An apparatus for measurements of thermal conductivity and thermal expansion based on GM cryocooler

Huiming Liu$^{1,2}$, Dong Xu$^{1,2}$, Peng Xu$^{1,2}$, Rongjin Huang$^1$, Xiangdong Xu$^1$, Laifeng Li$^1$, Linghui Gong$^1$

$^1$Key Laboratory of Cryogenics, Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, China
$^2$Graduate University of Chinese Academy of Sciences, Beijing 100049, China

E-mail: huimingliu@mail.ipc.ac.cn

Abstract. The thermophysical properties of matters are extremely important for engineering and materials science. This paper describes a multifunctional apparatus based on GM cryocooler for measurement of thermal conductivity and thermal expansion via steady-state longitudinal heat flow method and strain gauge technique respectively. The apparatus consists of a removable sample test rod on which bulk samples can easily be mounted and placed in the measurement device. Besides, the sample holder is easy to be replaced so that it suits various needs. All measurements are efficiently and accurately carried out at different temperatures by following a set of stability criteria the setup of the apparatus has been calibrated with sample stainless steel and copper, which gives an error within 6% around the published results in the literatures.

1. Introduction

Thermal properties such as thermal conductivity and thermal expansion in low temperatures are very important parameters to applications. However, reliable thermal parameters of new structural materials in low temperature are in shortage due to the lack of apparatus for efficient and accurate measurements in low temperature. Therefore, such an apparatus to measure thermal properties in low temperature is always in urge for many laboratories.

In the past, most apparatus for thermal conductivity measurement are based on liquid cryogen[1, 2]. These instruments not only require expensive cryogens, such as helium, which is quite scarce resources, but also require complex contraption for reducing consumption of liquid helium. On the other hand, many researchers began to design measurement apparatus based on cryocooler[3, 4], but, these instruments are inconvenient to alternate sample due to their design. Therefore designing new instruments without liquid cryogens becomes more and more urgent.

In this paper, we report the design and measurement specification for our novel measurement device which is based on a commercial cryocooler. It consists of a removable sample test rod on which bulk samples can easily be mounted and then placed in the measurement device. The sample holder is easy to be replaced for different needs. A unique sample holder is equipped for measurement of thermal conductivity and thermal expansion respectively. Samples of stainless steel and copper are
measured for calibration and the results agree well within a 6% error around the standard reference data.

2. Techniques
In this paper the techniques to measure thermal conductivity and thermal expansion are longitudinal heat flow method and strain gauge technique respectively.

2.1. Thermal conductivity
The steady-state method determines thermal conductivity by measuring heat flux and temperature gradient upon reaching equilibrium. There is heat flux only in the axial direction of a rod specimen. The radial direction is well isolated thermally. Then thermal conductivity can be determined by the one-dimensional Fourier-Biot heat-conduction equation\\[5\\].

$$\lambda = \frac{q \Delta x}{S \Delta T}$$  (1)

Where $\lambda$ is the average thermal conductivity at the temperature, $T_{sample}=(T_1+T_2)/2$. $\Delta T=T_1-T_2$ is the temperature difference, $q$ is the heat flux, $S$ is the cross-sectional area of the sample along the direction of heat flux, and $\Delta x$ is the spatial distance between temperature measuring points for $T_1$ and $T_2 (T_1>T_2)$. To improve the accuracy of the results, the heat loss must be minimized or calculated to reduce the uncertainty. This is done by using small diameter wires with low thermal conductance, high vacuum to reduce convection, gas conduction loss and radiation loss.

2.2. Thermal expansion
Among various techniques\\[6\\], we adopted the strain gage technique to measure thermal expansion because it is simple and efficient. The technique makes use of two well-matched strain gages, with one bonded to a sample of the reference material, and the other to a sample of the test material. The samples can be of any size or shape compatible with the available equipment for heating and cooling. Under stress-free conditions, the differential output between the gages on the two samples, at any common temperature, is equal to the differential unit expansion. When a resistance strain gage is installed on a stress-free sample of any test material under varying temperature, the output of the gage varies correspondingly. The net temperature induces apparent strain as follows\\[7-9\\]:

$$\varepsilon = \left(\frac{\beta_g}{F_g} + (\alpha_s - \alpha_g)\right) \Delta T$$  (2)

With $\varepsilon$ being the apparent strain of the strain gage, $\beta_g$ being thermal coefficient of resistivity of the grid material, $F_g$ being gage factor of the strain gage, $\alpha_s - \alpha_g$ being difference in thermal expansion coefficients between sample and grid respectively, $\Delta T$ is the temperature change from arbitrary initial reference temperature. For two same types of strain gages installed on test sample ($\alpha_s$) and reference sample ($\alpha_r$) respectively, the apparent strains of them are:

$$\varepsilon_s = \left(\frac{\beta_g}{F_g} + (\alpha_s - \alpha_g)\right) \Delta T$$  (3a)

$$\varepsilon_r = \left(\frac{\beta_g}{F_g} + (\alpha_r - \alpha_g)\right) \Delta T$$  (3b)

Subtracting Equation (3b) from (3a), and rearranging,

$$\alpha_s = \left(\varepsilon_s - \varepsilon_r\right)/\Delta T + \alpha_r$$  (4)

$\alpha_r$ is a well-known thermal expansion of reference material, $\varepsilon_s - \varepsilon_r$ and $\Delta T$ can be accurately measured by instruments, so the thermal expansion of test sample can be obtained.

Using the strain gage technique, Reference sample with well known thermal expansion is needed. In the paper fused-silica is used for two reasons. For one thing, thermal expansion data of Copper, tungsten, and fused-silica is well referenced in NBS office of Standard Reference Materials\\[10-12\\].
for another, it is often advantageous to select a material with expansion properties close to zero as much as possible[7].

3. Description of experimental setup

3.1. The cryostat and the removable system

The schematic view of the cryostat setup is shown in figure 1. The configuration of the measurement system has been discussed in detail[13]. Comparing with traditional devices based on cryocooler, our apparatus has two main advantages: rapid sample replacement while the cryocooler is operating and Multifunctional properties measurements (e.g., thermal conductivity, thermal expansion, etc.). These advantages are made possible by using the cryocooler to cool a vertical test tube which is parallel with the cold head of the cryocooler. The sample, mounted in a long test rod, is inserted into the test tube for cooling. The heat transfers from the thermal couple ring of the test tube to the test rod, finally to the sample. Samples are exchanged by simply removing the sample test rod, switching samples on the rod, and reinserting the rod into the cryostat. The entire sequence takes only a few minutes and is performed while the cryocooler is operating. Thus the time to replace samples is greatly reduced as compared with traditional ones. The second advantage is the feature that the sample holder in the test rod is removable. So many thermal properties can be obtained by different sample holders.

![Figure 1. Scheme of removable apparatus for rapid measurement system.](image)

3.2. The removable sample holders

As mentioned above, the sample holder is removable, so different sample holders are designed for different measurements of different properties. Here the sample holders for thermal conductivity and thermal expansion are described.

The sample holder for thermal conductivity measurement is shown in figure 2. The sample is adhered to the sample holder by the silver glue. The heat flux is generated by heater 1 of a foil resistance, which is plastered to the sample with a very thin layer of the silver glue. Two silicon diode thermometers which are calibrated with an accuracy of ±12mK in 4.2K and ±32mK in 300K from Lakeshore Cryotronics Inc. are used to measure the temperature of the sample at two ends (\(T_H\) and \(T_L\)). The two thermometers are attached to the top of the sample and the sample holder by silver glue respectively. To minimize the radiation heat loss a copper adiabatic shield is placed surrounding the...
sample. Thermal anchor is set to reduce the conduction loss of wires[14]. More detailed configuration is discussed in[13].

Aside from the simplicity and efficiency of measuring thermal expansion by strain gage technique, it has another extraordinary advantage, requiring no specialized instruments. As shown in figure 3, the setup of the thermal expansion is very simple. Test samples and reference sample are placed on the sample holder stress-free. Same type of strain gages are bonded on them respectively. A silicon diode thermometer is plastered to reference sample to measure temperature of samples. Anther thermometer and foil resistance heater are attached to the bottom of the sample holder. They are designed to maintain constant temperature of the sample holder.

![Figure 2. Assembly drawing of thermal conductivity measurement.](image1)

![Figure 3. Assembly drawing of thermal expansion measurement.](image2)

4. Experimental procedure

Once the sample is mounted on the sample holder with all wires installed properly, the sample chamber is mounted to the flange base sealed by indium wire. Then, the test rod can be pumped to high vacuum. If there are several samples, the corresponding test rod can be assembled and pumped in advance. Subsequently, the test rod is plugged into the test tube coupling with quick connector. And the measurement starts. When the measurement is finished, one simply removes the test rod, switches samples on the rod, and reinserts the rod into the test tube.

The thermal conductivity measurement procedure was been discussed in detail[13]. The automated program first roughly control the temperature of the 2nd stage cold head by a temperature controller, then control the temperature of the sample holder within ±40 mK. Once the temperature is stable, the initial temperature difference ($\Delta T_i$) between ends of the sample is recorded, and then a small power is independently input into the heater 1 by a SourceMeter. The final temperature difference ($\Delta T_f$) is recorded again when the heat flow of the sample is uniform and stable. So the actual temperature difference caused by the heat is as follows: $\Delta T = T_f - T_i$. The temperature difference is limited in 1.00 to 2.00K.

To measure thermal expansion, two same type foil resistance strain gages from the same manufacturing lot are adhered to the samples. A commercial strain indicator system is used that has an estimated accuracy of ±1 microstrain. The procedure used in the thermal expansion measurement is to heat the samples continuously. The heating rate is slow enough, allowing thermal equilibrium to occur during the entire measurement. In addition, a room-temperature point (293K) is taken to obtain the zero of the digital strain indicator before the samples are cooling. When the measurement starts, the automated program controls the temperatures of the cold head and the samples to rise simultaneously at the rate of 0.4K/min. And the thermal output of the strain gages and the temperature are recorded.
5. Results and discussion

The main sources of the uncertainties in thermal conductivity measurement are associated with the measuring of the heat flux $q$, geometrical factor $g=S/\Delta x$ (sample cross-sectional area $S$ and effective length $\Delta x$), warm end temperature $T_1$ and cold end temperature $T_2$. The temperature difference $(T_1-T_2)$ during the test will vary when the heat flux is increased to elevate temperature. The temperature difference is limited in 1.00K to 2.00K in the measurement, therefore, the temperature uncertainty amounts to approximately less than $\pm 1.6\%$. In addition to the above factors, heat loss must be taken into account in the measurement. The total heat loss due to heat conduction through the wires of thermometers and heaters, convection by the residual gas and radiation heat exchange were estimated. It is estimated that the uncertainty for heat flux $q$ comes mainly from the uncertainty of the temperature measurement for estimating heat loss, which amounts to about $\pm 6.2\%$ in the whole measurement. The experimental uncertainties of thermal conductivity measurement are summarized in Table 1.

| Parameter measured       | Uncertainty |
|--------------------------|-------------|
| Heating current, I(A)    | Negligible  |
| Heating voltage, U(V)    | Negligible  |
| Sample diameter, D(mm)   | $\pm 0.02$  |
| Effective length, L(mm)  | $\pm 0.02$  |
| Temperature, T(mK)       | $\pm 12$ (<10K) |
|                          | $\pm 22$ (<77K) |
|                          | $\pm 32$ (<300K) |

| Parameter derived        | Uncertainty |
|--------------------------|-------------|
| Heat flux, $q$ (%)        | $\leq 6.2\%$ |
| Geometrical factor, $g=S/\Delta x$ (%) | $\leq 0.6\%$ |

To benchmark the accuracy of the thermal conductivity measurement, a standard AISI stainless steel 304 sample is measured between 8K and 300 K. Those values from National Institute of Standards and Technology (NIST)[15] and Touloukian[5] are compared with our experimental results. Figure 4 shows the comparison of thermal conductivity of experimental results and that of published results of standard stainless steel 304 in the temperature from 8K to 300 K. After eliminating corrections from radiation heat loss and conduction along the wires, we conclude that our data is accurate to an error within 6% around NIST data, and within 4% around Touloukian’s data.

![Figure 4. Comparison experimental and published values of thermal conductivity of stainless steel 304](image_url)
The accuracy of the thermal expansion measurement depends on the accuracy of the strain gages and the strain indicator, and the procedure of the gages installation. The thermal expansions of AISI stainless steel 304 and copper are measured between 20K and 300K on this apparatus. Because the lowest temperature of samples is 7K, beyond the accurate measuring capability (20K-300K) of the strain gages, we demonstrate the measurement only in the temperature range between 20K and 300K. The Comparisons between experimental and published values of thermal expansion of stainless steel 304[15] and copper[10] are shown in figure 5 and 6 respectively. The result of thermal expansion of stainless steel matched very well with reference data. The maximum error for instantaneous coefficients \( \alpha \) is \( 0.45 \times 10^{-6} \text{K}^{-1} \) (figure 5). The comparison results of copper deviates more than those of the stainless steel, but the temperature dependency is identical, and the maximum error for instantaneous coefficients \( \alpha \) is \( 1.1 \times 10^{-6} \text{K}^{-1} \) (figure 6). The unit thermal expansions (dL/L) of stainless steel and copper are all matched very well with the reference data. Experiments are repeated from 20K to 300K to verify the reproducibility of the results and found to be within ±2% variation, which is acceptable.

![Figure 5. Comparison experimental and published values of thermal expansion of stainless steel 304](image1)

![Figure 6. Comparison experimental and published values of thermal expansion of copper.](image2)
6. Conclusion
In this paper, we designed a novel measurement system. Samples are exchanged very quickly and simply by using a dismountable mounting rod. The entire sequence takes only a few minutes to change samples and is performed while the cryocooler is operating. Using different sample holders, different thermal properties can be measured. In this paper, thermal conductivity and thermal expansion have been measured. The techniques used are longitudinal heat flow method and strain gauge technique respectively, the results are convincible. Meanwhile this system can be applied on other thermal property measurements, such as specific heat and electric conductivity.

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