DOE/ID/13283--TI-App.C- Pt. 4

Draft of Final Report for the Aerocapacitor TRP

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 August 23, 1996

Summary

This report summarizes work done at LLNL for the development of the aerocapacitor in a collaborative effort between LLNL, PowerOne and PolyStor. The overall goal was to demonstrate a cost competitive manufacturing process for the use of carbon aerogels in a capacitor component suitable for incorporation in power supplies. Below, we feature several categories of work, including progress specifically in the areas of collaboration with our industrial partners, in the construction and testing of the aerocapacitors, and in the development and characterization of the aerogel materials.

Interactions with PolyStor and Power-One

- Developed milestone schedule in conjunction with industrial partners
- Delivered a series of Aerocaps to PowerOne and/or Polystor for testing: an off-the-shelf 2-cell Aerocap to Power-One for preliminary testing; a 5-cell Aerocap at 6.4 F, 0.05 mohms; and 6-cell Aerocaps (0.5 F; 0.25 ohm) to PolyStor and PowerOne
- Transferred aerogel synthesis & processing technology to Aerojet. This arrangement later proved unsatisfactory and a new business needed to be found. We later identified a small business (Ocellus Inc.) that can serve as a commercial supplier of carbon aerogel monoliths upon termination of the ARPA/TRP program
- Provided input on aerogel chemistry for joint publications with PolyStor and PowerOne
- A meeting with LLNL, Pantronix and Polystor resulted in an initial agreement to work on a proposal for funding through the California re-investment initiative program. Pantronix is willing to work with Polystor to assist in the high-volume packaging of capacitors. LLNL provided detailed input to the final proposal.
- Continue to supply PolyStor with carbon aerogel paper for building button cells and microspheres for thin-film coating and the construction of an organic electrolyte-based capacitor.
- Carbon aerogel monoliths (~0.8 g/cc) were provided to PolyStor for thin-film coating and the construction of an organic electrolyte-based capacitor; future scale-up will be required to meet the PolyStor demand. Provided ~150 g of carbon aerogel monoliths to PolyStor on a weekly basis for coating electrodes onto aluminum foil. This material is being ground and coated onto aluminum foil for the construction of organic electrolyte-based capacitors. The synthesis, processing, and characterization of carbon aerogel monoliths is now consuming the majority of our time. LLNL has become the primary supplier of this material as a result of inadequate funding at Aerojet. A comparison is being made between monoliths which were supercritically dried and those which were air-dried. Preliminary data from PolyStor indicates equivalent double layer capacitance for these materials. Thus far, carbon aerogels obtained from air-drying appear to provide the same capacitance as the material that has been supercritically dried.

Aerocaps Development and Testing

- Began cycle testing of Aerocaps
- An Inframetrix I.R. Camera used to record the thermal profile of an operating aerocapacitor revealed that there may be an insufficient thermal load to evaluate the profile using this technique.

Packaging development

- A variety of new packaging approaches were tested.

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- Efforts were made to find a compatible potting compound to work both as a sealant and as a vent valve. Packaging problems proved to be more challenging than anticipated. Samples were received from Dow Corning for evaluation.
- A conventional electronics connector(40 pin DIP) was attached to aerocapacitors to provide a prismatic package that is easily mounted on circuit boards. The first attempt at using a conventional electronics connector(40 pin DIP)to power a C-gel capacitor was made. Minor changes will be made for the next prototype. A second Aerocapacitor was attached to a 40 pin DIP package; this connector makes the Aerocapacitor compatible with conventional electronics.

Ni as contact material

- Evaluated new contact methods between Ni current collector and carbon aerogel paper (flame spray, conductive adhesives, sintered Ni flake, Ni-coated C paper)
- Demonstrated that Ni Exmet can be eliminated from Aerocapacitor
- Constructed 6-cell Aerocaps using Exmet and sintered Ni flake contact methods
- Delivered C aerogel electrodes with sintered Ni flake for button cell assembly at PolyStor
- Carbon aerogel samples were coated with nickel using e-beam evaporation. Success was obtained on coating both monoliths and aerogel paper to thicknesses ranging from 0.2-2.0 microns.
- Eleven carbon aerogel samples were coated with nickel using e-beam evaporation. Success was obtained on coating both monoliths and aerogel paper to thicknesses ranging from 0.2-2.0 microns.
- Eutectic bonding tests were performed using nickel foil and carbon aerogel samples. Two samples were coated with nickel as above and two were uncoated. The samples were then exposed to a reducing atmosphere at 1450 °C.
- Purchased a 1700 °C oven for sintering studies of carbon aerogel paper to Ni foil

Electrolytes development

- Evaluated alternative electrolytes that may provide advantages over 5M KOH
- Terminated work on prismatic capacitors using KOH electrolyte to begin development of organic electrolyte-based "jelly roll" capacitors.

Carbon aerogel paper

- Identified need to sort carbon aerogel paper based on mass

Coating

- Five carbon aerogel samples were coated with aluminum using e-beam evaporation. Success was obtained on coating aerogel paper to a thickness of approximately 2 microns. We attempted to sinter bond these materials to aluminum foil.

Aerocaps Built

- Investigated the possibility of using the LLNL plastic shop to inject mold cases for Aerocapacitor encapsulation.
- Built 12-cell Aerocap (0.25 F; 0.90 ohm)
- A total of eleven 8-cell and two 12-cell Aerocapacitors were constructed using a variety of new separator materials. These cells are operational at 6.5 V and 10 V, respectively.
- Demonstrated that 8-cell Aerocapacitors could be held at 6.5V for >24 hrs. at room temperature. Packaging problems caused electrolyte leakage at test temperatures greater than 50 °C.
- Built single-cell Aerocapacitors for PolyStor evaluation. The cells contained various combinations of Inconel®, nickel, and copper current collectors
- Built and tested nineteen 8-cell Aerocapacitors; identified a new porous polyamide as the separator of choice. Built and tested seven 8-cell Aerocapacitors using new polyamide separator; demonstrated that devices can be held at 6.5V for > 24 hrs. at room temperature; scaling problems are still hampering high temperature performance.

Development and Characterization of Aerogel Materials

Preparation of Carbon Aerogel

- Prepared carbon aerogel microspheres using an inverse emulsion process. These materials could potentially eliminate a grinding step prior to PolyStor's coating operations.

Air Drying of RF for Cost Reduction

- Demonstrated that resorcinol-formaldehyde gels can be air-dried from pentane to produce aerogels having densities of 0.50 g/cc. The ability to use *air-drying* rather than supercritical drying is a significant step toward the reduction of carbon aerogel production costs.
- Pyrolyzed the above aerogels and delivered the carbonized product to PolyStor for evaluation.
- We continue to optimize the conditions for air-drying resorcinol-monomoliths from pentane.

Characterizations

- Provided scanning electron microscopy on carbon aerogel paper and on various PolyStor samples.
- Performed BET surface area and pore size measurements on batches of carbon aerogels prepared. A database was established to evaluate quality control parameters. Used gas adsorption techniques to obtain surface area and pore size data on 8 different batches of carbon aerogels. The surface area ranged from 500-600 m²/g while the pore size ranged from 5-6 nm. These data indicate good quality control during the production of carbon aerogels and demonstrates that the first air-dried samples have surface areas and pore sizes comparable to their supercritically-dried counterparts.
- Investigated the change in mechanical properties for carbon aerogels as a function of pyrolysis temperature. Performed acoustic measurements on wet resorcinol-formaldehyde gels to investigate their mechanical properties prior to drying operations. Established an acoustic technique for measuring the mechanical properties of gels both before and after drying.

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