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## **CRYOGENIC THERMAL PERFORMANCE TESTING OF BULK-FILL AND AEROGEL INSULATION MATERIALS**

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### **ABSTRACT**

The research testing and demonstration of new bulk-fill materials for cryogenic thermal insulation systems was performed by the Cryogenics Test Laboratory at NASA Kennedy Space Center. Thermal conductivity testing under actual-use cryogenic conditions is a key to understanding the total system performance encompassing engineering, economics, and materials factors. A number of bulk fill insulation materials, including aerogel beads, glass bubbles, and perlite powder, were tested using a new cylindrical cryostat. Boundary temperatures for the liquid nitrogen boil-off method were 293 K and 78 K. Tests were performed as a function of cold vacuum pressure from high vacuum to no vacuum conditions. Results are compared with other complementary test methods in the range of 300 K to 20 K. Various testing techniques are shown to be required to obtain a complete understanding of the operating performance of a material and to provide data for answers to design engineering questions.

**KEYWORDS:** Cryogenic tanks, thermal insulation, granular materials, aerogel, thermal conductivity, liquid nitrogen boil-off

**PACS:** 65.60.+a, Thermal properties of amorphous solids and glasses

## INTRODUCTION

Thermal performance of bulk-fill insulation materials under actual-use cryogenic conditions is important for understanding current systems and for developing new systems. Example systems include double-wall tanks, double-wall piping, cold boxes, and cavities. Difficult to insulate cavities are prevalent on launch vehicles and cryogenic propellant loading systems in areas such as umbilicals, quick-disconnects, pipe bellows, tank flanges, instrumentation feedthroughs, and numerous other critical components. Testing must be performed to meet requirements for both engineering and economics factors. The first step is to completely characterize the insulation materials in configurations and environments as they will be used in the field. The Cryogenics Test Laboratory at Kennedy Space Center has developed a family of test cryostats and standardized test methods according to this purpose. Several bulk-fill insulation materials, including aerogels, have been tested using the latest test apparatus, Cryostat-100. These comprehensive new sets of thermal performance data are being used for engineering design, thermo-economic analysis, and as the basis for new materials development. Development of new insulation materials requires complete understanding of the contributions of the different modes of heat transfer and how they vary with vacuum level. The results have already been applied to successful demonstrations of insulation solutions for the Space Shuttle External Tank, liquid hydrogen storage tanks, and other industrial applications.

## CRYOSTAT-100 TEST APPARATUS AND METHOD

The Cryostat-100 test apparatus, shown in Figure 1, is the replacement for Cryostat-1 [1, 2, 3]. The similar Cryostat-1 was used in earlier studies of bulk-fill materials [4, 5]. The system is a liquid nitrogen boil-off (evaporation) calorimeter which provides absolute data for the apparent thermal conductivity (k-value) of materials or systems. Mass flow of nitrogen gas under steady-state, energy-rate balanced conditions is the primary measurement. This new apparatus is capable of extremely stable boil-off rates over a very wide range of heat flux.

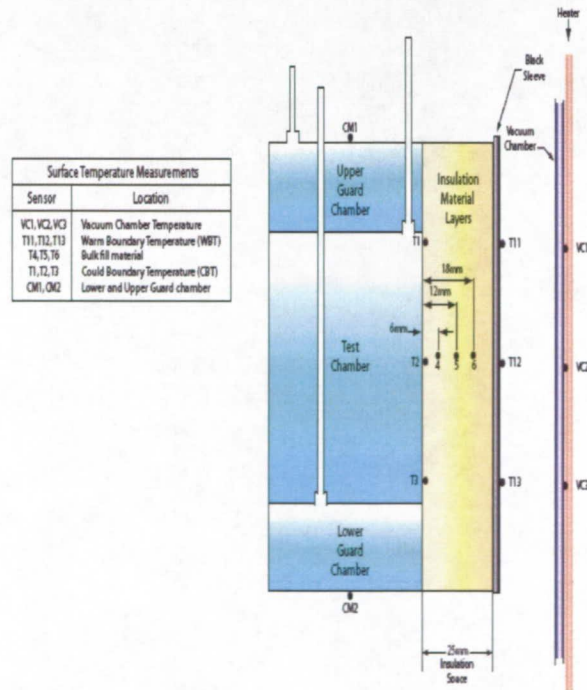
The cold mass cylindrical configuration is 167-mm by 1026-mm, including guarded ends. The standard thickness for test specimens is 25-mm. Bulk-fill materials are kept in place using a thin black sleeve. Test temperatures are as follows: cold-boundary temperature (CBT) 78 K, warm-boundary temperature (WBT) 293 K, temperature difference ( $\Delta T$ ) 216 K, and mean temperature 186K. Multiple temperature sensors are included for boundary and thickness layer temperatures as shown in Figure 2.

The test specimens are evacuated and heated to achieve a high vacuum level within the material prior to beginning a series of tests. After a suitable warm vacuum pressure (WVP) is obtained, typically below 1 millitorr, tests are conducted over the full range of cold vacuum pressures (CVP). Most engineering applications fall into one of three levels of thermal performance as designated by the following CVP: high vacuum (HV), below  $1 \times 10^{-4}$  torr; soft vacuum (SV),  $\sim 1$  torr; and no vacuum (NV), 760 torr. A typical test series consists of a minimum of eight CVP starting at HV and increasing decade by decade to NV. Nitrogen is the residual gas for all tests reported here. Liquid nitrogen is supplied to the three chambers independently. The boil-off mass flow rates from all three chambers, after system cool down and thermal stabilization is complete, are continuously measured. **The boil-off flow from the test**

chamber is averaged over the period of time when the test chamber is between 92 and 88% full of liquid nitrogen, and used to calculate the k-value.



FIGURE 1. Overall view of the Cryostat-100 test apparatus and system for determining the absolute k-value for a cryogenic thermal insulation system.



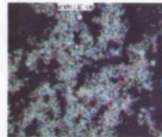
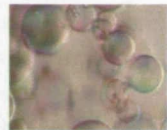
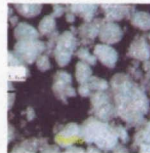
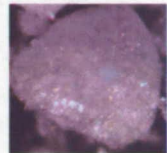
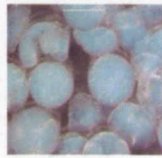

**FIGURE 2.** Schematic of Cryostat-100 showing cold mass chambers, insulation material, black sleeve, and location of temperature sensors.

## BULK-FILL INSULATION TEST MATERIALS

Three different bulk-fill insulation materials—perlite powder, glass bubbles, and aerogel beads—were tested in the Cryostat-100 test apparatus. The perlite powder was an evacuated cryogenic grade perlite processed by Silbrico Corp. under the trade name Ryolex grade no. 39. The glass bubbles material is manufactured by 3M under the name 3M Scotchlite Type K1 Glass Bubbles. The aerogel beads are commercially available from Cabot Corporation under the trade name Nanogel. All three materials appear to be a similar white powder to the naked eye. However, noticeable differences at a microscopic level are shown in Table 1. The combination of morphology, microstructure, and submicroscopic features of a given material determine how the heat energy will be transmitted through the bulk of the material at a particular vacuum level. Further details have been reported [4-6].

Each material was carefully measured and poured cup by cup into the black sleeve around the cold mass. Thickness and density details are shown in Table 2. Each material was heated above 340K and evacuated to approximately 1 millitorr or lower WVP.

**TABLE 1.** Microscope comparison of the three white bulk-fill insulations. Note: Glass Bubbles are shown at 300x.

	10x	100x
<b>Glass Bubbles</b> ~65 $\mu$ m		
<b>Perlite Powder</b> ~600 $\mu$ m		
<b>Aerogel Beads</b> ~2000 $\mu$ m		

**TABLE 2.** Installed densities for insulation test materials.

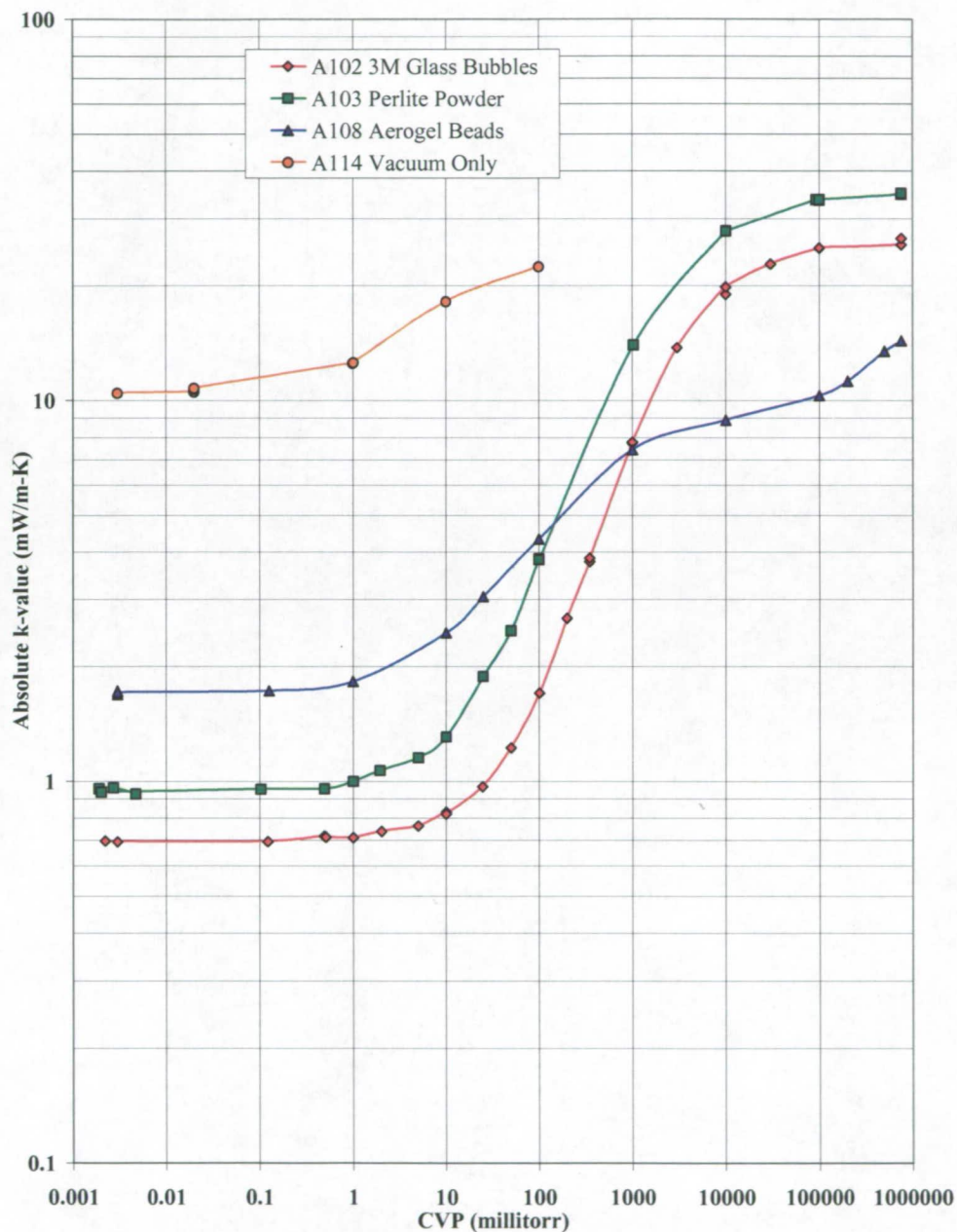
Ref. No.	Material	Thickness	Mass	Tap Volume	Tap Density
		mm	g	cm <sup>3</sup>	g/cc
A102	Glass Bubbles	25.4	885.6	11073	0.080
A103	Perlite	25.4	1875	11268	0.166
A108	Aerogel Beads	25.4	967	11268	0.086

## TEST RESULTS

A total of 83 tests of 3 material systems are reported here. An additional test series of 15 tests were run without any insulation material inside the black sleeve. The corresponding steady-state run time on Cryostat-100 was over 500 hours. Variation of absolute k-value with CVP is presented in Figure 4. The variation of heat flux with CVP is given in Figure 5. The k-values rose sharply in the soft-vacuum range as gas conduction began to dominate the heat transfer. The glass bubbles had much better thermal performance than perlite for all vacuum levels. The glass bubbles were also better than aerogel beads at high vacuum up to soft vacuum. Above 1000 microns the aerogel beads perform significantly better than glass bubbles and perlite. The

vacuum only test is included to illustrate the reduction in heat flux through the system by using an insulation material. This data provides a basis for the heat transfer analysis of the materials.

An example test (aerogel beads at 1 millitorr) is presented in Figures 6. The final  $k$ -value and heat flux are taken from the averaged test chamber boil-off flow rate when the liquid nitrogen level is between 88% to 92% full (or between about 17 and 21 hours in the example case). The standard requirement is to achieve a fine thermal equilibrium that is coincident with a liquid level of about 90%. As Figure 6 shows, one or two refills are sometimes required beyond the initial cooldown phase.



**FIGURE 4.** Variation of apparent thermal conductivity (absolute  $k$ -value) with cold vacuum pressure for bulk-fill cryogenic insulation materials. The boundary temperatures are approximately 78 K and 293 K. The residual gas is nitrogen.

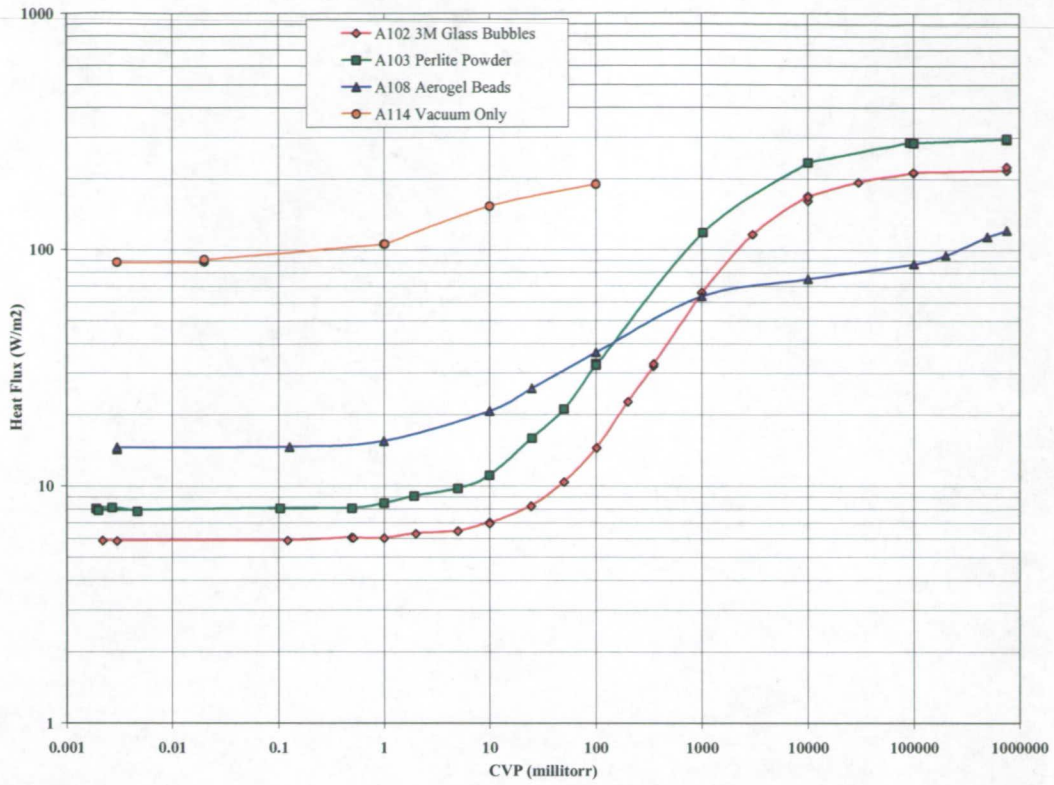


FIGURE 5. Variation of heat flux with cold vacuum pressure for bulk-fill cryogenic insulation materials. The boundary temperatures are approximately 78 K and 290 K. The residual gas is nitrogen.

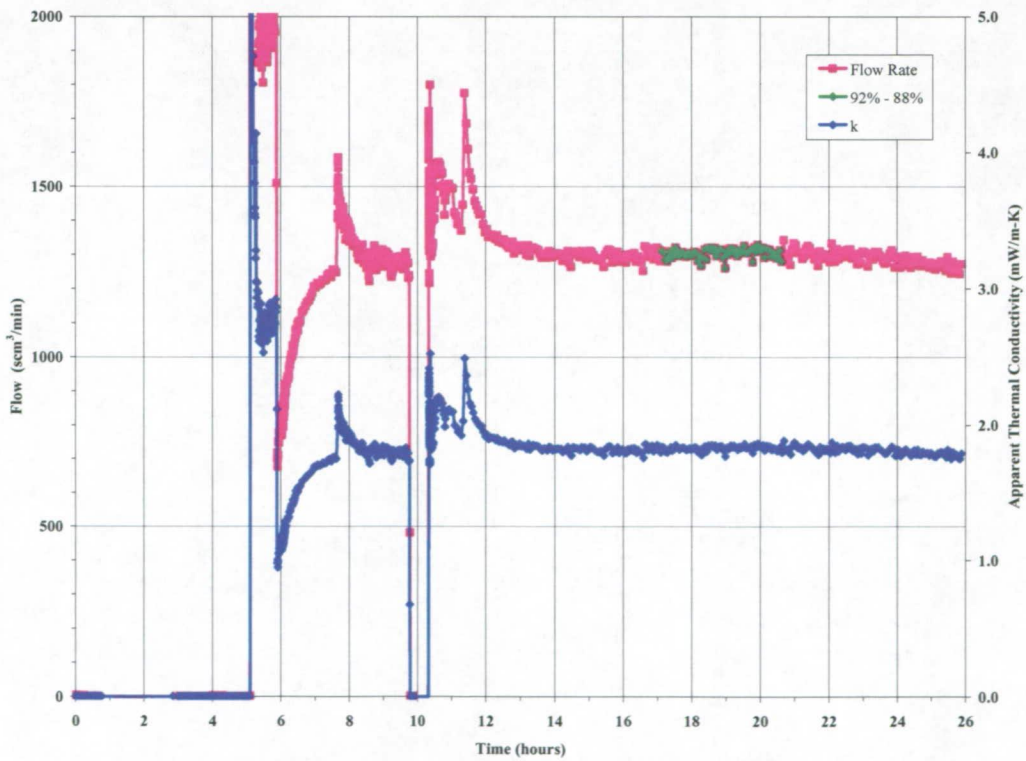


FIGURE 6. Nitrogen boil-off flow rate and the calculated k-value show stabilization in the 92-88% full range.



## ANALYSIS AND DISCUSSION

Complementary test methods are necessary for establishing a complete understanding of the heat transfer properties for a given material system and to establish credibility of the heat measurement results. This fact is illustrated in Table 3 by comparing the Cryostat-100 results for perlite and aerogel beads at high vacuum to the results of the in-house CESAT project [6], Fulk [7], and our research partners at the FSU/NHMFL [8]. Extrapolating the data to the Cryostat-100 average temperature gives a good correspondence of the results.

**TABLE 3.** Comparison of Cryostat-100 and other test methods at high vacuum. Notes: \*  $k_{\text{eff}}$  value, \*\*average

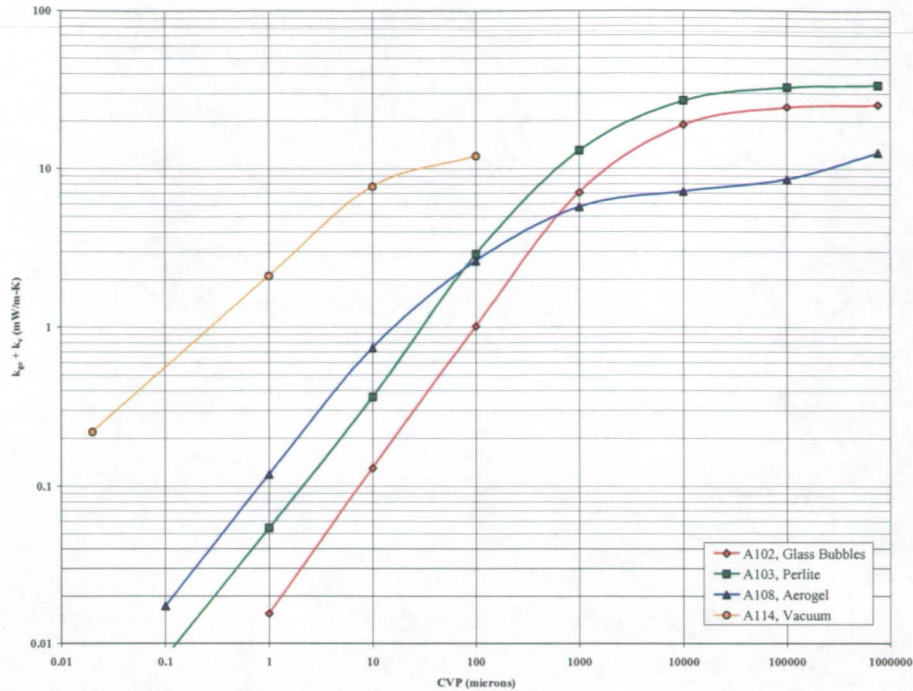
Material	Test	WBT K	CBT K	Average T K	k-value mW/m-K
Perlite	A103	293	78	186	0.94
	CESAT	296	20	158	1.03*
	Fulk	304	20	162	0.65
	Fulk	76	4	40	0.08
Aerogel	A108	293	78	186	1.71
	Fulk	304	76	190	2.10
	Fulk	76	20	48	0.20
	FSU/NHMFL			29	0.16**

The vacuum test series and the literature were used to determine the effect of the three bulk-fill insulation materials on the different modes of heat transfer. The total thermal conductivity is the sum of the thermal conductivity of each mode of heat transfer ( $k_T = k_{sc} + k_{gc} + k_c + k_r$ , where  $k_{sc}$  is the solid conduction thermal conductivity,  $k_{gc}$  is the gas conduction thermal conductivity,  $k_c$  is the convection thermal conductivity, and  $k_r$  is radiation thermal conductivity). At high vacuum, gas conduction and convection are neglected, while it is assumed that solid thermal conduction and radiation are constant at all vacuum levels. Figure 7 illustrates the thermal conductivity of gas conduction and convection for the three bulk-fill materials calculated by using the data from Cryostat-100 and extrapolating data in the literature [8, 9, 10]. Although adding a bulk-fill insulation material to a vacuum system introduces solid conduction, the materials improve thermal performance by reducing heat transfer by radiation, convection, and gas conduction.

## CONCLUSIONS

Thermal performance testing of perlite powder, glass bubbles, and aerogel beads was successfully completed at the Cryogenics Test Laboratory using Cryostat-100. However, a number of tests under different conditions are needed to completely understand and characterize insulation materials for the full vacuum pressure range. These results will be applied to a number of different projects in the area of energy-efficient cryogenics for space vehicles, space launch, and industry.

Aerogel granules by Cabot and aerogel composite beads and blankets by Aspen Aerogels are some of the newer materials currently being studied for cryogenic applications. Current experimental work also includes evacuation and vacuum retention rates, flammability and fire compatibility, and the effects of different residual gases. **Current work** also includes a focus on non-vacuum cryogenic insulation systems. A new test apparatus using industry piping equipment will provide for laboratory scale field demonstrations and allow practical insulation solutions to old problems using new materials.



**Figure 7.** Calculated gas conduction and convection thermal conductivities for each material as a function of CVP. The boundary temperatures are approximately 78 K and 293 K. The residual gas is nitrogen.

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