## THERMAL PERFORMANCE TESTING OF GLASS MICROSPHERES UNDER CRYOGENIC VACUUM CONDITIONS

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# ABSTRACT

A key element of space launch vehicles and systems is thermal insulation for cryogenic tanks and piping. Glass microspheres, or glass bubbles, represent an alternative insulation material for a number of applications. Composite materials and engineered thermal insulation systems are also being developed based on the use of glass bubbles as the main constituent material. Commonly used materials, such as spray-on foam insulation, or SOFI, for vehicle tanks and perlite powder for ground storage tanks, are targeted for replacement with the new-technology systems that use glass bubbles. Complete thermal characterization of the glass bubbles is the first step toward producing the engineering solutions required for the energy-efficient, low-maintenance cryogenic systems of the future. Thermal performance testing of the glass microsphere material was successfully completed at the Cryogenics Test Laboratory of NASA Kennedy Space Center. The test measurements were made at the full temperature difference (typical boundary temperatures of 78 kelvin [K] and 293 K) and included the full cold-vacuum pressure range. The results are reported in apparent thermal conductivity (k-value) and mean heat flux.

### **INTRODUCTION**

A key element of space launch vehicles and systems is thermal insulation for cryogenic tanks and piping. Glass microspheres, or glass bubbles, represent an alternative insulation material for a number of applications. Composite materials and engineered thermal insulation systems are also being developed based on the use of glass bubbles as the main constituent

material. Commonly used materials, such as spray-on foam insulation, or SOFI, for vehicle tanks and perlite powder for ground storage tanks, are targeted for replacement with the new-technology systems that use glass bubbles. Complete thermal characterization of the glass bubbles under actual-use cryogenic vacuum conditions is the first step toward producing the engineering solutions required for the energy-efficient, low-maintenance cryogenic systems of the future.

### EXPERIMENTAL

The steady-state liquid nitrogen boiloff (evaporation rate) calorimeter methods established by the Cryogenics Test Laboratory of NASA Kennedy Space Center were used to determine apparent thermal conductivity (k-value) of insulation material systems [1,2]. The cylindrical test apparatus Cryostat-1 was used. This apparatus, shown in FIGURE 1, includes a cold mass of overall dimensions 167-mm diameter by 900-mm length and provides absolute kvalues for a 25-mm-thick specimen. A thin aluminum sleeve, painted black on the outside, was used to contain the powder-type insulation material and support the temperature sensors located inside. A simplified schematic of the insulation test article is given in FIGURE 2.

A cryostat test series begins with specimen preparation, installation, and then a vacuum pumping and heating cycle to obtain the initial high-vacuum condition. A test is defined as the steady-state heat leak rate (watts) 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 stable vacuum level.

The liquid nitrogen cold mass maintained the CBT at approximately 78 K (-319 °F). The WBT was maintained at approximately 293 K (+68 °F) using an external heater with electronic controller. The temperature difference was therefore 215 K (387 °F), while the mean temperature was about 186 K (-125 °F). Vacuum environments, or cold vacuum pressures (CVP), included the following three basic cases: high vacuum (HV) (below  $1 \times 10^{-4}$  torr), soft vacuum (SV) (1 torr), and no vacuum (NV) (760 torr). Additional tests were performed at cold vacuum pressures from  $1 \times 10^{-4}$  torr to 760 torr. Nitrogen was the residual gas within the vacuum chamber for all tests.

### INSULATION TEST MATERIAL

The insulation test material was glass microspheres in bulk form. The material is manufactured by 3M under the name 3M Scotchlite<sup>TM</sup> Type K1 Glass Bubbles. The glass bubbles are white and are the most economical glass microsphere product available from 3M. The cost is approximately \$0.37 per liter, which is similar to the bulk cost of perlite powder. These glass bubbles are hollow and are formulated for a high strength-to-weight ratio. The target isostatic strength, or crush strength, of Type K1 glass bubbles is 1.7 megapascals (MPa) (250 pounds per square inch [psi]). The actual bulk density is 0.064 grams per cubic centimeter (g/cm<sup>3</sup>), while the density of the individual spheres is 0.125 g/cm<sup>3</sup>. The particle size distribution ranges from 30 to 110 micrometers ( $\mu$ m) with a mean particle size of 65  $\mu$ m. For test series C137, the bulk density (as installed) was measured to be 0.060 g/cm<sup>3</sup>. A photograph of the material, shown in comparison to perlite powder and aerogel beads, is given in FIGURE 3. Further information on perlite powder and aerogel beads can be found in the paper by Fesmire et al. [3].



FIGURE 1. Cryogenic insulation test apparatus for cylindrical specimens with vacuum can removed.

		Cold-Mass Assembly ● <sup>4</sup>	Insulation	Black Cylinder	Heater │  │
Surface Temperature Measurement		Upper Guard Chamber		Vacuum Chamber	
4, 5 8, 9, 10 11, 13, 15 1, 2, 3 4, 5, 6	Location Lower and Upper Guard Chambers 3M Glass Bubbles Material Black Cylinder Outer Surface Vaccuum Chamber Vacuum Chamber, Opposite Side	Test Chamber 6 mm→	Insulation Material 12 mm 12 mm 8 9 10	• 11 • 13	• 1 • 4 • 2 • 5
		Lower Guard Chamber 5	3M Glass Bubbles 25 mm	<b>●</b> 15	• 3 • 6
			Total Insulation	า	



Thickness



FIGURE 3. Different cryogenic insulation materials in the bulk-fill form.

## **RESULTS AND DISCUSSION**

The glass microspheres (glass bubbles) were tested using Cryostat-1. The summary chart of the k-value as a function of the cold vacuum pressure (CVP) is presented in FIGURE 4. Similar curves for other bulk-fill materials (perlite and aerogel beads) are also included for general reference. This k-value is the apparent thermal conductivity of the material between the boundary temperatures of approximately 78 K and 293 K. The heat leak rate in milliwatts is computed directly from the boiloff flow rate of the nitrogen gas. The k-value in milliwatts per meter-kelvin (mW/m-K) is then calculated from Fourier's law of heat conduction through a cylinder. We report k-values of 0.65 mW/m-K at 0.2 millitorr (high vacuum), 7.8 mW/m-K at 1 torr (soft vacuum), and 22.1 mW/m-K at 760 torr (no vacuum).

The test conditions were representative of the actual-use conditions for cryogenic insulation. As expected, the k-values rose sharply in the soft-vacuum range as gas conduction began to dominate the rate of 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 about 300 millitorr.

The material was also tested under ambient pressure and temperature conditions using the standard method of ASTM C 518 and the Netzsch 2300F test machine. The thermal conductivity was measured to be 34.9 mW/m-K at a mean temperature of 294 K and a pressure of 1020 millibar (765 torr).

The variation of the mean heat flux with CVP is presented in FIGURE 5. The mean heat transfer area for the cylindrical insulation test article is  $0.345 \text{ m}^2$ . We report heat flux values from 5.7 W/m<sup>2</sup> at high vacuum to 197 W/m<sup>2</sup> at no vacuum.

Cunnington and Tien studied a similar glass microsphere product from 3M in the 1970s [4]. Experimental k-values for the high-vacuum test condition of about  $1 \times 10^{-6}$  torr are given. A k-value of 0.594 mW/m-K is reported for the boundary temperatures of 303 K and 78 K. These data are therefore in excellent agreement with C137 data at the high-vacuum condition.



**FIGURE 4.** Variation of k-value with cold vacuum pressure for glass bubbles in comparison with perlite powder and aerogel beads. Boundary temperatures are approximately 293 K and 77 K. Residual gas is nitrogen.



**FIGURE 5.** Variation of mean heat flux and total heat transfer with cold vacuum pressure for glass bubbles. Boundary temperatures are approximately 293 K and 77 K. Residual gas is nitrogen.

### CONCLUSIONS

Thermal performance testing of the glass microsphere material was successfully completed at the Cryogenics Test Laboratory. Fourteen tests were performed using the cryogenic insulation test apparatus for cylindrical specimens, Cryostat-1. The test series C137 included different vacuum levels from high vacuum to no vacuum conditions. The test measurements were made at the full temperature difference (typical boundary temperatures of 78 K and 293 K) and included the full vacuum pressure range. The results are reported in k-value (apparent thermal conductivity) and mean heat flux.

This research testing showed that a number of tests under different conditions are needed to understand and characterize the glass microsphere material 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.

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