THERMAL CONDUCTIVITY OF POWDER INSULATIONS FOR CRYOGENIC STORAGE VESSELS

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ABSTRACT

The objective of the present work was to develop a precise instrument for measuring the thermal conductivity of powder insulating materials over a temperature range from 20 K to near room temperature. The instrument consists of two concentric copper cylinders with the annular space filled with the insulating material. The outer cylinder is thermally anchored to the coldhead of a single stage Gifford-McMahon cryocooler, while the inner copper cylinder is used for generating uniform heat flux through the insulating material. The temperature of both cylinders is measured at several locations to ensure uniform boundary conditions. The entire apparatus is wrapped in multi-layer insulation and suspended in a vacuum cryostat that provides an insulating environment. For a supplied heat flux, the temperature difference between the two cylinders is measured in steady state, from which the thermal conductivity of powder insulation is calculated and compared with published results.

KEYWORDS: Thermal conductivity, Powder insulations, Cryostats **PACS:** 44.10.+i

INTRODUCTION

The various types of thermal insulation, such as the multilayer insulation (MLI), powder insulation, and foam insulation, are used in the cryogenic storage vessels. Among

these insulations, evacuated powders have received considerable attention for many practical applications in cryogenic systems because of their thermal performance, low cost, light weight, durability and low maintenance [1-7]. However, the apparent thermal conductivity of these powder insulations is not always well characterized and can vary according to factors such as temperature, vacuum level or residual gas pressure, and packing density. Therefore, one of the best means to compare these different types of insulating materials is a direct measurement of the thermal conductivity as a function of the relevant sensitivity parameters.

Measurements of the thermal conductivity of powder insulations have been successfully carried out by a number of groups. Tien et al. [3] have studied the effective thermal conductivity of glass microspheres in the temperature range between 80 K and 300 K. This work concentrated on making precision measurements using a concentric sphere apparatus and understanding the results based on a combination of conduction and radiation heat transfer. Rettelbach et al. [4,5] have similarly performed measurements of the effective thermal conductivity of silica aerogel over the temperature range 10 K to 275 K. In this research, a guarded parallel plate experimental configuration was used. The effect of packing pressure and introduction of carbon black within the material were also studied in that work. Recently, Fesmire et al. [6,7] have made extensive measurements of the average thermal conductivity of powder insulations. These measurements have primarily involved determining the apparent thermal conductivity of different materials averaged between 77 K and 293 K.

To obtain data for the temperature dependence of the thermal conductivity of porous insulating materials, we have developed a new measurement instrument. This instrument is capable of measuring the thermal conductivity over the temperature range between 20 K and 300 K, allowing measurements as a function of residual gas pressure and the introduction of different powder insulating materials. A Gifford-McMahon (GM) cryocooler is employed as a heat sink for the experiment in order to meet these requirements. This thermal conductivity facility is a precision device capable of conducting similar measurements as new materials are developed. In this paper, we describe the instrument in detail and present preliminary powder insulation thermal conductivity results measured during cool-down in a liquid nitrogen bath. In addition, the effect of averaged temperature on the thermal conductivity of powder insulation is investigated.

EXPERIMENTAL APPARATUS

FIGURE 1 shows the assembly schematic of thermal conductivity of powder insulation experiment. The main components of the apparatus are two concentric oxygen-free high conductivity copper cylinders, a vacuum cryostat and a GM cryocooler. The cryocooler is mounted directly at the top plate of the cryostat and thermally anchored to the outer cylinder, which is located at the center of the cryostat. The inner cylinder for generating a uniform heat flux is supported from the outer cylinder at a uniform distance so that the powder insulations can be installed in an annular space between the two cylinders.

A single-stage GM cryocooler (Cryomech model AL63) provides the cooling to the experiment. The flexible tinned copper braids are used for a thermal connection between the coldhead and the outer cylinder as well as a protection of the coldhead from thermal contraction. A flat ThermofoilTM heater locating at the bottom of inner cylinder is sandwiched between two identical copper plates and cryogenic epoxy is applied to ensure good contact between the heater and the copper plate. The heater supplies a constant heat flux and the heating power is regulated with a DC power supply (HP 6253A 0-30V/0-3A).

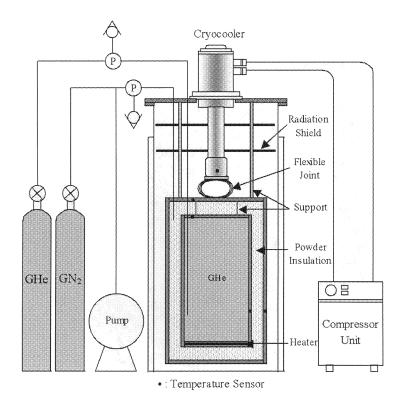
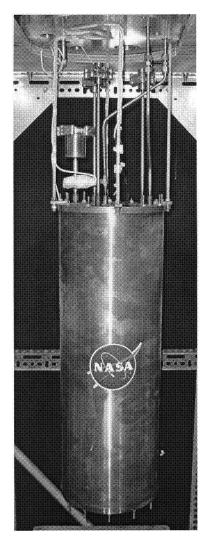
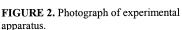


FIGURE 1. Schematic of thermal conductivity of powder insulation experiment

A photograph of the experimental set up and a detailed drawing of copper cylinders are shown in FIGURES 2 and 3, respectively. The outer cylinder is suspended at the top plate of cryostat with four gravitational supports made of threaded GFRP rod. The inner cylinder is supported from the outer cylinder by three low thermal conductivity tension members, 1.6 mm diameter GFRP rods. In the steady state experiment, all the heat generated by the inner cylinder passes through the insulation powder, the lead wires and the inner cylinder supports. In order to minimizing the heat loss through the lead wires, all leads are thermally anchored at the top of the outer cylinder. Also, as shown in FIGURE 3, the inner cylinder supports are housed in long vacuum insulated support tubes to minimize heat conduction loss.

The inner cylinder is filled with helium gas and contains two temperature sensors. The outer cylinder is also vacuum-tight so that the annular space can contain insulation material and a residual gas pressure. The outer cylinder is designed to be removable so that the insulation material may be changed. In conformance with ASTM standards, the inner surfaces of both cylinders are painted flat black to provide the recommended high-emissivity boundaries [4]. TABLE 1 shows the dimensions of the thermal conductivity experiment.





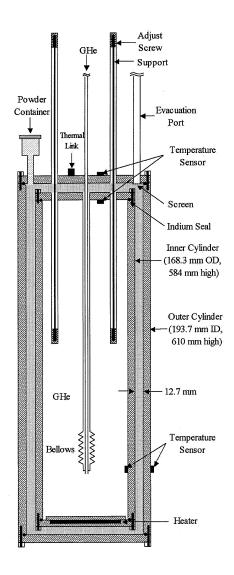


FIGURE 3. Cross section view of experiment.

The insulation test material in the present study consists of aerogel beads, which are manufactured by the CABOT Corporation. The powder is white and the size distribution ranges from 1 \sim 4 mm in diameter. The bulk density of this powder is 80 \sim 150 kg/m³, while the density of the aerogel material is 1900 \sim 2200 kg/m³. To achieve the evacuation of the powder insulation within the measurement apparatus, a vacuum port with 9.5 mm diameter is provided. A 600×600 mesh stainless steel filter cloth is installed at the entrance of this port to prevent the powder from being removed during the purge and vacuum

TABLE 1. Dimensions of thermal conductivity experiment

Outer Cylinder	Inner Diameter	193.7 mm
	Outer Diameter	219.1 mm
	Height	609.6 mm
	Weight	45 kg
Inner Cylinder	Inner Diameter	146.1 mm
	Outer Diameter	168.3 mm
	Height	558.8 mm
	Weight	30 kg
Outer Cylinder Support	Diameter	6.35 mm
	Length	305 mm
Inner Cylinder Support	Diameter	1.6 mm
	Length	609 mm
Heating Source	Heater	Thermofoil TM Heater
	Heating Power	0 ~ 1.7 W

pumping procedure. A container on the outer copper cylinder stores extra powder to make up for any depletion in the powder level due to settling during the experiment.

A gaseous helium supply tube penetrates both cylinders and carries the instrumentation wires. As with the inner cylinder support, the stand-off structure is also employed with this tube in order to minimize the conduction load through the probe. Since the probe is welded at both ends, the bellows tube is installed in the middle of the probe in order to adjust the gap distance between two cylinders.

The coldhead temperature of the GM cryocooler is measured with silicon diodes (Lakeshore DT-470-SD). The temperatures of inner and outer cylinders are measured with platinum thermometers (Lakeshore PT 111) and Cernox TM thin film resistance temperature sensors (Lakeshore CX-1070) at a number of locations as shown in FIGURE 3. These temperature sensors are embedded in the copper cylinder wall or plate and calibrated against a commercial standard. A relief valve set at 105 kPa is installed to maintain the pressure of inner copper cylinder constant.

At the initial phase of the experiment, the annular space between two copper cylinders is filled with powder and evacuated several times in order to ensure the complete settling of powder. After inserting the powder, the experimental apparatus is immersed in liquid nitrogen bath and cooled down to near the liquid nitrogen temperature. During cool-down, temperatures at each cylinder are measured, from which the effective thermal conductivity of powder is calculated using the net heat transfer rate. All temperatures in the experiment are recorded every 10 minutes with a data acquisition system operated through LabViewTM software. Variables in the experiment are the average temperature and the gas pressure in powder insulation.

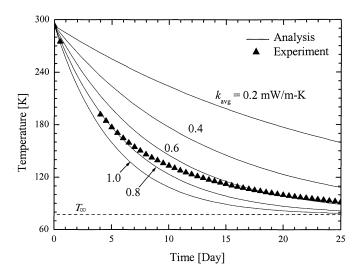


FIGURE 4. Measured temperature history of inner copper cylinder in comparison with lumped capacitance analysis.

RESULTS AND DISCUSSION

In the experiment, the temperature of inner copper cylinder is spatially uniform within \pm 0.3 K variation at any instant during the cool-down process and gradually decreases for time t > 0 until it eventually reaches $T_{\infty} = 77$ K. Based on these conditions, we assume a measurement of the thermal conductivity can be obtained using the lumped capacitance model [8]. FIGURE 4 shows the measured temperature history of the inner copper cylinder after the experimental apparatus was immersed in liquid nitrogen bath. These results are compared to analysis based on the lumped capacitance model. The specific heat of copper cylinder evaluated at the average temperature between room temperature (300 K) and liquid nitrogen (77 K) was used in this analysis. The elapsed time of the temperature from 300 K to 77 K increases as the average thermal conductivity of powder decreases. In other words, the higher thermal conductivity case has a steeper slope than the lower one.

For temperatures near 180 K, good agreement is observed between experiment and analysis for an average thermal conductivity of 0.8 mW/m-K. For longer times, the measured temperature becomes greater than the analysis based on an average thermal conductivity of 0.8 mW/m-K. This discrepancy can be explained by the temperature-dependent thermal conductivity of aerogel and specific heat of copper. As the thermal conductivity of the aerogel decreases with temperature, the slope of temperature profile would become flatter as seen in FIGURE 4. The details of the temperature-dependent thermal conductivity of aerogel will be discussed later. The specific heat of copper also decreases with temperature. The cool-down could be accelerated as temperature decreases

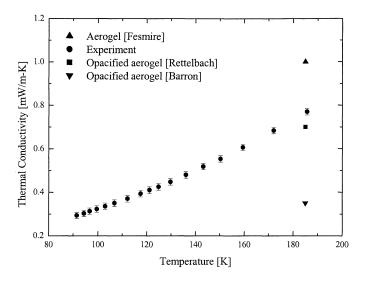


FIGURE 5. Effective thermal conductivity of aerogel

if the other conditions are same. However, the rate of decreasing temperature is getting slow because the temperature dependent thermal conductivity of aerogel is one of the parameters that determines the cool-down time. The pressure in powder annular space was approximately 5×10^{-4} millibar during experimental measurement.

During the cool-down, the temperature decreases very slowly, at approximately 5 mK/min, which makes the quasi-steady state assumption reasonable. Thus, the effective thermal conductivity of powder, k_{eff} , can be estimated from the net heat transfer rate during the cool-down.

$$Q = mC \left(-\frac{dT}{dt} \right) \approx k_{eff} A \frac{T - T_{\infty}}{L}$$
 (1)

where m, A, and L denote the mass of inner copper cylinder, the average surface area of copper cylinder, and the thickness of powder, respectively. The specific heat of copper, C, is temperature dependent and determined from CRYOCOMP[®] [9].

FIGURE 5 shows our measured values with error bars for the effective thermal conductivity of aerogel for the temperature range between 90 K and 200 K, showing an increasing value over the given temperature range from 0.3 ~ 0.8 mW/m-K. As predicted above, the effective thermal conductivity of aerogel decreases with temperature, which is the same trend as in the previous results [4,5]. Comparing to the apparent thermal conductivity of aerogel when the boundary temperatures are 293 K and 77 K [7], we note that our experimental data is about 20 % lower than previous result. Thermal conductivity of aerogel mixed with carbon black is smaller than that of aerogel [5], and it can be reduced

to about 0.35 mW/m-K by using the optimum amount of opacifier [10]. Further experimental data in steady state is required to increase our experimental confidence.

CONCLUSIONS

A new concept of measurement apparatus is successfully developed in order to measure the thermal conductivity of powder insulation material. The apparatus employing a GM cryocooler as a heat sink is capable of measuring the thermal conductivity as a function of temperature and residual gas pressure. This thermal conductivity facility is a precision measurement instrument capable of conducting similar measurements as new materials are developed. Utilizing this new apparatus, the preliminary data obtained during cooldown in a liquid nitrogen bath is measured, from which the effective thermal conductivity of powder insulation material is evaluated. The thermal conductivity of aerogel decreases with temperature, and it is in the range of 0.3 ~ 0.8 mW/m-K for temperature between 90 K and 200 K. Further experiments not only in steady state but also with various powder insulation materials are planned.

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