Standardization in Cryogenic Insulation Testing and Performance Data

James E. Fesmire

Cryogenics Test Laboratory
NASA Kennedy Space Center, FL USA

International Cryogenic Engineering Conference and
International Cryogenic Materials Conference
University of Twente, Enschede, Netherlands
July 7-11 2014
Background

- Thermal insulation provides:
  - energy savings over time,
  - system control,
  - and/or process safety.

- Adverse effects of sudden loss of vacuum, flow induced vibrations, pressure transients, transient structural loads, or inability to achieve certain conditions.

- What thermal insulation system is the best for a given application depends on the operational environment, mechanical design, and insulation materials.

- Economic objectives underscore the technical approach to be taken: *thermal performance must justify the cost*.

- Standard sets of thermal data are needed to make proper design trade-offs and come up with the best solution.

- But we first need *standard ways of testing and reporting* those data.
Electrical Performance

- Circuit boards below are for the brains of the Tesla automobile.
- Measure electrical resistance of a wire and then determine the electrical performance of the boards? **No!**
Thermal Performance

- The tank below is for a truck’s LNG fuel supply.
- Measure **thermal resistance** of a piece of material and then determine the thermal performance of the tank? **No!**
Motivation for Standardization

- Broader use of cryogenic systems in commercial enterprises worldwide.
- One area in particular is the emerging cryofuels usage for transportation.
- From the future passenger car, commercial space launch vehicle, to the floating liquefied natural gas platform, the successful proliferation of these efforts will be partly enabled by the adequate isolation of liquid hydrogen or liquefied natural gas from the ambient environment.
- The end-user applications involving transportation necessarily include many transient operations.
- Transient, or on and off, cold and warm, operations are a challenge for the design of robust, safe systems that meet their overall cost objectives.
Key Terminology

- **Cold boundary temperature (CBT)** — the cold temperature imposed on cold-side surface of the insulation system by the cold mass (K).
  - *May be cooled by a cryogen or a cryocooler.*

- **Warm boundary temperature (WBT)** — the warm temperature imposed on the warm-side surface of the insulation system by the warm mass (K).
  - *May be heated by an electrical heater, liquid bath heat exchanger, or ambient environment.*

- **Cold vacuum pressure (CVP)** — the steady-state vacuum pressure level within the insulation system achieved after cooldown (Pa or millitorr; 1 millitorr = 0.133 Pa).
  - *Can be any pressure from high vacuum to no vacuum, with or without a residual gas.*

- **Warm vacuum pressure (WVP)** — the vacuum pressure level within the insulation system before cooldown.

- **Heat flow rate (Q)** — quantity of heat energy transferred to or from the insulation system in a unit of time (W).

- **Heat flux (q)** — heat flow rate, under steady-state conditions, in a direction perpendicular to the plane of the insulation system (W/m²).
  - *Based on the effective heat transfer area (Aₑ)*

- **Effective thermal conductivity (kₑ)** — the thermal conductivity through the total thickness of the insulation system between the reported boundary temperatures and in a specified environment (mW/m-K).
  - *The insulation system may be one material, homogeneous non-homogeneous, or a combination of materials.*

- **System thermal conductivity (kₛ)** — the thermal conductivity through the total thickness of the insulation system plus any ancillary elements such as packaging, supports, getter packages, enclosure, outer jacket, etc. (mW/m-K).
Three categories of CVP using the example of heat flux through an MLI system (boundary conditions are 77 K and 293 K with residual gas of nitrogen). The MLI system is aluminum foil and micro-fiberglass paper (40 layers at a density of 3.6 layers per millimeter).
Technical Consensus Standards

• Through ASTM International, Committee C16 on Thermal Insulation, two new standards were recently published:

• Both standards are comprehensive guides that provide the necessary terminology, analytical approaches, and reporting requirements for the technology area of cryogenic insulation systems.

• Advances in test apparatus, methods, and materials have provided a foundation for these new standards.
Guide to Cryogenic Thermal Performance Testing

• Covers six different testing approaches/apparatus including:
  – Boiloff calorimetry and electrical power methods.
  – Absolute and comparative methods.
• Applicable to a wide variety of specimens, from opaque solids to porous or transparent materials to composite systems:
  – A “system” may be composed of one or more materials that may be homogeneous or non-homogeneous.
  – Flat, cylindrical, or spherical geometries.
  – Highly anisotropic materials and layered systems such as multilayer insulation (MLI).
• Includes a wide range of environmental conditions:
  – Including various gases and over a range of pressures.
  – Boundary conditions from 4 to 400 K.
  – Environments from high vacuum to an ambient pressure of air or residual gas.
• Laboratory measurement and calculations of the steady-state thermal transmission properties and heat flux.
• A key aspect of this guide is the notion of an insulation system, not an insulation material. *Under the practical use environment of most cryogenic applications, even a single-material system can be a complex insulation system.*
Typical characteristics of boiloff calorimeter instruments (cryostats):

<table>
<thead>
<tr>
<th>Type</th>
<th>Name</th>
<th>Heat Flux ($q$)</th>
<th>$k_e$</th>
<th>Specimen Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cylindrical Absolute</td>
<td>Cryostat-100</td>
<td>0.1 to 500</td>
<td>0.01 to 60</td>
<td>1-m length; up to 50-mm thickness</td>
</tr>
<tr>
<td>Cylindrical Comparative</td>
<td>Cryostat-200</td>
<td>1 to 500</td>
<td>0.1 to 60</td>
<td>0.5-m length; up to 30-mm thickness</td>
</tr>
<tr>
<td>Flat Plate Absolute</td>
<td>Cryostat-500</td>
<td>1 to 1,000</td>
<td>0.05 to 100</td>
<td>200-mm diameter; up to 30-mm thickness</td>
</tr>
<tr>
<td>Flat Plate Comparative</td>
<td>Cryostat-400</td>
<td>10 to 1,000</td>
<td>0.5 to 100</td>
<td>200-mm diameter; up to 30-mm thickness</td>
</tr>
</tbody>
</table>
Guide to Cryogenic Thermal Performance Testing

General arrangement of a flat plate boiloff apparatus (left) and a cylindrical boiloff apparatus (right):
Guide to Cryogenic Multilayer Insulation Systems

- Covers the heat flux or thermal conductivity data, performance considerations, typical applications, manufacturing methods, material specification, and safety considerations:
  - Warm boundary temperatures of 300 K or higher and cold boundary temperatures from 4 K to 111 K, but any temperature below ambient is applicable.
  - Typically used in a high vacuum environment (evacuated), but soft vacuum or no vacuum environments are also applicable.

- Heat flux values well below 1 W/m² are achievable in high vacuum MLI systems. Other evacuated insulations (bulk-fill materials) can provide heat flux values in the range of 5 to 20 W/m².

- For comparison among different systems, or for space & weight considerations, the effective thermal conductivity of the system can be calculated for a specific total thickness:
  - Effective thermal conductivities of less than 1 mW/m-K [R-value 143] are typical.
  - Values on the order of 0.01 mW/m-K have been achieved [R-value 14,300].

- Applications categories: storage, transfer, thermal protection, and low-temperature processes. Very low temperature (4 K and below) refrigeration is a major technical capability for basic physics research world-wide.
Cylindrical Absolute Boiloff Test Apparatus

- Cylindrical absolute boiloff test apparatus (Cryostat-100): Lift mechanism, vacuum-pressure regulation ports, and funnel filling assembly (left); schematic, guard chambers, and temperature sensor locations (right).
The steady-state heat flow rate \(Q\) is the basis for calculating the thermal properties including effective thermal conductivity \(k_e\), or system thermal conductivity \(k_s\), and heat flux \(q\). This heat flow rate through the insulation test specimen and into the cold-mass tank is directly proportional to the liquid nitrogen boiloff rate.

The heat flux \(q\) is calculated by dividing the total heat transfer rate by the effective area for heat transfer:

\[
q = \frac{Q}{A_e}
\]

Calculations of \(k_e\) are highly sensitive to the thickness of the test specimen:

\[
k_e = \frac{Q x}{A_e T}
\]

For Cylindrical geometry:

\[
k_e = \frac{Q \ln\left(\frac{d_o}{d_i}\right)}{2 L_e T}
\]

The steady-state condition is reached when the boiloff flow rates from all three chambers are stabilized, the temperature profile through the thickness is stabilized, and the liquid level in the test chamber is at least 90% full.

The total test duration may be several hours to several days depending on the range of heat flow involved.
Benchmark data summary from Cryostat-100 testing with LN₂:

- Boiloff flow rate (m-dot)
- Heat (Q)
- Heat Flux (q)
- Effective thermal conductivity ($k_e$)

Materials include:
- Vacuum Only (black sleeve)
- Three different MIL systems
- Aerogel blanket
- Fiberglass blanket
- Glass bubbles
- Perlite powder
- Aerogel particles
- Spray-on foam

### Table: Thermal Performance – Data Summary

<table>
<thead>
<tr>
<th>Test Specimen</th>
<th>CVP millitorr</th>
<th>WBT K</th>
<th>Flow Q W</th>
<th>$q$ W/m²</th>
<th>$k_e$ W/m-K</th>
<th>$x$ m</th>
<th>$n$ µm</th>
<th>$A$ mm²</th>
<th>$\rho$ kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>A114 Vacuum Only</td>
<td>100000</td>
<td>165</td>
<td>1141</td>
<td>30.8</td>
<td>100.5</td>
<td>10.49</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Black sleeve</td>
<td>100000</td>
<td>125</td>
<td>1321</td>
<td>36.9</td>
<td>105.9</td>
<td>12.02</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- 100000</td>
<td>192.8</td>
<td>1321</td>
<td>36.9</td>
<td>105.8</td>
<td>12.02</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td>A154 MLI Mylar-Net (49, 10, 42)</td>
<td>100000</td>
<td>293.0</td>
<td>78</td>
<td>0.312</td>
<td>0.097</td>
<td>0.023</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Double-aliunized Mylar and polyester net space</td>
<td>100000</td>
<td>293.2</td>
<td>78</td>
<td>0.312</td>
<td>0.097</td>
<td>0.023</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Layer by layer installation</td>
<td>100000</td>
<td>293.3</td>
<td>78</td>
<td>0.312</td>
<td>0.097</td>
<td>0.023</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>A125 MLI Mylar-Net (16, 40, -)</td>
<td>100000</td>
<td>31.4</td>
<td>32.3</td>
<td>0.22</td>
<td>0.065</td>
<td>0.028</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Double-aliunized Mylar and polyester net space</td>
<td>100000</td>
<td>293.5</td>
<td>78</td>
<td>0.312</td>
<td>0.097</td>
<td>0.023</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Layer by layer installation</td>
<td>100000</td>
<td>293.6</td>
<td>78</td>
<td>0.312</td>
<td>0.097</td>
<td>0.023</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>A128 MLI Foil-Paper (21, 80, -)</td>
<td>100000</td>
<td>293.0</td>
<td>33.9</td>
<td>0.22</td>
<td>0.065</td>
<td>0.028</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Aluminum foil/continuous rolled and microfiber glass paper space</td>
<td>100000</td>
<td>293.0</td>
<td>33.9</td>
<td>0.22</td>
<td>0.065</td>
<td>0.028</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Continuous-rolled installation</td>
<td>100000</td>
<td>293.0</td>
<td>33.9</td>
<td>0.22</td>
<td>0.065</td>
<td>0.028</td>
<td>1.25</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>A112 Aerogel Blanket (23, 2, 133)</td>
<td>100000</td>
<td>1060</td>
<td>4.800</td>
<td>12.4</td>
<td>1.47</td>
<td>0.04</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>- Cryogel aerogel composite</td>
<td>100000</td>
<td>1060</td>
<td>4.800</td>
<td>12.4</td>
<td>1.47</td>
<td>0.04</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>- Microfiber glass batt</td>
<td>100000</td>
<td>1060</td>
<td>4.800</td>
<td>12.4</td>
<td>1.47</td>
<td>0.04</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>A151 Fiberglass (49, 2, 16)</td>
<td>100000</td>
<td>1000</td>
<td>1.36</td>
<td>26.2</td>
<td>6.53</td>
<td>12.8</td>
<td>0.04</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>- Microfiber glass batt</td>
<td>100000</td>
<td>1000</td>
<td>1.36</td>
<td>26.2</td>
<td>6.53</td>
<td>12.8</td>
<td>0.04</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>A102 Glass Bubbles (25, 1, 65)</td>
<td>100000</td>
<td>1072</td>
<td>44.4</td>
<td>115.0</td>
<td>25.4</td>
<td>8.06</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Type K1 hollow microspheres</td>
<td>100000</td>
<td>1072</td>
<td>44.4</td>
<td>115.0</td>
<td>25.4</td>
<td>8.06</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Black sleeve</td>
<td>100000</td>
<td>1072</td>
<td>44.4</td>
<td>115.0</td>
<td>25.4</td>
<td>8.06</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>A103 Perlite Powder (25, 1, 132)</td>
<td>100000</td>
<td>297.2</td>
<td>7620</td>
<td>31.5</td>
<td>98.5</td>
<td>10.49</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- High density</td>
<td>100000</td>
<td>297.2</td>
<td>7620</td>
<td>31.5</td>
<td>98.5</td>
<td>10.49</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>- Black sleeve</td>
<td>100000</td>
<td>297.2</td>
<td>7620</td>
<td>31.5</td>
<td>98.5</td>
<td>10.49</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
<tr>
<td>A108 Aerogel Particles (25, 1, 80)</td>
<td>100000</td>
<td>297.2</td>
<td>7620</td>
<td>31.5</td>
<td>98.5</td>
<td>10.49</td>
<td>1.26</td>
<td>8.99</td>
<td>0.1</td>
</tr>
</tbody>
</table>

**Materials include:**

- Vacuum Only (black sleeve)
- Three different MIL systems
- Aerogel blanket
- Fiberglass blanket
- Glass bubbles
- Perlite powder
- Aerogel particles
- Spray-on foam

**Notes:**

- CVP: Critical Vacuum Pressure
- WBT: Water Black Temperature
- Flow Q W: Flow Rate in Watts
- $q$: Heat Flux in Watts/m²
- $k_e$: Effective Thermal Conductivity in W/m-K
- $x$, $n$, $A$: Dimensions
- $\rho$: Density in kg/m³
Variation of effective thermal conductivity ($k_e$) with CVP for different cryogenic insulation systems and materials. Boundary temperatures: 78 K and 293 K. Residual gas is nitrogen.

Notes:
1. Boundary Temperatures approximately 78 K & 293 K.
2. Residual gas nitrogen.
3. Legend data (25, 40, 55) means: 25 mm thickness, 40 layers, and 55 kg/m$^3$ bulk density [x, n, ρ].
Variation of heat flux ($q$) with CVP for different cryogenic insulation systems and materials. Boundary temperatures: 78 K and 293 K. Residual gas is nitrogen.

Notes:
1. Boundary Temperatures approximately 78 K & 293 K.
2. Residual gas nitrogen.
3. Legend data (25, 40, 55) means: 25 mm thickness, 40 layers, and 55 kg/m$^3$ bulk density [$x$, $n$, $\rho$].
Cryogenic Insulation Standards:
- ISO 21014 Cryogenic Vessels: Cryogenic Insulation Performance
- ASTM C740 Standard Guide for Evacuated Reflective Insulation in Cryogenic Service

Insulation Materials Standards with Mention of Cryogenic Temperatures:
- ASTM C1728 Standard Specification for Flexible Aerogel Insulation
- ASTM C534 Standard Specification for Preformed Flexible Elastomeric Cellular Thermal Insulation in Sheet and Tubular Form
- ASTM C549 Standard Specification for Perlite Loose Fill Insulation
- ASTM C552 Standard Specification for Cellular Glass Thermal Insulation
- ASTM C1482 Standard Specification for Polyimide Flexible Cellular Thermal & Sound Absorbing Insulation
- ASTM C1594 Standard Specification for Polyimide Rigid Cellular Thermal Insulation
• Future technical consensus standards are envisioned for both test methods and materials practices.
• Specific test methods would be formulated for cylindrical and flat plate geometries covering absolute and comparative approaches, as required by mutual industry needs. Also:
  – Tanks and Pipelines
  – Water Vapor Sorption and Evacuation/Purging/Heating
• Materials of interest include, for example:
  – Non-Vacuum Multilayer Insulation Systems in Cryogenic Service
  – Glass Microspheres Loose-Fill Insulation Materials in Cryogenic Service
  – Aerogel Loose-Fill Insulation Materials in Cryogenic Service
  – Aerogel Blanket Insulation Materials in Cryogenic Service
  – Low-Density Cellular Foam Insulation in Cryogenic Service
• Standard data sets for specific materials would then be produced through a round robin of cryogenic testing among laboratories.
Preservation of the Cold

ΔT = 500 °F

ΔT = 500 °F

H₂O

LH₂
• Energy efficiency, system control, and operational safety are inter-related aspects of deciding the best thermal insulation system for a particular application.

• Emerging cryofuels enterprises including LNG and LH2 are a particular challenge due to the transient operational processes to be addressed and the competitive economic targets to be met.

• New ASTM standards (C1774 and C740) are one step toward providing thermal performance data and materials specifications for the cryogenic industry.

• Cryostat-100 insulation test apparatus has provided thermal performance data for a wide range of different materials under relevant conditions.

• Benchmark thermal performance data can be used to calibrate comparative instruments and support detailed studies of insulation system designs for specific applications.

• Future technical consensus standards are envisioned for both test methods and materials practices to support the cryogenic industry and further the proliferation of new industrial opportunities in the areas of transportation and energy.
Standardization in Cryogenic Insulation Testing and Performance Data


