Experimental Thermal Performance Testing of Cryogenic Tank Systems and Materials

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A comparative study was conducted to collect and analyze thermal conductivity data on a wide variety of low density materials, as well as thermal performance data on a number of vacuum-jacketed cryogenic tank systems. Although a vast number of these types of materials and cryogenic tank systems exist, the thermal conductivity of insulation materials and the thermal performance of cryogenic tank systems is often difficult to compare because many industrial methods and experimental conditions are available and utilized. The availability of a new thermal conductivity measurement device, the Macroflash Cup Cryostat, which is applicable for assessing a variety of materials, is accessible at NASA's Cryogenic Test Laboratory (CTL) at the Kennedy Space Center (KSC). The convenience of this device has resulted in the ability to rapidly measure the thermal conductivity properties of these materials by using a flat-plate liquid nitrogen (LN_2) boiloff technique that employs a guarded heat flow test methodology in order to determine the effective thermal conductivity (k_e) of a test specimen. As the thermal conductivities are measured at cryogenic temperatures, materials suitable for both future space missions and cryogenic tank systems can be identified and experimentally analyzed. Also recognizable are materials which may help increase energy efficiency by limiting the thermal losses encountered under various environmental conditions. The overall focus of this work consisted of two parts. One part, was to produce and analyze thermal conductivity data on a wide variety of materials with suitable properties conducive to those needed to aid in the production of a calibration curve for the "low end" of the Macroflash instrument. (Low end meaning materials with a thermal conductivity rating below 100 mW/m-K). The second part was to collect and analyze heat transfer data for a variety of small vacuum-jacketed vessels (cryogenic tank systems) in order to compare the thermal performance between them.

Nomenclature

Q	=	Heat flow rate (W)
$\dot{m}$	=	Mass flow rate (g/s)
h_{fg}	=	Heat of vaporization (J/g)
q	=	Heat flux (W/m^2)
A_e	=	Effective heat transfer area (m^2)
k_e	=	Effective thermal conductivity (mW/m-K)
x	=	Thickness of specimen (m)
ΔT	=	Temperature difference (K)
Z	=	Volumetric boiloff rate (W/L)
ρ	=	Density (g/cm^3)
σ_c	=	Compressive strength

I. Introduction

The Cryogenics Test Laboratory at Kennedy Space Center was established to develop thermally efficient technologies for a wide range of below-ambient temperature applications. Its overall discipline areas include the study of heat management, energy efficiency, thermal insulation systems, and novel materials. Both novel and conventional means of refrigeration and heat control are being investigated to develop the most suitable thermal management and control systems to meet the needs for propellant process systems, superconducting power device refrigeration, rocket vehicle protection systems, human exploration habitats, cold chain shipping, and biomedical research platforms, to name a few.

In general, one cannot measure how much heat is present in an object, but rather only how much energy is transferred between objects at different temperatures, hot and cold. Early attempts at temperature measurement include the experiments of Greek physician Galen in Ad 170, followed many centuries later by Fahrenheit's description of the first modern temperature scale in 1724. A few decades later, J. Black devised an ice calorimeter

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based on his discovery of hidden heat. And by 1822, J. Fourier had published The Analytical Theory of Heat, a work that remains the basis of our notions of heat, thermal energy and temperature. The technological development of large-scale liquid hydrogen in the US in the 1950's gave rise to the demand for high performance thermal insulation systems. To meet this demand and enable the development of multilayer insulation (MLI) and evacuated perlite powder systems, engineers devised different apparatuses to directly measure heat flows from a few milliwatts and up using evaporation— or "boiloff" calorimetry. The use of boiloff calorimetry to measure the effects of thermal energy (or heat) dates back to the early 1900's [1, 2]. It has become a practical and useful tool to measure, in a direct way, the thermal insulating performances of materials and systems of materials. Gas flow rates measured using boiloff calorimetry enable direct calculation of quantities such as heat flux and thermal conductivity. A particularly useful approach is to use nitrogen for the heat measurement fluid as it is readily available, inert and generally safe to use. 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 also provides a convenient way to establish the sub-ambient test conditions represented in the wide range of end-use applications. The temperature range from normal boiling point (77.4 K) to ambient (approximately 300 K) represents a wide range of particular needs in construction, transportation, food and beverage, pharmaceuticals, electrical power, electronics, medical imaging, aerospace, industrial processes and so forth, touching on virtually all aspects of modern life.[3]

One of the greatest advantages of using liquid nitrogen boiloff calorimetry is its ultimate simplicity and provision of a direct energy measurement. The liquid provides a stable cold boundary temperature and serves as a sort of power meter. The approach also lends itself to testing under representative conditions (i.e., those that reflect the actual-use or field-installed conditions) afforded by the very large temperature difference established by the liquid nitrogen. Cryogenic boiloff methods provide the means to reliably test the thermal conductivity of materials and the thermal performance of cryogenic tank systems. This method is a direct measurement of the flow of heat and enables the testing of complex materials and systems over a very wide range of conditions. This type of testing will be used for experimental laboratory investigations of both materials and cryogenic tanks. The thermal conductivities of materials will be tested using a Macroflash (Cup Cryostat). The total system thermal performance of small tanks will be tested using a custom developed laboratory methodology.

II. Macroflash Testing of Materials

A. Test Apparatus

The Macroflash Cup Cryostat instrument (shown in Figure 1) is a cryogenic boiloff calorimeter whose data is recorded by a National Instruments LabVIEW Data Acquisition Program. The Macroflash houses a cold mass test chamber centered directly over the test specimen, and wrapped in multiple layers of aerogel blankets to ensure the thermal isolation and stability necessary for accurate steady-state boiloff measurements. The Macroflash is a comparative, flat-plate apparatus that tests at a large ΔT (such as~187K) and determines effective thermal conductivity (k_e) in accordance with ASTM 1774-13 [4]. Boiloff calorimetry provides a way to directly measure the heat flow rate through the test specimen. In Figure 1, the Macroflash system can be seen positioned on a high-sensitivity scale used for measuring the mass change of cryogenic fluid as fluid boil off occurs. The Macroflash system is connected to a heat controller for the hot plate and nitrogen gas for purging of the specimen throughout testing. The nitrogen gas is used to maintain an inert and dry environment, which prevents any moisture condensation on the test specimen. An adjustable compression loading system provides 0.5, 1.5, or 4.5 psi of applied force, ensuring full consolidation and contact between the test specimen and cold/hot plate boundaries, preventing any void spots that would otherwise alter thermal conductivity results.

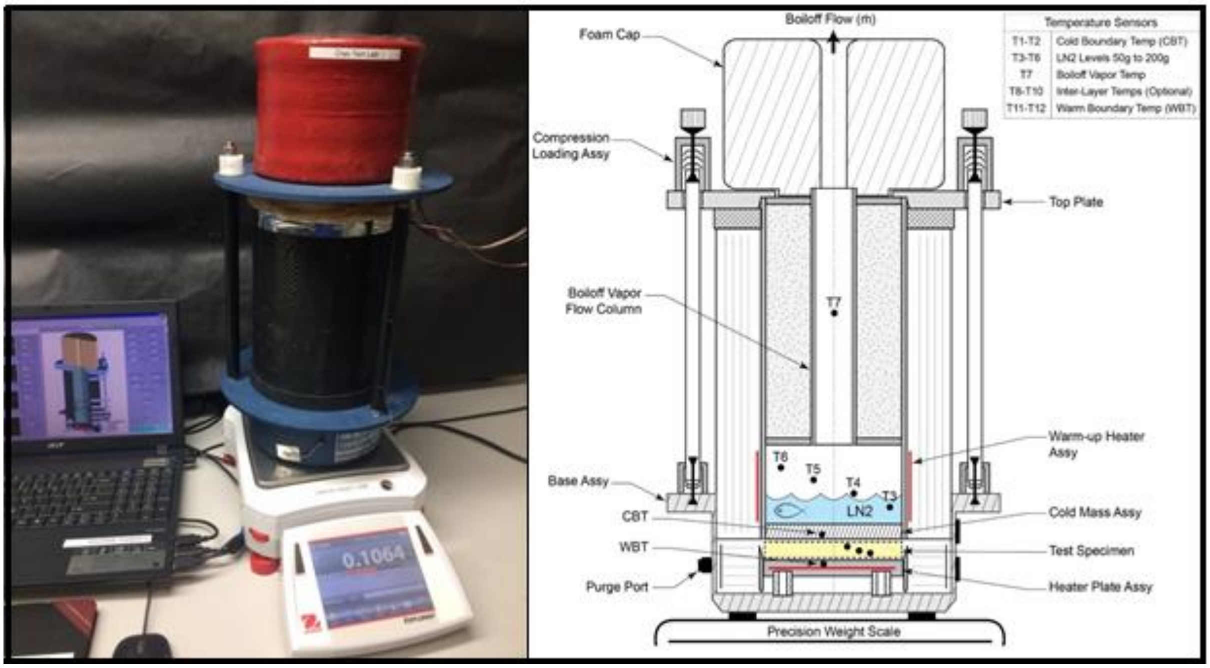


Figure 1. (Left) Assembled Macroflash setup. (Right) Cross sectional schematic for the Macroflash thermal test instrument.

B. Test Method

The test method is comparative and therefore requires calibration with materials of known thermal conductivity data. However, finding these materials with the necessary standard reference data is a difficult challenge. This project builds on the previous five years of work by different colleagues of the NASA Cryogenics Test Laboratory including scientists, engineers, and university researchers and brings in additional testing for a specific range of the spectrum of materials [5, 6].

The test conditions are representative of actual-use cryogenic applications with a warm boundary temperature (WBT) of approximately 293 K and a cold boundary temperature (CBT) of approximately 78 K. The test measurement principle is liquid nitrogen boil-off calorimetry where the mass flow rate of nitrogen gas is directly related to the rate of heat energy transmitted through the material. All tests are performed at an ambient pressure gaseous nitrogen (GN2) condition for consistency. A boiloff calorimetry test is conducted by filling a test chamber with a liquid cryogen which then boils/evaporates at room temperature. A test specimen of pre-determined geometry is affixed to the bottom of the test chamber and put in an environmental apparatus that provides the desired test conditions. The flow of heat through the test sample is directly proportional to the cryogenic fluid boiloff flow rate, measured by a weight scale or mass flow meter, with energy transfer measured as the heat flow rate. This cryogen boiloff rate, or gas flow rate (which can also be measured as mass loss) is directly used for the calculation of heat flux and effective thermal conductivity. Under steady-state flow conditions, the rate of heat flow through the test specimen is constant at all points through the thickness of the specimen. The thermal conductivity can then be easily calculated using a series of equations:

$$Q = \dot{m} h_{fg} \tag{1}$$

$$q = \frac{Q}{A_e} \tag{2}$$

$$k_e = \frac{Qx}{(A_e \Delta T)} \tag{3}$$

The heat flow rate (Q) is the product of boiloff mass flow rate ($\dot{m}$) and enthalpy of vaporization of liquid nitrogen (h_{fg}) as shown in the equation above. Knowing the boundary temperatures on either side of the specimen in a set test environment, the heat flux (q) can be calculated. Since the specimen area is also known (A_e), as well as the thickness of the specimen (x) and the temperature differential between cold and hot boundaries (ΔT), the effective thermal conductivity (k_e) can then be calculated.

For this testing, liquid nitrogen (LN_2) was used as the cryogen, allowing for a temperature differential from 77 K (nominal boiling point of liquid nitrogen) to 293 K (room temperature) to be created across the thickness of the test specimen (Figure 2). The LN_2 provides the cooling (refrigeration) required, acts as an "energy meter", and produces the temperature differential (i.e., the change in temperature from one side of the specimen, chilled by LN_2, to the other side, maintained at room temperature by a heat controller. Boiloff calorimetry provides the ability to test both simple uniform materials and complicated non-homogeneous, anisotropic, composite materials with equal ease [7]. This method also inherently presents a temperature differential where thermal conductivity is calculated by the mass flow rate of cryogenic fluid boiloff, which is measured through either the system mass loss or boiloff flow rate. The extent of the temperature differential is therefore dependent on the cryogenic fluid and the h eat source used, providing high sensitivity for the accurate measurement of highly thermally insulating materials or structural materials alike. Intermediate temperature sensors can also be employed for obtaining thermal conductivity data at different mean temperatures up to the ambient.

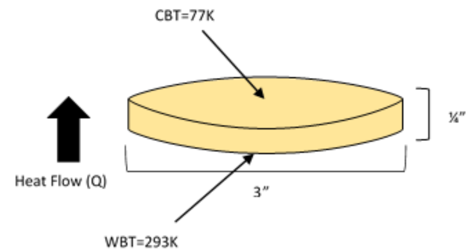


Figure 2. Boundary temperatures on the faces of a test specimen.

C. Materials (Test Specimens)

The test specimens (examples shown in Figure 3) are typically 3" diameter by ¼" thickness and should be flat and smooth-faced or easily compressible to insure good thermal contact between the heater assembly and the cold mass. The specimen is placed in the test section between two plates that are maintained at different temperatures during the test. For commercial samples, the compressive properties were obtained from the supplier technical data. For in-house research samples, compression properties were evaluated according to ASTM D695. A complete list of specimens tested and their properties is given in Table 1.

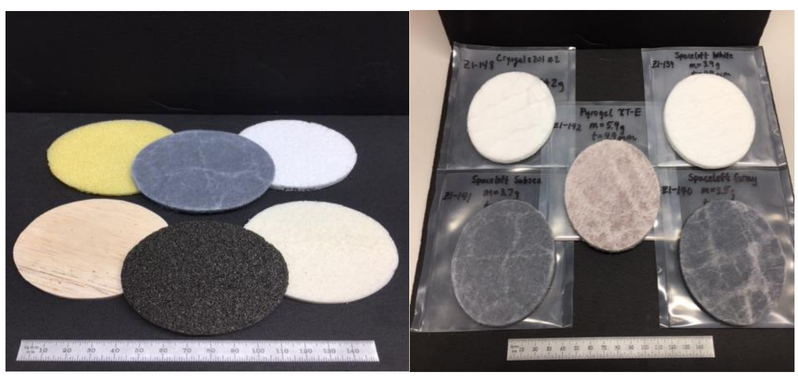


Figure 3. Example test specimens prepared for Macroflash testing: a variety of different thermal insulation materials (left) and five aerogel blanket materials (right).

Table 1. Summary and Properties of Materials Tested

Test Series	Matl Code	Material	Thick mm	Dia. mm	Mass g	Density g/cm^3	Load psi	Grease Y or N	Comp. Strength MPa
Z1-138	A	Cryogel x201	4.80	76.2	3.6	0.175	0	N	0
Z1-139	A	Spaceloft White	4.90	76.2	3.9	0.175	0	N	0
Z1-140a	A	Spaceloft Grey	4.90	76.2	3.5	0.157	0	N	0
Z1-141	A	Spaceloft Subsea	5.00	76.2	3.7	0.162	0	N	0
Z1-142	A	Pyrogel XT-E	4.90	76.2	5.4	0.242	0	N	0
Z1-143	L	Polyglas SBS Asphalt Aerogel Blanket	6.00	76.2	22.4	0.819	0	N	0
Z1-144	A	Cryogel x201 2-layers	8.40	76.2	6.5	0.175	0	N	0
Z1-145	P	Aeroplastic Versify Sample-A 22-stack	6.10	76.2	23.8	0.896	5	N	0
Z1-146	P	Aeroplastic Versify Sample-B 13-stack	6.50	76.2	26.2	0.884	5	N	0
Z1-147	P	Aeroplastic Versify Sample-C 14-stack	6.70	76.2	27.4	0.857	5	N	0
Z1-148	A	Cryogel x201 #2 (1 Layer)	4.90	76.2	3.6	0.161	0	N	0
Z1-149	A	Cryogel x201 #3 (1 Layer)	4.90	76.2	3.7	0.166	0	N	0
Z1-138a	A	Cryogel x201 (1 layer)	4.80	76.2	3.6	0.164	0	N	0
Z1-150	F	SOFI #2	6.60	76.2	1.0	0.036	2	N	0.4
Z1-151	W	Balsa (in-plane) 7.3mm	7.31	76.6	5.6	0.166	5	N	7
Z1-152	W	Balsa (in-plane) 6.4mm	6.40	76.6	4.9	0.166	5	N	7
Z1-153	F	Polyimide Foam Solimide	6.30	76.2	0.2	0.080	0	N	0
Z1-154	F	FoamGlass #1 broken	6.18	76.2	3.3	0.116	5	N	0.8
Z1-155	A	ULD Aerogel	7.48	76.2	2.1	0.118	0	N	0.8
Z1-156	F	FoamGlass #2	6.29	76.2	3.4	0.119	5	N	0.8
Z1-157a	F	FoamGlass #3	6.34	76.2	3.4	0.118	5	N	0.8
Z1-158	B	Glass Bubbles, low density 0.038	6.35	76.2	1.1	0.038	0	N	1.72
Z1-158a	B	Glass Bubbles, nominal tap density	6.35	76.2	2.1	0.073	0	N	1.72
Z1-158b	B	Glass Bubbles, nominal tap density	6.35	76.2	2.1	0.073	0	N	1.72
Z1-158c	B	Glass Bubbles, nominal tap density	6.35	76.2	2.1	0.073	0	N	1.72

D. Test Results

The thermal conductivities of a large number of test specimens have been evaluated using the Macroflash, as described above. The specimens tested covered a wide variety of materials that met the thermal conductivity specification for the "low end calibration", and thus have a similar range in bulk density, but a varying range in material composition. A total of 25 test specimens representing approximately 130 boiloff test runs were performed as part of the current project. The test results are listed in Table 2.

Table 2. Macroflash thermal conductivity test data summary

Test Series	Matl Code	Material	Test Date	Test Runs	Mass Flow g/s Boiloff	Q W Heat	*q* W/m^2 Heat Flux	K_e - comp mW/m-K Measured	Std Dev %	k_e mW/m-K Calibrated	*cal* L or H	Comp. Strength MPa	Figure Of Merit K-m-s/g
Z1-138	**A**	Cryogel x201	12/11/2017	5	0.02981	5.936	1302	27.3	0.2	9.7	L	0	0
Z1-139	**A**	Spaceloft White	12/12/2017	5	0.02651	5.279	1158	26.4	1.1	8.8	L	0	0
Z1-140a	**A**	Spaceloft Grey	1/17/2018	5	0.02707	5.390	1182	27.0	0.7	9.4	L	0	0
Z1-141	**A**	Spaceloft Subsea	12/20/2017	7	0.02420	4.818	1056	24.6	0.9	6.8	L	0	0
Z1-142	**A**	Pyrogel XT-E	1/8/2018	5	0.02928	5.830	1278	29.2	0.6	11.8	L	0	0
Z1-143	**L**	Polyglas SBS Asphalt Aerogel Blanket	12/19/2017	5	0.03832	7.630	1673	46.7	0.2	30.6	L	0	0
Z1-144	**A**	Cryogel x201 2-layers	1/26/2018	3			0	37.0		20.2	L	0	0
Z1-145	**P**	Aeroplastic Versify Sample-A 22-stack	1/31/2018	5	0.09489	18.893	4143	117.6	0.2	153		0	0
Z1-146	**P**	Aeroplastic Versify Sample-B 13-stack	1/31/2018	5	0.09887	19.685	4317	130.6	0.1	174		0	0
Z1-147	**P**	Aeroplastic Versify Sample-C 14-stack	2/1/2018	5	0.09384	18.684	4097	127.7	0.5	170		0	0
Z1-148	**A**	Cryogel x201 #2 (1 Layer)	2/7/2018	5	0.03071	6.114	1341	30.5	0.5	13.2	L	0	0
Z1-149	**A**	Cryogel x201 #3 (1 Layer)	2/9/2018	5	0.03000	5.973	1310	29.8	0.6	12.4	L	0	0
Z1-138a	**A**	Cryogel x201 (1 layer)	2/2/2018	5	0.03074	6.120	1342	30.0	0.4	12.6	L	0	0
Z1-150	**F**	SOFI #2	2/12/2018	5	0.02916	5.805	1273	39.1	1.4	22.5	L	0.4	495
Z1-151	**W**	Balsa (in-plane) 7.3mm	2/13/2018	5	0.04153	8.264	1812	61.0	0.6	46.0	L	7	916
Z1-152	**W**	Balsa (in-plane) 6.4mm	2/13/2018	5	0.05086	10.121	2219	60.8	1.0	45.8	L	7	920
Z1-153	**F**	Polyimide Foam Solimide	2/14/2018	5	0.04080	8.119	1780	52.2	0.4	36.6	L	0	0
Z1-154	**F**	FoamGlass #1 broken	2/14/2018	7	0.04092	8.143	1786	51.4	1.0	35.7	L	0.8	193
Z1-155	**A**	ULD Aerogel	2/16/2018	7	0.02337	4.651	1020	35.5	1.0	18.6	L	0.8	365
Z1-156	**F**	FoamGlass #2	2/21/2018	7	0.03842	7.646	1677	49.1	0.7	33.2	L	0.8	202
Z1-157a	**F**	FoamGlass #3	2/23/2018	6	0.03746	7.455	1635	48.2	0.4	32.3	L	0.8	210
Z1-158	**B**	Glass Bubbles, low density 0.038	3/6/2018	5	0.04944	9.839	2157	63.7	0.3	49.0	L	1.72	925
Z1-158a	**B**	Glass Bubbles, nominal tap density	3/8/2018	5	0.03626	7.216	1582	46.7	0.2	30.6	L	1.72	769
Z1-158b	**B**	Glass Bubbles, nominal tap density	3/12/2018	5	0.03553	7.070	1550	45.8	0.1	29.7	L	1.72	794
Z1-158c	**B**	Glass Bubbles, nominal tap density	3/15/2018	5	0.03579	7.122	1562	46.1	0.3	30.0	L	1.72	786

For example of a range of data for materials with exceptionally low thermal conductivity, Figure 4 shows a comparison of various commercial aerogel insulation materials, which have a thermal conductivity less than 50 mW/m-K, which is specific to the needs of the work to accomplish a special "low end" calibration. The work of the current project begins with test specimen Z1-148 and goes through Z1-158.to the low end calibration.

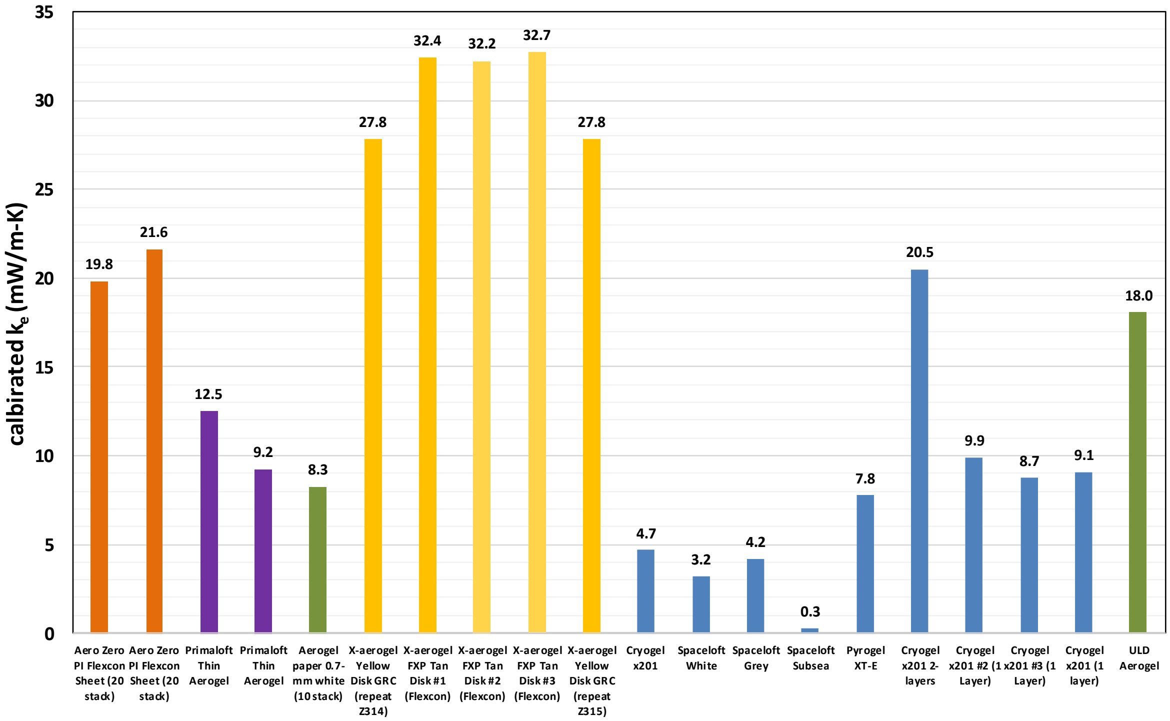


Figure 4. Comparison of various commercial and research Aerogel insulation materials

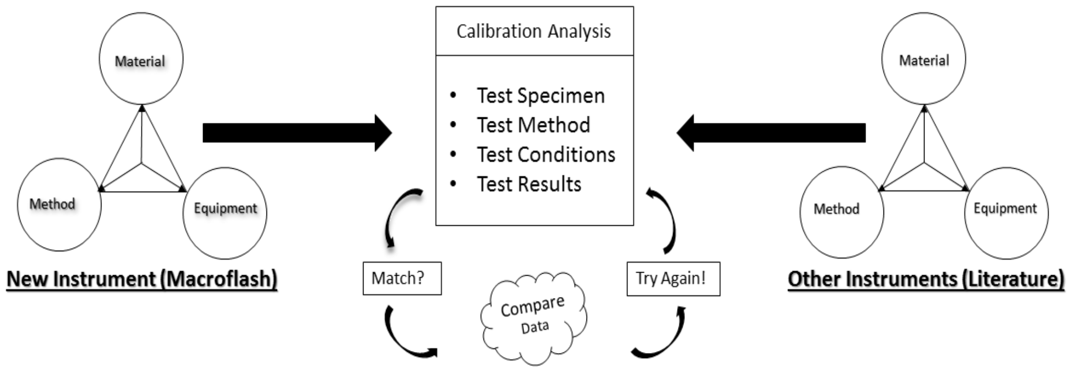
Additionally, the compressive strength and density were recorded for each material so that a "Figure of Merit" (FOM) could be derived as a representation of the overall performance. These results are summarized by various categories in Table 2, and the FOM was calculated according to the provided FOM equation:

$$FOM = \left(\frac{\sigma_c}{k_e \cdot \rho}\right) \cdot 10^3 \quad (4)$$

Using the compressive strength (σ_c), effective thermal conductivity (k_e), and density (ρ), the FOM equation generates values (with units of K·m·s/g) in which larger values are indicative of potential candidates for structural thermal insulation materials. Since low values of thermal conductivity and density are desired, these parameters were placed in the denominator of the FOM equation. The compressive strength was placed in the numerator since large strengths are desired. Combining the desired values in this way allows for a rapidly obtained, quantitative screening parameter to identify potentially high performing structural thermal insulation candidates.

III. Macroflash Analysis and Calibration

Initial Macroflash comparative k_e measurements are reported as raw data that is then calibrated to give the effective k_e values reported in the tables above. The calibration is based off of a linear fit of measured material thermal conductivities to standard reference data for commercial material with a known k_e, such as: Cryogel, SOFI, FoamGlas, Polytetrafluoroethylene (PTFE), and Glass Fiber Reinforced Plastic (G-10CR). The problem here is that the manufacturers reported values for thermal conductivity are not at the necessary warm and cold boundary temperatures of 293 K and 78K, respectively. Most typically, these data are taken at ambient temperature (around 293K) with only a very small temperature difference imposed by the commercial test instruments generally used. The standard reference data therefore have to be derived by carful research and certain selection from the available scientific peer-reviewed published literature of the world – some of it from the NASA CTL itself. Figure 5 provides a general overview of the process and parameters involved in calibrating a comparative type thermal apparatus. Many factors must match up including test specimen type, method, condition, etc.



1) Test Specimen: material, density, size, shape, surface finish, etc.
2) Test Method: type of apparatus, principal of heat flow calculation, steady state or transient, etc.
3) Test Conditions: WBT, CBT, ΔT, calculated T_m, thermal paste, compression load, gas environment, etc.
4) Test Results: type of thermal conductivity, large ΔT or small ΔT, T_m, method of calculating thermal conductivity (λ, λ_e, k-value, k_a, k_e, etc.)

Figure 5. Guide for the Calibration of a Comparative Thermal Test Apparatus.

Standard methods of test specimen preparation, testing, thermal performance calculations, data recording and data reporting are all essential. As shown previously in Table 1 and Table 2, the key parameters include density, compression, specimen thickness, diameter, and whether or not thermal contact paste was used.

A. Explanation of Full Range and New Low Range

From the prior work of the NASA CTL over the last 5 years, researchers determined that a full range curve fit worked well except for materials with a Ke < 50 mW/m-K. This work therefore picks up with the need to add a special low range calibration curve and, if reasonable, update the full range curve as well.

A summary of the special low-end calibration data is given in Table 3 and graphically presented in Figure 6. Together, these five materials through dozens of tests have produced the necessary data sets for bringing a higher fidelity to the Macroflash testing of materials with absolute values of effective thermal conductivity below 50 mW/m-K. The baseline test conditions for this calibration remain as follows: 293 K and 78 K boundary temperatures; ambient pressure gaseous nitrogen environment.

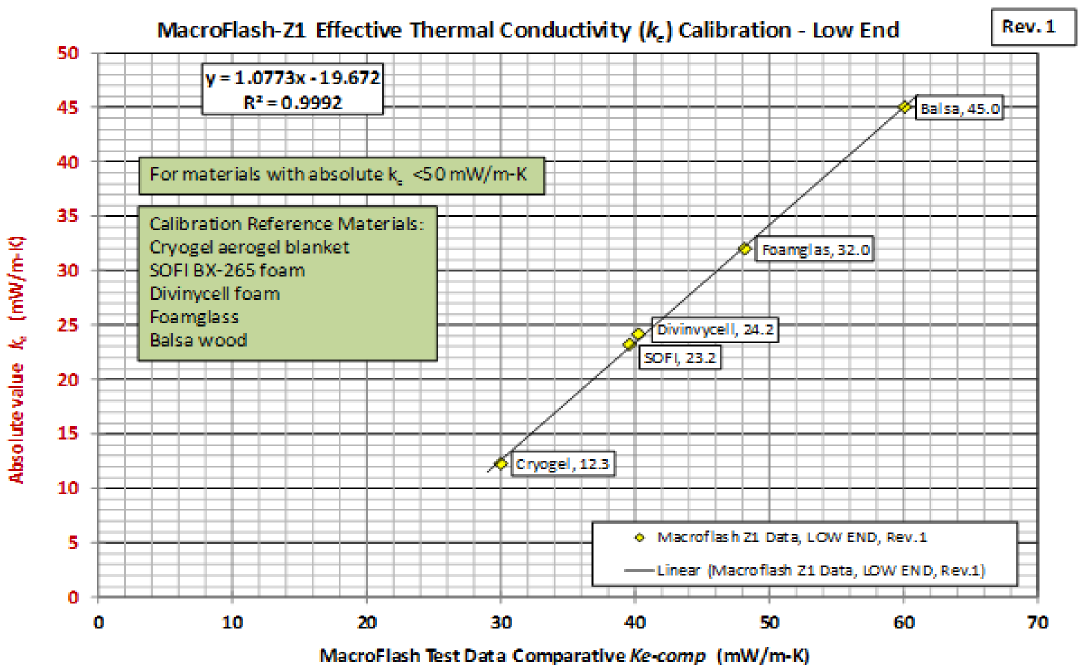


Figure 6. Macroflash- Z1 Effective Thermal Conductivity (k_e) Calibration- Low End.

Table 3. Macroflash Calibration Data- Low End Range

Reference Material	Density	Macroflash Test Data	Absolute Value	Reference
		comparative-Ke	k_e	
	kg/m^3	mW/m-K	mW/m-K	
Aerogel Blanket (Cryogel)	177	30.0	12.3	1
Polyiso Spray Foam (SOFI BX-265)	36	39.6	23.2	2
Divinycell Foam H45	50	40.3	24.2	3
Cellular Glass	118	48.2	32.0	4
Balsa wood (in plane)	166	60.1	45.0	5

According to the last column of Table 3, the references for the absolute thermal conductivity data for these materials being used as reference materials for the special low-end calibration are listed as follows:

1. Fesmire J, Ancipink J, Swanger A, White S, and Yarbrough D, "Thermal conductivity of aerogel blanket insulation under cryogenic-vacuum conditions in different gas environments," Advances in Cryogenic Engineering, IOP 278 (2017) 012198.
2. Fesmire, J. E., Coffman, B. E., Meneghelli, B. J., Heckle, K. W., "Spray-On Foam Insulations for Launch Vehicle Cryogenic Tanks," Cryogenics, doi:10.1016/j.cryogenics.2012.01.018.
3. Fesmire, J., et al., "Thermal Performance Testing of Candidate Tank Insulation Materials," Space Launch Initiative / 2nd Generation Launch Vehicles, Final Report to NASA-LaRC, Boeing, Northrup Grumman, May 30, 2002.
4. Flynn, Thomas, Cryogenic Engineering, 2nd edition, Marcel Dekker, New York, 2005.

5. National Institute of Standards, NIST Cryogenic Materials Property Database Index, https://www.nist.gov/mml/acmd/nist-cryogenic-materials-property-database-index

The low-end is of course only part of the full range of materials from insulators to plastics to composites with thermal conductivities as high as approximately 800 mW/m-K. For completeness, the full range calibration is presented in Figure 7.

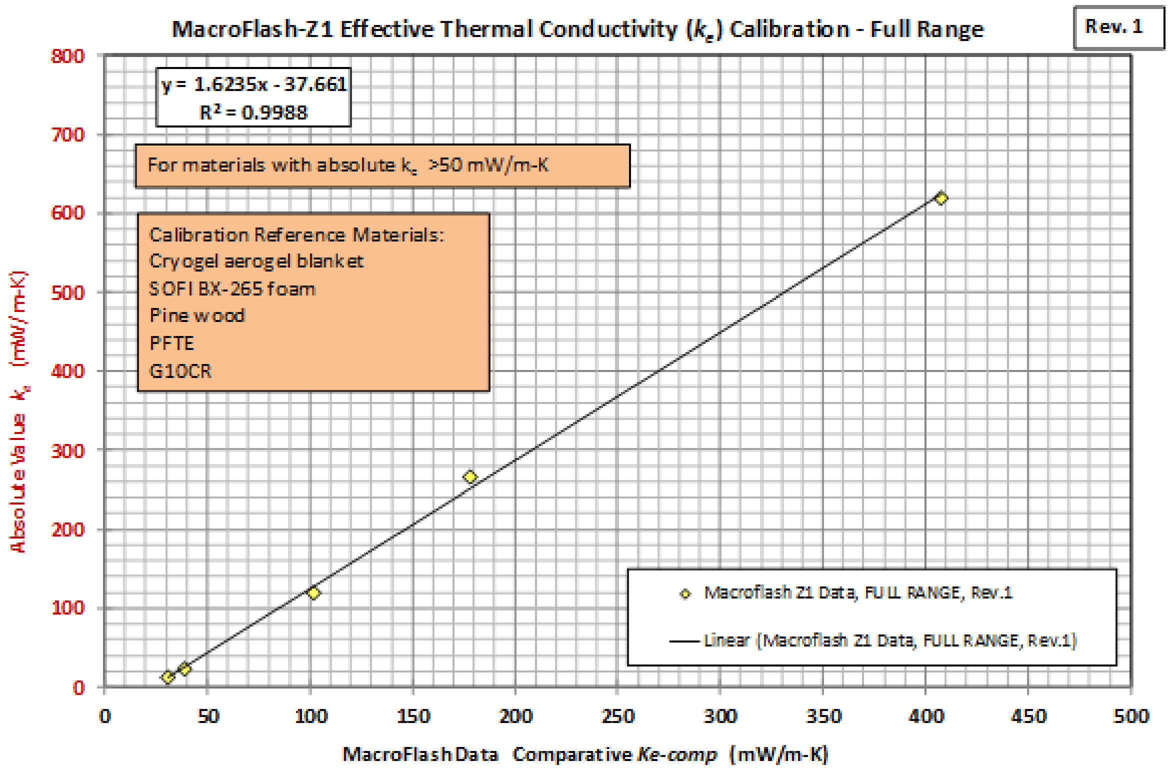


Figure 7. Macroflash-Z1 Effective Thermal Conductivity (k_e) Calibration – Full Range.

B. Software Revisions

Putting all the data together, including the full range of materials for testing over the last five years, the Macroflash software revisions are listed in Table 4. Included are the revised full range equation and the new low-end equation. These equations will be applied to the LabView software of the Macroflash test system. The user will select, up front before each given test, if the material is to be subject to calibration by the full range (above 50 mW/m-K) or low-end (below 50 mW/m-K) calibration. The automated report will be generated accordingly with notice as to which equation was applied for the calibrated data. The Macroflash uses custom designed LabView software by National Instruments. The software will be revised to include the special low-range curve fit as well as an update (from Rev 0 to Rev. 1) of the full range curve fit. The old and new equations are listed as follows:

Table 4. New low-end and updated full range calibration equations for the Macroflash test system.

Macroflash Calibration Curve-Fit Equations:					Special Low-End Calibration:		
Rev	**Date**	**m**	**b**	**R^2**	**m**	**b**	**R^2**
0	10/6/2015	1.6301	-39.827	0.9988	n/a	n/a	n/a
1	3/31/2018	1.6235	-37.661	0.9988	1.0773	-19.672	0.9992

With these new equations as a result from the testing and analysis of these low density materials, the calibration columns on the master data sheets have been successfully updated which will result in a k_e measurement that more accurately reflects the literature value. This information will enable more accurate comparisons among materials, whether they be thermal insulation materials or composite structural materials, in each category of research interest. The information also provides valuable data for engineering calculations in the design and construction of future cryogenic tank systems.

IV. Cryogenic Testing of Tanks

A technology demonstration test was conducted as a portion of this project to provide comparative thermal performance data for a variety of small vacuum insulated vessels (cryogenic tank systems) in order to establish good methodology for test and evaluation as well as guide and inform the development of high efficiency, light weight cryogenic tanks of the future. The test articles, shown in Figure 8, range from 8 oz. flasks to 10 liter lab dewars that were custom designed and built to serve as storage tanks for cryogenic fluid. Evaporative (boil-off) calorimeter test protocols using LN_2 were established to provide tank test conditions characteristic of the large storage tanks such as those that support Space Launch System (SLS) hardware and flight operations. The following section provides comparative thermal performance test results for the vessels listed in table 4.

Figure 8. Cryogenic vacuum-jacketed test vessels.

Table 4. Summary and Properties of Vessels Tested.

Tank	Description	Volume l	Design Details OD mm	ID mm	Le mm	Construction
International Cryogenics Inc. Dewar	Large Lab Grade Dewar	10.000	286	235	356	Aluminum Alloy
MVE Lab Dewar	Large Lab Grade Dewar	10.000	260	210	333	5156 Aluminum Alloy
Standard Lab Flask	Lab Grade Flask	1.082	85	70	295	silvered glass
Stanley Classic	Classic VJ bottle, 1.1 qt, purchased in 2017	1.058	96	89	263	SST
Hydroflask	40 oz White powdercoated exterior	1.118	91	88	216	SST
Yeti Bottle 30 oz.	30 oz VJ bottle, stanless steel exterior	0.887	95	76	178	SST w/Cu Shield
NASA Slim Flask	8 oz VJ slender flask, stainless steel exterior	0.237	45	39	197	SST
Yeti 18 oz. Cup	18 oz Travel Mug, stainless steel exterior	0.563	76	68	159	SST
"Char-Vac" Stanley Classic	Classic VJ bottle with "Char-Vac" Technology	0.968	96	83	222	SST
Stanley Cup	12 oz Travel Mug, classic Stanley Green exterior	0.355	74	62	108	SST

A. Explanation of "Total System" Thermal Performance

Thermal performance as a function of cryogenic commodity (nitrogen), vacuum pressure, insulation fill level, tank liquid level, and thermal cycles will be presented. Thermal performance is greatly dependent on the quality of fabrication of the vessel and the vacuum level maintained during operation. The thermal performance of the vessels was calculated from the heat transfer rate, and the tank geometry data. One of the primary measurements of comparative thermal performance in this work is the heat flow (Q) per volume, or "Z-number", which is a highly useful parameter that supplies rapid insight for cryogenic tank insulation system comparison purposes.

B. Method

The method used for measuring the heat flow (Q) in each case was boiloff calorimetry using LN_2 as the cryogen and the mass measured overtime with a precision scale. This method was most appropriate as the weight loss due to the boiloff of nitrogen gas is proportional to the total heat leak into the inner vessel. The heat transfer rate into the test vessel was calculated from the flow rate using the heat of vaporization of LN_2 (199 J/g). Vessels with the thermal capability to hold LN_2 at a level more than 25% of their total volume for a 24 hour period were cold soaked for 24 hours prior to testing. For vessels that did not have the thermal capability to maintain a 25% level of LN_2 overnight, a standard 8 hour cold soak was utilized prior to testing. The larger industrial vessels, which are specifically constructed to hold cryogenic fluid, were tested over a period of 72 hours after having been cold soaked for an undetermined amount of time. Vessels were filled to approximately 75% of their total volume for testing and fit with a lid or loosely attached plug to allow for ventilation of cryogen. A total of three runs were performed on all vessels with a volume less than five liters. Each run was 15 minutes in duration and the data points were averaged from a 10 minute window within the 15 minute time frame. The ambient conditions (temperature, barometric pressure, and humidity) were also monitored and recorded.

C. Results

The performance of a given cryogenic tank insulation system has as much to do with engineering and manufacturing as it does with materials and heat transfer properties. This research study was entitled "comparative" to acknowledge all experimental methods (preparation, and testing sequences) must always be performed as close to the same way as possible. The results from the tests are given in Table 5. The Z numbers ranged from as low as 0.02 W/L up to 7 W/L. The two very low numbers (highest thermal performance) are attributed to the fact that these dewars included approximately 40 layers of multilayer insulation (40 reflection layers) within the vacuum space. The next best vessel was the standard laboratory flask with its silvered glass reflection layer within its vacuum space. The remaining small flasks have only the metal surfaces of the inner and outer surfaces of the vacuum annular space which could be polished or not polished, stainless steel only or copper coated. In vacuum, the dominate heat transfer is by radiation thus making the type of number of radiation shields a crucial factor (in addition to a high level of vacuum and a good overall design that minimizes the heat transfer that occurs by solid conduction).

Table 5. Cryogenic Vacuum-Jacketed Tank Test Results

Tank	Description	Q_{total} W	? g/s	Test Notes Dates	Runs	Protocol	*A_e m^2	q_{total} W/m^2	†q_{rad} W/m^2	^q_{cond} W/m^2	Z W/L	Normal Evaporation Rate %	Time to Empty hours
International Cryogenics Inc. Dewar	Large Lab Grade Dewar	0.230	0.0012	3/19/2018	1	Tested over a 72 hour period.	0.08663	3		2.655	0.02	1.2	1935.5
MVE Lab Dewar	Large Lab Grade Dewar	0.353	0.0018	3/26/2018	1	Tested over a 72 hour period.	0.07613	5		4.636	0.04	1.9	1263.2
Standard Lab Flask	Lab Grade Flask	0.862	0.0043	3/2/2018	3	Standar, 24+ hr cold soak	0.01060	81	10	71	0.80	42.9	56.0
Stanley Classic	Classic VJ bottle, 1.1 qt, purchased in 2017	1.327	0.0066	3/3/2018	3	standard, 24+ hr cold soak	0.00809	164		164.03	1.25	67.6	35.5
Hydroflask	40 oz White powdercoated exterior	1.901	0.0095	3/4/2018	3	standard, 24+ hr cold soak	0.00669	284		284.07	1.7	90.97	26.38
Yeti Bottle 30 oz.	30 oz VJ bottle, stanless steel exterior	2.156	0.0105	3/5/2018	3	standard, 24+ hr cold soak	0.01212	178	20	157.89	2.43	127.51	18.8
NASA Slim Flask	8 oz VJ slender flask, stainless steel exterior	1.15	0.00578	3/6/2018	3	8+ hr cold soak	0.00295	390		389.83	4.85	261.43	9.18
Yeti 18 oz. Cup	18 oz Travel Mug, stainless steel exterior	2.852	0.0143	3/7/2018	3	8+ hr cold soak	0.00554	515	20	494.62	5.07	272.82	8.8
"Char-Vac" Stanley Classic	Classic VJ bottle with "Char-Vac" Technology	5.207	0.0262	3/8/2018	3	standard, 24+ hr cold soak	0.01026	508		507.5	5.38	289.3	8.29
Stanley Cup	12 oz Travel Mug, classic Stanley Green exterior	2.489	0.0125	3/9/2018	3	8+ hr cold soak	0.00579	430		430.15	7.01	377.46	6.36

V. Discussion and Recommendations

From the test methodology for materials and tanks, and from the test results and analysis of each, we can begin to inform and guide the design for high efficiency tanks of the future. Structural materials, insulation materials, design and construction must all work together in order to form the basis for a well-made cryogenic storage tank.

IV. Conclusion

A study of the thermal conductivity of materials was performed in order to improve the accuracy of the calibration for the low-end range of the Macroflash thermal test instrument. As a secondary study at the Cryogenics Test Laboratory, the thermal performance testing of different cryogenic tank systems was performed according to a new standard laboratory methodology. A new performance metric called the Z-number, or volumetric boiloff, was developed to compare the wide range of tank test results. Combining aspects of materials science and cryogenic engineering, the experimental data sets include thermal performance comparisons between vacuum-jacketed vessels (cryogenic tank systems) and the effective thermal conductivities of a variety of low density materials. Through multiple liquid nitrogen boiloff calorimetry tests on an extensive array of low density, thermal insulation materials tested using the Macroflash Cup Cryostat a new low-end calibration has been set in place as an updated revision that has improved the data analysis for the test results. From the thermal performance data on the 10 different vacuum-jacketed vessels tested using liquid nitrogen boiloff calorimetry, better informed decisions can be made moving forward on the construction, design and configuration of future cryogenic tank systems.

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