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Measurement of thermal conductivity of materials down to 4.5 K for development of cryosorption pumps

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Abstract. Cryosorption pumps belong to the class of entrapment or capture vacuum pumps and they retain the gas molecules by sorption and / or by condensation on its internal surfaces. An important aspect in their development is the proper adhesion of the activated carbon granules onto the metallic panel and their cooling to the lowest possible temperature by using high thermal conductivity adhesives for adhering the activated carbons. Hence, the thermal conductivity data of the select adhesives and activated carbons down to 4.5 K are quite essential, but they are not available in open literature. Towards this, an experimental setup has been developed to measure the thermal conductivities of samples with high or low thermal conductivities from 300 K to 4.5 K, with liquid helium using a Janis SuperVariTemp cryostat. This paper presents the details of the experimental setup and the results of our studies on (i) standard samples and (ii) epoxy based adhesives samples. The above studies will enable to make the right choice of adhesives for the development of cryosorption pumps.

1. Introduction

Thermal conductivity is one of the fundamental thermal properties of any material. This crucial inherent property indicates the rate at which heat energy can propagate through a material at a particular temperature.

In any field of science and technology where the measurement of heat transfer plays an important part, the precise values of thermal conductivity of associated materials must be known beforehand. Thermal conductivity of materials play significant role for design of various components and systems. Especially in low temperature technology and space science, measurement of thermal conductivity holds the utmost importance. Measurement of thermal conductivity is an important experimental method of modern thermo physics which provide the necessary reference data for engineering applications. Thermal conductivity of any material depends on temperature, crystal structure, density of states, Fermi energy, impurities etc. Furthermore, with the advances in the technology now days new compound materials are being developed for low temperature applications and the thermal conductivity values of these materials are still unknown. Therefore measurement of thermal conductivity of those materials fills the blank of low temperature thermal property data base. Epoxy is
one such a material whose low temperature thermal properties are unknown. It is an essential component in the design of high efficiency cryosorption pumps, the only possible vacuum pump used in fusion reactors. The epoxy used for the cryosorption pump not will adhere the activated carbons on the SS panel where liquid helium flows through the panel but also transfer the cold of the liquid helium to the activated carbons for high rate of adsorption of the helium and neutrons liberated during the fusion process. Therefore the epoxy should have high thermal conductivity such that it transfers maximum cold from liquid helium to activated carbons via SS panel. The thermal conductivity of the commercial available adhesives like Styecast 2850 FT and G10 for use with liquid helium temperature are very low [1-2]. A special adhesive has been developed to use with the cryopanels of the cryosorption pumps, whose thermal conductivity is very high in comparison with the commercially available adhesives used at liquid helium temperature.

The photographic view of the stainless steel liquid helium panel and the stainless steel panel coated with activated carbon by epoxy adhesive is shown in figure 1 and figure 2 respectively.

![Figure 1. Stainless steel cryopanel.](image1)

![Figure 2. Stainless steel panel coated with activated carbon by epoxy adhesive.](image2)

This paper presents the development of an experimental system to measure the thermal conductivity of various materials down to 4.5 K and measurement of thermal conductivity of some specific materials like epoxy which will be useful towards the development of cryosorption pumps.

2. Experimental principle

The thermal conductivity of solid at low temperatures can be measured either by steady state or transient method depending on whether the sample has a high or low thermal conductivity. We have used the steady state method to measure the thermal conductivity both for aluminium 2024 T4 and the developed epoxy adhesive [3]. The thermal conductivity was measured by longitudinal steady heat flow method on the basis of one dimensional Fourier heat conduction law in this measurement.

The sample in the sample chamber is kept under high vacuum to minimize the conduction and radiation shields to reduce the radiation heat transfer. A heating power Q is supplied to one end of the sample to establish a temperature difference $\Delta T$ along the axial direction. When steady state is reached, the thermal conductivity of the sample is measured by Fourier law of heat conduction,

$$Q = - k A \left( \frac{\Delta T}{\Delta x} \right)$$

where,

$Q$ = heating power, Watt

$k$ = thermal conductivity, Watt per meter Kelvin

$\Delta T$ = temperature gradient, Kelvin

$\Delta x$ = effective length of sample across which temperature is measured, Meter

$A$ = cross sectional area of sample, square meter
3. Experimental set up and procedure

The experimental set up developed for the thermal conductivity of materials down to 4.5 K consists of the Janis liquid helium cryostat (Model: SVT-200T-5), the turbo pumps (Model: Varian-V301-AG) along with necessary instrumentation for data acquisition. The schematic and photographic view of the experimental set up is shown in figure 3 and figure 4 respectively.

![Schematic of the experimental set up.](image)

The sample along with the sample chamber (made of OFHC copper) is kept in the sample mount of the cryostat. The schematic and the photographic view of the sample chamber are shown in figure 5 and figure 6 respectively. Liquid nitrogen is transferred to the liquid nitrogen reservoir of the cryostat after evacuating the vacuum chamber of the cryostat to about $1.3 \times 10^{-5}$ mbar and kept for 30 minutes to cool down the helium chamber to liquid nitrogen temperature. Then liquid helium from the storage dewar is transferred to the helium chamber of the cryostat. The level of the liquid helium in the cryostat is measured by liquid helium level meter (Model: AMI-110A). The sample chamber is evacuated to around $9.1 \times 10^{-6}$ mbar. Liquid helium from liquid helium reservoir of the cryostat is transferred to outside of the sample chamber through the capillary tube and the vaporizer. The temperature of the sample / sample chamber is controlled by the rate of flow of liquid helium along with the heater mounted on the top of the sample chamber and controlled by Lakeshore 332 temperature controller. Depending on the desired temperature of the sample chamber the rate of liquid
helium flow is controlled accordingly. After maintaining the desired temperature of the sample / sample chamber, a known

![Figure 4. Photographic view of the experimental set up.](image1)

![Figure 5. Schematic of sample chamber.](image2)

temperature gradient along the axial length of the sample is monitored through the temperature sensors (Model: Si410B) mounted on the sample. When steady state is reached, the temperature gradient between the two sensors along the length of the sample is measured. The necessary electrical connections for the heaters and sensors are connected through the feed through. The photographic view of the total assembly of the sample chamber and the feed through is shown in figure 7. The data acquisition is done using LabVIEW 2011 software.

![Figure 6. Photographic view of sample chamber.](image3)

![Figure 7. Photographic view of sample chamber along with the feed through.](image4)

4. Results and discussion

The experimental set up has been standardized by measuring the thermal conductivity of Aluminium 2024 T4 sample in the range of 300 K to 4.5 K. The obtained data have been compared with the published data [4,5] as shown in figure 8. The results are in very good agreement with the published data with accuracy of almost 90%.

Studies have been conducted to measure the thermal conductivity of the developed epoxy from room temperature to 4.2 K. The results are given in table 1 and graphically represented in figure 9.
The thermal conductivity data of the developed epoxy is compared with commercial available epoxies like Stycast 2850 FT and G10 Cryocomp. The comparison is shown in figure 10. It can be seen that the thermal conductivity of the developed epoxy is quite high especially in the temperature range of 4.2 K to 7 K as compared to Stycast 2850 FT and G10 cryocomp. The thermal conductivity data of the above mentioned temperature range is of primary interest to the cryopanels of the cryosorption pumps.

![Figure 8. Comparison of thermal conductivity of Al 2024 T4 with published data [4,5].](image)

**Table 1.** Thermal conductivity of the developed epoxy.

| Temperature (K) | Thermal conductivity (W/m-K) |
|-----------------|-----------------------------|
| 4.2             | 0.382                       |
| 4.5             | 0.388                       |
| 7               | 0.403                       |
| 20              | 0.432                       |
| 40              | 0.541                       |
| 60              | 0.767                       |
| 80              | 0.898                       |
| 100             | 1.002                       |
| 150             | 1.004                       |
| 200             | 1.021                       |
| 250             | 1.040                       |
| 293             | 1.052                       |
Figure 9. Thermal conductivity of epoxy.

Figure 10. Comparison of thermal conductivity of epoxy with Stycast 2850 FT and G10.
5. Conclusion
An experimental set up has been developed to measure the thermal conductivity of materials in the range of 300 K to 4.5 K based on Janis SuperVariTemp. liquid helium cryostat. The set up has been standardized with the known sample namely Aluminium 2024 T4 and the results are in good agreement with the published data. The thermal conductivity of the developed epoxy for use with the cryopanels of the cryosorption pumps have been measured down to 4.2 K. The thermal conductivity of the developed epoxy is much higher in magnitude in comparison with the commercial epoxy adhesives like Stycast 2850 FT and G10 Cryocomp in the temperature range of 4.2 K to 7 K, the operating temperature range of the cryosorption pumps. The measured data of the developed epoxy will be very useful for the development of the cryosorption pumps.

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