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Specific Heat Measurement of Insulating Material using Heat Diffusion Method

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Abstract— **The objective of the present work is to develop a precise instrument for measuring the thermal property of insulating material over a temperature range from 30 K to near room temperature by utilizing a cryocooler. The instrument consists of two thermal links, a test sample, heat sink, heat source and vacuum vessel. The cold head of the cryocooler as a heat sink is thermally anchored to the thermal link and used to bring the apparatus to a desired temperature in a vacuum chamber. An electric heater as a heat source is placed in the middle of test sample for generating uniform heat flux. The entire apparatus is covered by thermal shields and wrapped in multi-layer insulation to minimize thermal radiation in a vacuum chamber. For a supplied heat flux the temperature distribution in the insulating material is measured in steady and transient state. The thermal conductivity of insulating material is measured from temperature difference for a given heat flux. In addition, the specific heat of insulating material is obtained by solving one-dimensional heat diffusion equation.**

1. INTRODUCTION

Various types of insulating materials are used in a high temperature superconductor (HTS) power application, operating at around liquid nitrogen temperature. Among these materials, polypropylene layered paper (PPLP) has received considerable attention for many practical applications in HTS device, especially HTS power cable, because of its insulation performance and low dielectric loss [1,2]. The thermal properties of PPLP therefore are significant because heat is transferred through PPLP between conductor and liquid nitrogen in an HTS cable cryostat. However, the thermal properties of this material are not characterized and can vary according to factors such as temperature, pressure and packing force. Therefore, one of the best means to obtain these properties is a direct measurement as a function of the relevant sensitivity parameters [3].

Several papers have reported the measurements of thermal conductivity for nonmetallic materials, and liquid helium or nitrogen was most widely used as a heat sink in these studies [4-7]. Recently, the development of cryocoolers [8,9] opened new opportunities in thermal property measurement, mainly because continuous measurement was possible without any replenishment of cryogenic liquid. In addition a wide temperature range in measurement system was available using a cryocooler.

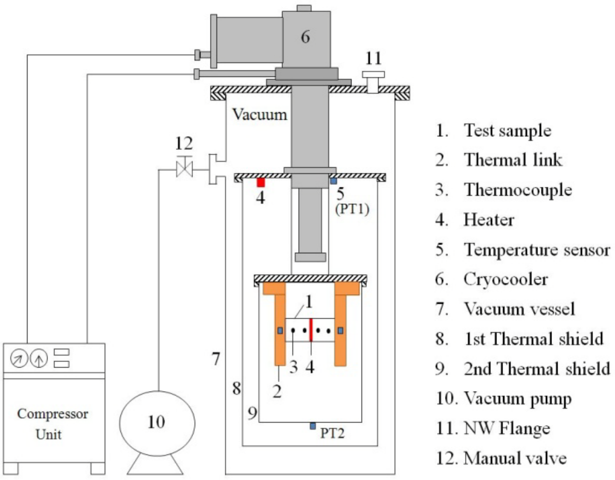

Fig. 1. Schematic overview of thermal property measurement system.

To obtain data for the temperature dependence of the thermal properties of insulating materials, we have developed a new measurement instrument. The instrument is capable of measuring the thermal property over the temperature range between 30 K and near room temperature, allowing measurements as a function of temperature using a cryocooler. A Gifford-McMahon (GM) cryocooler is employed as a heat sink for the experiment in order to meet there requirements. Moreover, the instrument is optimized to minimize the heat invasion from room temperature to cryogenic temperature section. In this paper, we describe the instrument in detail and the experimental result during initial cool-down and steady state is presented. In addition the specific heat of PPLP is reported resulting from the solution of heat diffusion equation.

2. EXPERIMENTAL SETUP

Fig. 1 shows the assembly schematic of thermal property measurement system. The main components of the apparatus are basically symmetric copper thermal link, vacuum vessel, thermal shields and GM cryocooler. A cryocooler is mounted directly at the top plate of vacuum vessel and thermally anchored to the copper link, which is

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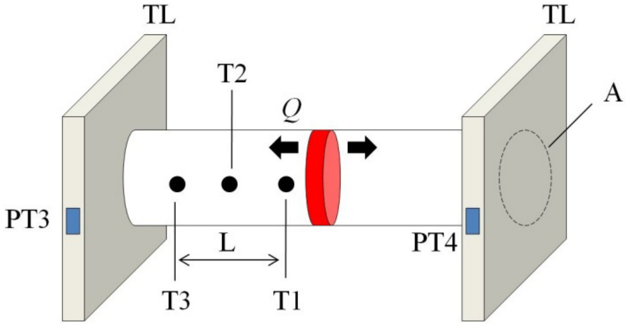


Fig. 2. Detailed drawing of test sample, heater and thermal link (T's: Thermocouples).

Fig. 3. Picture of PPLP sample used for measurement.

located at the center of vacuum vessel. Two pieces of copper thermal link are attached to the cold head of cryocooler in order to minimize temperature gradient from the heat sink. The test sample is located between two copper thermal links as shown in Fig. 1. The manganese wire heater is sandwiched between two identical copper plates and is located at the middle of test sample as a heat source, supplying a constant heat flux per unit area to the test sample. The heating power is regulated with a portable DC power supply.

The temperatures of cold head, thermal shield and copper thermal link are measured with platinum resistance thermometers by four-wire method. A number of T-type (copper-constantan) sheathed thermocouples are used to measure the absolute temperature as well as temperature distribution in the test sample. The test sample was made by stacking 10 mm diameter PPLP disks between heater plate and thermal link plate. The total length of test sample was 12.4 mm, made out of 155 layers of PPLP disk. While the first thermocouple was inserted after the 5th layer of PPLP from the heater, the second one was inserted after 61st layer and the third one was inserted after 105th layer of PPLP from the heater for measuring temperature distribution. Fig. 2 is a detailed figure of test sample, heater and thermal link, and Fig. 3 is the picture of PPLP sample used for the measurement. The specification of PPLP test sample is summarized in Table I.

TABLE I
SPECIFICATION OF PPLP SAMPLE FOR SPECIFIC HEAT MEASUREMENT.

Parameter	Value	Unit
Material	PPLP	-
Thickness (One layer)	0.08	mm
Diameter (D)	10	mm
Test length (L)	8	mm
Number of PPLP	155	layer

In most cryogenic temperature applications, heat to the sample could be occurred by conduction, convection and radiation [10,11]. Therefore, thermal isolation of the sample from its surroundings is required. Thermal anchor of instrumental wire, evacuation of cryostat and double layers of thermal shield with multi-layer insulation (MLI) are employed in this instrument, eliminating heat invasion into the test sample.

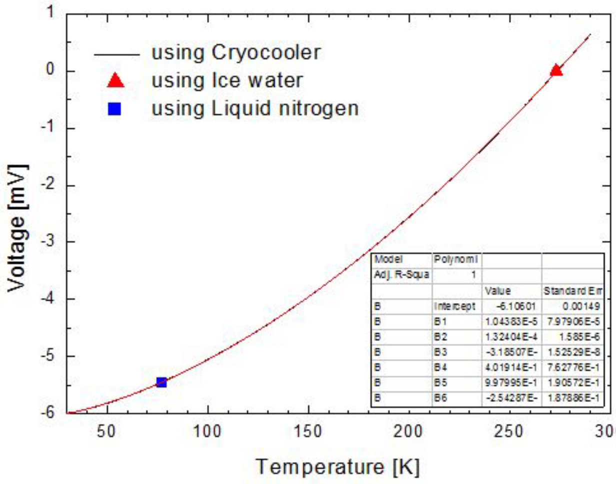
At the initial phase of the experiment, the cryostat is pumped down to the range of 2×10^{-3} Torr and then it is cooled down by a cryocooler. Once the cryostat is cooled down, a uniform heat flux is supplied so that heat flows from the source through test sample. The temperature distribution along the sample is measured during transient process, from which the specific heat is calculated from heat diffusion equation.

3. RESULTS AND DISCUSSION

The calibration of thermocouple is carried out individually against the commercially available platinum resistance thermometer [12]. Several thermocouples are inserted into the holes on the copper block thermally connected to the cold head of a cryocooler. To have good thermal contact between the thermocouple and the copper block, the hole is filled with high thermal conductivity silver epoxy before plugging the junction of thermocouple in. The representative voltage curve of thermocouple with respect to the temperature is plotted in Fig. 4, showing that the results of calibration agree well with those in ice water and liquid nitrogen.

The accuracy of this instrument is already checked using a test sample, Teflon, and reported in our previous publication [13]. Good agreement of thermal conductivity has been observed between our measurements and data from NIST. Fig. 5 shows the temperature history of cold head (PT1), thermal shield (PT2) and thermal links (PT3, PT4) after turning on the cryocooler. During the initial cool-down process, the temperature decreased almost at a constant rate, requiring approximately 4 hours for the cold head of cryocooler to reach 50 K. Once it was stabilized, electric power was then supplied to the manganese heater so that heat was conducted through the test sample. When the heater was on, the temperature of test sample increased gradually and the temperature difference between warm and cold side became measurable.

How temperature varies with position in test sample could be used to estimate the thermal diffusivity, the ratio of the thermal conductivity to the heat capacity. When the heating power is supplied to the test sample, the



Model	Polynoml		
Adj. R-Squa	1		
		Value	Standard Err
B	Intercept	-6.10601	0.00149
B	B1	1.04383E-5	7.97906E-5
B	B2	1.32404E-4	1.585E-6
B	B3	-3.18507E-	1.52529E-8
B	B4	4.01914E-1	7.62776E-1
B	B5	9.97995E-1	1.90572E-1
B	B6	-2.54287E-	1.87886E-1

Fig. 4. Representative calibration curve of thermocouple used in measurement.

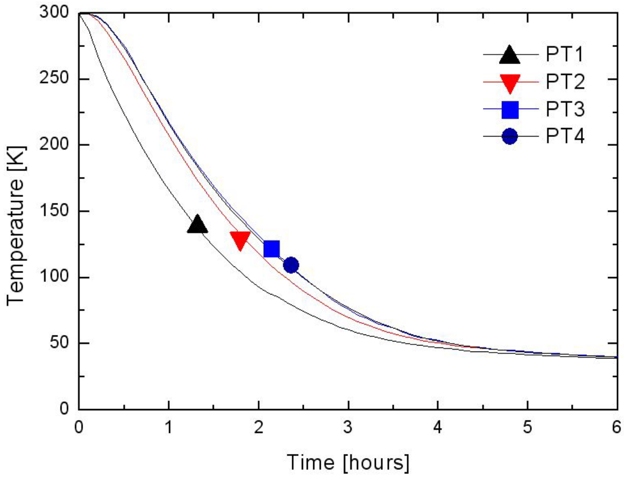


Fig. 5. Initial cool-down of specific heat measurement system using a cryocooler (Positions of PT's are indicated in Fig.1 and Fig.2).

temperature distribution along the axial direction is governed by the one dimensional heat diffusion equation if the thermal property is a constant [14].

$$\frac{\partial T(x,t)}{\partial t} = \alpha \frac{\partial^2 T(x,t)}{\partial x^2} \quad where \ \alpha = \frac{k}{\rho C_p} \tag{1}$$

where ρ and C_P are density and specific heat, respectively. In Eq. (1), T is the temperature of the sample, x is axial coordinate and t is the heating time. Since the Eq. (1) is second order in the spatial coordinate and first order in time, there must be two boundary conditions for x direction and one initial condition for time. The test sample is at a uniform temperature, T_L, before the supplying of heating power, so the initial condition is

$$T(x, 0) = T_L \tag{2}$$

While the temperature at the cold surface corresponds to the low temperature or thermal link temperature, that at the heat surface corresponds to the warm temperature or heater temperature, which is fixed with time. The boundary conditions therefore are

$$T(0, t) = T_L \tag{3}$$

$$T(L, t) = T_H \tag{4}$$

The temperature distribution along the axial direction is solved by a numerical analysis and plotted in Fig. 6 with respect to the non-dimensional distance from the thermal link as a function of time. As shown in Fig. 6, it takes time to reach thermal equilibrium and the thermal diffusivity is a dominant factor to determine the equilibrium time.

The temperatures in the test sample after supplying heating power are presented in Fig. 7 when the temperature of thermal link is 70.2 K. When the electric heater in the middle of test sample was on, the temperature at each position increased gradually and then stabilized. The temperature variations with respect to the sensor position are plotted in Fig. 8 as a function of elapsed time. As time increased the temperature distribution along the axial

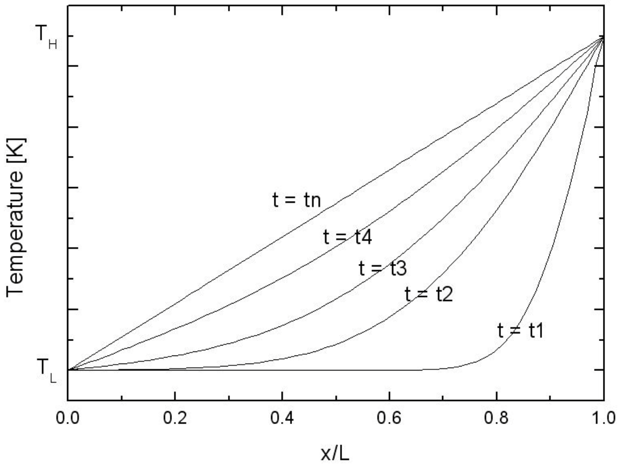


Fig. 6. Temperature distribution along the axial direction solved by numerical method.

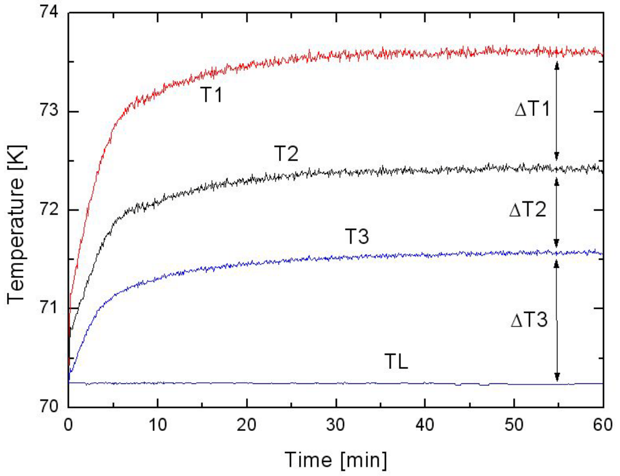


Fig. 7. Temperatures of PPLP after supplying heating power.

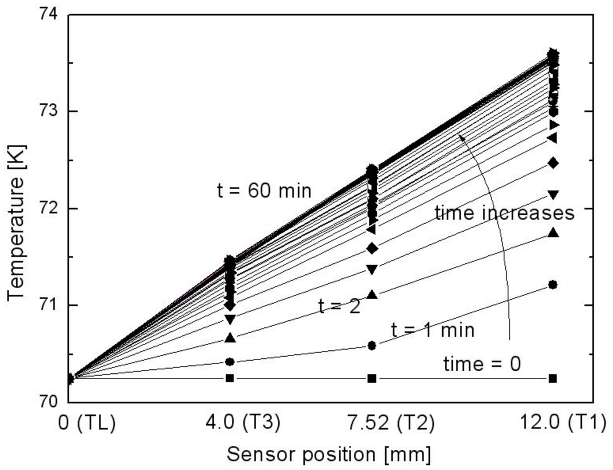


Fig. 8. Temperature variations with respect to the sensor position as a function of elapsed time.

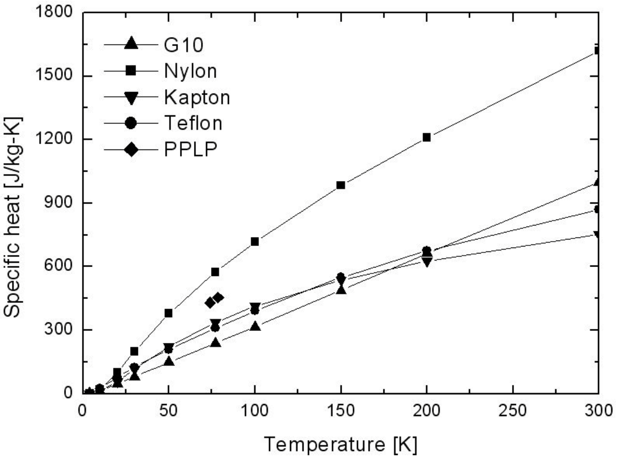


Fig. 9. Specific heat of PPLP compared with other materials.

direction became linear. In other words, the temperature distribution was linear under the steady-state conditions.

The specific heat of PPLP is obtained by fitting the solution of Eq. (1) into the measured temperature distribution along the distance from the thermal link. The time to reach steady state was determined by monitoring the temperature at each position until the time rate of change was less than 0.06 K/min. The density of PPLP was 1098 kg/m^3 averaged from our three individual measurements. In the calculation, we assume that the thermal conductivity is constant so the temperature distribution is only a function of the specific heat of PPLP. The specific heat of PPLP is plotted in Fig. 9 and compared with those of other materials [11,12]. As shown in Fig. 9, the specific heat of PPLP is 430 J/kg-K at 74 K and slightly increases with temperature.

4. CONCLUSION

A new concept of measurement apparatus is successfully developed in order to estimate the thermal properties of insulating material. The apparatus employing a cryocooler as a heat sink is capable of measuring the thermal properties as a function of mean temperature. This thermal property facility is a precision measurement instrument capable of conducting similar measurements as new materials are developed. Utilizing this new apparatus, the temperature distribution in PPLP is measured for a given heat flux, from which the specific heat is calculated by solving heat diffusion equation. The results will be used for thermal stability analysis of a cryogenic system for high temperature superconducting power applications.

ACKNOWLEDGMENT

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