IOP Conference Series: Materials Science and Engineering

PAPER • **OPEN ACCESS**

Comparison tests of cellular glass insulation for the development of cryogenic insulation standards

To cite this article: J A Demko *et al* 2015 *IOP Conf. Ser.: Mater. Sci. Eng.* **101** 012016

View the article online for updates and enhancements.

Comparison tests of cellular glass insulation for the development of cryogenic insulation standards

J A. Demko[1], J. E. Fesmire[2], J. Dookie[1], J. Bickley[1], and S. Kraft[1]

[1]LeTourneau University2100 South Mobberly Avenue, Longview, TX 75607 USA
[2]NASA Kennedy Space Center, Cryogenics Test Laboratory, NE-F6, KSC, FL 32899 USA

jonathandemko@letu.edu

Abstract. Standards for thermal insulation used in applications between ambient and low temperatures, below 100 K, require test data under relevant conditions and by different laboratories to develop data sets for the proper comparisons of materials. This critically important technology is needed to provide reliable data and methodologies for industrial energy efficiency and energy conservation. Under ASTM International's Committee C16 on Thermal Insulation, two standards have been issued on cryogenic thermal insulation systems. Thermal conductivity data sets have been taken using identical flat-plate boiloff calorimeter instruments independently operated at the Cryogenics Test Laboratory of NASA Kennedy Space Center (KSC) and the Thermal Energy Laboratory of LeTourneau University (LETU). Precision specimens of cellular glass insulation were produced for both laboratories to provide the necessary comparisons to validate the thermal measurements and test methodologies. Additional specimens of commercial cellular glass pipe insulation were tested at LETU to compare with the flat plate results. The test data are discussed in relation to the experimental approach, test methods, and manner of reporting the thermal performance data. This initial Inter-Laboratory Study (ILS) of insulation materials for sub-ambient temperature applications provides a foundation for further ILS work to produce standard data sets for several key commercial materials.

1. Introduction

There is a need for standard thermal data for cryogenic insulation materials or standard test methods and instrumentation in this area. Standard test methods are needed to produce standard data for conduction of heat by materials in the sub-ambient temperature range. An initial step has been taken for producing standard thermal data through a preliminary Inter-Laboratory Study (ILS) initiated by NASA Kennedy Space Center and LeTourneau University. A major objective of this initial research is to explore the suitability of new methodologies for producing standard data for effective thermal conductivity (k_e(T)) and heat flux (q(T)) for commonly used cryogenic insulation materials. Other objectives of this effort are to pave the way for establishing standard data sets for use by engineers, to develop accepted baselines for the performance of novel materials under development, and to provide technical insight on the nature of low heat flows through low-density materials. Knowledge of the dependence of heat transfer related properties on temperature for materials is extremely important because even materials that are considered homogenous may be complicated when applied in systems having temperature differences of several hundred degrees imposed across its thickness.

This study grew out of interaction and collaborations with the members of the ASTM International Committee C16 on Thermal Insulation including the National Institute for Standards and Technology and a number of industry experts. Two Task Groups under C16 recently completed the development of two new standards on the subject of cryogenic insulation. The new standards include ASTM C1774 *Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems* and ASTM C740 *Standard Guide for Evacuated Reflective Insulation in Cryogenic Service* [1-2]. The standard guide to testing covers a number of different methods including boil-off calorimetry.

2. Test Facilities and Materials

This ILS effort is based on comparative testing using an identical testing apparatus, the 'Cryostat-400' [3-4]. The Cryostat-400 test apparatus is schematically shown in figure 1, is a comparative, flat-plate instrument, with a cryogenic boil-off calorimeter. It was chosen for this effort for three reasons. The first is the relative simplicity and ease of operation of this apparatus compared to other insulation test cryostats. Another reason is the ready availability of two identical Cryostat-400 units. A third important reason is the suitability for testing rigid, flat disk type materials in an ambient pressure environment.

Test facilities for ILS include the Cryogenics Test Laboratory (CTL) at the NASA Kennedy Space Center and the Universal Thermal Energy Laboratory (UTEL) at LeTourneau University. The CTL team has been developing experimental techniques and test methodologies in the area of cryogenic thermal insulation system for 20 years. The newly established UTEL includes academic expertise and connects to a number of different industries which are involved in cryogenics and the advancement of the technology for subjects such as energy storage, energy efficiency, and novel thermal material systems targeted for sub-ambient temperature applications. Figures 2 and 3 show the Cryostat-400 in operation at LeTourneau. The UTEL at LeTourneau became operational performing tests using the Cryostat-400 during the summer of 2013 [5].

The round of testing in this paper is limited to cellular glass foam. The test conditions are focused on the ambient pressure (dry nitrogen-no vacuum) case. The standard test specimens are machined to the following nominal dimensions: 203-mm diameter by 25.4-mm thickness by the CTL and supplied to LeTourneau University for testing. A photograph of a typical cellular glass foam sample is shown in figure 4. Individual specimen dimensions and weights are also recorded. The average density of twenty specimens is measured to be approximately 118 kg/m^3. Additional materials and conditions, including vacuum environments, are envisioned for future work with additional laboratory facilities.

The testing in both laboratories was performed in accordance with the standard laboratory practices of the CTL. The overall test methodology and data reporting follow the guidance set in applicable portions of the new ASTM C1774, Annex A3.

2.1. *Brief discussion of the measurement technique*

The measurement technique is described in detail in [3-5]. A brief overview is provided here. The heat flow rate (Q) is the basis for calculating thermal properties of the samples. Thermal properties of interest include: effective thermal conductivity (k_e(T)) and heat flux (q). Calculations of k_e(T) are highly sensitive to the thickness of the test specimen. The thickness, as-tested, must be carefully measured or calculated with explanations of any assumptions taken. The symbols used for calculation of thermal properties from boil-off testing are given in TABLE 1.

The heat load Q, is determined directly from the experiment. The effective heat transfer area of the specimen, A_e, is measured, so once a suitable fit for the temperature distribution can be determined, the first derivative can be found and the thermal conductivity as a function of temperature can be determined. In these initial tests, only the cold boundary temperature (*CBT*) and warm boundary temperature (*WBT*) were measured for simplicity in the current measurement system. These sensors provide sufficient data to determine the thermal conductivity and make the necessary data comparisons.

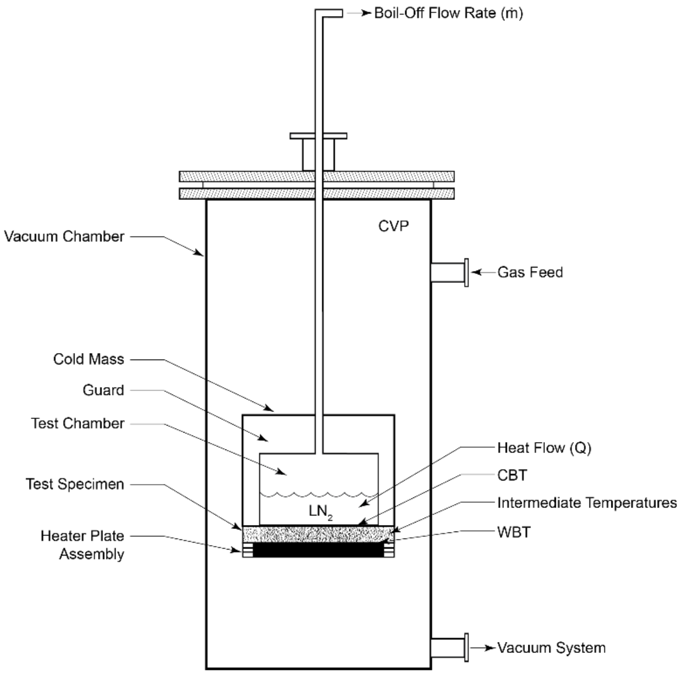

Figure 1. Basic schematic of cryogenic boil-off testing apparatus, flat plate configuration.

TABLE (1). Symbols used for calculation of thermal properties from boil-off testing.

Symbol	Description	Unit
m	Volumetric Flow Rate (at STP)	m^3/s
ρ	Density of Gaseous Nitrogen	kg/m^3
h_{fg}	Heat of Vaporization	J/g
x	Thickness of insulation system	m
d_e	Diameter, effective heat transfer (flat plate)	m
A_e	Area, effective heat transfer area	m^2
ΔT	Temperature difference *(WBT – CBT)*	K

The rate of heat flow (Q) through the insulation system into the cold mass vessel is directly proportional to the liquid nitrogen boil-off rate as shown by Equation (1).

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

Steady-state measurement is achieved by the liquid mass being stratified and stable (a condition promoted by the design features of the cold mass assembly). Measurements of Q are made as the nitrogen is evaporated, with the final test measurement being taken when the vessel is nearly empty. This minimizes any edge effects and ensures a very high level of repeatability in operation.

The value of k_e for heat conduction through a flat plate, is determined from Fourier's law as given by Equation (2). The calculated value of k_e is a comparative one for the Cryostat-400 instrument.

$$k_e = \frac{4Q\Delta L}{\pi d_e^2 \Delta T} \quad (2)$$

IOP Conf. Series: Materials Science and Engineering **101** (2015) 012016 doi:10.1088/1757-899X/101/1/012016

Figure 2. Insulation test instrument Cryostat-400 in the laboratory setting at LeTourneau University.

Figure 3. Insulation test instrument Cryostat-400 in the laboratory setting at LeTourneau University.

Figure 4. Typical test specimen of a cellular glass material.

Measurements were made at the UTEL at LeTourneau University of three standard test specimens of cellular glass insulation supplied by the CTL. Each specimen was installed in the Cryostat-400 unit and, after securing the vacuum chamber lid, it was filled with bulk fill aerogel beads (Cabot Nanogel) to a level just covering the cold mass assembly. The bulk fill insulation distributes around the specimen and reduces the edge effects as prescribed by the CTL test procedure. The Cryostat-400 is a comparative instrument, but provides a stable and repeatable platform for a precise and direct measure of the total heat flow.

3. Inter-laboratory Comparison of Three Cellular Glass Specimens

Provided here is a brief description of the initial series of test runs at the UTEL. Specimen L101 had four test runs, L102 had three test runs, and L103 had two test runs. Each test run resulted in a determination of the effective thermal conductivity (comparative), k_e, for the specimen. The ambient temperature in the room was measured to vary between 27 ^{o}C in the morning reaching 38 ^{o}C by late afternoon.

Measurements of the boil-off and temperatures for a typical run are presented in figures 5 and 6. The boil-off flow is seen to approach a constant value after 3 to 5 hours after filling with liquid nitrogen. After this time that it is assumed that the system has reached thermal equilibrium. Using a point near the end of the run, the conditions are selected that are used to calculate the k_e for the run. The measured WBT was used to determine the value of k_e.

Testing at the CTL maintains the WBT of approximately 293 K. At the UTEL attempts were made to reach this target but the heater system and controls were not fully adequate for this initial round of testing. After discussions, it was decided that obtaining data for the three specimens was more important than investing more time in upgrading and learning the heater system. The results at a slightly lower WBT in the UTEL data but are reasonably close to the CTL value. For the UTEL data in figure 7, the WBT varied between 276 K and 290 K. An adjusted value for k_e was determined using the measured heat flux and assuming the WBT = 293 K. Figure 8 presents results for the CTL measurements. There is reasonable agreement between the two laboratories The UTEL data (9 runs) averaged 67.1 mW/m-K with a variation of 2.6%. The CTL data (24 runs) averaged 73.4 mW/m-K with a variation of 2.0%. The offset of 6.3 mW/m-K (8.6%) is considered to be primarily attributed to different warm boundary temperatures between the two laboratories.

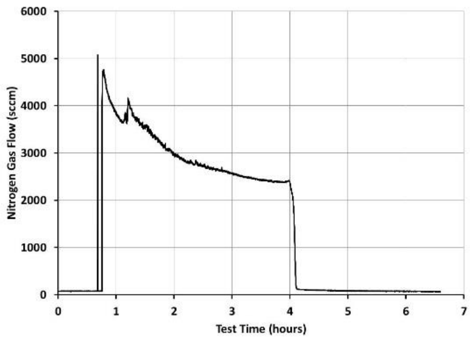

Figure 5. Boil-off flow measurements from L101 Foam glass 8 Specimen #2 Test 2 .

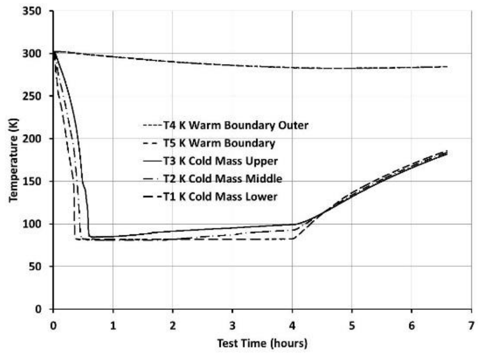

Figure 6. Specimen temperature measurements from L101 Foam glass 8 Specimen #2 Test 2.

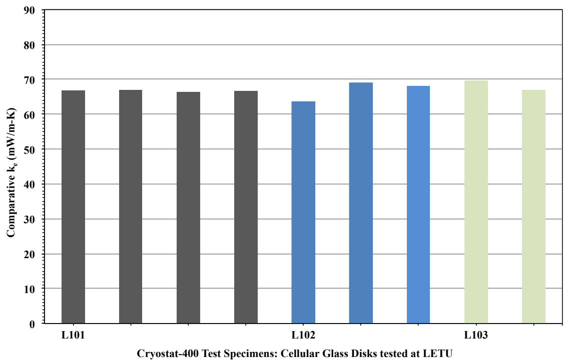

Figure 7. Cryostat-400 results for comparative k_e of cellular glass foam disks tested at LETU: No Vacuum (760 torr) cold vacuum pressure, residual gas nitrogen, boundary temperatures approximately 78 K and WBT that varied between 276 K and 290 K.

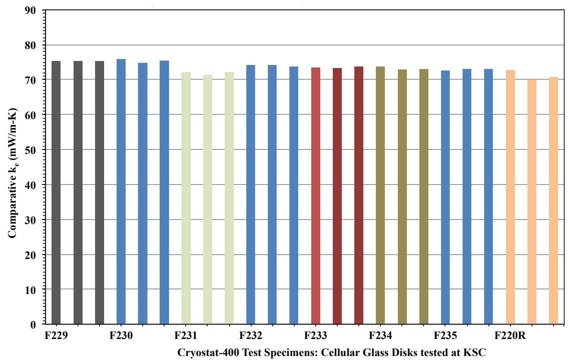

Figure 8. Cryostat-400 results for comparative k_e of cellular glass foam disks tested at KSC: No Vacuum (760 torr) cold vacuum pressure, residual gas nitrogen, boundary temperatures approximately 78 K and 293 K.

4. Initial Development and Testing of Cylindrical (Pipe) Geometry

Testing using the Cryostat-400 required flat specimens. Many applications are for insulating pipe that has a cylindrical geometry. There can also be some practical difference in results, depending on the application. A simple cylindrical pipe geometry test apparatus was therefore conceived and a demonstration prototype was built. The test results are then compared with data from the supplier.

4.1. Experimental Set-up
The apparatus is being developed for making measurements of thermal conductivity by immersing the apparatus in a bath at a controlled temperature (e.g. liquid nitrogen, ice water). The initial testing was conducted at the ambient temperature of the test facility. The apparatus consists of a 609.6 mm long G-10 rod with a 50.8 mm OD. A 304.8 mm long heater is centered between the ends. It was made by wrapping a nichrome wire around the rod and covering with aluminum tape to evenly distribute the heat. The Foamglas (Pittsburgh-Corning) test specimen was obtained from Grainger Industrial Supply. The test specimen has a 50.8 mm ID and is 25.4 mm thick. It came with a vapor barrier that was left on the sample. The length of the specimen was 304.8 mm and it is placed over the heater with a thermal conducting compound in order to minimize contact resistance between the specimen and the heater.

The instrumentation consists of thermometers on the surface of the heater. One is centered on the heater with the other two thermometers equally spaced on either side 76 mm from the center. On the outside of the insulation test article is a fourth RTD that is used to measure the outer surface temperature. A photograph of the assembly is provided in Figure 9.

The ends of the G-10 rod are insulated with foil backed fiberglass in this photograph. The insulation minimizes heat loss from the specimen during a test. The mounted specimen can be enclosed in an aluminum housing so that it can be immersed into different temperature baths (ice water, liquid nitrogen), but the initial results are at room temperature while this system was under development. The pipe insulation comes as two half cylinders. In order to attempt to prevent leakage along the gap between the two parts, polyurethane foam was used in the gap and is seen bulging out at the seam.

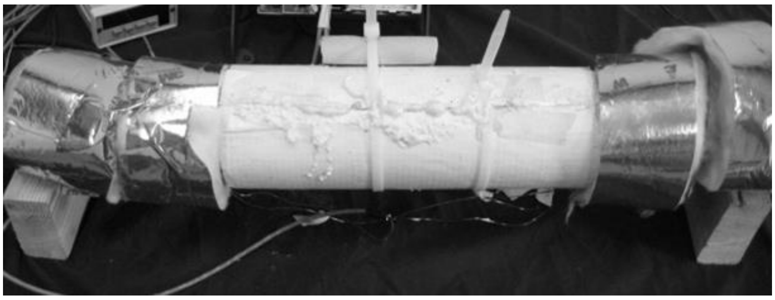

Figure 9. Pipe thermal test apparatus.

The heat load to the specimen was developed by supplying DC current to the nichrome heater wire. The voltage across the heater and the current supplied to the heater are measured to determine the heater power, *Q*, applied to the sample. Then the thermal conductivity can be determined from:

$$k_{measured} = \frac{Q \ln(D_{outer}/D_{inner})}{2\pi L(T_{outer} - T_{inner})}$$

4.2. Initial Results for the Foamglass pipe Sample
The pipe insulation has an advertised R-value ≈ 3.45. According to ASTM C168, this value is equivalent to a thermal conductivity of 41.8 mW/m-K. As described earlier, the 304.8 mm long section was tested using the pipe test apparatus at room temperature. The temperatures for one run are shown in figure 10. The lowest temperature is the measured outer surface temperature.

The testing was conducted in a building that does not have conditioned space and so the start of the test run is in the evening and continues all night. The increase in temperature at around 780 minutes is due to solar heating of the facility. Following the peak at around 1000 minutes, the heater power is turned off and the sample cools down as seen in the temperature plot. There is a discrepancy between the two end temperatures that is not yet resolved. Therefore, the thermal conductivity result must be regarded to have a high uncertainty associated with it at this time.

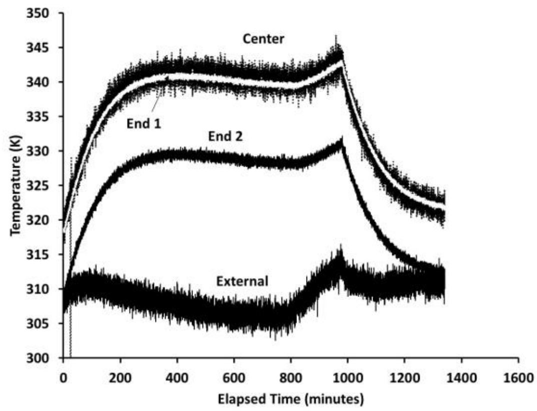

Figure 10. Typical measured temperatures during a pipe thermal conductivity measurement.

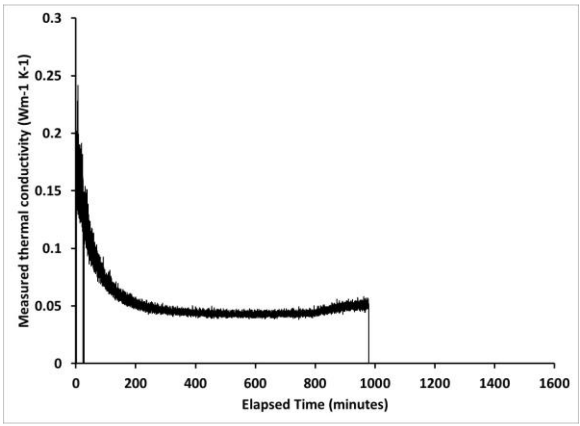

Figure 11. Instantaneous thermal conductivity during a measurement.

The thermal conductivity calculated instantaneously is shown in figure 11 for this test run. After about 400 minutes, the measurement is constant. The value of k_e = 43 mW/m-K compares reasonably well with the vendor data. This result is promising, but the use of more reliable thermometry is necessary to make an accurate determination.

A tendency is seen for the thermal conductivity to increase after about 780 minutes as the room heats up. Future tests in a bath of liquid nitrogen would not be as sensitive to the changing environment (other than perhaps a change in barometric pressure which would change the boiling point).

5. Conclusions and Recommendations for Future Work

Extending some recent developments in the area of technical consensus standards for cryogenic thermal insulation systems, a preliminary Inter-Laboratory Study of insulation materials was initiated by NASA Kennedy Space Center and LeTourneau University. The initial focus is ambient pressure cryogenic boil off testing using the Cryostat-400 flat-plate instrument. Boiloff testing technology is not just for cryogenic testing but is a cost-effective, field-representative methodology to test any material or system for applications at sub-ambient temperatures. The results from measurements of three cellular glass disks compared indicating good repeatability for each laboratory and reasonable agreement between the two laboratories. Initial measurements were at boundary temperatures of approximately of 78 K for the cold boundary and the range of 275 K to 295 K for the warm boundary.

A pipe thermal test apparatus was devised and built. Preliminary results show the method to provide measurement of thermal conductivity, but increased temperature accuracy is needed to reduce uncertainty. This device is important for cylindrical shapes and wide range of temperatures when the apparatus can be immersed in an isothermal bath. The plan is to conduct additional testing in an ice bath (273 K) and in a liquid nitrogen bath (80 K).

A growing need for energy efficiency and cryogenic applications is creating a worldwide demand for improved thermal insulation systems for low temperatures. The need for thermal characterization of these systems and materials raises a corresponding need for insulation test standards and thermal data targeted for cryogenic-vacuum applications. Such standards have a strong correlation to energy, transportation, and environment and the advancement of new materials technologies in these areas.

Acknowledgments
Research sponsored in part by the National Aeronautics and Space Administration (NASA) Kennedy Space Center, Cryogenics Test Laboratory through Grant / Cooperative Agreement Number NNX13AJ14G. We would like to thank LeTourneau University School of Engineering and Engineering Technology for providing facilities, materials, and laboratory equipment to develop the pipe test apparatus.

References
[1] ASTM C1774 - *Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems*. ASTM International, West Conshohocken, PA, USA (2013).
[2] ASTM C740 - *Standard Guide for Evacuated Reflective Cryogenic Insulation*. ASTM International, West Conshohocken, PA, USA (2013).
[3] Fesmire, J.E., Johnson, W.L., Kelly, A.O., Meneghelli, B.J., and Swanger, A.S., “Flat Plate Boiloff Calorimeters for Testing of Thermal Insulation Systems,” Cryogenic Engineering Conference, Tucson, Arizona, July 2015.
[4] Fesmire, J.E. and S.D. Augustynowicz. “Thermal Insulation Testing Method and Apparatus,” U.S. Patent 6,824,306, issued November 30, 2004.
[5] Demko, J.A., Fesmire, J.E., Johnson, W.L. and Swanger, A. M., "Cryogenic Insulation Standard Data and Methodologies," Advances in Cryogenic Engineering: Transactions of the Cryogenic Engineering Conference - CEC, American Institute of Physics, Vol. 59A, pp. 463-470, 2014.
[6] Demko, J.A., Daniels, C. , DeKastle, C. Heitmann, P. and Fesmire, J. E., “Core Technical Capability Special Studies: Cryogenic Insulation Standard Data And Methodologies,” Final Report, NASA Award Number, NNX13AJ14G, March 2014.
[7] ASTM C168, “Standard Terminology Relating to Thermal Insulation,” ASTM International, West Conshohocken, PA, USA (2014).