Aerogel Hybrid Composite Materials: Designs and Testing for Multifunctional Applications

Dr. Martha K. Williams
Mr. James E. Fesmire

NASA Kennedy Space Center
Exploration Research and Technology Programs
Science and Technology
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NASA Kennedy Space Center Exploration Research works to provide *practical solutions to thermal management problems* while focusing on long-term technology targets for the *energy-efficient* systems on Earth and in space. We develop new technologies including composite materials, insulation systems, and testing methodologies devoted to meeting the unique thermal requirements of the aerospace industry and space exploration, while providing technical solutions for national and global needs.
Thermal Materials and Systems Focus Areas

Technology Focus Areas:

✓ Thermal and structural insulation systems
✓ Novel materials and components
✓ Propellant storage, refrigeration, and transfer systems
✓ Low-temperature and extreme environments applications

Included are foam hybrid systems with polymeric and fiber aerogel composites for structural and thermal applications where reduction in heat transfer are required for both steady-state and transient thermal management. Development of novel methodologies for testing and evaluation of these unique systems are also important in covering the technology gaps in thermal materials development and utilization.
Overview

- Introduction and Background
- Aerogel Composite Systems
- AeroFoam - Hybrid Foam Systems
- AeroPlastic - Thermoplastic composites
- AeroFiber - Fiber Composites and Aerogel Laminate Systems
  - Aero-cover - Aerospace Application
- Summary
- Questions
What is Modern Aerogel?

• Aerogel materials used: generally silica based, light weight materials, fully breathable, and treated to be super-hydrophobic.

• Aerogel particles are free flowing, fills small cavities, does not compact, no preconditioning required, and can be molded or formed using binders.

• Aerogel particles by Cabot Corp.:
  – 90% porous with a mean pore diameter of 20 nm.
  – 700 sq meters per gram surface area
  – Particle bulk density \( \approx 80 \text{ kg/m}^3 \) (5 lbs/ft\(^3\)).
  – Individual beads are fragile (shear), but have high elastic compression of over 50% with no damage.
  – k-value \( \approx 18 \text{ mW/m-K} @ 25^\circ \text{C and 760 torr.} \)

• Aerogel blanket (Spaceloft®) by Aspen Aerogels:
  – Began in 1993 under an SBIR with Kennedy Space Center
  – Bulk density 10 lbs/ft\(^3\).
  – k-value \( \approx 14 \text{ mW/m-K} @ 25^\circ \text{C and 760 torr.} \)
  – Use temperature range -273°C to 200°C (-459°F to 390°F).

• Aerogel Pyrogel® blanket by Aspen Aerogels:
  – Silica Aerogel embedded into black reinforcing fiber
  – Flexible aerogel, nano-porous, insulation composite blanket designed for high-temperature applications (up to 650°C/1200°F).
Real-world problem-solving for Space Shuttle flights: deep investigation of specific, hard problems leads to practical knowledge, understanding, and new technologies.
Aerogel Hybrid Composites

**AeroFoam** is a new hybrid foam/aerogel composite

**AeroPlastic** is a new composite material of certain polymer and aerogel particle combinations

**AeroFiber** is a new hybrid laminate system composed of fiber composites and aerogel blankets

- All are tailorable and represent families of different approaches, designs, and combinations.
- All are available for licensing
AeroFoam: what is it?

- **AeroFoam** is a foam hybrid composite material
  - Component one is an organic polymeric cellular solid material
  - Component two is an inorganic or organic aerogel or xerogel filler that is physically held in place by the “foam”
- The organic foam material strengthens the aerogel
- The aerogel reduces the heat transfer within the foam
- Current examples of AeroFoam are TEEK polyimide foams with Cabot beads/granules or with Aspen aerogel blanket or the combination there of

![Estimate of damping log Decrement method for Brick samples](image)

![Estimate of damping time series .02-.06 seconds from hammer hit](image)
AeroFoam: what’s the benefit?

• Foam composites can be fabricated to target densities
  – High density foam composites are considered as structural foams
  – Low density foam composites are considered as flexible foams

• Heat transfer is reduced – function of aerogel loading
  – Aerogel loading is primary driver of heat transfer NOT density
  – More aerogel added results in reduced heat transfer through foam composite
  – Density affects on heat transfer are limited
  – Higher density foams typically have higher heat transfer.
  – Aerogel blanket composites have most significant reduction in heat transfer.

• Improved acoustic insulation and vibration attenuation

• Increased cryogenic storage capability

• TEEK foams and TEEK foam composites are inherently flame retardant
AeroPlastic – What is it?

- AeroPlastic is a *new composite material* with *properties* which are not necessarily all present in the respective or the pure components.
- A method to reduce the thermal conductivity and peak heat releases rates of base polymer.
  - 20%-50% reduction of heat flow
  - Maintains or enhances mechanical properties
- Aerogel reduces heat transfer and work with commodity grade and engineered grade polymers using current extrusion and injection molding processes.
AeroPlastic Technology Summary

- Most effective way to significantly reduce heat transfer using an additive melt processing method*
- Can be combined with other additives
- Retention of mechanical properties, no significant reduction in tensile strength
- Applicable to multiple markets using commodity and engineered polymers
  - Polyamides (MXD6, PA6,6), polyolefins, polystyrene, and Ultem™ prototypes
  - Suitable for molded and extruded product forms, film and fiber products
  - Expands the use of thermoplastics at cryogenics or lower temperatures
  - Applicable to aerospace, cryogenics, oil and gas, automotive, electronics, military, wood plastics, medical, food packaging and textile markets

*Glass Bubbles have been used in melt processed materials with reported $k$-value reductions to up to 20%
AeroPlastic - New Composite Materials

- Perhaps the only additive with such results in significant reduction of heat transfer.
- Very limited approaches for reducing the heat transfer of thermoplastic composites.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal conductivity (W/mK)</th>
<th>Thermal conductivity reduction from neat</th>
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<tbody>
<tr>
<td>MXD6 neat, sample 1</td>
<td>0.217</td>
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<tr>
<td>5% aerogel-MXD6, sample 1</td>
<td>0.115</td>
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<tr>
<td>MXD6 neat, sample 2</td>
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<td>PA66 neat</td>
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<td>5% aerogel-PA66</td>
<td>0.216</td>
<td>26%</td>
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</table>
AeroFiber: what is it?

• **AeroFiber** is a hybrid laminate system made of fiber composites and aerogel blankets
• Aerogel and fiber composites is integrated into unique lay-ups
• Tailorable properties with thermal and mechanical energy absorption capabilities
• Vacuum infusion for fiber composites
• Adhesive system for lamination can be tailored for application, e.g. cold versus hot
• Prototypes in multiple textiles and combinations thereof
AeroFiber: Thermal Conductivity

Cryostat-500 - cryogenic thermal performance - panels - 760 torr tests

- G118 panel SCPCS XC106 (2.7, 783)
- G119 panel SPS XC108 (3.5, 580)
- G120 panel KCPCK XC109 (2.9, 766)
- G121 panel KPK XC110 (3.0, 729)
- G122 panel CPC XC103 (3.2, 718)
- G123 panel Pyrogel XC101 (2.2, 182)
- G124 panel CopperPyrogel XC104 (1.98, 856)
- G125 panel SilverPyrogel XC105 (2.52, 637)
THERMAL INSULATING PERFORMANCE IN K-VALUE OF VARIOUS MATERIALS

- Pure Copper
- Stainless Steel
- Ice
- Concrete
- Water
- Whale Blubber
- Oak Board
- Cork
- Cellular Glass
- Polyurethane Foam
- Fiberglass
- LCX
- Aerogel Blanket
- LCI System @ SV
- LCI System @ HV
- Perlite Powder @ HV
- Glass Bubbles @ HV

Thermal Conductivity or K-value (milliWatt per meter-Kelvin)

k-value = 20 mW/m-K
Examples of reduced thermal conductivity in laminates

Legend
C: Carbon
P: Polyester
I: Innegra S
S: Spectra 1000
K: Kevlar
Number after letter is the number of layers of the Fabric
Example (6) indicates that the composite has 6 layers of fabric

k-value (mW/m-k) vs Thickness (mm)
Mechanical Evaluation of Properties

Mechanical Testing

Tensile, maximum load

Tensile (psi) vs. Thickness (mm)

- Red line: tensile (psi)
- Blue line: Thickness (mm)

Axes:
- Tensile (psi) on the y-axis
- Thickness (mm) on the x-axis

Data points:
- K (6)
- C1K1C1C1
- C1K1PK1C1
- K2PK2

Values for Tensile (psi):
- K (6): 60000
- C1K1C1C1: 50000
- C1K1PK1C1: 40000
- K2PK2: 30000
Cryo-Impact Testing - Sample Holding Fixture

- Compare mechanical properties for fiber composites with hybrid laminates

- Visual observation, data indicate increase energy absorption of the aerogel-fiber composites laminates compared to fiber composites alone

- Thickness, combination of lay-ups and adhesive system effects

- Cryo-impact testing was carried out using holder designed and built by NASA/KSC
  - For LN$_2$ (77 K) or ambient temperature testing.
  - Fits 3-inch round or 4-inch square specimens.
  - Impact Energy (Joules): 10, 25, or 50 J
  - Clamping force (torque): 0, 10, or 15 ft-lb

*Collaboration with Dr. David Sypeck at Embry Riddle Aeronautical University (ERAU)*
Comparison of Cryo-Impact Results

Carbon fiber composite after cryo-impact

Carbon Aero Fiber composite after cryo-impact
Cone calorimeter calculates peak heat release rates (correlates to size of fire generated)
- Heat release is a key measurement used in fire resistant materials research development and performance testing
- Allows for calculating heat release rates in thermal protection multi-layered systems
Aero-cover, an Aerospace Application

A application demonstration of the technology. Goals were to carry the design and development of the aerodynamic composite cover or “shroud” from cradle to grave including materials research, purchasing, design, fabrication, testing, analysis and hardware demonstration product.
Aerodynamic Cover Demo Design

- Represents a 0.6-meter segment of the 10-meter circumference of a concept heavy lift vehicle architecture.

- A concept was developed for realistic attach methods to a launch vehicle.

- The cover was designed to be mounted on a display stand and slipped into upper and lower brackets.

- There are many possible concepts on how to attach the aerodynamic cover to an actual vehicle structure.

- Load analyses were performed only on the demo solid composite Aero-cover.
Non-Destructive Testing Evaluation

- Flash thermography imaging of the front and back side of each composite panel.
- Uses a heat source to heat the composite part that is being inspected. As the composite part is cooled down, an infrared camera with digital processing monitors the temperature distribution across the surface for voids.
- TWI Flash Thermography System - temperature (R data), rate of temperature change in time (1D Data), and the rate of the rate of temperature change in time (2D data).
## Mechanical Testing Evaluation

<table>
<thead>
<tr>
<th>Specimen Label</th>
<th>Load at Tensile Strength (Pound Force - lbf)</th>
<th>Tensile stress at Tensile Strength (Pounds Per Square Inch - psi)</th>
<th>Layup Scheme</th>
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<tbody>
<tr>
<td>A-1</td>
<td>65.5</td>
<td>16.4</td>
<td>#12, 8-ply, [0,45,90,-45]s with Aerogel felt</td>
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<tr>
<td>A-3</td>
<td>66.9</td>
<td>16.7</td>
<td>#12, 8-ply, [0,45,90,-45]s with Aerogel felt</td>
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<tr>
<td>A-2</td>
<td>69.7</td>
<td>17.4</td>
<td>#12, 8-ply, [0,45,90,-45]s with Aerogel felt</td>
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<tr>
<td>A-4</td>
<td>53.2</td>
<td>13.3</td>
<td>#12, 8-ply, [0,45,90,-45]s with Aerogel felt</td>
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<tr>
<td>A-5</td>
<td>58.1</td>
<td>14.5</td>
<td>#12, 8-ply, [0,45,90,-45]s with Aerogel felt</td>
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<tr>
<td><strong>Average</strong></td>
<td><strong>62.68</strong></td>
<td><strong>15.66</strong></td>
<td><strong>8-ply [0,45,90,-45]s Aerogel, Axiom, Aeropyox</strong></td>
</tr>
<tr>
<td>B-1</td>
<td>71.6</td>
<td>17.9</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<td>B-2</td>
<td>83.4</td>
<td>20.8</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<td>C-2</td>
<td>43.1</td>
<td>10.8</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<td>B-3</td>
<td>85.7</td>
<td>21.4</td>
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<td>B-4</td>
<td>72.2</td>
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<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<tr>
<td>B-5</td>
<td>82.6</td>
<td>20.6</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<tr>
<td>C-4</td>
<td>55.1</td>
<td>13.8</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<tr>
<td>C-5</td>
<td>50.6</td>
<td>12.6</td>
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<tr>
<td>C-1</td>
<td>24.7</td>
<td>6.2</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<tr>
<td>C-3</td>
<td>15.9</td>
<td>4</td>
<td>4-ply, [0,45,90,-45] with Aerogel felt</td>
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<tr>
<td><strong>Average</strong></td>
<td><strong>58.5</strong></td>
<td><strong>14.6</strong></td>
<td><strong>4-ply [0,45,90,-45] Aerogel, Axiom, Aeropyox</strong></td>
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<tr>
<td><strong>Average (C-1, C-3 removed)</strong></td>
<td><strong>68.0</strong></td>
<td><strong>17.0</strong></td>
<td><strong>4-ply [0,45,90,-45] Aerogel, Axiom, Aeropyox</strong></td>
</tr>
</tbody>
</table>
Mechanical Testing Evaluation
AeroFiber Composites Summary

• Built on KSC’s knowledge base of aerogel composites
• Composites included carbon, Innegra, Spectra and Kevlar and combinations thereof
• **Insulative** by tailoring hybrid laminate composite architecture provides for 25-75% reduction in heat transfer compared to a fiber-only composite system
• **Lightweight**, use of this composite sandwich structure can offer substantial weight savings compared to metals, metal alloys and some fiber alone composite systems
• **Tailorable Designs**, enables unique combinations of properties in one architectural system
• Analyses included thermal conductivity on Macroflash and Cryostat-500, physical and mechanical analyses
• Cryo-impact data indicated increased energy absorption
• Demonstrated the Aerocover concept design and prototype for a heavy lift vehicle interface
Aerogel Hybrid Composites

**AeroFoam** is a new hybrid foam/aerogel composite

**AeroPlastic** is a new composite material of thermoplastics and aerogel particle combinations

**AeroFiber** is a new hybrid laminate system composed of fiber composites and aerogel blankets

- All are tailorable and represent families of different approaches, designs, and combinations
- All are available for licensing
• All technologies have commercial industries and aerospace/space exploration applications

• **AeroFoam** is a hybrid foam/aerogel composite that is multi-functional for reducing heat transfer, improved attenuation properties, fire resistant and cryogenic storage capabilities

• **AeroPlastic** is a new composite material of thermoplastics and aerogel particle combinations
  – Most effective approach of reducing heat transfer in thermoplastics, a science/an art
  – Expands the use of high engineered polymers in cryogenic systems
  – Recognized for innovative approaches by Techconnect Showcase and Innovation Award in 2015

• **AeroFiber** systems provide a tunable system that provides both thermal and structural properties with its integrated/layered approach
Thank you for your attention!

Dr. Martha K. Williams
Lead Polymer Scientist,
Sr. Principal Investigator, UB-R3
1.321. 867.4554
Martha.K.Williams@nasa.gov

James E. Fesmire
Sr. Principal Investigator, UB-R1
Cryogenics Test Laboratory
1.321.867.7557
james.e.fesmire@nasa.gov
Exploration and Technology
NASA Kennedy Space Center, FL 32899  USA

Questions on technology transfer or commercial licensing opportunities?
Please contact:
Jeff Kohler
jeffrey.a.kohler@nasa.gov
1.321.861.7158

Acknowledgement of Other Inventors:
AeroPlastic includes: Trent Smith, Dr. Luke Roberson and Dr. LaNetra Tate
Aerofoam includes: Trent Smith and Dr. Erik Weiser (Langley Research Center) and Jared Sass
Aerofiber includes: Dr. Luke Roberson, Judith McFall, Anne Caraccio-Meier, Dr. LaNetra Tate and Chad Brown


Back-up Slides
NASA has a history of collaboration with industry for the development of aerogels in different forms. Aerogel composite blanket development began in 1993 through a Small Business Innovation Research (SBIR) program between Aspen Systems, Inc. (Marlborough, MA) and NASA Kennedy Space Center (KSC, FL). The motivation was to enable new and improved thermal insulation systems for cryogenic and other high-performance needs. Scientists at KSC have continued to expand the design, development, and applicability of using aerogels by developing advanced composite materials and systems with multi-functional capabilities. Hybrid composite systems include aerogel/polymer composites (AeroPlastic), aerogel/foam composites (AeroFoam), and aerogel/fiber laminate systems (AeroFiber).
Abstract

Aerogel Hybrid Composite Materials: Designs and Testing for Multifunctional Applications

This webinar will introduce the broad spectrum of aerogel composites and their diverse performance properties such as reduced heat transfer to energy storage, and expands specifically on the aerogel/fiber laminate systems and testing methodologies. The multi-functional laminate composite system, AeroFiber, and its construction is designed by varying the type of fiber (e.g. polyester, carbon, Kevlar®, Spectra® or Innegra™ and combinations thereof), the aerogel panel type and thickness, and overall layup configuration. The combination and design of materials may be customized and tailored to achieve a range of desired properties in the resulting laminate system. Multi-functional properties include structural strength, impact resistance, reduction in heat transfer, increased fire resistance, mechanical energy absorption, and acoustic energy dampening. Applications include aerospace, aircraft, automotive, boating, building and construction, lightweight portable structures, liquefied natural gas, cryogenics, transportation and energy, sporting equipment, and military protective gear industries.
1. Thermal isolation is needed for energy savings, system control, and/or safety/reliability.

2. Thermal insulation is often an afterthought or something to be dealt with later in the design process.

3. Different working fluids need thermal isolation: chilled water, cold air, Freon, CO₂, LO₂, LN₂, LNG (or LCH₄), LH₂, or LHe, etc.

4. Mechanical complexity for below-ambient systems is often the norm and challenges are increased multifold for:
   - Mechanical/vibration loads
   - Weathering environments
   - Accessibility/maintenance

5. Thermal insulation systems must also be lightweight and meet a wide range of fire, compatibility, outgassing, and other physical and chemical requirements.
   - Thermal conductivity is important, but usually is not at the top of the list!
Aerogel Composites Summary

AeroFoam = polyimide foam + aerogel

AeroPlastic = thermoplastic + aerogel
Extruded process, composite reducing heat transfer by 40-60%. Cryogen storage/transfer applications such as piping and seal. Also in wood plastics, and oil and gas. Patents: US 7,790,787 B2 and divisional

Aerofiber Laminate Composites (Fiber/Textile Composites + aerogel)
structural and thermal composites, patent pending

Aerofoam - vibration attenuation testing

AeroPlastic demo testing on cryo-piping system
Dianhydride + Alcohol
Alkyl Alcohol

Agent

Dialkylester-Diacid
complexed by an Agent

Diamine

Stable Mixture at Ambient Conditions (Stablized by the Agent)

Agent, Water, & Alcohol By-Products

Polyimide Foam

4,4’-oxydiphthalic anhydride / 3,4’-ODA

3,4’-oxydianiline

TEEK-HH (0.082 g/cm³) and TEEK-IL (0.032 g/cm³), ODPA/3,4’-ODA

Heat Plate
Upper Graphite Plate
Mold Chamber
Expandable Powder
Lower Graphite Plate
Heat Plate
## TEEK/Aero Foam-K-values

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Material</th>
<th>Date Fabricated</th>
<th>Density (lb/ft³)</th>
<th>k-value (mW/m-K)</th>
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<td>28</td>
<td>N096</td>
<td>Horizontal Strips (8)</td>
<td>3/31/2006</td>
<td>2.55</td>
</tr>
<tr>
<td>29</td>
<td>N097</td>
<td>Vertical Strips (6)</td>
<td>3/29/2006</td>
<td>2.53</td>
</tr>
<tr>
<td>30</td>
<td>N132</td>
<td>Single Layer 10% Beads</td>
<td>7/26/2006</td>
<td>2.88</td>
</tr>
<tr>
<td>31</td>
<td>N133</td>
<td>Diagonal Strip (9) 30% Beads</td>
<td>8/7/2006</td>
<td>2.34</td>
</tr>
<tr>
<td>32</td>
<td>N134</td>
<td>Single Layer 30% Beads</td>
<td>8/2/2006</td>
<td>3.45</td>
</tr>
</tbody>
</table>
Aerogel and Fiber Composite Laminates

<table>
<thead>
<tr>
<th>Material</th>
<th>Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>5.7 oz/yd²</td>
</tr>
<tr>
<td>Innegra™ S</td>
<td>4.0 oz/yd²</td>
</tr>
<tr>
<td>Innegra™ S</td>
<td>6.0 oz/yd²</td>
</tr>
<tr>
<td>Kevlar®</td>
<td>5.0 oz/yd²</td>
</tr>
<tr>
<td>Trevira Core 150</td>
<td>12.3 oz/yd²</td>
</tr>
<tr>
<td>Spectra® 1000</td>
<td>3.5 oz/yd²</td>
</tr>
</tbody>
</table>

Legend
C: Carbon
P: Polyester
I: Innegra S
S: Spectra 1000
K: Kevlar

Number after letter is the number of layers of the Fabric

(6) Indicates the total number of layers in the composite

Example (6) indicates that the composite has 6 layers of fabric

• Vacuum infusion for fiber composites
• Adhesive lamination

Cryo-Impact Results
Thermal Conductivity Testing

- Cryostat-100: absolute, cylindrical
- Cryostat-200: comparative, cylindrical
- Cryostat-500/600: absolute, disk
- Cryostat-400: comparative, disk
- Macroflash Cup Cryostat: comparative, disk
- Netzsch Heat Flow Meter: calibrated, disk
- Anter Quick Line 10 Thermal Instrument: calibrated, disk
- Anter Quick Line 30 Thermal Analyzer: calibrated, probe

Thermal Performance Testing

- Cryogenic Moisture Uptake (CMU) Apparatus
- Spherical Cryostat (1000-liter)
- Cryogenic Pipeline Test Apparatus (CPTA)
- Sub-scale tanks and 10-liter dewar kits
- Thermal cycling, expansion & contraction, loads & vibration effects
Boundary temperatures 78 K & 293 K; nitrogen residual gas. See ASTM C740 or C1774.


The Cryostat-500 insulation test instrument provides:

- Testing 204-mm diameter, 25-mm thick specimens under representative-use conditions.
- Direct energy rate measurement by LN$_2$ boiloff calorimetry.
- Reliable testing of non-homogenous, non-isotropic thermal insulation systems.
- ASTM C1774, Annex A3
Testing and Evaluation of Thin Layered Composites

• Transient Thermal Tester
Aeroplastic Market Size/Impact

- Engineered composites with improved thermal efficiency for the aerospace, cryogenics, oil and gas, automotive, electronics, military, wood plastics, medical, food packaging and textile markets

- **Market volume of thermoplastic piping oil and gas applications** CAGR of 5.5%, to $75B from now to 2020
  
  [http://www.researchandmarkets.com/research/8pvqpz/thermoplastic](http://www.researchandmarkets.com/research/8pvqpz/thermoplastic)

- Wood plastics composites alone expected to grow to ~$5B in 2019
  

- Polyamide 6 volume market expected to reach 4.6M tons by 2018, US$16.5 billion by 2018.
  
Void Content Analysis

- Should be less than 1%

\[
T = \frac{100}{(R/D + r/d)} \quad (1)
\]
\[
V = \frac{100(T_d - M_d)}{T_d} \quad (2)
\]

Where:

- \( T \) = Theoretical density
- \( R \) = Resin in composite, weight %
- \( D \) = Density of composite
- \( r \) = reinforcement in composite, weight %
- \( d \) = density of reinforcement
- \( V \) = Void content, volume %
- \( T_d \) = Theoretical Composite density
- \( M_d \) = Measured composite density

Table 1: Void Content Analysis

<table>
<thead>
<tr>
<th>Void Content, Volume %</th>
<th>3k [0, 45, 90, -45]s</th>
<th>3k [-45, 90, 45, 0]</th>
<th>3k [0, 45, 90, -45]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>Sample 2</td>
<td>Sample 1</td>
<td>Sample 2</td>
</tr>
<tr>
<td>1.12</td>
<td>3.14</td>
<td>12.04</td>
<td>9.87</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10.39</td>
<td>12.67</td>
</tr>
</tbody>
</table>