the Manufacturing R&D program was $2 million for FY 2013, and $4 million was requested for FY 2014. The FY 2014 request-level funding will continue existing Manufacturing R&D projects and provide funding for new projects through a competitive solicitation, subject to appropriations.



Majority of Reviewer Comments and Recommendations:

Four Manufacturing R&D projects were reviewed and the maximum, minimum, and average scores for the projects were 3.6, 3.0, and 3.3, respectively. All projects were judged to be highly relevant to the DOE Hydrogen and Fuel Cells Program activities, with very good technical approaches. Project progress and accomplishments were judged to be extremely good. Project teams were judged to be strong; participation and contribution from industry partners were judged to be useful and coordinated. For most of the projects, reviewers felt that more details needed to be presented for future work.

The highest-ranked project (3.6) was considered by the reviewers to be highly relevant, with an excellent approach, outstanding accomplishments, and strong technology transfer and collaborations. Reviewers noted that the benefits of the technique developed in the project with the lowest score (3.0) were not clear compared to XRF and were possibly not compelling.

Fuel Cell MEA Manufacturing: Three projects were reviewed in the area of fuel cell MEA manufacturing, with an average score of 3.4. Reviewers noted that the approach for each of these projects was good and commended coordination with industry to improve manufacturing methods. The projects yielded performance gains and cost savings. The reviewers recommended that sensitivity be improved for all techniques and applications and recommended that the techniques developed be licensed/transferred to other organizations.

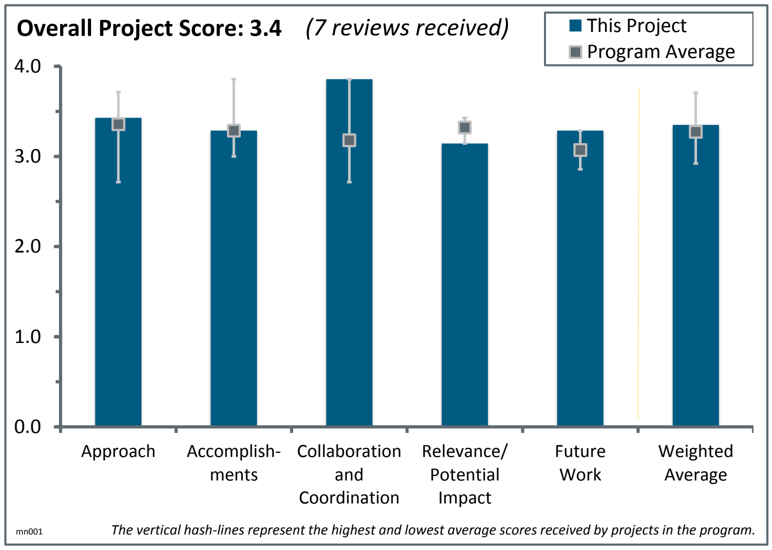
Metrology for Fuel Cell Manufacturing: The one project reviewed regarding metrology for fuel cell manufacturing received a score of 3.0. Reviewers approved of the general approach to this project but noted that it was unclear how surface changes due to normal manufacturing variation will impact measurement, and whether this variation can be accommodated in the processing. It was recommended that the researchers look into Mie scattering and some acousto- and electro-optical methods for flexibility.

Project # MN-001: Fuel Cell MEA Manufacturing R&D

Michael Ulsh; National Renewable Energy Laboratory

Brief Summary of Project:

The aim of this project is to develop the capabilities and acquire the knowledge for in-line quality control during fuel cell manufacturing. Industry partners and forums provide input to understand quality control needs. The project has highlighted the relevance of crosscutting quality control development by estimating the cost of poor quality analysis. Research in the past year focused on exploratory studies to improve the sensitivity of current techniques, expand techniques to new materials, explore the feasibility of new diagnostic concepts, and develop new in situ techniques and capabilities.



Question 1: Approach to performing the work

This project was rated **3.4** for its approach.

- The approach is good; the principal investigators are developing techniques based on various excitation methods to detect defects in the fuel cell assembly process.
- The National Renewable Energy Laboratory (NREL) employs a good combination of input from industry and modeling to direct experimental efforts.
- NREL coordinates with industry to develop and improve diagnostics that are critical to fuel cell membrane electrode assembly (MEA) and gas diffusion layers (GDL) manufacturing.
- The first step taken is to understand suppliers' diagnostic and quality control (QC) needs. Early involvement of different component suppliers for different fuel cell types is a strong point. The approach covers identification of needs and possible solutions, development of techniques, and in-line validation. The intent to transfer the technology to industry is positive. Study of the effects of defects on cell performance will guide diagnostic development and define the defect envelope needing detection.
- There is a new approach planned that will focus more on exploratory studies; however, the researchers should continue to do "field evaluation" on other already "developed" techniques, such as optical observation of defects (such as GDLs, black on black, etc.).
- The approach is relatively broad based and covers a number of defects that can occur in MEA manufacturing processes. Input from component developers has helped shape the project objectives. Installing successful diagnostics on actual manufacturing lines, such as at Ion Power, is necessary to confirm the promise of these techniques to detect defects in industrial settings. There is still a need to demonstrate that these techniques can detect defects that occur during actual manufacturing runs rather than just detect defects in samples known to include them. This may be difficult to accomplish without detailed knowledge of the manufacturing processes in use by component suppliers.

Question 2: Accomplishments and progress toward overall project and U.S. Department of Energy (DOE) goals

This project was rated **3.3** for its accomplishments and progress.

- Development of methods to detect electrode coatings on GDLs has been very encouraging. Further detection of local membrane failures is also encouraging.

- The optical diagnostic technique was demonstrated on polymer electrolyte membrane fuel cell (PEMFC) gas diffusion electrodes (GDEs) and solid oxide fuel cell tubes. However, sensitivity needs significant improvement in all cases.
- The project changed focus somewhat this year and the new areas of investigation are showing some promising results. NREL should examine the need for variable or multiple non-uniform applications of the non-uniform GDL/catalyst coated membrane (CCM) electrical excitation.
- The optical diagnostic (mainly reflectance), IR/DC, and IR/RFT are all performing very well, and suggested improvements will increase their value. Using IR/RFT with higher hydrogen should prove to be very valuable. It is not clear whether the "gas knife" design is optimum for measurement in an open environment for that process. There are many configuration choices available for that version of IR/RFT.
- Reasonable progress has been made since the last DOE Hydrogen and Fuel Cells Program Annual Merit Review. However, optical detection of defects on CCMs and GDEs was difficult for black surfaces typical of fuel cell MEAs; defects of approximately 1 mm were detected. It is not clear if these defects had a significant effect on performance. The IR/DC diagnostic is the furthest along in development in terms of detecting surface cuts and scratches. Even so, GDL/CCM cracks in line with the electric field were difficult to detect using the technique. Lawrence Berkeley National Laboratory (LBNL) modeling was used to good effect to optimize excitation geometry, and the diagnostic will be installed on an Ion Power commercial coating line. The other techniques presented need further development before their usefulness can be established. A new diagnostic to measure the ionomer/carbon ratio was described. It was unclear what variations can be detected and how they impact performance. The presentation did not indicate a strong industry need for this diagnostic.
- The project continues to make progress related to QC of MEA manufacturing. This year, the researchers have an agreement with Ion Power to deploy on their catalyst coating line. The team also developed a dual light source to improve sensitivity to low-emissivity materials, as well as demonstrated IR/RFT in an open environment. The next step is to demonstrate with a moving substrate and a dry-open air environment to measure I:C ratio – electronic capacitance.

Question 3: Collaboration and coordination with other institutions

This project was rated **3.9** for its collaboration and coordination.

- This project features excellent collaboration with relevant industry partners.
- This project is well coordinated, from gleaning industry needs for measurements to deploying early developed diagnostic systems with industry partners.
- Collaborators from industry are essential for the nature of this work. This project has a good mix of these collaborations in addition to laboratory and academic partners.
- The agreement for deployment with Ion Power is excellent; one suggestion is that it should include recording of other experimental conditions, including relative humidity (RH), temperature, etc.
- Reasonable collaborations are evident with a number of the DOE manufacturing projects. LBNL modeling has been used to optimize the IR/DC and IR/RFT diagnostics.
- A formal collaboration with a manufacturer of fuel cell components, such as GDLs or MEAs, would strengthen the team. This small weakness is mitigated by testing materials provided by component suppliers and the early involvement of components suppliers in project planning and prioritization as well as diagnostics development and validation.

Question 4: Relevance/potential impact on advancing progress toward DOE research, development, and demonstration (RD&D) goals

This project was rated **3.1** for its relevance/potential impact.

- The development of crosscutting component QC diagnostics is cost effective. Very high defect-free manufacturing yields are needed for fuel cell operation and durability. This project addresses this issue.
- The output of this project when implemented will have a significant impact toward addressing the highlighted barriers. While identification of defects is good, the project should focus on permitting the quantification of these defects to have a higher impact.

- All of the measurement methods developed in this project have value in aiding in fuel cell manufacture; some have significant value.
- MEA quality improvements (leading to cost reduction) offer opportunities for PEMFC cost reduction.
- It is worthwhile to develop improved diagnostics to detect defects in MEAs and GDLs that can be implemented on actual manufacturing coating lines. NREL has developed several techniques; the most successful is IR/DC, which can detect known pre-fabricated defects down to around 1 mm size at realistic line speeds. Because this technique will be installed on an Ion Power coating line, researchers will be able to determine the usefulness of the technique for detecting unknown defects on a full-scale line. The other techniques being investigated at NREL require further development before their usefulness can be assessed. Perception of relevance would be enhanced if there were clearer indications that the NREL project closely aligned with component suppliers' recommendations on diagnostic development needs.
- The potential impact could be good; however, it is dependent on whether the manufacturing developers are willing to use these techniques. Thus, a substantial portion of this project's future efforts should ensure that manufacturers are deploying the techniques or are willing to do so.

Question 5: Proposed future work

This project was rated **3.3** for its proposed future work.

- All of the future plans are important to fuel cell manufacturing. Deployment of the diagnostics in industry to ensure valuable feedback for optimization is very good. Hopefully there will be a chance to deploy the IR/RFT in a real in-line manufacturing arrangement.
- The overall plans for future work are well thought out. Deployment efforts and partner studies should yield valuable information regarding the ultimate usefulness for industry.
- The proposed future work features a logical progression of efforts. Important work on improving sensitivity is planned.
- The future work plans are good—the project team should continue developing these techniques to improve their performance in real-world applications. Schedule information and go/no-go decision points for the various diagnostics would be helpful. Decision metrics based on the expected impacts of various defects on performance are important and should be presented.
- The next steps appear reasonable, given the progress to date.
- The proposed future work lacks details on quantification; i.e., how these techniques can be used in a feedback process loop control. Further, the impact of these strategies on cost reduction is unclear.
- Much more can potentially be done with the capacitance measurement; the project should expand beyond I/C ratios. Work on deployment looks to be initiated with Ion Power. This work should demonstrate whether the industry developers are willing to incorporate the measurements into their lines. The value in the spatial failure studies in relation to MEA defects is unclear. This is not really a manufacturing/QC methodology, and it is probably not likely to be beneficial in relation to the goals of this project.

Project strengths:

- A strength of this project is the manner in which NREL applies new techniques to solve production process controls in fuel cell manufacturing.
- This project features a strong collaborative team.
- The project is making good progress on multiple approaches and is working well with industry for guidance and deployment.
- NREL is using commercially available hardware from other industries and adapting the hardware for use in fuel cell manufacturing processes that occur at high line speeds. There is good synergy between NREL and the other DOE manufacturing projects in terms of developing and implementing these diagnostic techniques.

Project weaknesses:

- Improved sensitivity is strongly required for all techniques and applications.
- The value of the segmented cell operation to detect MEA defects is not clear in terms of QC.

- This project seems more qualitative; it is not quantitative enough.
- Still to be resolved is the correlation between the prearranged defects that are used to develop the diagnostics and the defects encountered on high-speed production lines.

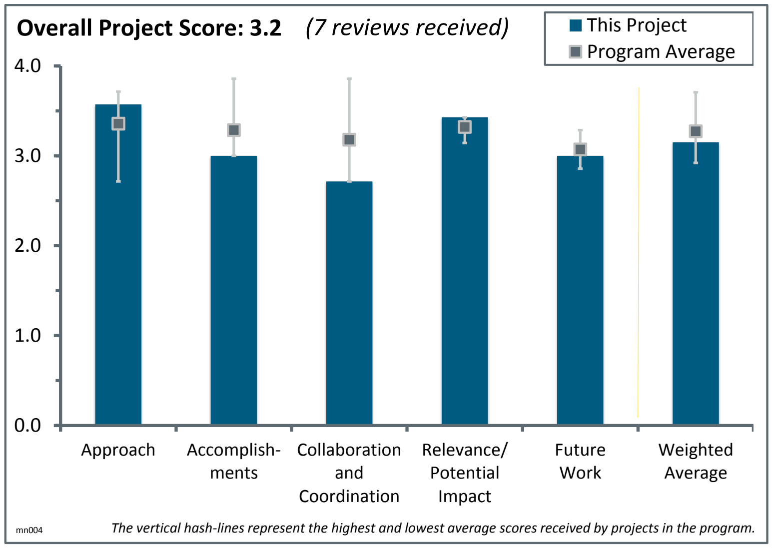
Recommendations for additions/deletions to project scope:

- Researchers should develop methods to quantify catalyst loading on the GDL after electrode coating application.
- Researchers need to further develop the relationships between defects and failures and cost.
- The project team should focus on diagnostics for manufacturing processes that have potential for implementation. For instance, segmented cell studies are not likely to be used in actual manufacturing environments. Decision points and go/no-go points should be established for each diagnostic along with decision metrics.
- The project team should consider other image processing algorithms to enhance information and increase signal to noise. For the reflectance, new information could be produced from these techniques with the same raw data. Suggestions may be two-dimensional auto correlations and cross correlations, among other software methods.
- The capacitance measurement could be interesting and useful. For the catalyst layers, researchers should refine this to reasonable carbon/ionomer ratios to see if they can distinguish anything for the small variations expected in manufacturing. They should also evaluate this as a function of RH; while RH does not vary at NREL, it certainly does in other places, such as Delaware, for example. The researchers should also evaluate the use of capacitance as a quick QC method for catalysts and catalyst supports. For example, they could compare MEAs with carbon black and graphitized carbon and vary the platinum loading on these. This technique might be more useful for catalyst QC (including Pt/C loading, carbon surface area, and catalyst loading on the MEA) than for I/C ratio monitoring. The modeling from LBNL looks interesting in terms of further utilizing the data and improving capabilities; however, it is unclear if and how this is being used. This analysis should be included in the demonstrations (such as at Ion Power). The researchers are concentrating on the visible spectrum of light for low-emissivity materials. They should try other wavelengths, such as ultraviolet.

Project # MN-004: Manufacturing of Low-Cost, Durable Membrane Electrode Assemblies Engineered for Rapid Conditioning

Colin Busby; W.L. Gore

Brief Summary of Project:



The overall objective of this project is to develop unique, high-volume manufacturing processes that will produce low-cost, durable, high-power-density, five-layer membrane electrode assemblies (MEAs) that minimize stack conditioning. Manufacturing processes must be scalable to fuel cell industry MEA volumes of at least 500,000 systems/year and must be consistent with achieving the U.S. Department of Energy (DOE) 2017 automotive MEA cost target. The products manufactured should be at least as durable as the MEAs produced using current manufacturing processes for relevant automotive duty cycling test protocols and must demonstrate power density greater or equal to that of MEAs made using the current processes.

Question 1: Approach to performing the work

This project was rated **3.6** for its approach.

- W.L. Gore is using a well-designed combination of modeling, experiments, and in-situ validation in the effort.
- The project is well organized with objectives to reduce MEA and stack costs, as well as increase the durability and power density of the fuel cell stack. The approach combines component, manufacturing optimization, and manufacturing process development with mechanical modeling. The very systematic and directed approach increases the probability for success.
- The approach is good. W.L. Gore is developing methods to produce fuel cell MEAs in volume. Little attention has been given to the integration of the gas diffusion layer with the MEA from W.L. Gore. It is unclear whether this will be a significant cost adder. W.L. Gore should also focus on roll-to-roll lamination and integration of all layers.
- W.L. Gore is developing a direct-coating process to reduce the cost of MEA fabrication. The W.L. Gore project addresses one of the critical barriers to fuel cell introduction—cost. The approach is logical and, if successful, is expected to lower the cost of MEA fabrication to the DOE target of $9/kW by 2017.
- The manufacture of low-cost, durable MEAs for high-volume processes is an important objective for DOE. The 2009 Process Waste Map helped inform efforts and project that up to a 25% reduction in MEA cost could be achieved. W.L. Gore identified six very relevant sub-objectives for this effort, including scalable manufacturing processes, reduction in stack break-in time, maintaining or improving power density for relevant automotive operating conditions, and making progress toward achieving DOE’s 2017 cost target of $9/kW MEA.

Question 2: Accomplishments and progress toward overall project and U.S. Department of Energy (DOE) goals

This project was rated **3.0** for its accomplishments and progress.

- The project appears to be on track to achieve its overall goal of reducing MEA cost by 25%.

- The accomplishments have been very good; W.L. Gore has met and/or completed the majority of the project milestones. New 5 µ and 10 µ reinforced membranes have been incorporated into the primary development path. Previously modeled process improvements have been incorporated that indicate a potential 25% reduction in high-volume, three-layer (3-L) MEA cost. There is good potential to meet automotive power density and durability targets. W.L. Gore is seeking a new partner for stack testing; UTC Power is no longer the test partner after its sale to ClearEdge Power, Inc.
- Process and material improvement work with identified cost reductions appears to be on track to exceed original targets. The cost analysis support from Strategic Analysis, Inc. (SA) is a helpful addition to the team.
- The project passed its go/no-go decision. The University of Delaware (UD) work on parametric analysis went from 50% to 80% complete. The fatigue analysis work went from 0% to 30% complete and will be finished this summer. The low-cost backer task is 100% complete this period. Cathode layer work is 95% complete this period and the principal investigator stated it "is as good as or better than current MEA." The reinforced membrane layer was demonstrated on a roll-to-roll process. Cost analysis went from 0% to 100% complete this period. Improvements have contributed to W.L. Gore going from 18 µ to developing a 5–10 µ MEA.
- W.L. Gore reports success in the development of low-cost backer material. W.L. Gore's data on direct-coated cathodes demonstrates performance consistent with control materials. W.L. Gore developed a state-of-the-art thin, durable, reinforced membrane that has enhanced performance at high current density characteristics. The thickness has been reduced by up to 75%. The membrane was operated more than 80,000 cycles in the relative humidity (RH) cycling test. UD has successfully modeled the strains in the single cell associated with RH cycling. The model allows selective evaluation of RH effects on either the anode or the cathode and can separate those factors. Working with SA, W.L. Gore has established a cost model for a 3-L MEA manufacturing process. W.L. Gore's analyses identify the three top cost uncertainties for the MEA: ePTFE cost, maximum coating speed, and ionomer cost. The analyses did not include the cost of the platinum group metal (PGM) catalyst and it must be assumed (and should be confirmed) that the cost of PGM was considered to be identical for all modeling conditions. The W.L. Gore/SA model predicts similar costs for the W.L. Gore MEAs and the 3M nanostructured thin film/membrane catalyst coated membrane. This is a surprising input and the catalyst loading for the two MEA structures was not identified.
- It is unclear what progress has been made since last year other than selecting a low-cost backer. The cost sensitivity shows the highest uncertainty due to ePTFE cost; it is not clear why this was not the focus. Further, there is no quantification of defect and fallout rates and their impact on cost sensitivity.
- This was, in essence, a repeat of the fiscal year (FY) 2012 presentation. It featured almost all of the same slides as FY 2012. The presenter showed the SA slides, which really feature SA modeling and not work from this project; otherwise, only two slides were new: slide 14 and slide 17. Even the summary slides were essentially exactly the same as last year. In addition, no modeling results were shown from the University of Tennessee, Knoxville, (UTK) and there was no indication of how these modeling results are being incorporated into this project.

Question 3: Collaboration and coordination with other institutions

This project was rated **2.7** for its collaboration and coordination.

- The team would have been outstanding had not UTC Power withdrawn from the fuel cell activity. Depending on the replacement, the team could be rated outstanding.
- Appropriate collaborations are in place, albeit the project needs a new partner for stack testing.
- Losing UTC Power is a concern and replacing it will be important to the overall success of project.
- The project is making good use of outside cost analysis and academic modeling inputs. A replacement needs to be identified for UTC Power for stack testing.
- Collaboration among the remaining partners has been focused on modeling process improvements as well as a manufacturing cost analysis of the new process. Once the roll-to-roll process for the 3-L MEA has been demonstrated, the modeling focus will shift to the optimization of the process. Interaction among the partners appears to be effective.

- W.L. Gore has provided data to SA, which has clearly used the data in its project. This is a benefit to the DOE Hydrogen and Fuel Cell Technologies Program (the Program); however, it appears that this was a one-way transfer of information. The presenter discussed modeling from UD and UTK; no results from UTK are evident. There is no evidence that the UD modeling results are being used in this project, aside from it being a stand-alone modeling project at UD.
- UD completed its mechanical model work and it appears W.L. Gore will utilize this model once the new process is stabilized. It will be validated with in-situ nitrogen cycling testing. UTK heat and water management modeling work is complete. The UD/UTK publication of modeling work can inform the general public/industry/academia beyond just W.L. Gore, which no longer has UTRC as a partner for stack testing. W.L. Gore is looking for a new partner. UD work identified integral response as a critical parameter for fatigue life of the MEA. The National Renewable Energy Laboratory (NREL) is working with W.L. Gore on in-line quality control (QC). This will help W.L. Gore and also provide NREL with another industry partner from which to evaluate the efficacy of the in-line QC tools being demonstrated.

Question 4: Relevance/potential impact on advancing progress toward DOE research, development, and demonstration (RD&D) goals

This project was rated **3.4** for its relevance/potential impact.

- The project addresses one of the key barriers in the Program—cost in high-volume manufacturing. If successful, the project is expected to meet the DOE target of $9/kW for a 3-L MEA product.
- The project is addressing a number of important barriers to meeting the Program's objectives.
- The development of a low-cost, high-performance MEA is critical for the polymer electrolyte membrane fuel cell system. The reduction of the stack break-in time to 4 hours would be a significant improvement.
- The new 3-L MEA process exploration is "leading the charge." W.L. Gore will utilize UTK/UD models during the future process optimization phase. New raw material formulations have been developed for the 3-L MEA process; it is not clear how these process improvements will translate beyond W.L. Gore. The cost model developed jointly with SA may help other U.S. MEA firms better understand cost drivers (e.g., 3M). This model identified the top three cost uncertainties as being ePTFE cost, maximum coating speed, and ionomer cost.
- The stated objectives are relevant; however, the approach and progress suggest that minimal impact has been made on addressing the barriers, contrary to the researchers' claim. The project goal was to reduce the cost of MEA manufacturing, not the material cost that would have been used in the MEA.

Question 5: Proposed future work

This project was rated **3.0** for its proposed future work.

- The proposed future work seems partially relevant.
- The remaining activities are appropriate and important (particularly scale-up and stack validation) to complete the project.
- The proposed future work is organized and consistent with the progress of the project. The schedule for the future work is reasonable and consistent with the capability of the team.
- Future work plans include optimization of the process once roll-to-roll fabrication is demonstrated and stack testing of the MEA package is performed. A new stack testing partner needs to be identified in short order to meet the project timeline.
- The proposed future work is consistent with what is appropriate, given the time and funding left on this project. Stack testing is important to validate results of this effort; however, W.L. Gore no longer has UTC Power to do this function.
- Future work includes MEA conditioning, process scale-up, and stack validation, which are all good tasks. However, no details were given on how the conditioning methodology will be conducted. Stack validation is probably straightforward and being conducted at UTC Power. It is unclear at what point in time the modeling at UD and UTK will be incorporated into this project; it does not seem to really appear in the project plans.

Project strengths:

- This is a well-designed effort that is exceeding expectations.
- This project features a strong project lead and partners.
- W.L. Gore’s expertise and innovation are strengths of this project. The project also features great modeling support by the partners.
- W.L. Gore’s experience and knowledge of membrane and MEA processing is the strength of this project. UD and UTK modeling expertise has been a significant help in predicting the impact of potential modifications to the manufacturing process.
- The strength of this project is the quality of experience W.L. Gore brings to the project. The MEA manufacturing capability of W.L. Gore is world class. The cost analysis capability of SA is well recognized.
- MEA costs are a large fuel cell cost driver and this effort addressed a multiple-percent cost reduction. The 3-L MEA process was informed by this effort, and when transitioned to production, it should provide significant cost and durability benefits. Inclusion of two academic institutions and NREL provides a greater degree of transparency, at least for the modeling and in-line QC efforts. MEA cost reduction efforts have made significant progress in recent years and this well-thought-out and well-executed effort has certainly been one of the key contributions.

Project weaknesses:

- There is a lack of focus on the areas of highest impact.
- There were not any visible inputs from NREL.
- The manufacturing improvements that W.L. Gore realizes from this project will likely apply only to W.L. Gore and not have any general applicability to the larger fuel cell developer community.
- It is not clear if the 25% reduction in high-volume 3-L MEA manufacturing costs will in fact be achieved. W.L. Gore has only indicated the potential for these reductions. In its defense, W.L. Gore is still in lab-scaled development of the 3-L MEA process.
- This project showed limited-to-no progress from FY 2012 to FY 2013; the cause of this is unclear. It featured almost all of the same slides as FY 2012. Even the summary slides were essentially exactly the same. For example, the 2012 Key Accomplishments include “lab scale development of the new 3-L MEA process is nearing completion,” whereas the 2013 Key Accomplishments include “Lab scale development of the new 3-L MEA process is nearing completion.”

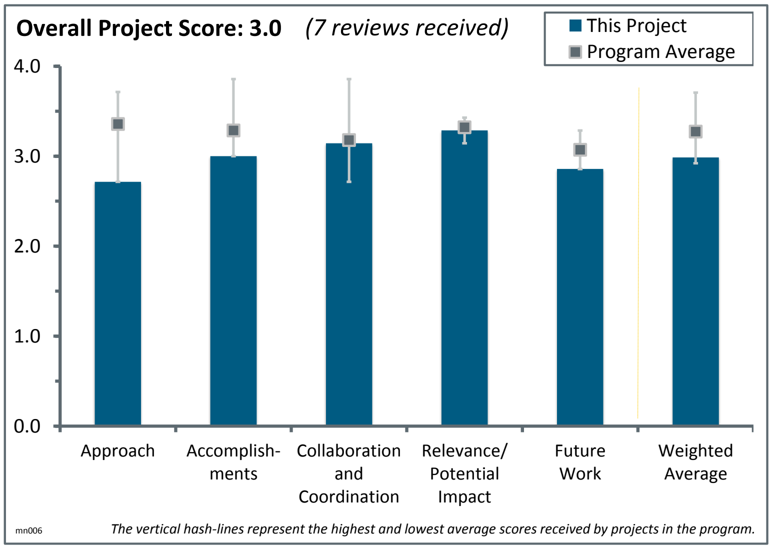
Recommendations for additions/deletions to project scope:

- The project team should conduct a cost model analysis of manufacturing MEAs through electrode coating on diffusion media approach.
- Researchers should seek to make the results of the project more broadly applicable to other MEA manufacturers.
- A “major” portion of the cost reduction of this project is moving to low-cost backing materials for the MEA. The project team should explore a reusable backing material instead of a replaceable, low-cost backing material. That would make for more substantial cost reductions. More clarity is needed on how the UD modeling and UTK modeling will actually be used. It is hard to know how the MEA/stack conditioning is going to work with no details. The researchers should show a comparison of performance direct coating versus the previous coating methodology.

Project # MN-006: Metrology for Fuel Cell Manufacturing

Michael Stocker; National Institute of Standards and Technology

Brief Summary of Project:



This project is focused on developing high-throughput optical process control methods to develop an accurate optical metrology infrastructure for semiconductors and nanomanufacturing. Using catalyst-coated samples provided by manufacturers with variations in critical parameters (e.g., platinum and platinum alloy catalyst loading) and including various types of defects characterized using standard methods, this project evaluates the sensitivity of optical scatterfield metrology to these parameters. A custom optical metrology tool, the Large Aperture Projection Scatterometer (LAPS), was designed and built to further understand the underlying measurement science and develop the necessary rigor.

Question 1: Approach to performing the work

This project was rated **2.7** for its approach.

- The general approach to the effort was appropriate. Additional input from other researchers at laboratories or in academia may have been useful.
- This year’s effort was primarily focused on the LAPS. The approach was direct in that the LAPS was used as it would normally be used for many material surfaces.
- The approach so far appears to have concentrated on samples with different nominal values. Researchers need to understand how the technique responds to spatial variability within a sample. Testing the LAPS under motion of the substrate is good to see, though 4 ft/min is quite slow. Researchers need to understand performance at higher speeds. Also, there does not appear to have been studies on gas diffusion electrodes (GDEs), though applicability is stated.
- The product is just now coming online, so it is difficult to fully assess the quality of the effort. The process it took to get to this stage is reasonable in regard to the approach.
- The project seeks to adapt existing equipment and procedures from semiconductors to fuel cell components and the development of new equipment. Some of the techniques do not appear useful on rough surfaces, such as 3M’s nanostructured thin-film (NSTF) materials. More discussion of the applicability and capability of the techniques would be beneficial.
- The development of optical scatterfield metrology for determination of fuel cell catalyst layer properties is well focused on proving that the technique measures real catalyst properties, adapting the technique from a small sample measurement to larger sample-size measurements, and expanding the capability of the technique to “high-speed” production lines. The project approach addresses the first two of these issues. The third issue, high-speed production applications, was not addressed in this presentation— even though it is a critical component to success. The researchers introduce spectroscopic ellipsometry to the project. Ellipsometry is a well-known methodology used by industry (measuring the protective coating on metal used in the manufacture of soup cans), and it is not clear why the project team incorporated an existing technology. This incorporation needs a better explanation.
- The project approach appears to be “trial and error,” as three iterations of instrumentation have been evaluated. A priori modeling, calculations, or other analysis on similar substrates may have helped narrow the choices.

Question 2: Accomplishments and progress toward overall project and U.S. Department of Energy (DOE) goals

This project was rated **3.0** for its accomplishments and progress.

- When this equipment is finally tested, this project's progress could be upgraded to outstanding.
- The project team has achieved a good proof-of-principle on 3M NSTF and W.L. Gore catalyst coated membranes (CCMs).
- The LAPS device has some potential, but there still seem to be some process control issues to resolve, particularly samples that are not perfectly flat. Additional work is required to develop a full array and there will certainly be unanticipated hurdles bringing several of these devices together in a coordinated way.
- The development of LAPS appears to provide a strong advance over the original optical scatterfield system. LAPS has larger spot size—approximately 10 mm wide and at least that long (the spot is an ellipse, with length dependent on the illumination beam angle of incidence with the sample). It is unclear how this will enable detection of small defects. Also, it would appear that this large of a spot size, unless the response time of the measurement is extremely fast, would lead to significant smearing of the data as the substrate is moving, in process.
- The success of increasing the spot size for large data averaging is very impressive. However, the size of the spot is still very small compared to the width/length of a membrane electrode assembly (MEA) manufactured in a continuous manner. Researchers need to explain how to use the "larger" spot. The demonstration of platinum in real samples is impressive. Still, the technique is limited to a local, albeit larger, spot. Measurement of the diffraction pattern is impressive and the need to develop the technique for a greater understanding of how to apply the diffraction pattern measurement is important.
- The LAPS diffraction pattern (slide 10) contains information corresponding to the platinum loading, but it also looks like a possible forward Mie scatter pattern with intensity lobes. The Mie scattering would correspond to particle size, shape, and diameter ratio with laser wavelength. The measurement shown is likely diffraction with a correlation to loading (mg/cm^2); however, it may be worthwhile to double check that the measurement is not also Mie scattering, indicating particle dimensions that could also correlate to Pt mass loading. Judging by the optics horsepower within the team, however, the presented conclusions are correct. The principal investigator is correct (summary slide) in that significant work remains, but other than time, there should be few showstoppers.
- The relatively low budget precludes major advances. Most of this year's progress has been equipment building and procedure development. Some preliminary data have been obtained on W.L. Gore (at line velocity) and 3M samples.

Question 3: Collaboration and coordination with other institutions

This project was rated **3.1** for its collaboration and coordination.

- The project team is working with leading fuel cell MEA manufacturers that could most benefit from this technique.
- W.L. Gore and 3M are providing samples and insights into possible defects and their characteristics.
- The selection of the two CCM partners to represent the range of materials expected for measurement was very astute.
- This project features good collaboration with industry in test samples and measurement needs. The addition of research and development with a metrology toolmaker could prove to be very valuable in this field, which is rich in nuances in optical techniques.
- Current collaborators consist of sample suppliers. Additional collaborators may be helpful in moving along the characterization and validation of novel measurement techniques.
- The level of involvement from 3M and W.L. Gore is not clear. Also, there does not appear to be any involvement by a GDE company, though the applicability of the technique to GDEs is stated.

Question 4: Relevance/potential impact on advancing progress toward DOE research, development, and demonstration (RD&D) goals

This project was rated **3.3** for its relevance/potential impact.

- This project fits directly with DOE's directive for advanced quality control (QC) analytical methods. Measuring Pt on both sides of the MEA independently is a great accomplishment.
- If proven, the techniques developed in this project are very important to fuel cell manufacturing.
- In-line, real-time measurement of fuel cell component properties will definitely facilitate commercialization.
- This reviewer hopes that the testing of this product is successful.
- There could be potential for in-line Pt loading measurement to lead to cost savings, but the linkage is not clear.
- The technique(s) need to be demonstrated to cover the full width of a continuous MEA manufacturing process. The technique provides a local spot on the moving MEA.
- Uniformity of the catalyst layer is of high importance to scale up and reduce costs of MEAs. However, other than the benefit of being able to measure two-sided coated membranes (which has not been shown), it is not clear how this technique offers significant benefits over X-ray fluorescence (XRF) technique, which has already been funded and demonstrated under another DOE manufacturing project. Also, the widespread applicability of this benefit is not clear; all GDE structures are one-sided, and this reviewer is not aware of any simultaneous two-sided coatings, although it is a possible direction.

Question 5: Proposed future work

This project was rated **2.9** for its proposed future work.

- The proposed future work is a logical progression of what has been done so far. Measurements on large formats at web-line speed will be attempted.
- Researchers have identified considerable work to reach the project's goals, and it seems appropriate.
- The proposed future work builds well on prior discoveries/shortcomings, but it is unclear whether the current third path will work. The presenter noted that 3M NSTFs act as an optical grating, which may present issues.
- The "robust model" that permits flexibility and normalization among different measurement samples is a good idea. The multiple-beam LAPS is a necessity. Perhaps there are acousto-optical or electro-optical methods for scanning that could do the same as the multiple-beam approach.
- Until researchers establish the applicability of the technique to different types of electrodes, higher rates of motion, and samples with spatial variability, the value of spending a lot of effort on modeling is unclear. The objective of measuring membranes is unclear; it is not clear whether this is part of understanding the electrode measurement or a new measurement. The need for membrane measurement is also unclear—other techniques have been developed and demonstrated under DOE manufacturing funding. The plan to look at higher speeds is a good direction to pursue.
- The researchers are missing the application point and are more concerned with the development of a robust model for analysis. The researchers should consider how they can apply this technique, assuming the analytical methodology is complete (which it is not). They want to answer the analytical methodology question without addressing the application question.

Project strengths:

- The team is persistent in overcoming issues, able to measure both sides of CCM independently, and can meet the target data acquisition rate.
- Strengths of this project include NIST's expertise and the high-quality skills of the people working on this project.
- The project seeks to address a key QC need: Pt loading. The metrology capabilities at NIST are world class.

- This project features a great capability in advanced optical methodology; probably one of the top two or three in the world.

Project weaknesses:

- The slides are too dense.
- It is unclear how surface changes due to normal manufacturing variation will impact measurement, and whether this variation can be accommodated in the processing.
- The potential cost savings resulting from the techniques under development are not clear. There does not seem to be sufficient time before the project ends to complete the identified future work.
- The researchers need to get better input from industry. The question of how to apply the technique needs to be answered. If the technique cannot be applied, it is not clear why it should be developed.
- Overall, the benefits of this technique compared to XRF are not clear and possibly not compelling. The ability to measure two-sided coatings simultaneously is definitely something XRF cannot easily do, but the need for this benefit—i.e., how many companies actually will need a simultaneous measurement—is not clear and does not appear to be very widespread. The project team has not looked at defects yet.

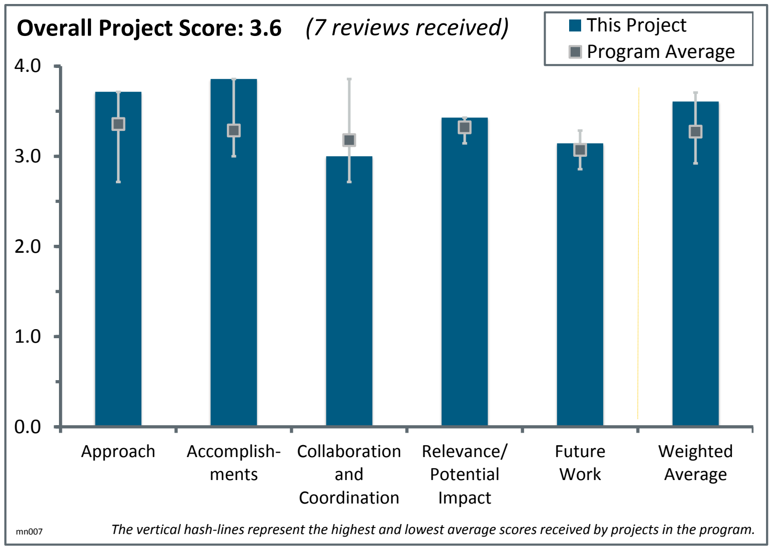
Recommendations for additions/deletions to project scope:

- Researchers should look into Mie scattering and some acousto- and electro-optical methods for flexibility.
- The future work should include testing of GDE samples.
- The project team needs to add a strong modeling component to understand whether the commonly found range of morphological differences will impact the signal/noise ratio. The team may have to focus on fixturing to keep moving web in a flat orientation to avoid spurious signals.

Project # MN-007: High-Speed, Low-Cost Fabrication of Gas Diffusion Electrodes for Membrane Electrode Assemblies

Emory De Castro; BASF

Brief Summary of Project:



The objectives of this project are to (1) reduce costs in fabricating gas diffusion electrodes (GDEs), focusing on GDEs used for combined heat and power (CHP) generation; (2) relate manufacturing variations to actual fuel cell performance to establish a cost-effective product specification within six-sigma guidelines; and (3) develop advanced quality control (QC) methods to guide realization of these two objectives. BASF also strives to further decrease costs with paper substrates, single-pass applications, and by exploiting stable links to promote platinum thrift.

Question 1: Approach to performing the work

This project was rated **3.7** for its approach.

- The project systematically addresses the barriers to GDE production by building on past accomplishments and progress.
- The project has been making steady progress since 2009. The awardee laid out and implemented a methodical approach to addressing the problems in the statement of work (SOW). The approach was well thought out to achieve the goals, and the methodology has allowed the researchers to perform thorough root cause analysis for identifying issues for improvement of the GDE. An additional task was even added in 2012 and the awardee has been making progress on this task. Goals have been set and achieved throughout the project.
- Membrane electrode assemblies (MEAs) are a big cost driver in the production of affordable fuel cells. The GDE represents an important part of the MEA, and as stated, there is a lack of existing high-volume MEA processes. This approach is aimed directly at improving GDE production so that it is high speed and low cost. Having a GDE supplier (BASF) leading this effort makes it more likely that the resulting technology will transition. This effort has already led to new product development. The objective during this period of further decreasing cost with paper substrates and single-pass applications—including exploiting stable inks to promote platinum thrift—is considered a sound approach for this stage of the project. Specifically, BASF hoped to increase the solids content of ink without a loss of stability.
- This project is working to develop online diagnostics for QC that have good speed and are non-destructive.
- This project concentrates on the various aspects of throughput improvement—ink formulation, ink coating, maintaining quality, increasing width, and the effects of substrate.
- The only information presented on the approach was a list of milestones. It is assumed that the approach was valid because all of the milestones were met.

Question 2: Accomplishments and progress toward overall project and U.S. Department of Energy (DOE) goals

This project was rated **3.9** for its accomplishments and progress.

- Milestones were met. Throughput (line speed) was significantly increased; the paper gas diffusion layer (GDL) cost goal was met; and variability now meets an acceptable level.
- This project has developed a technique that has led to a reduction in manufacturing variability.
- This project features impressive improvement in platinum variation, reducing costs and improving performance.
- The awardee has made significant progress on all of the goals of the project. Researchers have met many of the goals to reduce the costs of the GDE. They have accomplished ink cost savings and obtained performance gain of the GDE. They have worked with stabilizing the ink and switched from carbon cloth to carbon paper for cost savings. The project has reduced the base material costs. A new task was added in 2012 to achieve performance gain and reduce precious metal (PM) content, and both of these goals are being accomplished.
- Researchers have met or exceeded project throughput targets, leading to reduction in labor costs by 75%. It would be useful to see the cost breakdown between labor, machinery, and materials. Although a reduction in platinum loading was not an objective of the project, new inks, processes, and QC allowed less platinum to be coated and resulted in greater performance.
- The creation of a six-sigma specification for the production of GDEs will reduce the variability and minimize yield loss. This develops a control zone for the manufacturing process by carefully monitoring properties of manufacturing lots and results in performance enhancement. The use of carbon paper substrates with the development of a controlled micro-porous layer (MPL) increases the performance characteristics of the fuel cell. For a high-temperature membrane system, the MPL flattens the catalyst layer, but the high temperature precludes the benefits of water removal. The presenter did not report whether the MPL influences acid transport out of the high-temperature membrane. The catalyst thrift program of less than one year demonstrated savings in the catalyst. This is an outstanding result for a small effort.
- Task 2, subtask 2.2, creating six-sigma specification, was largely met. Reaching six-sigma in one year was considered a “'stretch goal.” BASF is currently at approximately three sigma, which is much further along than it was in the past, when high-performing material was hand selected or “cherry picked.” The fact that this new method accounts for variability as well as performance, is a key accomplishment. The principal investigator (PI) gave an example of variability differences detected that led to the discovery of a heating coil that was failing and causing the excess variability. For Task 5, carbon paper substrates, BASF scaled single-pass MPL to production coating machining at 1/2 width. This is a 28% cost improvement compared to using carbon cloth (based on 3,000 5-kW systems). For the new task, exploring PM thrift, BASF successfully demonstrated a 30% cost reduction of PM at the anode with materials made on a production-scale coating machine. The PI commented that “this was impossible with pre-program inks.” The PI in 2012, however, had stated that this new task would address both anode and cathode coating. It does not appear any progress was made on the cathode. The levels of platinum used in this effort were significantly more than what would be anticipated in production.

Question 3: Collaboration and coordination with other institutions

This project was rated **3.0** for its collaboration and coordination.

- BASF is working with a leader in high-temperature membrane fuel cells, which is a great benefit. The National Renewable Energy Laboratory (NREL) collaboration can benefit manufacturing QC.
- BASF shows good collaboration with NREL on GDE defect detection (see project MN-001). ClearEdge Power will evaluate materials for use in micro-combined-heat-and-power (micro-CHP) applications at the full stack level. This project is applicable to a minimal number of U.S. firms, namely BASF and its customer base.

- The project partners have been working together to meet the goals. NREL provided GDEs for part of the research. Currently, ClearEdge Power is evaluating the PM levels to accomplish the new goal established in 2012. There is good coordination and teamwork throughout the project.
- This appears to be mostly a BASF effort.
- A national laboratory provided samples with known defects to aid in the development of advanced defect detection techniques. MEA samples with thrifted platinum were sent to a micro-CHP stack integrator for stack testing. Results were not presented on the slides.
- The main collaboration in this project appears to be XOS; otherwise, there are limited collaborations.
- Case Western Reserve University (CWRU) and XOS are listed as partners, but not mentioned at all in the presentation. The XOS X-ray fluorescence (XRF) system has already been installed—it is unclear if there are any relevant improvements or future work. The status of the CWRU modeling work is unclear. NREL and ClearEdge Power were listed as collaborators, but nothing was shown regarding outputs from their collaboration. There are no stack results yet.

Question 4: Relevance/potential impact on advancing progress toward DOE research, development, and demonstration (RD&D) goals

This project was rated **3.4** for its relevance/potential impact.

- This research is important for the fuel cell community and aligns very well with the DOE Hydrogen and Fuel Cell Technologies Program goals. Because of this research, a new product was brought to market, supporting DOE's goal to help the U.S. economy/industry.
- Reducing PM content is key to the cost competitiveness of fuel cells.
- A new MEA product was released (March 2012), demonstrating the positive impact of this project. High-temperature fuel cells are an important part of the fuel cell portfolio and are critical to micro-CHP applications. ClearEdge Power is the only U.S.-based micro-CHP manufacturer. This effort will directly impact its systems. The XRF automated defect detection technique will provide for greater throughput at a larger scale of MEA manufacturing, and with NREL involvement, it could lead to industry-wide adoption.
- The project is very beneficial to micro-CHP and other high-temperature polymer electrolyte membrane (HTPEM) applications. In general, HTPEM has not been a strong focus of the DOE Fuel Cell Technologies Office, but it is work that is beneficial to the industry and overall market development/cost reduction.
- This is an important project for demonstrating manufacturing cost reductions for fuel cells. DOE should test to determine if the techniques developed in this project translate to low-temperature polymer electrolyte membrane manufacturing.
- This project works to improve the manufacturing and quality of GDEs for CHP. There are, however, only a limited number of developers who are looking to manufacture GDEs. If this project could be expanded beyond GDEs, it would have a greater impact in the fuel cell community.
- The presenter said very little about the applicability of the techniques to other fuel cell types.

Question 5: Proposed future work

This project was rated **3.1** for its proposed future work.

- The proposed work of scaling the catalyst coating to production level demonstrates efforts to build on work that was accomplished. Gains made at the pilot scale will be translated to actual systems. New work beyond this effort will include improvement toward more corrosion-resistant carbons.
- The project shut down is well organized. The researchers are not trying to push the project beyond reasonable limits.
- The presenter only briefly touched on future work, but the results seem to indicate that more development of these products could be achieved in the future. The project is closing out in June 2013.
- The project is virtually over. Scaling to production level is planned. After the project is completed, the knowledge should be applied to corrosion-resistant carbons.
- This project is essentially over; therefore, the future work does not seem relevant for much discussion.
- There are only a few months left in the project.

- The proposed next steps are in line with the discussed project pathway; i.e., applying advancements made on non-woven substrates to woven substrates, for potential additional cost savings. It would be useful to see the projected cost breakdown/savings of going to woven and an expected performance/durability comparison. Also, it is unclear whether the process will need to be modified to handle woven, which is more brittle and typically thinner.

Project strengths:

- This was a strong effort from start to finish.
- BASF is the major leader in HTPEM GDL manufacturing. The team brings in many years of experience in manufacturing.
- This project addresses key areas of MEA fabrication. Significant (30%) cost reductions have been achieved. The effort to move from expensive carbon cloth to paper-based materials appears to have been successful.
- The very structured nature of this project allowed it to meet the goals laid out in the SOW. This is a well-performed research project with strong results. There was good collaboration with partners. Overall, it was a very good project that was well run and well executed.
- The project addresses key process variables in a structured way to improve throughput. Advancements leveraged corporate BASF competencies beyond the fuel cell group. BASF is a leader in HTPEM MEA production, so it is valuable for DOE to support the advancement of this technology.

Project weaknesses:

- The results and benefits of collaborations (other than XOS) are not very clear. Neither the stack data validating performance nor the modeling results were shown.
- Researchers did not address the major issue of acid movement and how to manufacture components to control acid movement.
- It appears that the project team’s technique shows +/- 0.4 g Pt/m^2, which is 0.04 mg Pt/cm^2. This is still a rather large variation, considering that the DOE target loadings are moving toward 0.125 mg Pt/cm^2 (i.e., this would be a 33% error in loading). The technique has a large depth of penetration; therefore, it appears that it is only applicable to one-sided MEAs or GDEs.

Recommendations for additions/deletions to project scope:

- It would be nice to see that the results of this project help the overall community more.
- The project is wrapping up in June 2013 and is on track.