

DOE-EERE Durability Working Group (DWG) Meeting
Thursday, October 13, 2011
Boston, MA
Meeting Notes
D. Myers and R. Borup

Meeting Agenda

- 6:30 pm** **Welcome and introductory comments**
 Debbie Myers (Argonne) and Rod Borup (Los Alamos) – co-chairs
- 6:40-7:45 pm** **Descriptions and summaries of results of modeling efforts in durability projects**
 Dane Morgan, University of Wisconsin-Madison
 Srikanth Arisetty, ANL
 Adam Weber, LBNL
 Shanna Knights, Ballard
 Sergei Burlatsky, UTC Power
 Jean St-Pierre, University of Hawaii – Manoa
- 7:45 pm** **Discussion of Accelerated Stress Tests: Start-Up/Shut-down and Membrane mechanical stress – relative humidity cycling**
 Rod Borup
- 8:15 pm** **Follow-up Discussion of Accelerated Stress Tests Proposed at the Spring, 2011 DWG meeting**
 Debbie Myers, Summary of Protocols
 KC Neyerlin, NREL, “Durability Protocols for non-Carbon Supports”
- 8:25 pm** **Wrap-up and action items**
 Debbie Myers and Rod Borup
- 8:30 pm** **Adjourn**

Summary of progress on action items from Spring, 2011 DWG meeting

1. List and prioritization of degradation effects, Presented by R. Borup
Action items: Develop failure mode analysis approach using a tree structure. Set up Wiki site for input. Assigned to D. Myers, D. Harvey, and M. Gummalla
Progress as of 10/11: Action item has been re-directed to be based on document distributed by G. Tsoitridis after the European Union Durability meeting which summarizes degradation modes and prioritizes research (see note below from the Fall, 2011 meeting).
2. Air-Air start-up protocol, Presented by G. James, Ballard
 - Action item: R. Borup and R. Mukundan (LANL) to test cells using proposed protocol and provide results and comments to Ballard for comparison with Ballard’s results.
 - Progress as of 10/11: See R. Borup’s presentation from this meeting for a detailed description of the progress.
 - Summary of progress:
 - The start-up/shut-down protocol was primarily developed by Ballard and was presented by G. James at the Spring, 2011 DWG meeting. This protocol was circulated to the DWG for comment.
 - R. Borup presented this protocol to the Fuel Cell Technical Team on Sept. 14, 2011. Tech Team comments were:

- ✓ Anode/Cathode flow must be constant and valve switched (e.g., using a gas chromatograph valve) otherwise mass flow controller response times vary and will make duplication impossible
- ✓ Test stand inlet line lengths vary and will be impossible to duplicate lab-to-lab measurements
- ✓ Recommend direct CO₂ measurements (NDIR)

LANL is collaborating with Nancy-Université (Lorraine, France) on investigating start-up/shut-down. Nancy-Université is using a segmented cell to determine local current densities during start-up. Their goal is to find operating points which could minimize the deteriorations during start-ups without using any external devices.

3. Accelerated stress test for oxide supports, Presented by S. Kocha, NREL

- Action item: V. Ramani (IIT), E. Brosna (LANL), and Nissan Technical Center North America (NTCNA) to test Pt/C and their support materials using proposed durability protocol for comparison with S. Kocha's results.
- Progress as of 10/11: See K.C. Neyerlin's presentation from this meeting for details on the progress on this action item.
- Summary of progress:
 - NTCNA evaluated support durability using Nissan, NREL and DOE (support protocol – 1.2 V hold) protocols in rotating disk electrode (RDE) experiments for Tanaka baseline catalyst (TEC10E50E) in 0.1M HClO₄ electrolyte.

Nissan protocol: 60°C, 500 mV/s triangle wave, 1.0 to 1.5 V, 5000 cycles.

NREL protocol: Room temperature, 100 mV/s triangle wave, 1.0 to 1.6 V, 6000 cycles.

DOE protocol: Room temperature, 1.2 V Hold, No cycling

NTCNA results show that the Nissan and NREL protocols show similar Pt electrochemically-active surface area loss, but Nissan protocol only takes ~8 hrs and NREL protocol takes ~24 hrs. Advantage of NREL protocol is room temperature operation which is attractive from a safety standpoint and that there's less poisoning due to decomposition of HClO₄ to Cl⁻ and less loss of electrolyte. The DOE protocol requires ~400 hrs and results in lower loss of ECA, oxygen reduction reaction area specific and mass activity.

- NREL applied their protocol to the oxide supports under development in their laboratory and found their supports to be more resistant to ECA loss than standard carbon supports.
- LANL has started using NREL protocol to test the durability of their nitride supports. They also commented that one protocol may not be appropriate for all support materials and that the NREL protocol should specify ink formulations and quantity deposited on electrode.

4. Electrochemical equivalent of start-up/shut-down, Presented by R. Atanasoski (3M)

Action item: Solicit opinions on upper potential limit for 5 mC/cm² hold

Progress as of 10/11: D. Myers and R. Atanasoski have had multiple conference calls and the protocol has been modified to allow upper potential limit to reach a maximum of 1.6 V and to increase frequency of potential sweep and hold down to 0.65 V to every 10 cycles rather than every 50 cycles.

5. Applicability of aqueous electrolyte testing to MEA test results, Presented by D. Myers

Action item: Draft aqueous RDE protocol for PGM catalysts

Progress as of 10/11: A conference call was held between P. Atanassov, R. Atanasoski, and D. Myers regarding protocols. Draft protocol was written by D. Myers and has been circulated to R. Borup and S. Kocha for comment. Comments have been received from S. Kocha. Draft protocol will be circulated to entire DWG.

Notes from Fall, 2011 DWG meeting

- A comment was made by R. Borup that a new membrane durability protocol may be necessary that addresses both mechanical and chemical stresses (e.g., combination of humidity cycling and potential protocols)
- Need a summary of all new and developing membrane durability protocols.
 - Action item: assembly of a summary of protocols. Assigned to R. Mukund by R. Borup
 - Action item: **all** those having protocols are to send them to R. Mukund
 - DuPont protocol – **R. Perry** (DuPont)
 - U.S. Fuel Cell Council protocol – **J. Kopasz** (ANL)
 - Fuel Cell Tech Team protocol – **C. Gittleman** (GM)
 - General Motors protocol – **C. Gittleman** (offered by K. O’Leary, GM)
 - Fuel Cell Commercialization of Japan – **J. Kopasz** (ANL) and **C. Gittleman**
 - **Action item: R. Borup, R. Mukundan, J. Kopasz, and C. Gittleman** to have a follow-up conference call.
- Comments on presentation of catalyst, support, and start-up/shut-down protocols:
 - Platinum group metal aqueous electrolyte protocol
 - Need to specify electrolyte volume and electrochemically-active surface area
 - May need to cycle to lower potential limits to capture degradation of non-noble component of PGM-base metal alloy catalysts
 - Non-platinum group metal aqueous electrolyte
 - Why is an upper potential limit of 1.1 V used as opposed to 1.0 V limit used currently for fuel cell catalyst cycling tests (D. Myers)?
 - ✓ Answer from P. Atanassov: Open circuit potential of non-PGM catalysts in fuel cell testing can be higher than 1.0 V
 - Catalyst support protocol
 - May miss reduction of oxide support when not cathodic potential cycling limit is too high. (R. Borup)
 - Should rotate electrode before doing ECA measurement since bubbles may be present on electrode surface due to oxygen evolution at high potentials of protocol.
 - Need statement in support protocol regarding lower potentials if appropriate for the material being developed (e.g., for oxides).
 - Start-up/shut-down protocol
 - Action item: Distribute protocol / present results by 2012 DOE Annual Merit Review.** Action item assigned to **R. Borup.**
 - Comments on all protocols: The Fuel Cell Commercialization of Japan (FCCJ) has an MEA working group that has undertaken the task of developing protocols, very similar to the task this DWG task. This is headed up by Atsushi Ohma from Nissan. These protocols were presented at the Fall, 2011 Electrochemical Society (ECS) Meeting in Boston.
 - Action items: D. Myers** to circulate A. Ohma’s 2011 ECS Transaction Paper that describes these protocols. **D. Myers** to check with D. Ho (DOE) to see if A. Ohma could be invited to the 2012 DOE Annual Merit Review and present the FCCJ protocols to the DWG.
 - **Action item: D. Myers** to circulate Draft protocols to DWG for comments. Draft protocols to be finalized **early in 2012** and distributed to DWG.
- It was proposed at the Spring, 2011 DWG meeting that there is a need for a list of the failure mechanisms of fuel cell components, R&D needs, and grading of importance and that this would be a worthwhile task for the DWG. At this DWG meeting, R. Borup suggested that the European Union is undertaking a similar task as an outcome of the 2nd INTERNATIONAL WORKSHOP ON DEGRADATION ISSUES OF FUEL CELLS, held on September 21-23, 2011 in Thessaloniki, Greece (attended by R. Borup and others from the DWG). This effort is headed by

Georgios Tsotridis, JRC, for the European Commission. R. Borup suggested that we use the template circulated by G. Tsotridis, with his permission, as the starting point for the compilation/assembly of our own list.

- **Action items: D. Myers** to distribute G. Tsotridis's template to DWG members and to establish a Wiki site for DWG members to begin to populate the template.