# VI.7 Cause and Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance

#### Eric Stanfield

National Institute of Standards and Technology (NIST) 100 Bureau Drive, MS 8211 Gaithersburg, MD 20899-8211 Phone: (301) 975-4882 Email: eric.stanfield@nist.gov

#### DOE Managers

HQ: Nancy Garland Phone: (202) 586-5673 Email: Nancy.Garland@ee.doe.gov GO: Jesse Adams Phone: (720) 356-1421 Email: Jesse.Adams@go.doe.gov

#### Contract Number: DE-EE0001047

Subcontractor: Los Alamos National Laboratory (LANL), Los Alamos, NM

Project Start Date: October 1, 2007 Project End Date: September 30, 2012

# Fiscal Year (FY) 2012 Objectives

- Fabricate and verify replacement experimental plate 5C in order to conclude back pressure sensitivity experiment.
- Complete statistical analysis of polarization curve measurements for each of the 10 experimental cathode plates.
- Incorporate NIST Neutron Center for Neutron Research (NCNR) imaging experiment to visually quantify differences in water management between poor and optimal performing experimental plates, as well as, the nominal design plates made with minimal dimensional variations.

### **Technical Barriers**

This project addresses the following technical barriers from the Manufacturing R&D section of the Fuel Cell Technologies Program Multi-Year Research, Development and Demonstration Plan:

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

## Contribution to Achievement of DOE Technology Manufacturing R&D Milestones

This project will contribute to achievement of the following DOE milestones from the Manufacturing R&D section of the Fuel Cell Technologies Program Multi-Year Research, Development and Demonstration Plan:

• Milestone 2.3: Develop manufacturing processes for polymer electrolyte membrane bipolar plates that cost <\$3/kW while meeting all technical targets. (1Q, 2018).

### FY 2012 Accomplishments

- Completed a statistical analysis of LANL's polarization measurements obtained from each of the ten experimental plates.
- Submitted and received approval for a NIST NCNR beam experiment to visualize the water content within the channels for a subset of the experimental plates representing the best, worst, and nominal designs.
- Fabricated and dimensionally verified the replacement experimental plate 5C to enable completion of the backpressure sensitivity experiment.
- Fabricated and dimensionally verified all experimental plates and end plate hardware to facilitate the NCNR beam experiment (material substitutions were required for optimal imaging).
- Acquired the assistance of Dr. Jeffery Allen from Michigan Technological University who is an expert in two-phase flow with specific application to fuel cells to help in the understanding of the underlying physics and to provide guidance in the development of the imaging experiment objectives.



# Introduction

This project originated conceptually as a result of a workshop organized by the Center of Automobile Research and NIST in December of 2004, where industry bipolar plate manufacturers identified a need for engineering data that relate geometric bipolar plate tolerances to fuel cell performance. This need is in response to pressure from fuel cell designers to produce lower cost plates that potentially require quality related trade-offs, by plate manufacturers, to achieve desired cost targets. Manufacturers questioned the relevance of stated tolerances on dimensional features of bipolar plates and expressed a desire for published engineering data relating performance and dimensional quality of the plates. This project was proposed to address this need and was partially funded through the NIST Advanced Technology Program Intramural Competition for a period of three years (FY 2005-FY 2007). The Advanced Technology Program funding was also intended to aid NIST with the development and validation of a single-cell testing laboratory. In 2008, DOE recognized the potential value in the outcome of this project, subsequently adding it to their portfolio of fuel cell manufacturing related projects and provided funding in an attempt to ensure successful completion.

This experiment focuses on introducing very precisely controlled dimensional variations within the flow field channels of the cathode side flow field plate where reactiongenerated water often interferes with the supply of oxygen (via air). Through the fabrication of multiple cathode plates with specific dimensional variations with different magnitudes and through substitution of these plates in a fuel cell, we hope to observe measureable differences in the output (performance) of the fuel cell. Using these differences, we then hope to statistically determine which single factors or two-factor interactions are most important. The most challenging aspect of this experiment comes from choosing the proper factors to vary and their corresponding magnitudes along with controlling and limiting all other sources of variability, such as those related to materials or the experimental testing procedure

### Approach

Using a statistically based design-of-experiments (Figure 1), NIST fabricated experimental "cathode" side flow field plates with various well-defined combinations of flow



#### Polymer Electrolyte Membrane (PEM) Fuel Cell

FIGURE 1. Concept - Reference Single Cell and NIST Fabricated Cathode Flow-Field Plates

field channel dimensional variations (Table 1). Then through single-cell fuel cell performance testing, using a well-defined protocol, NIST quantified any performance effects and correlated these results into required dimensional fabrication tolerance levels.

#### **Results**

Prior work included several performance testing protocol revisions necessary to achieve improved repeatability and to highlight the mass transport differences between the experimental cathode plates. Protocol optimization was validated through repeatability testing and results were commensurate with our benchmarks. LANL evaluated the performance of all the experimental plates by integrating each one in the reference single cell full cell then running multiple polarization curves. The DOE Annual Merit Review process combined with industry interactions have been invaluable with regards to the incorporation of additional experiments that validate the results over a broader range of applicability (back pressure sensitivity experiment) and the addition of alternative means to ensure intended channel perturbations are indeed the cause of the observed variability (neutron imaging experiment). All of the above work is described in detail in references [1-3] with the exception of the neutron imaging experiment, which is discussed in the following section.

In 2011 and 2012 we have focused on concluding the back pressure sensitivity test by fabricating and dimensionally verifying a replacement plate 5C and finalizing the project outcome with a rigorous statistical analysis of all the measurement results. During this time we received feedback from our peers regarding potential causes for operational differences that could be unrelated to the intended variations and that a lack of verification could limit the usefulness of the results. In response, we decided to explore the opportunity to perform neutron imaging on a subset of these experimental plates that represent the largest dispersion in output so that we can better understand the water management dynamics within the flow field channels. The submitted proposal was awarded beam time allotment in January 2012 and is currently scheduled for mid-September 2012. In preparation for this experiment, several material substitutions were required to optimize imaging; these included fabrication of the endplates from 6061 aluminum to replace the current stainless steel plates and fabrication of the experimental plate subset from a pure carbon material (POCO AXF-5Q) versus the same material but with a hydrocarbon material impregnation (POCO AXF-5QCF). The plate material substitution added additional processing and expense caused by the porosity of the plates needing to be sealed to prevent water uptake. Following the flow field fabrication at NIST, we are having POCO post-process the plates using their proprietary purifying and pyrosealing process. This process does not introduce any new materials

	2 <sup>4-1</sup> Fractiona	I Factorial Des	ign with replic	ated center	point (k=4,n=	10)			
	Sidewall Straightness	Sidewall Straightness	Bottom Straightness	Sidewall Taper					
	Amplitude	Phase	Amplitude			Sequence		Drawing	g
Part	X1	X2	<u>X3</u>	X4	Machining	Measuring	Perf. Testing	Cross-Section	Тор
9	0(25µm)	0(90)	0(25µm)	0(5)	1	1	1		
3	-1(0)	+1(180)	-1(0)	+1(10)	2	2	2		\$\$   {}
2	+1(50µm)	-1(0)	-1(0)	+1(10)	3	3	3		<b>\$\$ \$ \$</b>
4	+1(50µm)	+1(180)	-1(0)	-1(0)	4	4	4	1	\$\$   {}
8	+1(50µm)	+1(180)	+1(50µm)	+1(10)	5	5	5		\$\$   {}
5	-1(0)	-1(0)	+1(50µm)	+1(10)	6	6	6		
7	-1(0)	+1(180)	+1(50µm)	-1(0)	7	7	7		
10	0(25µm)	0(90)	0(25µm)	0(5)	8	8	8		
6	+1(50um)	-1(0)	+1(50µm)	-1(0)	9	9	9		<b>\\\</b>
1	-1(0)	-1(0)	-1(0)	-1(0)	10	10	10		

**TABLE 1.** Design of Experiment 2<sup>4-1</sup> Fractional Factorial Design...4 Parameters, 2 Levels, and Replicate Center Point

to the composition matrix. All end plates and experimental plates have been fabricated, verified, and are awaiting pyrosealing scheduled for July 2012. We decided that fabrication of only one 5C plate made from the alternative POCO AXF-5Q material, rather than two plates one from each material, would be necessary to meet all our objectives. With this decision, we intend to use this plate to confirm that the material substitution still produces the same polarization curve results at 25 psig as the original 5C plate. Then, assuming successful correlation of results, we will then use this plate to complete the back pressure sensitivity testing and for the neutron beam imaging experiment.

Though statistical analysis of the data did not yield significant conclusions, our statisticians classified them as strongly suggestive. To be statistically conclusive the F-distribution cumulative density function or probability needed to be 95% or greater for a main factor. Unfortunately, due to the fractional-factorial nature of the experiment this statistic could not be determined for the two-factor interactions because of their "confounded" nature. The fractional-factorial design-of-experiments alternative was chosen because it reduced the number of experimental plates needed and the associated testing. A further implication of this decision is that if a two-factor interaction is significant, there are not enough data to determine if the most important interaction, in the case of our experiment, is 1 & 2 or 3 & 4 or a combination of 1 & 2 and 3 & 4. The ranked order of significant factors and interactions is shown in Table 2 below. It is important to point out that (1) 3 and 4 factor interactions were not tested and (2) The "effect" in the table and subsequent plots represents the voltage difference multiplied by 100 for visualization purposes.

**TABLE 2.** Statistical Analysis Summary - Single Factors and Two-Factor

 Interaction Ranked by Order of Importance

Ranked Order										
Factor	Effect (V*100)	Rel. Eff %	Fcdf Stat %							
1 <mark>2 + 3</mark> 4	18.59			*						
3	16.17	106	82.4	**						
1	15.28	101	79.4	**						
4	6.02	40	36.1							
14 + 23	-5.67									
2	-3.71	24	22.6							
13 + 24	-2.74									

Although the first three results were not statistically significant, the main factors were not far off and thus can be classified as of strongly suggestive. Figures 2a and 2b show different graphical representations of the statistical results. In Figure 2a each factor and interaction has a corresponding box plot: the greater the slope, the more significant the factor or interaction is. The confounding for the two-factor interactions can be seen by identical plots for three different two-factor interactions. Figure 2b is more intuitive and clearly shows the experiment included a center



FIGURE 2. Statistical Plots

replication point to test for consistency, one of the intended characteristics of the design of experiments. Plates 9 and 10 were identical with all factors set mid-range, performing as expected, with both producing nearly the same output, with an output ranking near the mean relative to the other plates.

We consider the statistical analysis described as initial and we will continue to find new published methods to analyze the data to create more definitive conclusions.

# Conclusions

• The duration of this project has been longer than anticipated but the modifications that have been adopted, experiments added, and the care that has been taken to ensure every detail has been considered will hopefully go a long way towards ensuring confidence in the conclusions.

- Although not statistically conclusive, the analysis of the results based on the design of experiments strongly suggests that precisely controlled complex dimensional variability improves water management and yields improved performance. This is evident from the most important single factor and two factor interactions being side wall straightness, bottom straightness, and the interaction of sidewall straightness and phase of the side wall straight (variation in width). Applying intuition to unravel the confounded two-factor interactions might suggest the interaction of factors 1 and 2 being more important than 3 and 4; however it does not eliminate the possibility of three- and four-factor interactions.
- The preliminary statistical analysis dictated the need for a microfluidic two-phase flow expert; however, in hindsight, the project would have benefited greatly with this addition during the design stage of this project.

### **Future Directions**

- Complete all preparations for the imaging experiment, verify that with the substitute plate material the experimental results correlate with those initially obtained, and with the assistance of Dr. Allen finalize the imaging protocol.
- Complete the back pressure sensitivity experiment through testing at LANL using the replacement experiment cathode plate 5C.
- Continue working with Dr. Allen to obtain a better understanding of the physics that explain the results obtained.
- With the assistance of the NIST Statistical Engineering Division continue researching the application of different statistical analysis based data exploration techniques in an attempt to reveal the most rigorous conclusions. Incorporate, if possible, the physics based understanding in the statistical evaluation of the results as prior information.
- Deliver a detailed publication to disseminate our results to the fuel cell industry.

# Disclaimer

Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

# Acknowledgements

The work detailed in this report would not have been possible without the contributions of the following, all are NIST employees

unless designated otherwise: Tommy Rockward (LANL), Jeffery Allen (Michigan Technical University), James Filliben, Ted Doiron, Daniel Hussey, David Jacobson, Brian Pries.

## FY 2012 Publications/Presentations

**1.** E. Stanfield, "Cause and Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance," DOE Annual Merit Review Proceedings, MN011, May 16, 2012, http://www.hydrogen.energy.gov/pdfs/review12/mn011\_stanfield\_2012\_p.pdf

2. E. Stanfield, "Cause and Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance," 2011 DOE Hydrogen and Fuel Cells Program Annual Progress Report, November 2011, http://www.hydrogen.energy.gov/pdfs/progress11/vi\_6\_ stanfield\_2011.pdf

#### References

1. E.Stanfield, "Metrology for Fuel Cell Manufacturing," 2008 DOE Hydrogen and Fuel Cells Program Annual Progress Report, November 2008. http://www.hydrogen.energy.gov/pdfs/progress08/ vi\_3\_stanfield.pdf

**2.** E. Stanfield, "Cause and Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance," 2010 DOE Hydrogen and Fuel Cells Program Annual Progress Report, February 2011. http://www.hydrogen.energy.gov/pdfs/progress10/vi\_6\_stanfield.pdf

**3.** E. Stanfield, "Cause and Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance," 2011 DOE Hydrogen and Fuel Cells Program Annual Progress Report, November 2011. http://www.hydrogen.energy.gov/pdfs/progress11/vi\_6\_stanfield\_2011.pdf