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Insulation Chemistry on Earth and Beyond: Polyimide and Polyamide Aerogels

Mary Ann Meador
Senior Scientist, NASA Glenn Research Center

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Managing Editor, ACS Macro Letters, Biomacromolecules, Macromolecules and ACS Biomaterials Science & Engineering

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What are aerogels?

- Highly porous solids made by drying a wet gel without shrinking
- Pore sizes extremely small (typically 10-40 nm)—very good insulation
- 2-4 times better insulator than fiberglass under ambient pressure
- 10-15 times better in light vacuum
- Invented in 1930’s by Prof. Samuel Kistler of the College of the Pacific

Current aerogel products and market

Main players—silica aerogels
- Cabot
- Lumira Aerogel in skylights
  Grand Rapids, Mi
- Aspen Aerogels
  Pyrogel pipe insulation

Emerging players
- BASF
  Slentite panels
- Blueshift
  AeroZero
- Aerogel Technologies
  Airlof
Comparison of Aspen Aerogels blanket insulation with traditional insulation

Source: George Gould, Aspen Aerogels

Audience Survey Question
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

The current insulation market is worth about $44 billion. About what percentage of that is currently aerogel?

• 0.01 percent
• 1 percent
• 10 percent
• 30 percent
The current insulation market is worth about $44 billion. About what percentage of that is currently aerogel?

- 0.01 percent
- 1 percent
- 10 percent
- 30 percent

* Though aerogels are superior insulation, most current products are silica based which are limited to particulate or composite, which can be dusty. Emerging technologies are overcoming that but costs are still high compared to conventional insulation.

Monolithic silica aerogels out-perform particulate forms as insulation

However, silica aerogels are extremely fragile…

…and therefore limited to a few exotic NASA applications

Cosmic dust collector – Stardust Program

Insulation on rovers
Potential applications for durable aerogels in aeronautics and space exploration

- Cryotank insulation
- Fan engine containment (Ballistic protection)
- Antenna substrates
- Ultra-lightweight, multifunctional structures for habitats, rovers
- Insulation for EVA suits and habitats
- Sandwich structures
- Propellant tanks
- Heat shielding
- Inflatable aerodynamic decelerators

Durable aerogels by reinforcing silica aerogels with polymers

- Native
- Cross-linked
  - Two order of magnitude increase in strength at the same density
  - Does not change pore structure

- Versatile: cross-linking with variety of polymers
- Collaboration with Aspen Aerogels to scale up streamlined process

Polymer reinforced aerogels used to insulate cryotanks—collaboration with MSFC

Low density... to higher density, same aerogel pore structure
Hypersonic inflatable aerodynamic decelerator concept

- Hard aeroshells used to land rovers on Mars limit size of payload
- Inflatable structure overcomes this limitation
- Concept shown constructed from a series of stacked inflatable tori tied with a network of straps
- Flexible thermal protection system on fore body

Baseline insulation for HIAD was Pyrogel-2250

- Composite insulation: silica aerogel in batting
- Flexible but sheds dust particles on handling
- Begins to out gas at 380 °C
- In high heat flux testing, time to 300 °C of bottom thermocouple related to weight of insulation
- Lost 20-34 % weight during test
Polyimide aerogels

- Family of polymer aerogels made by cross-linking polyimide oligomers to form gel network
- Supercritical fluid extraction to remove liquid from gels


Typical PI Aerogel Process

Monomers mixed in NMP  Solution poured into mold  Extracted after gelation

Solvent exchange into acetone  Supercritical CO₂ extraction
**Polyimide 3D network using T8-POSS-first successful formulations**

- PI cross-linked with POSS
- Chemically imidized with pyridine/ acetic anhydride
- BPDA-(BAX-BPDA)n; n = 10 to 25
- Low shrinkage (~10 %)
- Density: ~0.1 g/cm³
- Porosity > 90 %


---

**Cross-linked polyimide aerogels cast as thin film are flexible**

- Collaboration with Prof. Miko Cakmak, University of Akron
- Density of film is similar to molded cylinder
- Middle picture is 9” x 13” pan; film is folded multiple times
- Currently can cast up to 18” inches wide, 8 ft. long

As-cast wet films  Dry aerogel
Polyimide Aerogel Development

- Two cross-linking approaches evaluated
- Over thirty different combinations of backbone chemistry studied
- Formulations identified with:
  - Best moisture resistance
  - Best mechanical properties at lowest density
  - Low thermal conductivity
  - Short term stability up to 400 °C
- 100s of enquiries from companies for commercial products

LCAT test—Saffil in combination with PI aerogel

- Layer of Saffil
- Two equivalent thicknesses PI aerogel
  - 50% DMBZ \ 50% ODA
- Test stopped after 247 s
  - Bottom TC reached 300 °C
- Top of PI stack ~590 °C max
- PI lost much less weight than Pyrogel
Multifunctional, Universal Thermal Insulation System

- Current multilayer insulation (MLI) only functions in vacuum
  - Layers of Mylar separated by scrim layers
- Aerogel is best insulation in gaseous environment
- MLI incorporating aerogel in place of scrim reduces TC by 23-37%
- Partnership with JSC and GRC

**Audience Survey Question**

ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

What properties do polyimide aerogels possess, besides being a great insulator that makes them important for aerospace applications?

- Good mechanical properties
- Low density
- High surface area
- Very low dielectric constant
- All of the above
What properties do polyimide aerogels possess, besides being a great insulator that makes them important for aerospace applications?

- Good mechanical properties
- Low density
- High surface area
- Very low dielectric constant
- All of the above

* Good mechanical properties combined with low density means they can be used as lightweight structures. The high surface area, low dielectric constant and superior insulation capability allow for the introduction of other functionality in the structure (insulation, sensing, etc.)
Polyimide aerogels are stronger than silica and polymer reinforced silica aerogels

Also compares favorably with polymer foams

One issue with polyimide aerogels is cost

- Cross-linkers used either not commercially available or expensive
- Solution:
  - Triacid chloride cross-linker with amine capped oligomers
- Properties similar to other cross-linkers
Polyamide aerogels

- Lower cost monomers and cross-linkers
- Slightly less thermally stable
- Stronger than PI aerogels at the same density

Aerogels also have low dielectric properties

- Dielectric properties of silica aerogels vary linearly with density
- Substrates with lower dielectric constants improve performance and reduce weight of antennas
How many antennas does a typical commercial aircraft have?

- Less than 10
- About 20
- About 50
- As many as 100

* Commercial and military aircraft can have up to 100 antennas for everything from ground communication to in-flight entertainment, taking up a large amount of room in the fuselage and weight of the aircraft.
Low dielectric properties of PI aerogel lead to lightweight antennas

- Relative dielectric constants of aerogels scale with density
- Better antenna performance than Duroid
- 77 % lighter weight

Summary

- PI aerogels produced as thin flexible films for use as insulation for inflatable decelerators or space suits
- Same aerogels as thicker parts are stiff and strong
- Lower cost options: new cross-linkers and other polymer chemistries (polyamide)
- Improved antenna performance with 77% lower mass
- Commercially available from Aerogel Technologies, LLC (molded shapes) and Blueshift (roll-to-roll films)
Acknowledgments

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- Director's Discretionary Fund

Additional Resources

- [http://technology.grc.nasa.gov/](http://technology.grc.nasa.gov/) - Information about technology available to license from NASA Glenn
- [http://www.aerogel.org/](http://www.aerogel.org/) - Learn more about aerogels
- [http://pubs.acs.org/doi/abs/10.1021/am507268c](http://pubs.acs.org/doi/abs/10.1021/am507268c) - Polyimide Aerogels with Amide Cross-Links: A Low Cost Alternative for Mechanically Strong Polymer Aerogels
- [http://pubs.acs.org/doi/abs/10.1021/cm5012313](http://pubs.acs.org/doi/abs/10.1021/cm5012313) - Synthesis and Properties of Step-Growth Polyamide Aerogels Cross-linked with Triacid Chlorides
- [http://pubs.acs.org/doi/abs/10.1021/acs.chemmater.6b00474](http://pubs.acs.org/doi/abs/10.1021/acs.chemmater.6b00474) - Thermoresponsive Shape-Memory Aerogels from Thiol–Ene Networks
- [http://pubs.acs.org/doi/abs/10.1021/am405106h](http://pubs.acs.org/doi/abs/10.1021/am405106h) - Dielectric and Other Properties of Polyimide Aerogels Containing Fluorinated Blocks
- [http://pubs.acs.org/doi/abs/10.1021/acs.jcim.6b00474](http://pubs.acs.org/doi/abs/10.1021/acs.jcim.6b00474) - Low Dielectric Polyimide Aerogels As Substrates for Lightweight Patch Antennas
- [http://pubs.acs.org/doi/abs/10.1021/am101123h](http://pubs.acs.org/doi/abs/10.1021/am101123h) - Tailoring Properties of Cross-Linked Polyimide Aerogels for Better Moisture Resistance, Flexibility, and Strength
- [http://pubs.acs.org/doi/abs/10.1021/am101123h](http://pubs.acs.org/doi/abs/10.1021/am101123h) - Mechanically Strong, Flexible Polyimide Aerogels Cross-Linked with Aromatic Triamine
- [http://pubs.acs.org/doi/abs/10.1021/am200007n](http://pubs.acs.org/doi/abs/10.1021/am200007n) - Tailoring Mechanical Properties of Aerogels for Aerospace Applications
- [http://pubs.acs.org/doi/abs/10.1021/am101123h](http://pubs.acs.org/doi/abs/10.1021/am101123h) - Polyimide Aerogels Cross-Linked through Amine Functionalized Polyoligomeric Silsesquioxane
Insulation Chemistry on Earth and Beyond:
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