Cylindrical boiloff calorimeters for testing of thermal insulation systems

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Abstract. Cryostats have been developed and standardized for laboratory testing of thermal insulation systems in a cylindrical configuration. Boiloff calorimetry is the measurement principle for determining the effective thermal conductivity (k_e) and heat flux (q) of a test specimen at a fixed environmental condition (boundary temperatures, cold vacuum pressure, and residual gas composition). Through its heat of vaporization, liquid nitrogen serves as the energy meter, but the design is adaptable for various cryogens. The main instrument, Cryostat-100, is thermally guarded and directly measures absolute thermal performance. A cold mass assembly and all fluid and instrumentation feedthroughs are suspended from a lid of the vacuum canister; and a custom lifting mechanism allows the assembly and specimen to be manipulated easily. Each of three chambers is filled and vented through a single feedthrough for minimum overall heat leakage. The cold mass design precludes direct, solid-conduction heat transfer (other than through the vessel's outer wall itself) from one liquid volume to another, which is critical for achieving very low heat measurements. The cryostat system design details and test methods are discussed, as well as results for select thermal insulation materials. Additional cylindrical boiloff calorimeters and progress toward a liquid hydrogen apparatus are also discussed.

1. Introduction

Evaporation or "boiloff" [1–2] continues to be a valuable technique for testing the thermal insulating performance of materials. In the general sense, boiling is associated with higher heat transfer rates and evaporation is associated with lower heat transfer rates. For the subject of cryogenic thermal insulation testing, the properties are well within the evaporation-only regime, with peak heat flux levels, at the most, only several hundred watts per square meter. The heat of vaporization (h_{fg}), also known as the enthalpy of vaporization or the heat of evaporation, is the energy required to transform a given quantity of a liquid into a gas at a given pressure.

Cryogenic insulation systems encompass a wide range of material combinations. An insulation test specimen is a system, composed of one or more materials (homogeneous or nonhomogeneous, with or without inclusion of a gas) whose thermal transmission properties are measured through its thickness under subambient temperatures.

Several cryostat instruments have been developed and standardized for laboratory testing of thermal insulation systems in a cylindrical configuration (table 1). Boiloff calorimetry is the measurement principle for determining the effective thermal conductivity (k_e) and heat flux (q) of a test specimen at a fixed environmental condition (boundary temperatures, cold vacuum pressure, and residual gas composition).

Through its heat of vaporization, liquid nitrogen (LN_2) or a different cryogen, serves as the energy meter to provide a direct measure of the heat flow rate. That is, the heat flow rate is directly proportional to the boiloff flow rate. Cryostat-100, a fully thermally guarded design, is an absolute (primary) type instrument of thermal transmission measurement. Absolute instruments produce the data by which other instruments, such as the comparative (secondary) Cryostat-200, can be calibrated.

Table 1. Insulation test cryostat instruments: cylindrical configurations.

Instrument	Type	Test Specimen Size	ASTM Test Standard	Environment	Heat Flux (W/m^2)
Cryostat-100 (1 unit)	Absolute	1 m long, 167 mm diameter, up to 50 mm thick	C1774 Annex A1	Full range vacuum 77 K–353 K	0.2–200
Cryostat-200 (2 units)	Comparative	0.5 m long, 132 mm diameter, up to 50 mm thick	C1774 Annex A2	Full range vacuum 77 K–353 K	1–200
Cold Pipeline Test Apparatus (1 unit)	Absolute	12 m long, 25 mm to 88 mm diameter	C335	No vacuum or vacuum-jacket 77 K–353 K	4–400

Although both cylindrical and flat-plate cryostats have been standardized for laboratory operation [3], cylindrical configurations are better at minimizing (or even eliminating) the unwanted lateral heat transfer or "end effects." Cylinders also align better with most applications, including tanks and piping. The vertically oriented cold mass assemblies of the cryostats can cause some convection problems when tests are conducted at ambient pressure, but otherwise, these assemblies provide a stable platform for testing over a wide range of heat flows.

The cold pipeline test apparatus also provides an absolute thermal measurement (both ends are guarded with cryogen reservoirs) for below-ambient temperature testing of insulated piping systems. This horizontal apparatus is sloped uniformly upward to provide a high point for the boiloff flow rate at the downstream end [4]. Two insulated pipelines, each 12.2 m in length, can be tested in parallel.

2. Test apparatus design and setup

The primary instrument for testing of cylindrical specimens is the Cryostat-100. The technology is built on the prior technology of cylindrical calorimeters developed by the Cryogenics Test Laboratory (CTL) [5–7]. This apparatus is guarded on top and bottom for absolute thermal performance measurement. The basic schematic and a photograph of the overall arrangement, including the mechanical lift mechanism, are shown in figure 1. A cold mass assembly, including the top and bottom guard chambers and a middle test chamber, is suspended from a domed lid atop the vacuum canister, as shown in figure 2.

Each of the three chambers is filled and vented through a single feedthrough (also connected from the lid) for easy operation and minimum overall heat leakage. A novel thermal break design between the liquid chambers [8] preclude direct, solid-conduction heat transfer from one liquid volume to another. Such isolation is critical for achieving very low heat measurements because even small temperatures variations between the liquids in the chambers can produce dramatic errors in results. The liquid within each individual chamber is allowed to stabilize in its natural stratified state, which makes the platform for heat flow measurement much more stable compared to destratified methods. All fluid and instrumentation feedthroughs are mounted and suspended from a top-domed lid for easy removal of the cold mass.

Cryostat-100 includes an external heating system for bakeout and high heat load tests, as well as an internal heater system for fine control of the warm boundary temperature (WBT). Three custom-designed funnel filling tubes (7.93-mm outside diameter) interface with the three LN_2 feedthroughs (12.7-mm outside diameter) and provide the means for cooldown, filling, and replenishment by pouring from a small nonpressurized dewar. The filling tubes are removed when not being used. Connected to the top ports of the LN_2 feedthroughs are the plastic tubing assemblies that route the boiloff flow from all three liquid chambers to their respective mass flow meters. Vacuum instrumentation typically includes two capacitance

manometers, an ion gage, and a full-range transducer for backup. The vacuum pumping system includes a directly connected turbopump and a separately plumbed mechanical pump. In addition, a gaseous nitrogen (GN$_2$) supply system provides purging and residual gas pressure control to vacuum levels as low at 5×10^{-5} torr. All instruments are connected to a customized LabVIEW data acquisition system for data recording and monitoring.

A custom lift mechanism, shown in figure 1, allows the cold mass assembly and insulation test specimen to be manipulated easily. The location of temperature feedthroughs on the lid allows the sensors to move with the cold mass assembly when insulation specimens are installed.
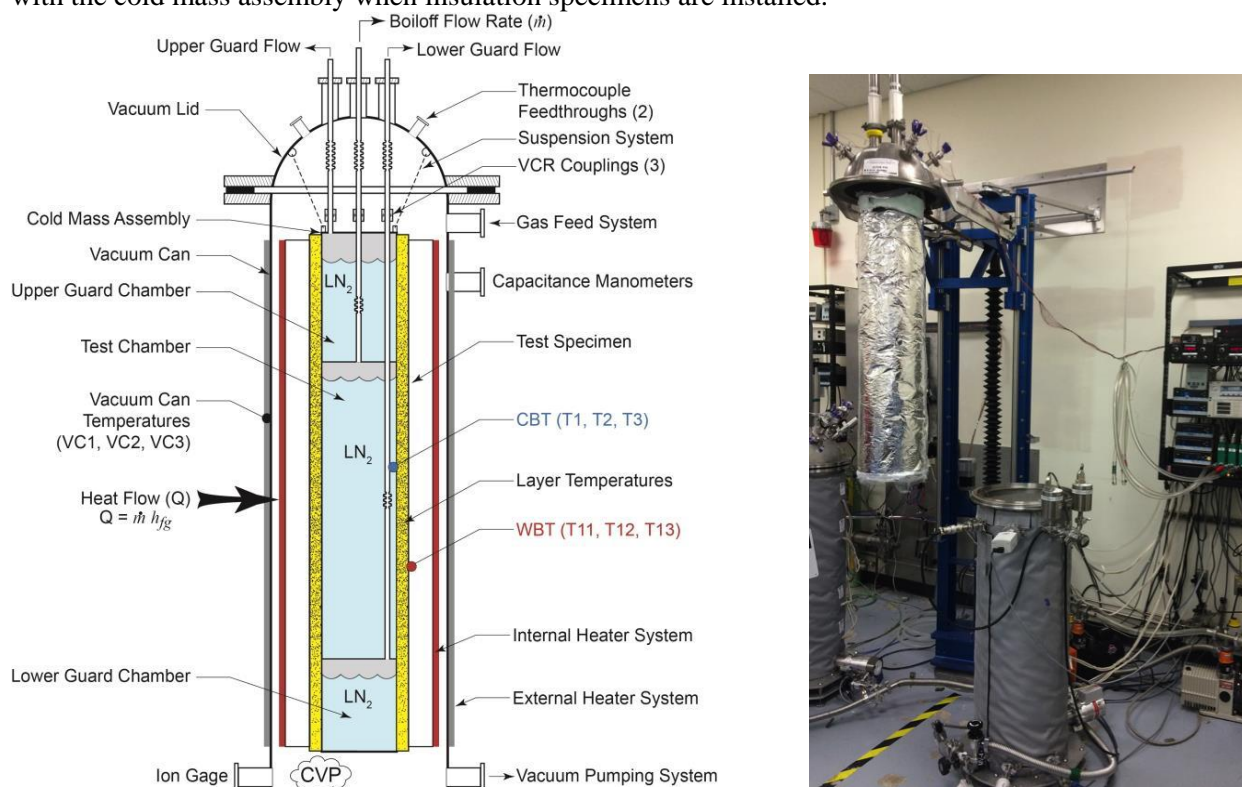

Figure 1. Cryostat-100: basic schematic (left) and overall arrangement with lift mechanism (right).

Figure 2. Overall system (left) and cold mass assembly (right).

3. Testing methodology

The principle of heat rate measurement for Cryostat-100 is based on LN_2 boiloff calorimetry, following the guidelines of ASTM C1774, Annex A1 [9]. 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). Any thermal performance test result is considered along with other results and other complementary methods [10]. The parameters of the test, the manner in which the test is set up and performed, and the key terminology for the reporting of data are properly documented to obtain an accurate technical evaluation.

Calculations of k_e are highly sensitive to the thickness of the test specimen. The thickness, as tested, is carefully measured or calculated, and any assumptions are explained. Thicknesses from 0 mm (bare cold mass) to approximately 50 mm can be tested on Cryostat-100.

Materials can be blanket, clamshell, molded, or bulk-fill. Blankets can be applied in individual layers or in various layering combinations as desired, and temperature sensors are placed between the blanket layers. Multilayer insulation (MLI) specimens can be installed in blanket, layer-by-layer, or continuously rolled fashion. The temperature sensors are typically Type E thermocouples, 30-gage size, with vacuum sides at least 2 m long. A sleeve assembly is installed for bulk-fill materials, providing a 25 mm-thick test specimen, with temperature sensors at discrete points within.

Test specimens are evacuated and heated according to approved standard laboratory procedure, which for MLI specimens includes five GN_2 purge cycles between 1 torr and 100 torr at a temperature up to 330 K. The WBT, defined by the heater shroud assembly inside the vacuum can, is typically set to 293 K for a test. The cold vacuum pressure (CVP) within the vacuum chamber is maintained in the range of 10^{-6} torr (high vacuum) by active vacuum pumping. Various vacuum pressures are produced and precisely maintained by active vacuum pumping in combination with a GN_2 supply system.

The test specimens are cooled down, stabilized, and tested according to approved standard laboratory procedure. For all tests, the cold boundary temperature (CBT) is approximately 78 K. 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. A stable state of the system is indicated by slight oscillation of the temperature sensors with no overall trend in their average value [9]. The total test duration may be hours to days, depending on the level of heat flow involved. All test data are ordered into standardized files, by test series, for processing and archival purposes.

The variation in boiloff flow rate is primarily determined by the states of the liquid masses in each of the cold mass chambers. The liquid can be stratified, mixed, or in transition. For steady-state measurement to be achieved, all liquid masses must be either stratified or mixed, and this condition is reached only by the inherent design of the cold mass assembly. Other important factors in boiloff flow rate stability are the regional variations and twice-daily fluctuations in atmospheric pressure that correlate to the atmospheric tides. Without systematic controls to counteract this effect, at very low heat flux rates, these fluctuations can influence the results by up to 20%. These fluctuations are eliminated by feeding the boiloff flow tubes into a custom plenum system set approximately 3 torr above the prevailing mean at atmospheric pressure and by controlling the back pressures of all three chambers within ±0.1 torr.

4. Uncertainty analysis

The rate of heat transfer through the insulation test specimen and into the side wall of the test chamber of the cold mass assembly (Q) is directly proportional to the LN_2 boiloff flow rate (V), as given by (1),

$$Q = V_{STP}\, \rho_{STP}\, h_{fg} \left(\frac{\rho_f}{\rho_{fg}} \right) \tag{1}$$

where STP = Standard Temperature and Pressure (0 °C and 760 torr) and the right hand term is the density correction, if any, between the liquid and the saturated liquid conditions. The value of k_e is determined from Fourier's law of heat conduction through a cylindrical wall, as given by (2).

$$k_e = \frac{Qx}{A_e \Delta T} = \frac{Q \ln\left(\dfrac{d_o}{d_i}\right)}{2\pi L_e \Delta T} \tag{2}$$

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 heat transfer area, as given by (3).

$$q = \frac{Q}{A_e} \tag{3}$$

The symbols and sources of error used for calculating thermal properties from boiloff testing with the cylindrical instrument, Cryostat-100, are given in table 2.

Table 2. Symbols and sources of error for the cylindrical calorimeter, Cryostat-100.

Symbol	Description	Unit	% Error
V	Volumetric flow rate (boiloff) at STP	m^3/s	0.500
ρ	Density of GN$_2$ (boiloff) [0.0012502 g/cm^3]	kg/m^3	n/a
h_{fg}	Heat of vaporization	J/g	2.37
d_o & d_i	Outer and inner diameters of insulation specimen	m	1.53 & 1.23
x	Thickness of insulation specimen	m	n/a
L_e	Length, effective heat transfer	m	0.730
A_e	Area, effective heat transfer area	m^2	n/a
ΔT	Temperature difference *(WBT – CBT)*	K	0.894

The error introduced by each parameter is taken into account for the calculation of the total error, in accordance with the "Error Analysis of Experiments" equation listed in *Perry's Chemical Engineers' Handbook* [11]. The total uncertainty in k_e is calculated to be 3.3% for the Cryostat-100. The uncertainty in heat flux q is 3.2% (the difference being that temperatures are not part of the heat flux calculation) [12]. Measurement of the boiloff flow rate is made using a mass flow meter that automatically compensates for gas densities in the range of 273 K to 323 K and renders the density error not applicable. The mass flow meter output is in terms of a volumetric flow rate at STP.

The overall error of k_e is estimated for the worst-case situation. The heat of vaporization of the cryogen is the largest source of uncertainty, typically 2% for LN$_2$ [13]. The repeatability should, of course, be high, and other factors, such as surface finish of the interior cold mass and overall cleanliness of the cryogen, are considered dominant. Physical measurement of the test specimen is robust because diameters and not thickness are part of the calculation. Thermal shrinkage effect is included in the analysis. In most testing situations, for a given series of tests, the overall repeatability is demonstrated to be within 2%.

The analysis assumes that all heat flow to the calorimeter goes to vaporizing the liquid and that none of it sensibly heats the vapor or the liquid. The nominal value for the heat of vaporization is 199.1 J/g based on a saturation pressure of approximately 765 torr (0.1 psig). The vapor heating effect can be neglected for LN$_2$ calorimeters with small ullage spaces (less than 20% of the total volume). The error attributable to vapor heating in nitrogen is estimated to be <0.1% when the results of the study by Jacobs [14] are applied.

5. Example test results and discussion
Testing technologies, methods, and experimental approaches go hand in hand with the research, development, and implementation of new thermal insulation systems and their high-performance material elements. The results of tests performed by the CTL with the cylindrical boiloff calorimeters are summarized in table 3. For cylindrical calorimeters, 174 material specimens have undergone more than 1,500 individual tests, representing roughly 5 years of continuous boiloff run time. Many of these results

have provided the baseline data for ASTM C740 and ASTM C1774 and continue to establish the benchmark of comparison for both new and old thermal insulation materials.

Table 3. Summary of cylindrical cryostat testing by number of material specimens and tests.

Apparatus	Number of specimens	Number of tests	Hours of run time[a]
Cryostat-100	132	~1,188	~35,640
Cryostat-200	42	~378	~7,560
Total	174	~1,566	~43,200

[a]Time does not include that required for evacuation and heating, purging, cooldown, or warmup.

The results from several select tests with Cryostat-100 are presented in figures 3 through 5. Figure 3 presents an example test result of a 60-layer insulation system at high vacuum. In this plot, the boiloff flow rates from all three chambers are shown over the 10-day duration of the test. The periodic oscillation of the test chamber flow rate, induced by atmospheric tides, is indicated by the regular 12-hour peaks. (Note: This test series did not include the atmospheric back-pressure control plenum.) Details of these and many other materials are given in the literature [9–10, 15].

Figure 4 presents the layer temperature profile for a 10-layer insulation system for various CVPs. The profile, with all 10 layers instrumented, shows the insulating effect of high vacuum as indicated by the sharp temperature rise in the first millimeter of thickness (about one layer). The sensitivity to CVP is also shown in this plot by the flattening of the temperature plots as the vacuum level is degraded.

Finally, figure 5 summarizes the Cryostat-100 test results for various thermal insulation systems and materials in terms of the variation of heat flux with CVP. Further details of these and many other materials are given in the literature [9–10, 15].

Future multilayer insulation systems are envisioned that will challenge the theoretical limit in thermal insulation performance (k_e <0.01 mW/m-K and/or q <0.1 W/m^2 for typical boundary conditions of 300 K / 77 K in vacuum). Boiloff technology to measure ultralow heat flow is at the heart of efforts to develop and prove such advancements. Ultralow heat flow systems are needed for superconducting power devices, long-duration storage of cryofuels, science instruments, space exploration craft, medical imaging equipment, and other performance-driven applications.
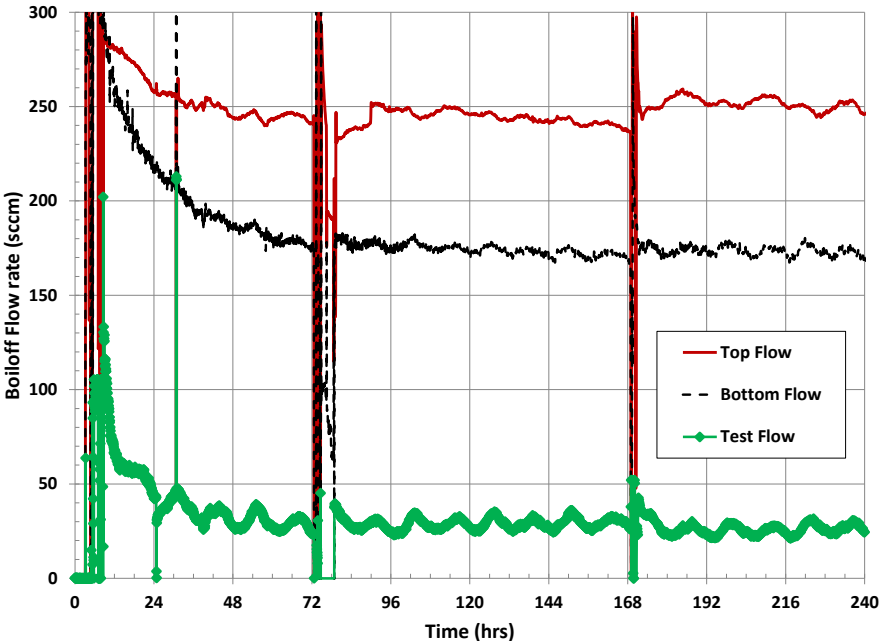

FIGURE 3. Example Cryostat-100 test result of A138 MLI (60 layers: Mylar/polyester net) at high vacuum: boiloff flow rates from all three chambers over 10 days. Boundary temperatures: 293 K/78 K. The periodic oscillation of the test chamber flow rate, induced by atmospheric tides, is indicated by the regular 12-hour peaks.
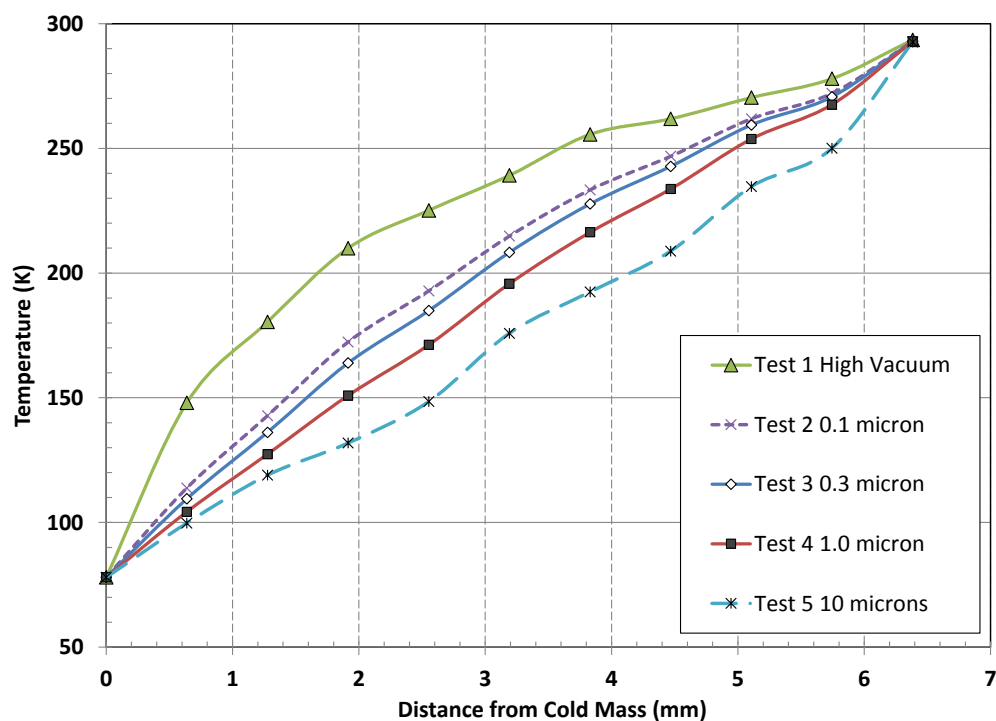
Figure 4. Temperature profile for A145 MLI (10 layers: Mylar and polyester fabric) for various CVPs. Boundary temperatures: 293 K/78 K; residual gas: nitrogen.
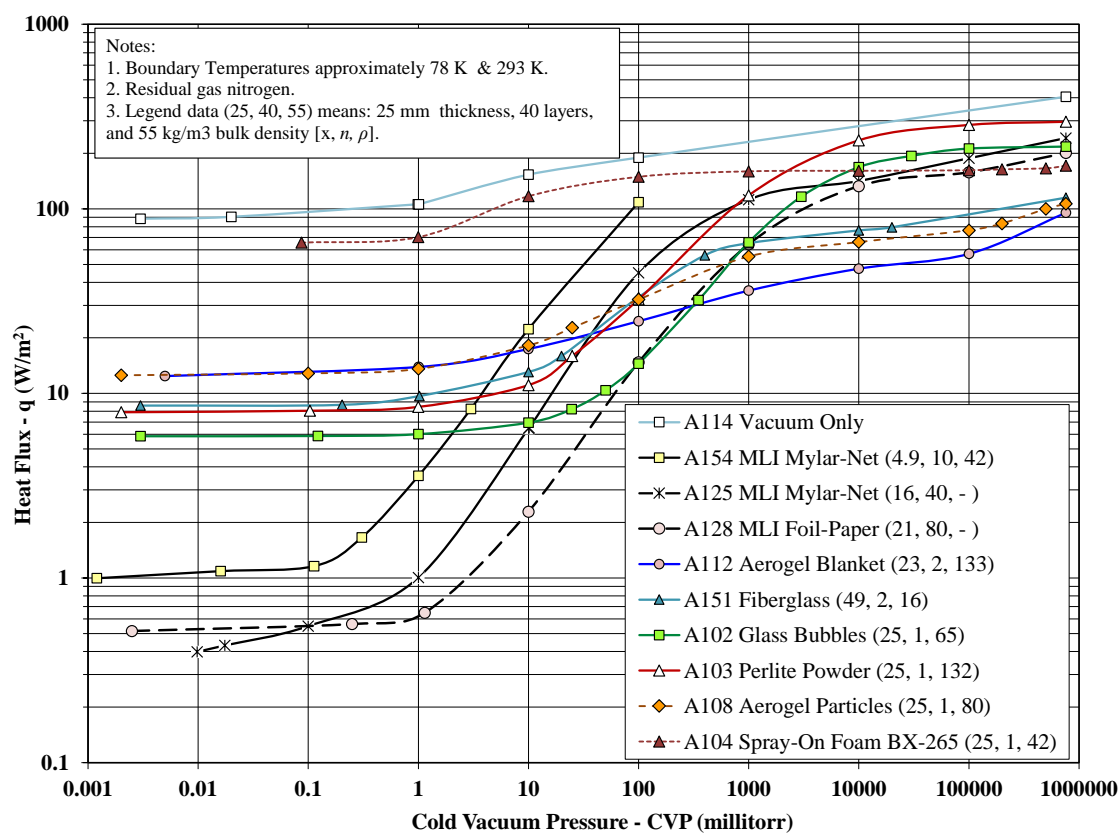

Figure 5. Summary of Cryostat-100 test results for various thermal insulation systems and materials: variation of heat flux with CVP. Boundary temperatures: 293 K/78 K; residual gas: nitrogen.

6. Conclusion

Based on boiloff calorimetry, new cylindrical cryostats and methods for testing thermal insulation systems have been successfully developed by the CTL at NASA Kennedy Space Center over the last 20 years. These boiloff instruments (or cryostats) are applicable to a wide range of materials and test conditions. Test measurements are generally made at large temperature differences (boundary temperatures of 293 K and 78 K are typical) and include the full vacuum pressure range. Results are generally reported in effective thermal conductivity (k_e) and mean heat flux (q) through the insulation system. The new cylindrical cryostat instruments provide an effective and reliable way to characterize the thermal performance of materials and systems under subambient conditions. Cryostat-100, with its thermal break cold mass design, stratified liquid approach, and single port filling/venting method, is an absolute calorimeter that has provided baseline data for dozens of materials and a foundation for future international standards for thermal insulation materials in cryogenic service. Proven through many hundreds of tests of different material systems, these insulation test cryostats have supported a wide range of aerospace, industry, and research projects.

7. Acknowledgements

8. References

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