COVER SHEET

Title: Thermal Performance Testing of Cryogenic Insulation Systems

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ABSTRACT

Efficient methods for characterizing thermal performance of materials under cryogenic and vacuum conditions have been developed. These methods provide thermal conductivity data on materials under actual-use conditions and are complementary to established methods. The actual-use environment of full temperature difference in combination with vacuum-pressure is essential for understanding insulation system performance. Test articles include solids, foams, powders, layered blankets, composite panels, and other materials. Test methodology and apparatus design for several insulation test cryostats are discussed. The measurement principle is liquid nitrogen boil-off calorimetry. Heat flux capability ranges from approximately 0.5 to 500 watts per square meter; corresponding apparent thermal conductivity values range from below 0.01 up to about 60 mW/m-K. Example data for different insulation materials are also presented. Upon further standardization work, these patented insulation test cryostats can be available to industry for a wide range of practical applications.

INTRODUCTION

Beginning in the early 1900's refrigeration has now matured into standard processes and goods through all industrial and consumer levels. A key part of our now refrigerated, air-conditioned age is the insulation testing and heat measurement methods that developed during the 20th century [1]. Rapid development of cryogenic insulation began in the 1950's in response to the need for large amounts of liquid hydrogen to support the development of thermonuclear weapons [2]. The work continued in the 1960's in response to the needs of the U.S. space program's goal of exploring the Moon. The cryogenic laboratories of NBS Boulder were leaders in both the measurement of thermophysical properties of cryogens and the test and development of evacuated insulation systems such as perlite and multilayer insulation [3]. An important test method was heat calorimetry by measuring the boil-off (or evaporation) of a liquid cryogen from a test vessel surrounded by the insulation system. Few research programs in this area were initiated in the 1970's through 1980's, but motivation for improved insulation systems and better understanding of thermal performance has been steadily increasing since the 1990's.

Cryogenics and the domain of very-low temperature refrigeration are taking a more and more significant role. Growing areas of application include food processing, cryosurgery, biological shipping, hydrogen fueled cars, computer equipment, cellular communications, superconducting power transmission, space launch vehicles, life-support systems, enormous facilities for particle physics research, and many energy-intensive industrial processes. All these applications require, directly or indirectly, that cryogens be stored, managed, and transferred among various locations. Based on the timetable for development of refrigeration, this technological demand for cryogenic applications coupled with increasing world demands for energy conservation, alternative energy sources, and improved environmental preservation would lead cryogenics into the mainstream of industrial and consumer products by about 2050.

The Case for Cryogenic Insulation

Concurrent with the need for more cryogenic engineering and application is the need for protecting the energy investment represented by the cryogenic fluid. The connection is born out of basic economics for reducing the power bill or minimizing the mass of a spacecraft. The needs for better thermal insulation are therefore integral with technological advancement on a wide scale. For advancement of thermal insulation systems, three areas of development must be concurrently performed: 1) increased knowledge of thermal performance, 2) improved materials, and 3) novel methods of application. Testing must be performed to minimize heat transfer into cryogenic tanks and piping and to provide adequate system control and safety. Thermal characterization of insulation materials is a key part of enabling the development of efficient, low-maintenance cryogenic systems.

Testing and evaluation of cryogenic thermal insulation systems is a focus of the Cryogenics Test Laboratory at NASA Kennedy Space Center. Complete understanding of the heat transfer mechanisms through novel insulation materials, such as aerogels and layered composites, led to the development of new equipment and methods for testing under actual-use, cryogenic-vacuum conditions. This new approach to cryogenic boil-off calorimetry is described in comparison with other test methods and with example thermal conductivity data for several materials.

BASIC TERMS FOR THERMAL CONDUCTIVITY

The cryogenic engineer must consider three categories of heat transfer performance. The first is *thermal conductivity*, k, which is the intensive property of a material that indicates its ability to conduct heat. Another term, *thermal transmittance*, incorporates the thermal conductance of a system along with heat transfer due to gaseous conduction, gaseous convection, solid conduction, and radiation. Zarf succinctly describes the "complicated combination" of heat transfer mechanisms involved with measurements of thermal insulation [4]. The problems with understanding the fundamental issues of heat transfer properties are further expanded by the extreme environmental conditions and large temperature differences associated with cryogenic systems. These problems are then amplified by the fact that many cryogenic insulation systems are typically high performance (well below 10 mW/m-K down to as low as 0.01 mW/m-K) which makes basic accuracy in measurement difficult to achieve. Thermal transmittance is termed herein as *apparent thermal conductivity* or *k-value*. A third term is the thermal performance of the overall system as it would be deployed in application. This measure is termed *overall k-value for actual field installation* or k_{oafi} [5]. These three terms, k, *k-value*, and k_{oafi} correspond to the three categories of testing, materials, systems of materials, and overall systems, required for the development of cryogenic insulation systems.

COMPARISON OF TEST METHODS

Three American Society for Testing Materials (ASTM) methods for flat-slab specimens are often used for insulation materials: C177 (absolute, guarded hot plate), C518 (comparative, heat flow meter), and C745 (absolute, boiloff calorimeter). Method C518 is used mostly for near-ambient temperature testing of materials such as building insulation [5]. Method C177 is difficult to apply for large temperature differences and for cryogenic vacuum conditions, for as it is stated, "Under vacuum conditions, especially at lower temperatures, serious errors can arise if care is not taken when installing heater and temperature sensor leads so as to minimize extraneous heat flow-rates and temperature measurement errors." [6] Method C745 is difficult in setup and execution in part because of the cryogenic guard vessel that is required [7]. A generalized comparison of ASTM methods and the newly established Cryostat methods is given in TABLE 1.

TABLE 1. Comparison of the ASTM Methods and the New Cryostat Methods

Method	Type	Sample	Delta Temp	Boundary Temp Range (K)	k-value Range (mW/m-K)	Heat Flux Range (W/m^2)
ASTM C518	Comparative, Heat Flow Meter	Flat, square	Small	273 to 383	5 to 500	---
ASTM C177	Absolute, Guarded Hot Plate	Flat, disk	Small	93 to 773	14 to 2000	---
ASTM C745	Absolute, Boiloff Calorimeter	Flat, disk	Large	250/670 and 20/300	---	0.3 to 30
Cryostat-100	Absolute, Boiloff Calorimeter	Cylindrical	Large	77 to 400	0.01 to 60	0.5 to 500
Cryostat-1	Absolute, Boiloff	Cylindrical	Large	77 to 300	0.03 to 30	0.8 to 120

	Calorimeter					
Cryostat-2	Comparative, Boiloff Calorimeter	Cylindrical	Large	77 to 350	0.1 to 50	2 to 400
Cryostat-4	Comparative, Boiloff Calorimeter	Flat, disk	Large	77 to 350	0.5 to 90	6 to 900

The complementary test methods are necessary for establishing a complete understanding of the heat transfer properties for a given material system. The Cryostat methods are motivated by specific application (system thermal performance evaluation) but are useful for materials research as well. The high-performance material systems used for cryogenic vacuum conditions are often impractical to test with existing ASTM methods, therefore presenting the need for development of additional standards.

The use of several overlapping test methods is important to establish credibility of the heat measurement results as illustrated by the following example for aerogel beads. From Cryostat-100 testing, we report a k-value of 1.7 mW/m-K at the high vacuum condition with boundary temperatures of 293 K and 78 K [99]. Our colleagues using a new liquid helium cooled apparatus report a thermal conductivity (k) of 0.14 mW/m-K at a mean temperature of 25 K [99]. Applying a temperature integral to the Cryostat data gives a good correspondence to the latter result. The data from different methods provide intersections in thermal conductivity values and promote understanding of the different mechanisms of heat transfer at work.

CRYOSTAT TEST METHODS

The new cryostats use steady-state liquid nitrogen boiloff (evaporation rate) calorimeter methods to determine apparent thermal conductivity (k-value) and heat flux. The advantages and disadvantages of using cryogen boiloff calorimeters are described in the literature [1,2,3,4]. Three new test methods and apparatus have been established to test insulation materials under the combination of full temperature difference (78 K – 293 K) and full-range vacuum conditions: Cryostat-1 (absolute, cylindrical), Cryostat-2 (comparative, cylindrical), and Cryostat-4 (comparative, flat disk). Cryostat testing is performed according to in-house standards with guidance from the noted ASTM methods, as applicable.

Test article preparation often includes the installation of temperature sensors through the thickness of the insulation. A cryostat test series begins with a vacuum pumping and heating cycle or a gaseous nitrogen purge, as required. A test is defined as the steady-state heat leak rate (watts [W]) through the specimen at a prescribed set of environmental conditions, including a stable warm-boundary temperature (WBT), a stable cold-boundary temperature (CBT), and a cold vacuum pressure (CVP).

A liquid nitrogen cold mass maintains the CBT at approximately 78 K. The WBT is maintained at approximately 293 K using an external heater with electronic controller. The delta and mean temperatures are therefore 215 K and 185 K, respectively. Vacuum levels cover the full range from high vacuum (HV) (below 1×10^{-4} torr) to soft vacuum (SV) (~1 torr) to no vacuum (NV) (760 torr). The residual gas is typically nitrogen, but other gases can be used as required.

The rate of the heat transfer, Q, through the insulation system into the cold-mass tank is directly proportional to the liquid nitrogen boiloff flow rate. The flow rate is measured by a mass flow meter or precision weight scale connected to a data acquisition system. In measuring the flow rate, the back-pressure from the flow meter and connecting tube is approximately 0.1 psig. This pressure, taken as the saturation pressure of the cryogen, corresponds to a heat of vaporization of 198.6 J/g for liquid nitrogen.

The apparent thermal conductivity, *k-value*, is determined from Fourier's law for heat conduction through a cylindrical wall or through a flat plate. The mean heat flux is calculated by dividing the total heat transfer rate by the effective area of heat transfer. Further details on the heat transfer calculations as well as uncertainty analyses for each apparatus have been previously reported [99]. The total error is approximately 3.2% with the accuracy of the heat of vaporization being the dominate factor at 2% estimated error. Further analytical refinements to be accomplished include considering the effect of ullage vapor heating for different test chamber geometries and the effect of barometric pressure variations on the heat of vaporization.

DESCRIPTION OF CRYOSTAT TEST EQUIPMENT

In prior experimental work using cryogenic boil-off calorimetry, the apparatus designs were dependent on thin-walled chambers and the goal of a uniform (de-stratified) cold mass temperature. Copper and copper wool materials were often used to achieve the practical minimum temperature gradients. The new cryostat test equipment is designed in just the opposite way. Heavy-wall stainless steel construction is used along with natural stratification of the cryogen to achieve improved thermal stability and repeatability. In this way several of the prior difficulties such as boil-off vapor condensation are avoided. The heat transfer due to slightly different liquid temperatures within the cold mass is minimized by the use of vapor gaps between chambers in combination with the method of thermal stabilization.

Cryostat-1 is a cylindrical test apparatus for direct measurement of the absolute thermal conductivity of a material system [99]. This apparatus, shown in FIGURE 1, includes a cold mass of overall dimensions 167-mm diameter by 900-mm length and provides absolute k-values for specimens up to 50 mm thick. A simplified schematic of the insulation test apparatus is given in FIGURE 1. A copper sleeve assembly can be used to facilitate the testing of continuously rolled insulation products such as multilayer insulation (MLI) [8].

Surface Temperature Measurement	
Sensor	**Location**
VC1, VC2, VC3	Vacuum Can Temperature
T11, T12, T13	Warm-Boundary Temperature (WBT)
T4 – T10	Insulation Layer Temperatures
T1, T2, T3	Cold-Boundary Temperature (CBT)
CM1, CM2	Cold-Mass Temperature

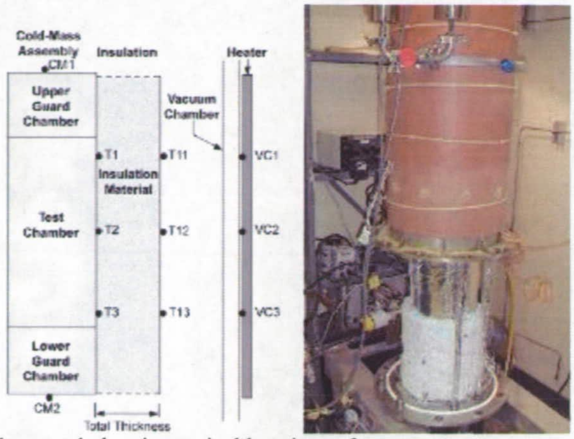

FIGURE 1. Simplified schematic of Cryostat-1 showing typical locations of temperature sensors. Photo on right shows cold mass assembly inside with the vacuum can assembly partly removed.

Cryostat-2 and its duplicate, Cryostat-3, are cylindrical test apparatuses for measurement of the comparative k-value [99]. These apparatus include a 132-mm-diameter-by-500-mm-long cold mass and accepts specimens up to 50 mm thick as shown in Figure 2. The entire cold-mass assembly is easily removed and mounted on a wrapping machine to facilitate the testing of MLI and other layered types of insulation materials.

Surface Temperature	
Sensor	Location
T1	Inside Layer (cold mass), CBT
T2, T3, T4	Middle Layers
T5	Outside Layer, WBT
VC1, VC2, VC3	Vacuum Chamber Exterior

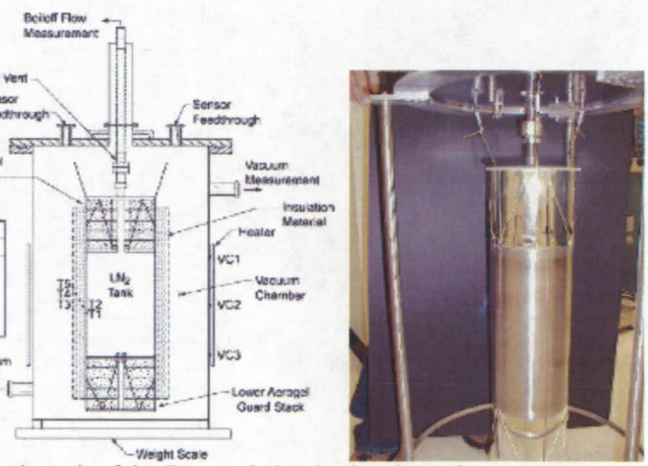

FIGURE 2. Simplified schematic of the Cryostat-2 showing locations of temperature sensors and equipment connections. Photo on right shows the cold-mass assembly mounted its work stand.

Cryostat-4 and its duplicate, Cryostat-5, are flat-plate test apparatuses used for comparative k-value measurements [99]. These apparatus accept test specimens 200 mm in diameter by up to 30 mm thick. A simplified schematic of the test apparatus showing the temperature sensor locations is given in FIGURE 3. Cryostat-4 can be used for a wide range of materials and conditions in addition to the full cryogenic vacuum testing. The cold-mass assembly can be configured for rigid or soft materials, with or without compressive loads applied. An optional load cell assembly is also provided for studying the effects of mechanical loading on a material.

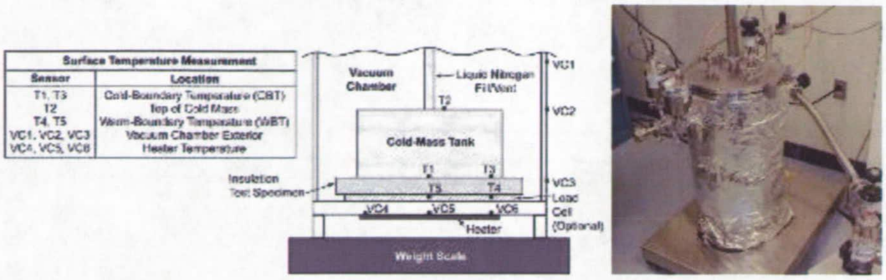

FIGURE 6. Simplified schematic of Cryostat-4 showing typical locations of temperature sensors. Photo on right shows overall view of the test apparatus.

Specimens may be tested in the form of blanket, bulk-fill, clam-shell, flat panel, multilayer, or continuously rolled. The new devices and methods have been proven through hundreds of tests as indicated in TABLE 2. A large library of experimental data and information has been produced through 10 years of research testing work. The newest apparatus, Cryostat-100, was activated in 2006 as the replacement for Cryostat-1. Cryostat-100 incorporates technology from the previous cryostats and provides for faster change-out of test specimens and improved accuracy over a wider range of heat flux. This apparatus was used extensively in the recently completed research studies of spray-on foam insulation (SOFI) and bulk-fill insulation materials for cryogenic tanks [x].

TABLE 2. Summary of Cryostat Testing by Number of Materials and Tests (as of May 2007)

Apparatus	Number of Materials	Number of Tests
Cryostat-1	39	372
Cryostat-2/3	27	208
Cryostat-4/5	101	687
Cryostat-100	14	309
Totals	181	1576

EXAMPLE CRYOSTAT TEST RESULTS

To illustrate the method and the utility of cryostat testing under actual cryogenic-vacuum conditions, some example experimental test results are presented here. A summary of the k-value results for the three test articles, spray-on foam insulation (SOFI), aerogel beads, and 60-layer multilayer insulation (MLI), is presented in Figure 4. The wide range of heat flux measured, several orders of magnitude, is evident through these test results.

For most materials, including the aerogel beads and the MLI, the test series begins with a heating and evacuation cycle to the ultimate vacuum level for that system. Testing then proceeds from high vacuum to no vacuum. For materials such as the SOFI that are mainly intended to operate at ambient pressure, the test series begins with a purge of gaseous nitrogen. Testing is performed at no vacuum and then at step-wise lower vacuum levels as required. Deeper knowledge of the heat transfer characteristics of a system can be obtained through full-range vacuum testing and discerning the interactions of the heat flow with the microstructure and density of the materials.

Some detailed measurements for a specific Cryostat-100 test of the aerogel beads at the 1 millitorr vacuum level is presented in Figures 5, 6, and 7. The final k-value and heat flux are taken from the averaged boil-off flow rate from 92 to 88% full level of the liquid nitrogen in the test chamber (or between about 17 and 21 hours in this example case as shown in Figure 6). The standard requirement is to achieve a fine thermal equilibrium that is coincident with a liquid level of about 90%. One or two refills, as can be seen in Figure 7 are sometimes required beyond the initial cooldown phase.

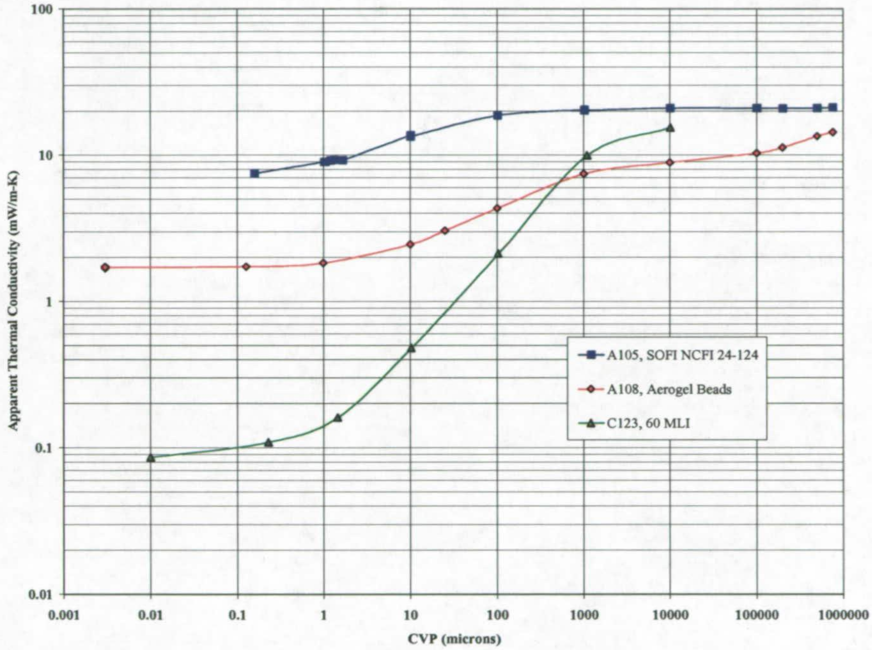

FIGURE 4. Variation of k-value with CVP for SOFI, aerogel beads, and MLI. Boundary temperatures are approximately 78 K and 293 K and the residual gas is nitrogen.

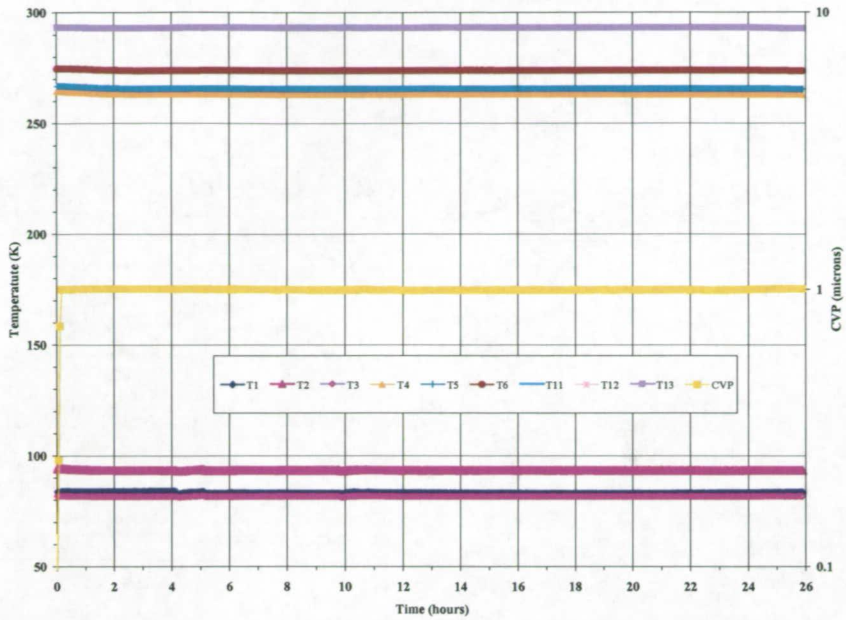

FIGURE 5. Temperature profile through the thickness of aerogel beads at 1 millitorr CVP.

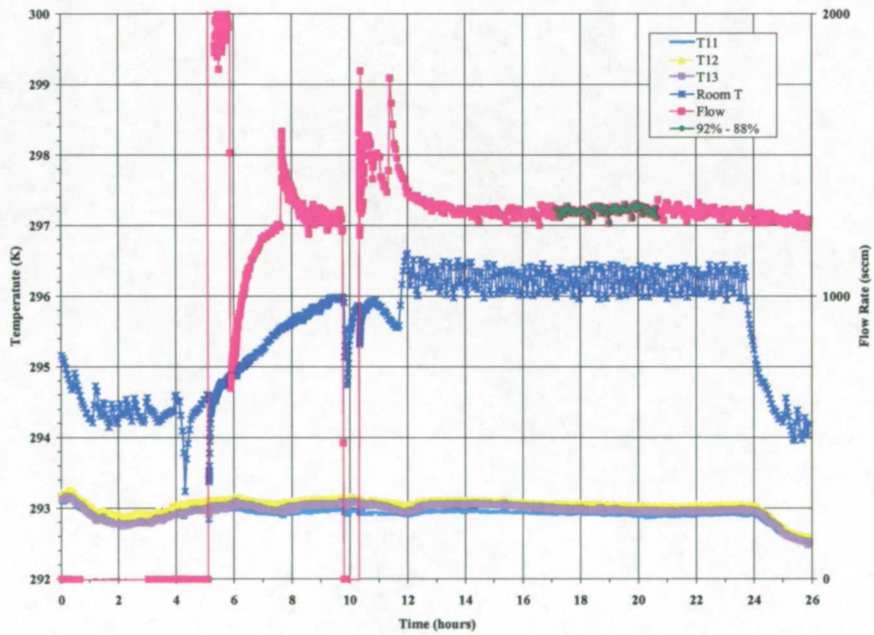

FIGURE 6. Nitrogen boil-off flow rate, WBTs (T11, T12, T13), and room temperature for a Cryostat-100 test of aerogel beads at 1 millitorr CVP.

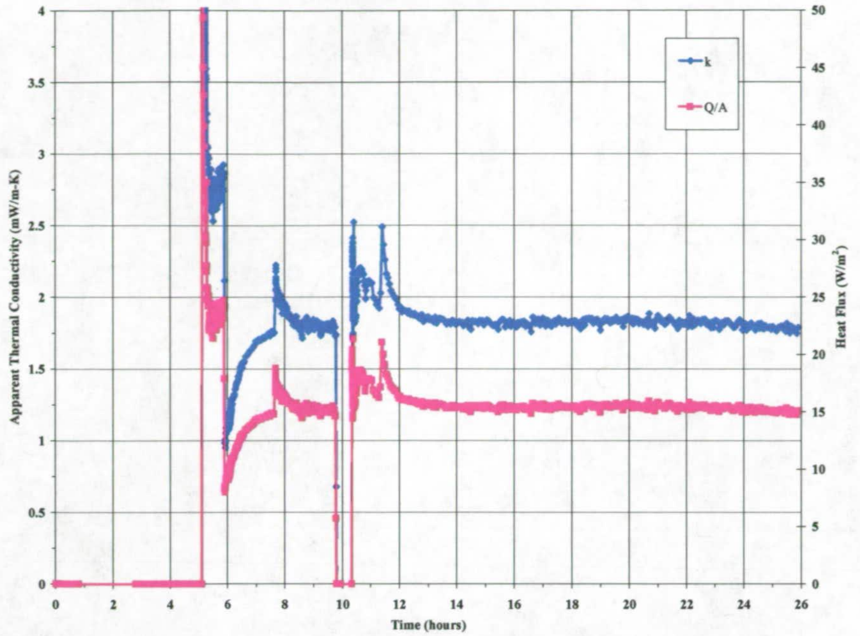

FIGURE 7. Results for k-value and heat flux for test of aerogel beads at 1 millitorr CVP.

CONCLUSION

New equipment and methods for testing cryogenic thermal insulation systems have been successfully developed. Test measurements are made at the full temperature difference (typical boundary temperatures of 78 K and 293 K) and include the full vacuum pressure range. The experimental heat transfer results are generally reported in apparent thermal conductivity (k-value) and mean heat flux through the insulation system. The three new cryostats provide an effective and reliable way to characterize the thermal performance of materials under cryogenic-vacuum conditions. These cryostats have been proven through over 1,500 tests of more than 150 material systems and have supported a wide range of aerospace and industry research projects [x]. The cryostat test methods are performed in accordance with well-developed in-house test standards.

Growing world-wide needs for energy efficiency and related cryogenic applications are creating a demand for improved thermal insulation systems for low temperatures. The corresponding need for the thermal characterization of these systems and materials raises the issue of insulation test standards that are targeted for the cryogenic-vacuum applications. A study of the motivation and arrangement of such standards, with connection to energy, technology, and environmental policies, is therefore recommended.

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14. ABSTRACT
Efficient ways to characterizing thermal performance of materials under cryogenic and vacuum conditions have been developed. These methods provide thermal conductivity data on materials under actual-use conditions and complement established methods. The actual-use environment of a large temperature difference across the insulation in combination with vacuum-pressure is essential for understanding how insulation systems perform. Test articles include solids, foams, powders, layered blankets, and composite panels. Test methodology and apparatus design for several insulation test cryostats are discussed. The measurement principle is liquid nitrogen boil-off calorimetry. Heat flux capability ranges from approximately 0.5 to 500 watts per square meter; corresponding apparent thermal conductivity values range from below 0.01 up to about 60 mW/m-K. Example data for different insulation materials are also presented. After further work to standardize test methods, these patented insulation test cryostats can be used in a wide range of industrial applications.

15. SUBJECT TERMS
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