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Standardization in cryogenic insulation systems testing and performance data

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Abstract

The close relationship between industrial energy use and cryogenics drives the need for optimized thermal insulation systems. Emerging cryofuels usage is enabled by adequate isolation of the liquid hydrogen or liquefied natural gas from the ambient environment. Thermal performance data for the total insulation system, as rendered, are essential for both engineering designs and cost-benefit decisions involving comparisons among alternatives. These data are obtained through rigorous testing with suitable apparatus and repeatable methods. Properly defined terminology, analysis, and reporting are also vital. Advances in cryogenic insulation test apparatus and methods have led to the recent addition of two new technical standards of ASTM International: C1774 - *Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems* and C740 - *Standard Guide for Evacuated Reflective Cryogenic Insulation*. Among the different techniques described in the new standards is the cylindrical boiloff calorimeter for absolute heat measurement over the full range of vacuum pressure conditions. The details of this apparatus, test method, and data analysis are given. Benchmark thermal performance data, including effective thermal conductivity (k_e) and heat flux (q) for the boundary temperatures of 293 K and 77 K, are given for a number of different multilayer insulation (MLI) systems in comparison with data for other commonly-used insulation systems including perlite powder, fiberglass, polyurethane foam, and aerogels.

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Keywords: Thermal insulation; boiloff calorimetry; thermal conductivity; heat flux; multilayer insulation; cryogenic testing; materials; standards.

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Thermal insulation plays a significant role in most cryogenic systems. Insulation provides net energy savings over time, sufficient system control and process safety, or often a combination of these two requirements. The system control and safety aspects of thermal insulation systems are often overlooked or taken for granted. Such aspects could involve the adverse effects of sudden loss of vacuum, flow induced vibrations, pressure transients, transient structural loads, or inability to achieve certain flow conditions within a given timeline. 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. The primary objective that *thermal performance must justify the cost* is discussed by Augustynowicz et al. (2000). Standard sets of thermal performance data for insulation materials are beneficial in making the proper design trade-offs and coming up with the best solution. But to have such standard data first requires that there be standard ways of testing and reporting those data.

Nome	Nomenclature					
h_{fg}	heat of vaporization, J/g					
ΔŤ	temperature difference, K					
A _e	effective heat transfer area, m ²					
Le	effective length of test specimen, m					
d _o , d _i	outer and inner diameters of test specimen, m					
Q	heat flow rate, W					
Q	heat flux, W/m^2					
k _e	effective thermal conductivity, W/m·K					

1.1. Applications and future growth

Beyond the basic needs for thermal insulation in field of cryogenics, there is growing movement toward 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 are necessarily include many transient operations. These transient, or on and off, cold and warm, operations are a particular challenge for the design of robust, safe systems that meet their overall cost objectives.

1.2. Operational environments

The operational environment is the first and foremost discriminator is deciding the best cryogenic insulation for the job. Three main categories can be defined by their dominant mechanisms of heat transfer: radiation, gas conduction, and convection. Each category has a range of residual gas pressure known as the cold vacuum pressure (CVP). Solid conduction is a constant and there is practical overlap among the three categories.

The total range of pressure is from 10^{-7} to 10^{+3} torr $(1.33^{-8}$ to 1.33^{+2} kPa) with different residual gases such as air, nitrogen or another purge gas. Corresponding to applications in cryogenic systems, the three categories of CVP are listed as follows: High Vacuum (HV) from $<10^{-6}$ to 10^{-3} torr $(1.333^{-4}$ to 0.133 Pa) [free molecular regime], Soft Vacuum (SV) from 10^{-2} to 10 torr (from 1.33 to 1,333 Pa) [transition regime], and No Vacuum (NV) from 100 to 1000 torr (13.3 to 133 kPa) [continuum regime].

Thermal performance can vary by four orders of magnitude over the vacuum pressure range. Effective thermal conductivities can range from 0.010 to 100 mW/m-K. The primary governing factor in thermal performance is the pressure of the test environment. High vacuum insulation systems are often in the range from 0.05 to 2 mW/m-K while non-vacuum systems are typically in the range from 10 to 30 mW/m-K. Soft vacuum systems generally perform in between these two extremes.

1.3. Key terminology

Cryogenic insulation systems encompass a wide range of material combinations. An insulation test specimen is composed of one or more materials, homogeneous or non-homogeneous, for which thermal transmission properties through its thickness are to be measured under sub-ambient conditions. The test specimen may consist of a single material, one type of material in several discrete elements, or a number of different materials working in a specialized design configuration. In reality, a test specimen is always a system, either a single material (with or without inclusion of a gas) or a combination of materials in different forms. The key terminology for thermal performance testing and reporting of data is listed as follows:

- *Cold boundary temperature* (CBT)—the cold temperature imposed on cold-side surface of the insulation system by the cold mass (K). The cold mass 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). The warm mass 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). The CVP 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, through the effective heat transfer area (A_e), in a direction perpendicular to the plane of the insulation system (W/m²).
- *Effective thermal conductivity* (k_e)—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 (k_s)—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).

As examined by Fesmire et al. (2008), the definition for k_e is distinguished from other terms for thermal conductivity. In accordance with ASTM C168 (2013), thermal conductivity (λ) is for a homogeneous material with a single mode of heat transfer and is generally independent of thickness. Apparent thermal conductivity (λ_a) is for a material that exhibits thermal transmission by several modes of heat transfer that often results from property variations such as thickness, surface emittance, and cellular or interstitial content. Test methods ASTM C518 and ASTM C177 use relatively small Δ T in the measurement of λ or λ_a . These issues for homogeneous materials under moderate conditions are much more pronounced in cryogenic-vacuum conditions and the low-density materials of interest. Practice C168 points out that it may not work for the following cases: (1) onset of convection, (2) abrupt change in phase of an insulation component such as a condensable gas, and (3) heat flow anomalies found in reflective insulations. These cases are often found in cryogenic insulation systems and large Δ T from 200 K to 300 K are typical in most applications. Technical consensus standards

Through ASTM International, Committee C16 on Thermal Insulation, two new standards are published: ASTM C1774 - *Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems* (2013) and ASTM C740 - *Standard Guide for Evacuated Reflective Cryogenic Insulation* (2014). Both standards provide the terminology, analytical approaches, and reporting requirements. Advances in test apparatus, methods, and materials have provided a foundation for these new standards.

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. Standard data sets for specific materials would then be produced through a round robin of cryogenic testing among laboratories, thus enabling new standard material practices.

2.1 Guide to cryogenic thermal performance testing

The Guide to cryogenic thermal performance testing covers different approaches to both boiloff calorimetry and electrical power methods for thermal performance measurement in sub-ambient temperature environments. The test apparatus designed for these purposes are divided into two categories: boiloff calorimetry and electrical power. Both absolute and comparative apparatuses methods are included.

These testing approaches methods are applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions including measurements conducted at extremes temperatures, with various gases and over a range of pressures. Of particular importance is the ability to test highly anisotropic materials and systems such as multilayer insulation (MLI) systems.

The guide provides information for the laboratory measurement of the steady-state thermal transmission properties and heat flux through thermal insulation systems under cryogenic conditions. Thermal insulation systems may be composed of one or more materials that may be homogeneous or non-homogeneous; flat, cylindrical, or spherical; at boundary conditions from 4 to 400 K; and in environments from high vacuum to an ambient pressure of air or residual gas. 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 still be a complex insulation system.

Boiloff calorimetry provides a straightforward approach for testing low-temperature thermal insulation systems that reflect the real-world conditions. These conditions are usually based on a large temperature difference and a particular environmental pressure. The geometry of the test materials, cylindrical or flat plate, can also be crucial for obtaining a representative test and thus the accurate thermal data. Typical characteristics of boiloff devices are given in ASTM C1774 along with simplified schematics for both cylindrical and flat plate designs.

2.2 Guide to cryogenic multilayer insulation systems

The Guide to cryogenic multilayer insulation systems covers the baseline heat flux or thermal conductivity data, performance considerations, typical applications, manufacturing methods, material specification, and safety considerations in the use of MLI systems in cryogenic service.

These systems often involve warm boundary temperatures in the range of 300 K or higher and cold boundary temperatures ranging from 4 K to 111 K, but any temperature below ambient is applicable. Insulation systems of this construction are used when heat flux values well below 10 W/m² are needed for an evacuated design. Heat flux values approaching 0.1 W/m² are also achievable. For comparison among different systems, as well as for space and weight considerations, the k_e of the system can be calculated for a total thickness. Effective thermal conductivities of less than 1 mW/m-K are typical and values on the order of 0.01 mW/m-K have been achieved. Thermal performance can also be described in terms of the effective emittance of the system, or E_e.

These systems are typically used in a high vacuum environment (evacuated), but soft vacuum or no vacuum environments are also applicable. A welded metal, vacuum-jacketed (VJ) enclosure is often used to provide the vacuum environment.

MLI systems are generally used when lower heat leakage rates than those obtained with other evacuated insulations are required. Other evacuated insulations include, for example, perlite powder, glass bubbles, or aerogel bulk-fill, which can provide heat flux values in the range of 5 to 20 W/m². The choice for an MLI system may be dictated by the value of the cryogenic fluid being isolated or by weight or thickness limitations imposed by the particular application. Applications generally fall into the following categories: storage, transfer, thermal protection, and low-temperature processes. Very low temperature (4 K and below) refrigeration for large-scale superconducting magnets, RF cavities, and other devices is a major technical capability for basic physics research world-wide.

2. Cylindrical absolute boiloff test apparatus

The use of boiloff calorimetry to measure the effects of thermal energy (or heat) dates back to the early 1900s. The gas flow rate enables direct calculation of thermal performance. Because heat does not flow through a material

as a function of temperature but according to a temperature difference, the use of a cryogen such as liquid nitrogen (LN2) also provides a convenient way to establish representative test conditions for a wide range of applications.

There are a number of advantages of using boiloff calorimetry to test thermal insulation materials and systems. The methodology and approach lends itself to testing under representative conditions (i.e., those that reflect the actual-use or field-installed conditions) as discussed by Fesmire and Augustynowicz (2004). Boiloff calorimetry provides the sensitivity needed to test high thermal resistance materials and systems. Another advantage in using boiloff calorimetry is its ultimate simplicity: the cryogen provides a stable cold boundary temperature and serves as the power meter. The boiloff flow rate is measured by using a gas flow meter or weight scale and is directly proportional to the energy transmitted through the test specimen by the heat of vaporization (h_{fg}).



Fig. 1. 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).

Among different techniques described in ASTM C1774 is the cylindrical boiloff calorimeter for absolute heat measurement. One such apparatus, Cryostat-100, is described by Fesmire and Dokos (2014) and shown in Fig 1. A custom lift mechanism allows quick change-out of test specimens and easy access for installation. The various supporting equipment such as heater controls, flow meters, and vacuum pumping system are also depicted. The liquid within each chamber of the cold mass assembly is allowed to stabilize in its preferred stratified state which makes for a much more stable platform for heat flow measurement. Interfaces between the test chamber liquid and the guard chamber liquid are designed to preclude any appreciable heat leak between the liquid masses. The sources of error have been previously reported by Fesmire et al. (2004) and estimated to be approximately 3.2%.

The steady-state heat flow rate (Q) is the basis for calculating the thermal properties including k_e and 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. Calculations of k_e are highly sensitive to the thickness of the test specimen. The thickness, astested, must be carefully measured or calculated with explanations of any assumptions taken. The value for k_e is determined from Fourier's law for heat conduction through a cylindrical wall as given by Eqn. (1):

$$k_{\rm e} = \frac{Q \ln \left(\frac{d_o}{d_i}\right)}{2\pi L_e \Delta T}$$

(1)

For cylindrical geometries, the effective heat transfer area (A_e) will be the mean area between the two concentric cylinders. The heat flux (q) is calculated by dividing the total heat transfer rate by the effective area for heat transfer. 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.

4. Benchmark thermal performance data

Benchmark thermal performance data from Cryostat-100, for the boundary temperatures of 293 K and 77 K, are given for different multilayer insulation (MLI) systems in comparison with other cryogenic insulation systems including fiberglass, aerogel composite blanket, perlite powder, glass bubbles, aerogel particles, spray-on polyurethane foam, and "vacuum only." The residual gas is nitrogen in all cases. The data are summarized by Fig. 2 and Table 1. The data shows a wide range of thermal performance from HV to SV to NV regions of CVP. The MLI systems are clearly the best performers in the HV range but caution is given that these data are for carefully controlled installation techniques and idealized laboratory conditions. Further caution is given against over-use and application of values of k_e in cryogenic system designs when it is often the values of q that are most important. Bulk-fill powder materials are next best in the HV range followed by the groups of aerogels, foams, and fiberglass blankets. Aerogels are the top performers in the SV to NV ranges. The data for "vacuum only" is given for reference and shows the dominance of radiation heat transfer in the HV range.

These benchmark data are a selected set from an extensive database of tests performed using the Cryostat-100. Further data on MLI systems are given by Johnson and Fesmire (2012) and Fesmire and Johnson (2013). The Bulk fill materials data are given by Scholtens et al. (2008). The glass bubbles are particularly useful as a calibration reference material due to their ease of handling and consistent performance over the full vacuum pressure range. Complete data sets on a number of aerogel composite blankets are given by Coffman et al. (2010). Finally, data on spray-on foam insulation materials are summarized by Fesmire et al. (2012).



Fig. 2. The variations of effective thermal conductivity (k_e) with cold vacuum pressure are shown for different cryogenic insulation systems and materials. The boundary temperatures are approximately 78 K and 293 K and the residual gas is nitrogen. The thicknesses, number of layers, and bulk density are also given.

Table 1. Summary of cryogenic thermal performance test data from Cryostat-100 for various insulation systems and materials.

Test Specimen	CVP millitorr	WBT K	Flow	Q W	q W/m ²	k _e mW/m-K	Specifications		ications	
A 114 Ve curum Only	0.003	293.1	7447	30.8	88.4	10.44	x	п	A _e	ρ
A114 vacuum Omy	0.02	292.9	7620	31.5	90.5	10.69	mm	-	m ²	kg/m ³
Black sleeve.	1	292.8	8912	36.9	105.8	12.52	-	-	0.304	-
	10	292.4	12907	53.4	153.3	18.16				
	760000	292.3	34094	141	404.8	62.37				
A154 MLI Mylar-Net (4.9, 10, 42)	0.001	293.0	75	0.312	0.997	0.023	x	п	A _e	ρ
	0.02	290.7 293.4	83 88	0.341	1.09	0.025	mm	-	m ²	kg/m ³
Double-aluminized Mylar	0.3	293.3	126	0.519	1.66	0.028	4.9	10	0.313	42
and polyester net spacer.	1	291.5	271	1.12	3.58 8.24	0.082				
Euger by uger instantion.	10	290.0	1688	6.98	22.3	0.512				
	100	285.3	8220	34.00	109	2.55	r			
A125 MLI Mylar-Net (16, 40, -)	0.02	293.1	35	0.132	0.431	0.020	~	"	2	P
Dauble administed Males	0.1	293.1	44	0.182	0.549	0.040	mm	-	m ²	kg/m [°]
and polyester net spacer.	1 10	292.8 293.3	518	2.14	6.46	0.072	15.5	40	0.332	-
Layer by layer installation.	10	292.9	521	2.16	6.51	0.468				
	100	292.5 292.8	3604 8983	14.9 37.2	45.0	3.24 8.06				
	10000	292.4	11341	46.9	142	10.2				
	100000 760000	293.0 292.2	15058	62.3 80.2	188	13.5				
A 129 MI LEoil Banar (21 80)	0.003	292.0	42	0.176	0.516	0.051	x	п	A _e	ρ
A126 WEA FOIl-1 aper (21, 60, -)	0.3	293.6	46	0.192	0.563	0.055	mm	-	m ²	kg/m ³
Aluminum foilContinuous rolled	1.1	295.1 294.0	54 188	0.221	2.29	0.064	21.1	80	0.341	-
and microfiberglass paper spacer.	100	293.0	1214	5.03	14.7	1.44				
Continuous-rolled installation.	1000	292.7 293.4	5295 10943	45.3	64.2 133	6.30				
	100000	293.2	13013	53.9	158	15.5				
	760000	291.7	16548	68.5	201	19.8	r	n	A	0
A112 Aerogel Blanket (23, 2, 133)	1	293.2	1299	5.377	13.9	1.64			2	P
Cruogal garagal composite	10	292.7	1626	6.729	17.4	2.06	23	-	m 0.344	133 Kg/m
cryoger acroger composae.	1000	292.7	3367	13.934	36.0	4.26	25	2	0.544	155
	10000	293.0	4427	18.318	47.4	5.60				
	760000	292.6 293.4	5328 8894	36.803	95.2	6.75				
A151 Fiberglass (49, 2, 16)	0.003	293.0	802	3.32	8.59	1.90	x	п	A _e	ρ
	0.2	294.1 294.0	809 903	3.35	8.67 9.68	1.95 2.13	mm	-	m ²	kg/m^3
Micro-fiberglass batt.	10	293.0	1219	5.05	13.1	2.89	48.6	2	0.386	16
	20	294.2 293.0	1487 3099	6.16 12.8	15.9	3.59 7.26				
	400	295.8	5227	21.6	56.0	12.51				
	1000	296.6	6080 7129	25.2	65.2 76.4	14.49				
	20000	293.6	7413	30.7	79.5	17.92				
	760000	293.0	10724	44.4	115	25.99			-	
A102 Glass Bubbles (25, 1, 65)	0.003	292.0	494	2.043	5.9	0.69	A	n	2	P
Toma K1 hallow missonshare	1	292.9	506	2.096	6.0	0.71	mm	-	m ²	kg/m
Black sleeve.	10 25	293.1 293.3	585 691	2.419 2.861	6.9 8.2	0.82	25.4	1	0.349	65
	50	293.6	875	3.620	10.4	1.22				
	100	293.8 293.5	1220 2696	5.048	14.5 32.0	1.70				
	1000	293.0	5547	22.953	65.9	7.78				
	3000	292.6 293.3	9795	40.535 58.602	116.3	13.76				
	30000	293.5	16294	67.427	193.5	22.80				
	100000 760000	292.7 293.6	17861 18308	73.913	212.1 217.4	25.09 25.61				
A103 Partita Pourlan (25 1 122)	0.002	292.6	666	2.756	7.9	0.94	x	n	A _e	ρ
A105 Terme Towaer (25, 1, 152)	0.1	292.7	679	2.808	8.1	0.95	mm	-	m ²	kg/m ³
High density.	1	292.9 293.5	935	2.945	8.5 11.1	1.00	25.4	1	0.349	132
Black sleeve	25	293.0	1342	5.555	15.9	1.88				
	100	293.2 292.7	2721 9961	41.220	32.3 118.3	3.81 13.99				
	10000	292.6	19792	81.903	235.0	27.82				
	100000 760000	292.7 293.3	23978 24954	99.227 103.265	284.7 296.3	33.68 34.95				

A108 Aerogel Particles (25, 1, 80)	0.002	293.0	1204	4.981	12.6	1.69	x	п	A _e	ρ
	0.1	293.2	1232	5.100	12.9	1.73			2	3
	1	293.0	1303	5.392	13.6	1.83	mm	-	m	kg/m
Nominal 1-mm diameter beads.	10	293.0	1746	7.226	18.2	2.45	25.4	1	0.349	80
Black sleeve.	25	293.3	2176	9.004	22.7	3.05				
	100	293.6	3092	12.796	32.2	4.33				
	1000	292.7	5292	21.901	55.2	7.44				
	10000	292.9	6332	26.203	66.0	8.89				
	100000	292.9	7334	30.350	76.5	10.29				
	200000	292.4	7986	33.046	83.3	11.23				
	500000	292.8	9579	39.638	99.9	13.45				
	760000	292.7	10207	42.240	106.5	14.34				
A 104 Sumary On Falam BY 265 (25, 1, 42)	0.1	293.0	5527	22.872	65.6	7.75	x	п	A_{e}	ρ
A104 Spray-On Foam BA-205 (25, 1, 42)	1	293.1	5924	24.513	70.3	8.68			,	3
	10	293.0	9861	40.805	117.1	14.46	mm	-	m	kg/m [°]
Machined; no rind.	100	293.0	12560	51.974	149.1	18.41	25.4	1	0.349	42
	1000	293.2	13443	55.628	159.6	19.69				
	10000	293.0	13534	56.004	160.7	19.85				
	100000	293.0	13621	56.364	161.7	19.97				
	200000	293.2	13792	57.074	163.8	20.20				
	500000	293.2	13957	57.755	165.7	20.44				
	760000	292.8	14424	59.690	171.3	21.17				

5. Conclusion

Thermal insulation plays a significant and sometimes enabling role in cryogenic systems. Energy efficiency, system control, and operational safety are inter-related aspects of deciding the best thermal insulation system for a particular application. The emerging cryofuels enterprises including LNG and LH₂ are a particular challenge due to the transient type operational processes to be addresses and the competitive economic targets to be met. New technical consensus standards on testing and MLI systems are but one step toward providing standard thermal data to support the design practices for cryogenic systems. The Cryostat-100 insulation test apparatus has provided thermal performance for a wide range of different materials under relevant conditions. Benchmark thermal performance data, including k_e and q, can ultimately be used to calibrate comparative type instruments and support detailed studies of insulation system designs for specific applications. Future standards are envisioned for both test methods and materials practices to support the cryogenic industry and further the proliferation of opportunities in the areas of transportation and energy.

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