

V.B.3 Low-Cost Durable Seals for PEMFCs

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Subcontractors:

- Freudenberg-NOK General Partnership, Plymouth, MI
- Henkel Corporation, Rocky Hill, CT
- Virginia Polytechnic Institute and State University, Blacksburg, VA

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 Project End Date: March 31, 2009

Objectives

- Develop a working material specification to guide the development of proton exchange membrane fuel cell (PEMFC) seal materials.
- Synthesize and compound materials that meet the requirements of the materials specification.
- Evaluate candidate materials through accelerated ex situ testing to predict whether the material will meet durability objectives given in Table 1.
- Validate the performance of the best performing material candidate through in-cell testing.

Technical Barriers

This project addresses the following technical barriers from the Fuel Cells section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

(A) Durability

Technical Targets

The aim of this project is to develop and evaluate new non-silicone liquid injection moldable (LIM) and dispensable materials to improve durability for both transportation and stationary applications while maintaining or improving on the cost benefits of LIM silicone materials.

TABLE 1. DOE Targets Receiving Focus as Part of This Project

Characteristic	Units	2008 Status	2010/2011 ^a
Durability	hours	Testing underway	5,000/40,000
Sealability at Low Temperature	°C	Testing to-date indicates success	-40/-35
Cost ^b	\$/kW	To be evaluated	1.70/4.16 ^c

^a DOE Transportation/Stationary targets

^b Based on high volume production (500,000 transportation systems per year/2,000 stationary units per year). *Note:* A cost target for seals is not currently carried in the Hydrogen, Fuel Cells & Infrastructure Technologies Program Multi-Year Research, Development, and Demonstration Plan. See footnote (c) below for an explanation of how this target was derived.

^c Derived from cost allocation for seals presented in Reference 1. For the transportation target, the value given in the reference was applied directly. To obtain a meaningful stationary target, cost vs. production volume was assumed to be reasonable while a change in material cost was made to account for greater durability requirements.

Accomplishments

- A working material specification has been completed and submitted to DOE. The specification addresses mechanical properties, processing parameters and interactions with the operating environment.
- Two materials have been developed for testing within the project. Initial testing indicates that they both meet all minimum beginning-of-life requirements given in the materials specification.
- Long-term test plans are in place. These include tests in various relevant liquid environments and in high temperature air.
- Fixtures required for long-term testing have been fabricated. These fixtures will be used to evaluate the useful life of the candidate seal materials based on testing in relevant environments.



Introduction

Seal durability is critical to achieving the 2010 DOE operational life goals for both stationary and transportation PEMFC stacks. The seal material must be chemically and mechanically stable in an environment consisting of aggressive operating temperatures, humidified gases, and acidic membranes. The seal must also be producible at low cost. Currently used seal materials do not meet all these requirements.

High consistency hydrocarbon-based rubber compounds that show promise for compatibility with the PEMFC environment are difficult to process in a way that leads to low-cost PEMFC production. Silicone-based LIM rubber compounds which are easy to process in ways leading to low-cost production are highly gas permeable and have been shown to be unstable in PEMFC applications. To produce PEMFC stacks which are both highly durable and low in cost, a seal material with the stability of high consistency hydrocarbon rubber and the processing ease of a LIM silicone is sought.

Approach

To accomplish the objectives of this project, the approach is to develop and evaluate non-silicone LIM seal materials that can meet the specialized mechanical, compatibility, and cost requirements inherent to the design and operation of PEMFCs. To guide material development, a working material specification was developed. Materials developed to this specification by Henkel will then be evaluated through out-of-cell testing at Virginia Tech in simulated environments. Using an appropriate set of accelerated testing techniques, an initial lifetime estimate will be made for the candidate materials. The best candidate or candidates will be selected for in-cell testing to validate the performance of the material in a PEMFC environment. Specimens for out-of-cell testing and full size prototypes for in-cell testing will be produced by Freudenberg-NOK.

The outcome of the project will benefit the PEMFC industry by providing a seal material specification, a material that satisfies it, and the verification that the specification and the material enable a low-cost and durable seal.

Results

With the specification completed early in the project, the work over the last year has been focused on three primary tasks:

- Synthesizing and formulating new materials.
- Developing test procedures and new test fixtures to support the work.
- Evaluating seal configurations for use in both out-of-cell and in-cell testing.

Over 100 lab-scale material development experiments were conducted over the last year. Some are still ongoing. Out of the experiments conducted, two materials were selected to evaluate. The first is a one-part resin. One advantage is that the material does not have to be mixed at the point-of-use. However, because of a low curing temperature of 110°C targeted by the formulation, the material does need to be frozen until point-of-use. The second material is a two-part resin which does require mixing at the point-of-use. An advantage over the one-part material is that it does not require freezing or even refrigeration prior to reaching the point-of-use. It also appears to have better cured mechanical properties than the one-part material and, potentially, a lower cure temperature. Both materials meet the minimum beginning-of-life requirements given in the materials specification. A comparison of some key properties is given in Figure 1.

Testing requirements were developed by reviewing the various potential seal-related failure modes that may be experienced in the fuel cell environment. These include such possibilities as viscoelastic rupture [2], physical and chemical stress relaxation, and the effects of seal erosion on the fuel cell environment. Sealing stress is generally considered to be one of the most important measures of seal performance. It is typically monitored using one of various compressive stress relaxation (CSR) procedures such as those described in ASTM D 6147. Physical relaxation, a largely reversible mode, is important. Even more important is irreversible chemical stress relaxation. This is a mode of stress relaxation that is driven by various degradation processes which can occur within the material. The methods developed for use in this project will emphasize these relaxation processes. In addition, the specific methods used should distinguish the primary mode of degradation, scission or cross-linking, in various environments. The method should also allow a correlation to be drawn

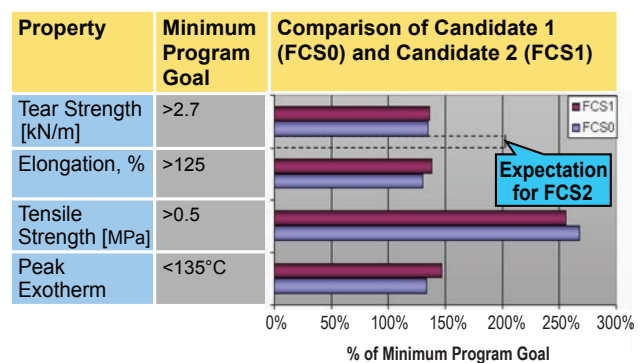


FIGURE 1. A comparison of some key properties for two materials evaluated, designated FCS0 and FCS1. Both meet or exceed all minimum project requirements as defined by the materials specification submitted to DOE previously. FCS1 is an improvement over FCS0 in three of the four key properties measured.

between CSR and compression set (CS) by drawing on the work of Tobolsky and later by Gillen [3,4]. This will be important in validating out-of-cell testing with in-cell testing. CS is readily measurable following an in-cell test. The correlation will allow these results to be related to in-cell sealing stress present at the end of the in-cell test. Figure 2 shows the configuration of a rig designed at Virginia Tech to collect all of the required data simultaneously without a need for periodic specimen removal. A separate set of rigs were fabricated to monitor seal leakage. This will allow correlation between seal failure and sealing stress.

Geometry of the compression specimen in CSR testing is important in interpreting the results. Differences in geometry lead to differences in the stress state within the material specimen. With this in mind, the specimens developed for use with the rig in Figure 2 are circular in form and have a cross-section that mimics the expected design of the full-size prototype seal.

References

1. Brian James, Jeffrey Kalinoski, "Mass Production Cost Estimation for Direct H₂ PEM Fuel Cell System for Automotive Applications", DOE Hydrogen Program 2007 Annual Merit Review Proceedings, Washington, D.C., 2007.
2. L.H. Sperling, "Introduction to Physical Polymer Science", 4th ed., John Wiley & Sons, Hoboken, New Jersey, 2006.
3. R.D. Andrews, A.V. Tobolsky, and E.E. Hanson, J. Appl. Phys., 17, 352 (1946).
4. K.T. Gillen, R. Bernstein, M.H. Wilson, Polym. Degrad. Stab., 87, 257 (2005).

Key Design Features

Concentric Shafts

- For independent compression of upper and lower seal stacks

Upper Seal Stack

- Uncompressed while aging
- For measurement of instantaneous compressive properties at the test temperature

Lower Seal Stack

- Maintained at constant compression
- For CSR measurement at the test temperature

Locking Nut and Spacer

- To set and maintain constant compression on the Lower Seal Stack

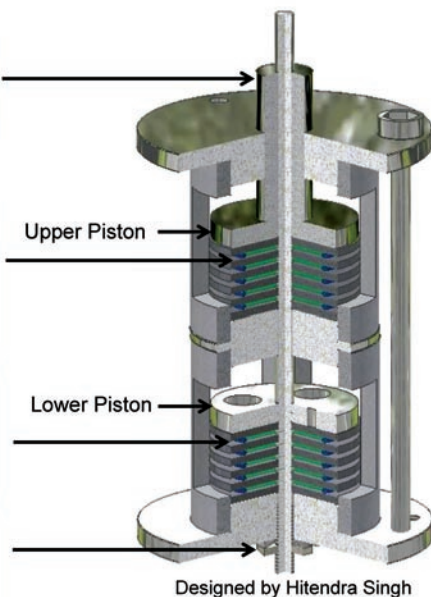


FIGURE 2. CSR Test Fixture This is the primary test apparatus which will be used to predict the useful lifetime of seal material candidates. The four key features shown allow for the simultaneous collection of instantaneous compressive properties and CSR.