Standard Operating Procedure

(SOP)

Compressibility of GDLs for Water Electrolyzers

Test ID # LTE-P-1

Rev 3

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Date: 12/03/2019

Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
03/22/2019	All	First Release
09/24/2019	All	Formatting, minor edits
12/3/2019	6 (section i)	Changed max load point from 219 to 500 psi (typical cell stack compressive load)

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1. Procedures

a. Scope and Applicability – This procedure is written for the standardization of *ex-situ* measurement of the compressibility of Gas Diffusion Layer (GDL) materials for use as electrode materials for applications such as fuel cells, electrolyzers, and batteries.

b. Summary of Method –
   1. Preparation of the samples
   2. Cleaning of the surface
   3. Zeroing the thickness device
   4. Performing initial thickness measurement as pressure (a)
   5. Compressing to measurement pressure (b)
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   7. Compressing to measurement pressure (c)
   8. Recording material thickness
   9. Returning to measurement pressure (b)
   10. Calculating compressibility
   11. Disposal of samples
   12. Recording of final values

c. Definitions –
   1. LVDT: linear variable displacement transducer
   2. FOD: foreign object debris

d. Health & Safety Warning – This procedure involves powerful equipment and high pressures. It is highly recommended that appropriate machine guards are in place and safety protocols are followed during operation. Do not attempt without proper training on safe operation of the equipment.

e. Cautions – Ensure that pressures are raised at controlled rates as described in the documentation during operation to prevent sample and equipment damage. If the sample is over-compressed at any point during the testing, discard the sample and re-test. Exposure to higher pressures can result in permanent displacement that will cause inaccurate results.

f. Interferences – Be careful when preparing samples to prevent any damage to the surface of the materials including scratches, bends / folds, or other issues that may affect the thickness of the parts.

g. Personnel Qualifications / Responsibilities – All operators should be adequately trained to operate the equipment in a safe manner to prevent personal injury and/or damage to equipment.

h. Equipment and Supplies –

1) Personal Protective Equipment
    a. Safety Glasses
    b. Latex or Nitrile Powder Free Gloves
2) Equipment
    a. Die cutter and/or razor blade
    b. Sample storage (plastic bags, paper folder, etc.)
    c. Tensile test machine with a moveable crosshead carrying a compression load cell
    d. 16 cm^2 (4.0 cm X 4.0 cm) graphite block test fixture. The graphite blocks (or platens) must be flat and parallel within a tolerance band than ensures even load distribution across the entire sample.
    e. LVDT readout/controller with compatible LVDT's (2) and a mounting fixture
    f. Optional: Load cell accurate in the range of 0 – 1500 lbf (0 – 6672 N)

i. Step by Step Procedure

- Instrument or Method Calibration and Standardization – Calibrate the travel distance on the tensile machine and verify force with a load sensor. Zero the 2 LVDT gauges with the graphite block test fixture with no sample at a pressure of 7.3 psi (0.05 N/mm^2) by applying a force of 18.6 lbf (83 N) and verifying that the readout is zero.

- Sample Collection – Carefully cut a test piece from the GDL material that measures 4.0 cm x 4.0 cm using a razor blade or die cutting system. Be careful not to damage the surface of the sample or to bend or otherwise mechanically compromise the sample during cutting. Weigh the sample with an analytical balance and reporting its basis weight (g/cm^2). Compare this to the published data sheet to determine if it is within the reported range.

- Sample Handling and Preservation – If the sample is not going to be tested immediately, carefully place the sample in a labeled holding container (plastic bag, paper folder, etc.) being careful not to fold, bend, scratch, or otherwise damage the material.

- Sample Test and Analysis –

    - Inspect the graphite block test fixture or other suitable platens ensuring they are clean and free of any foreign object debris (FOD).
    - Place the cut sample in the 16 cm^2 graphite block test fixture

- Increase the pressure to 7.3 psi (0.05 N/mm^2) by applying 18.1 lbf (80.5N) to the block by lowering the crosshead on the tensile machine making sure not to exceed 20 lbf /s.
- Record the average nominal thickness of the GDL sample (T_0) from the LVDT outputs on the record sheet
- Increase the pressure on the sample to 73 psi (0.5 N/mm^2) by applying 181 lbf (805 N) to the block by lowering the crosshead of the tensile machine, making sure not to exceed 20 lbf /s.
- Record the average thickness of the carbon sample (T_1) from the LVDT outputs on the record sheet.
- Increase the pressure on the sample to 500 psi (3.4 N/mm^2) by applying 1240 lbf (5516 N) to the block by lowering the crosshead of the tensile machine being sure not to exceed 20 lbf/s. *Note the 500 psi pressure was chosen since it is comparable to a typical low temperature electrolyzer cell stack compression load.*
- Record the thickness of the carbon sample (T_2) on the record sheet.
- Reduce the load to 7.3 psi at the end of the test and the repeat the thickness measurement to compare to the initial value. This will provide indication if the material remained within its elastic region or if there was any plastic deformation of the sample.

- Troubleshooting – N/A

- Data Acquisition, Calculations & Data Reduction Requirements –

  - To calculate the compressibility of the GDL material use the equation below:

$$\text{Compressibility (\%)} = \frac{T_0 - T_1}{T_0 - T_2} * 100$$

- Computer Hardware & Software – Various versions of computerized software can be used to the control the tensile test machine. These can be utilized but are not part of the standardization process.

j. Data and Records Management –Sample dimensions collected through manual measurement shall be recorded in a laboratory notebook. Data from the tensile tests can be obtained from the software and should be recorded both electronically and in laboratory notebook by the user so that compressibility values may be obtained.

4. Quality Control and Quality Assurance Section

   The tensile machine should be calibrated annually by a qualified technician for both travel distance of the head and pressure output. A load cell can be incorporated into the test setup to validate applied force for each test for improved accuracy, if desired.

5. Reference Section

   None applicable.