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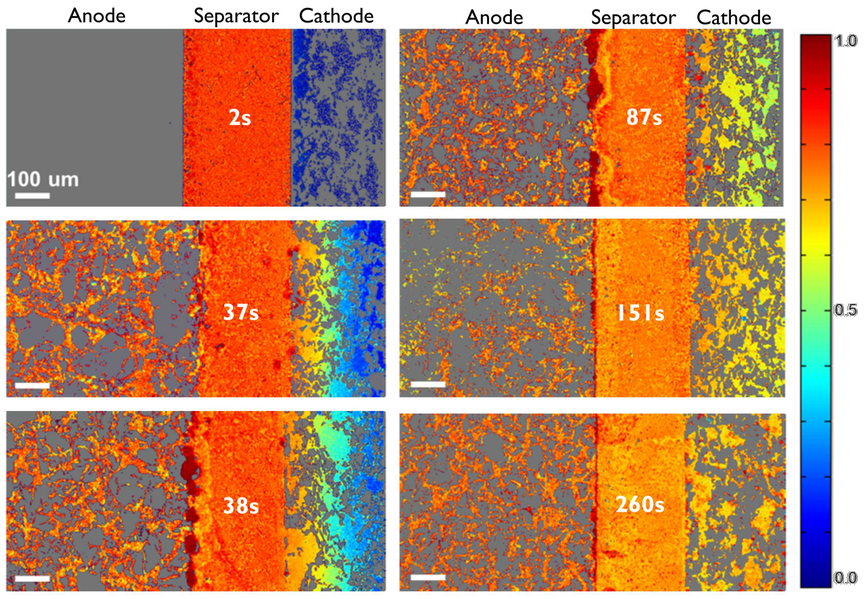
Capillary Pressure Versus Saturation Characterization of Molten-Salt Power Source Separator Materials

Alexander Headley, Martin Nemer, Christine Roberts, Ryan Solich
Sandia National Laboratories

Abstract
The Thermally Activated Battery Simulator (TABS) was developed to aid in battery design by modeling the thermal, mechanical, and electrochemical response of proposed batteries. While the model has been very successful, there is an ongoing effort to continue to improve its accuracy. One area of concern is the modeling of electrolyte flow between the separator and electrodes after battery activation, which may be influenced by capillary forces within the grain structure of the anode, cathode, and separator materials. This study focuses on characterizing the saturation versus capillary pressure of separator materials in conditions relevant to thermal battery operation. Two potential separator materials were tested while varying the initial saturation level and forming stress.

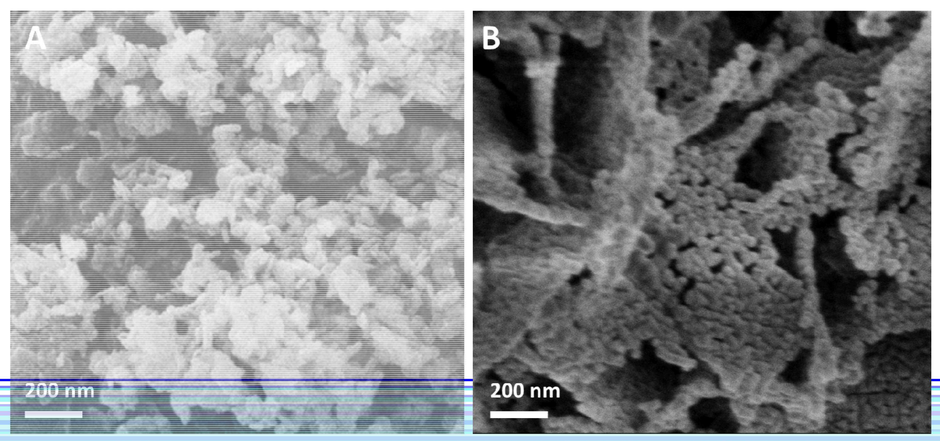
Model Requirements
The electrochemical impedance across an active battery cell is determined by the separator compression and electrolyte saturation distribution. Electrolyte flows until the fluid pressure equalizes among the electrodes. As such, the relationship between fluid pressure and electrolyte saturation in a separator needs to be known to properly model electrochemical transport.



Electrolyte migration tracked over time after activation with Br tracer. Sandia National Laboratories: Dan Wesolowski, Ashley Allen, Tom Humplik, Christine Roberts, Anne Grillet

Materials and Parameters of Interest
The amount of electrolyte affects the separator compaction during activation, so the separator thickness in an active battery depends on the amount of fluid it contains initially. Also, the pressure-saturation relationship for a material is a function of the binder microstructure as well as its packing density. Here, pressure-saturation data is measured for:

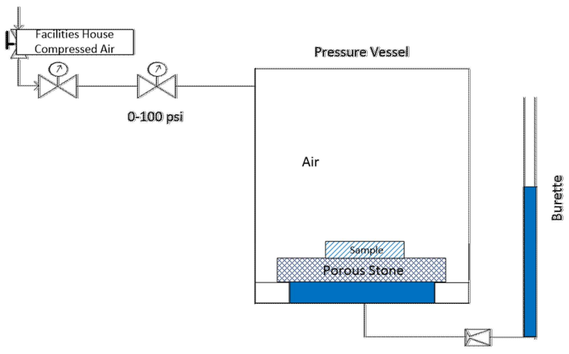
1) Two different MgO materials
2) Different packing pressures (8, 14, and 28 psi)
3) Different initial fluid saturations



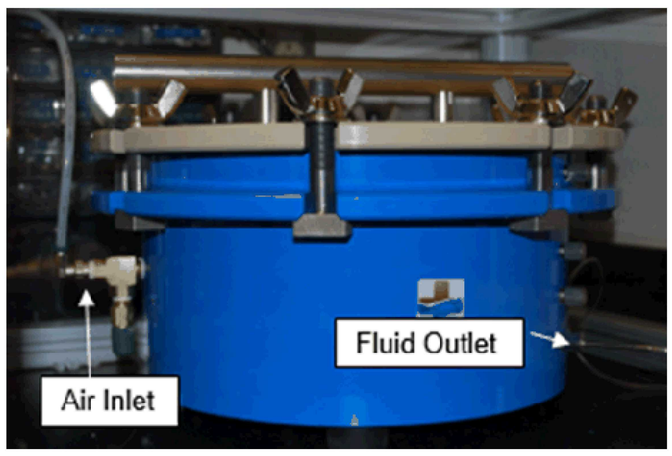
SEM images of separator materials tested: MgO-A (left) and MgO-B (right)

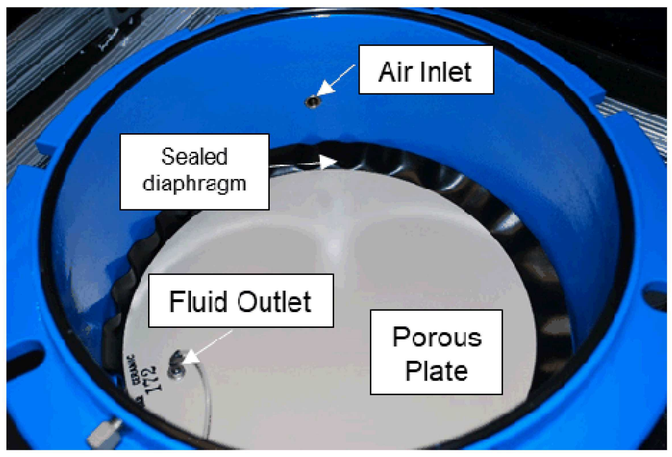
Eutectic electrolyte salt is difficult to use in pressure-saturation experiments due to its high melting temperature (352 °C) and corrosive nature. However, it is known that the salt thoroughly wets the MgO [4]. A surrogate fluid, 5 cSt polydimethyl siloxane (PDMS) was used as it also thoroughly wets the MgO.

Test Method
The porous plate method was used for this study. Samples are placed in capillary contact with a saturated, fine grain porous plate with a high air intrusion pressure inside a sealed pressure vessel. Known air pressures were applied in the head space of the vessel, pushing fluid from the sample and out of the system through the porous plate. The pressure was maintained until the fluid level in the sample reaches equilibrium, at which point the sample is weighed to determine the saturation of the sample. This process was repeated at increasing pressures to characterize the pressure-saturation curve for the sample.



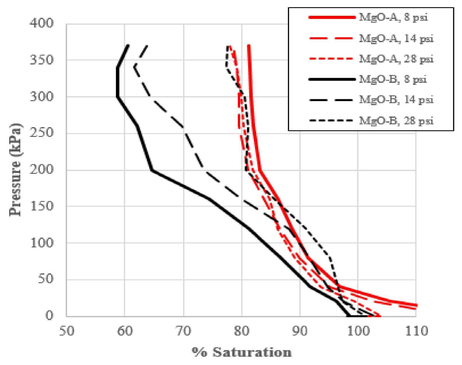
Schematic of a porous-plate apparatus

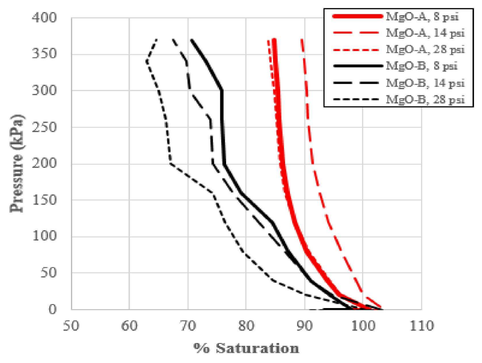




Porous-plate apparatus for determining capillary pressure

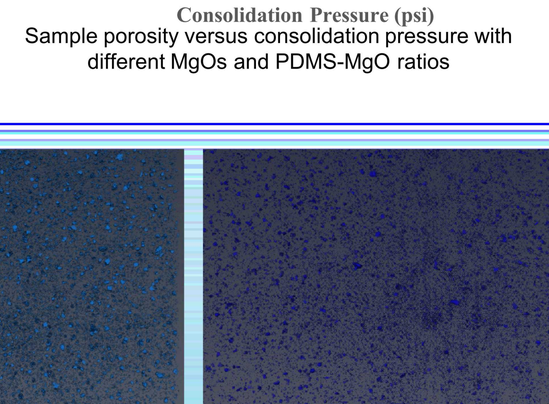
Results and Conclusions
MgO-A (small, disc-like primary particle shapes) consistently retained more fluid than MgO-B (larger, flake-like shapes). This is to be expected given the larger flow paths within the structure of MgO-B.



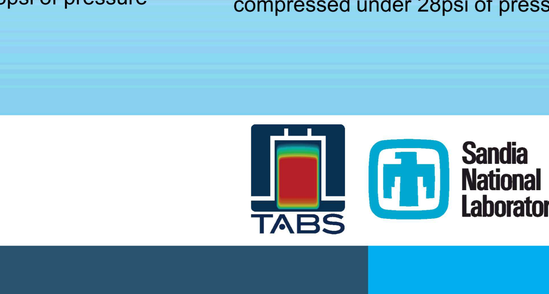


Measured pressure-saturation curve of PDMS fluid in MgO samples of varying initial compaction pressure. The initial ratio of fluid to MgO was 1.5:1 by mass (top) and 2:1 by mass (bottom)

Material packing efficiency also affects pressure-saturation characteristics. As the space between structures decreased (lower matrix porosity) less fluid was removed (higher saturation).However, the effect of the initial saturation on packing efficiency is not clear.



Sample porosity versus minimum saturation level with different MgOs

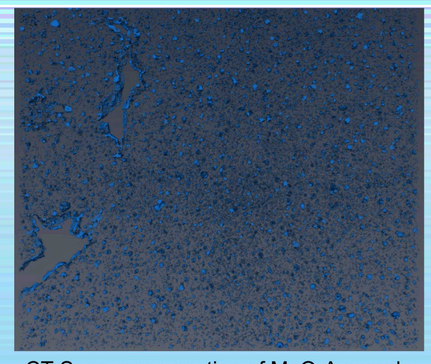


Sample porosity versus consolidation pressure with different MgOs and PDMS-MgO ratios

Ongoing Work
Material packing efficiency studies by CT scan changing saturation and consolidation pressure.

Porous plate testing of additional materials.

Air intrusion pressure investigation for fully saturated samples.



CT Scan cross-section of MgO-A sample compressed under 8psi of pressure

CT Scan cross-section of MgO-A sample compressed under 28psi of pressure

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