AN APPARATUS FOR THE SIMULTANEOUS MEASUREMENTS OF THERMAL CONDUCTIVITY, THERMAL EXPANSION AND THERMAL DIFFUSIVITY OF FRPs USING GM REFRIGERATOR

S.Kanagaraj and S.Pattanayak

Cryogenic Engineering Centre,
Indian Institute of Technology,
Kharagpur, West Bengal, 721 302, India

ABSTRACT

The applications of fibre reinforced plastic (FRP) materials in cryogenic engineering have stimulated keen interest in the investigation of its properties. The reliable design data generated by a precisely controlled setup at identical environment of its applications are extremely important. This paper describes an apparatus based on a GM refrigerator for the simultaneous measurements of thermal conductivity, thermal expansion and thermal diffusivity using a double-specimen guarded-hotplate, 3-terminal capacitance technique and Angstrom method respectively in the temperature range from 30 K to 300 K. An integrated and perfectly insulated sample holder is designed and fabricated in such a way that the simultaneous measurements of the above properties are conveniently and accurately carried out at different temperatures. A set of stability criteria has been followed during the measurements to ensure the accuracy of the experimental data. The setup is calibrated with stainless steel and copper and the experimental results are within 10 % of the published results given in the literatures.

INTRODUCTION

The selection of materials for low temperature application depends on its properties compatible to the applications. Such properties can be imparted into the FRPs using suitable fibres. However, newly emerging materials are required to be studied properly. The accurate measurement of thermo-physical data may reveal many hidden truths. In this paper, an effort has been made to measure the three properties, i.e. thermal diffusivity, thermal conductivity and thermal expansion of FRPs from 30 to 300 K simultaneously.

An experimental setup for the simultaneous measurements with different combinations of thermal conductivity, thermal diffusivity, thermal expansion and specific heat of materials using several techniques have been reported [1-12] at different range of

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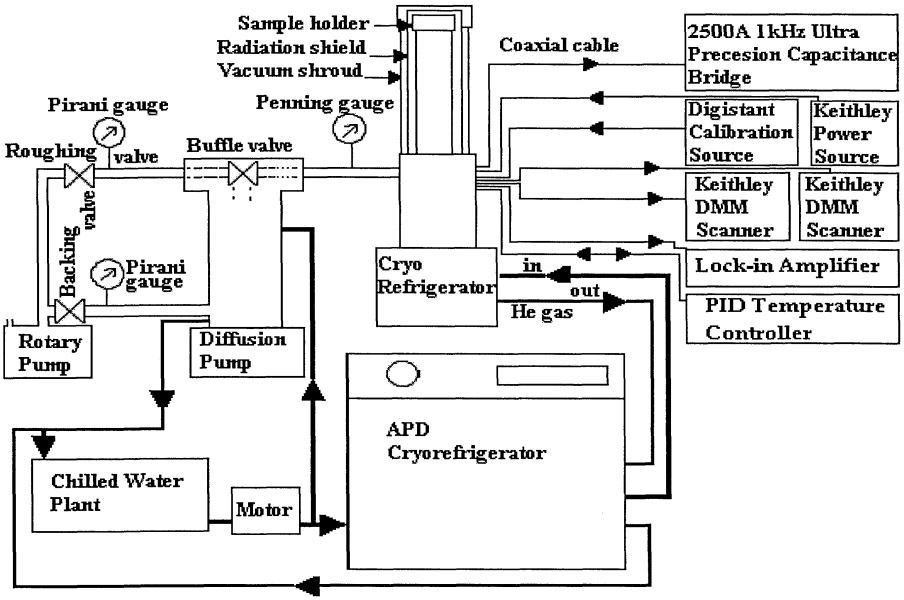

FIGURE 1 Schematic diagram of the experimental setup for the simultaneous measurement of thermal conductivity, thermal expansion and thermal diffusivity

temperatures under non-steady and steady state conditions. However, simultaneous measurements of all these properties together are not reported yet. Thus, an attempt has been made to develop such a setup to study the temperature dependence on thermal properties of fibre-reinforced plastics down to 30 K.

DESCRIPTION OF EXPERIMENTAL SETUP

The schematic views of the experimental setup and the integrated sample holder are shown in FIGURE 1 and 2 respectively. The main heater of 1800 ohm is sandwiched by two geometrically similar samples with the help of top and bottom hot plates, which are guarded by another heater of 3200 ohms. Heaters are made of canthalum wire and connected by thin 32-gauge phosphor-bronze wire. The temperature difference between these heaters is measured by a copper-constantan differential thermocouple. The surface temperature of the samples is measured by the platinum resistance thermometers, having 100 Ω at 273 K (PRT-100). Apiezon-N grease is applied at all the contacting surfaces to reduce the contact resistance. The heat leak by the connecting leads are minimized by using a thin 32-gauge copper wire of 40 cm length, and is thermally anchored at the cold head and a free length of 25 cm from the vacuum adopter, as suggested in [13]. The effect on dissimilar metal contacts in the connecting leads is also minimized by the thermal anchoring.

The capacitor plates for the measurement of thermal expansion are kept considerably away from the heater assembly of the thermal conductivity cell. The sample is gently fitted into the clamping head of the thermal conductivity cell. The high-terminal electrode is kept in close contact with sample without any air gap. The guard electrode is threaded down to

the sample holder till it shows electrical contact with high terminal electrode. The inner surface area of the guard electrode is coated with electrically insulating varnish. A teflon cap, which has a central hole for low terminal electrode with 0.1 mm radial gap between them, is threaded down for 2 mm into the sample holder. The low terminal electrode is tightened with the brass nut, which is kept on the teflon cap. The vertical movement of the low terminal electrode is adjusted by screwing the nut and kept just above the high terminal electrode. All electrodes, made of brass with high surface finish, are kept parallel to each other. It is also checked by passing light rays and observing the images. The sample temperature is measured using a copper-constantan thermocouple with reference to heat sink temperature. The physical dimensions of the cell, electrodes and the sample are measured by a precision micrometer with an accuracy of 0.01 mm.

The temperature wave in thermal diffusivity measurements is generated by a heater made of canthalum wire of 1800 Ω, i.e. the main heater of the thermal conductivity cell. The current is fed to the heater from a programmable voltage/current source by the pulsating voltage having square wave of peak voltage 8 V and current 35 mA. This is fed at an interval of 10 to 15 milliseconds, in case of stainless steel. The surface temperatures of the sample are monitored by PRT-100 sensors. The phase difference of the temperature wave at both the surfaces of the sample is measured with respect to a reference signal. All the electrical connections are made using coaxial cable, which is properly shielded and grounded.

The experiments were carried out in the temperature region from 30-300 K. Heat leak of this setup have been reduced by using 16 layers of mylar insulation [14] followed by stainless steel radiation shield. The conducting leads are long, thin and thermally anchored and kept at a vacuum of 10^{-7} kPa.

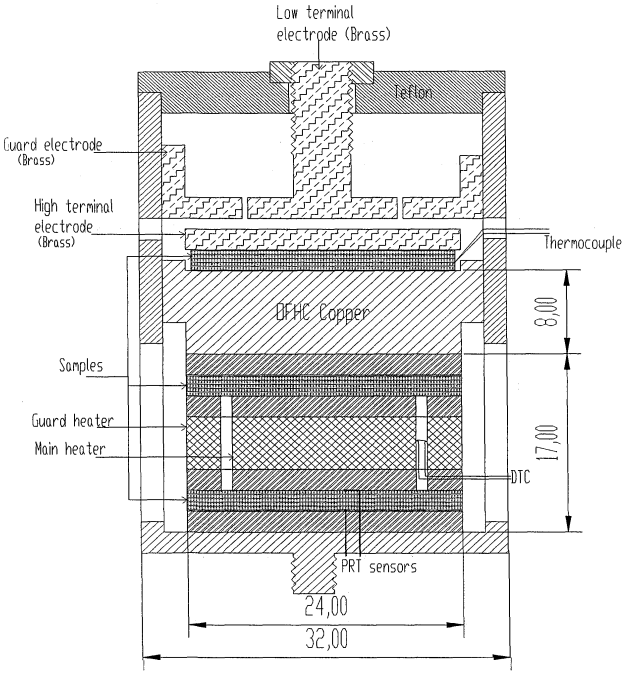

FIGURE 2 Cross-sectional view of the integrated sample holder with samples.

TECHNIQUES

The selection of a particular technique for the measurement of any thermal properties depends on the physical nature of the materials, temperature range, geometry of the sample, desired accuracy of the results and the experimental convenience. The techniques used for the simultaneous measurement of thermal conductivity and thermal expansion are guarded-hotplate method and three-terminal capacitance method respectively. Techniques for these measurements have been discussed in detail [12].

An attempt has been made to study the thermal diffusivity of the composite materials adopting Angstrom' method with this setup in the temperature ranges from 30-300 K. The basic principle of this method is that if one end of the sample is heated periodically, then the propagation of temperature wave along the sample also varies with the same period but with diminishing amplitude. Moreover, as the temperature wave travels along the sample with finite velocity, there is a relationship with varying phase. Thermal diffusivity is determined by measuring the phase difference across the sample surfaces, period of the wave and thickness of the sample [1]. The experimental setup for the individual measurement of thermal diffusivity using this technique is discussed elsewhere [15].

EXECUTION

In the thermal conductivity measurement, uniform heat flux from the main heater is applied normal to the samples. The temperature difference of 1.00 to 2.00 K across the sample thickness is maintained. To improve the accuracy of the results, the sample surface temperatures are kept nearer to the desired temperature. The power input to the guard heater is adjusted in such a way that the temperature difference between the main heater and guard heater is kept less than 0.05 K. The thermal conductivity is thus determined using one dimensional Fourier' heat conduction equation:

$$k_t = \frac{Q\,dx}{A(\Delta T)} = \frac{V\,I\,dx}{2\,A(\Delta T)} \tag{1}$$

where k_t, V, I, A and ΔT are the thermal conductivity (W/m-K) of the sample, applied voltage (V), current (A) passing through the heater wire, effective area (m^2) of the sample and temperature difference (K) across the sample thickness respectively. In the thermal expansion measurement, change of capacitance is measured at two different temperatures and thus sample expansion is determined with reference to 300 K. The inner surface area of the guard electrode is coated with electrically insulating varnish to avoid electrical contact with low terminal electrode. The Teflon cap, which holds the low terminal electrode, is used to avoid the electrical contact between sample holder and low terminal electrode. The change of sample thickness is given by:

$$\Delta l = d_2 - d_1 = \frac{-\varepsilon_0 \pi r^2 (C_2 - C_1)}{C_1 C_2} \tag{2}$$

where d_2, d_1, C_2, C_1 are the distance between the electrodes (m) and corresponding capacitance (F) at temperature T_2 and T_1 (K) respectively; ε_0 and r are permittivity of vacuum (8.854 pF/m) and effective radius (m) of the capacitor plate respectively. In the thermal diffusivity measurement, pulsating heat is applied with a defined frequency. The

surface temperature is monitored by PRT sensors and the phase difference across the sample is measured by a lock-in amplifier. The thermal diffusivity is thus calculated by:

$$TD = \frac{\pi l^2}{T(\Delta\varphi)^2} \tag{3}$$

where l, T and $\Delta\varphi$ are the thickness (m) of the sample, period of wave (s) and phase difference across (radian) the sample respectively.

EXPERIMENTAL ANALYSIS

The qualitative performance of any experimental setup can be specified in terms of inaccuracy. The error analysis estimates the limits of the probable system errors and expected inaccuracy in the measurement process. However, a more reliable estimate of the inaccuracy is obtained from the statistical analysis of the experimental results. In some cases, not only experiments are repeated, the entire process of reassembling the sample holder with sample and then carrying out the experiments are also executed. These investigations and the subsequent modification have resulted in a high degree of reliability. In temperature measurements, the conducting leads are thermally anchored at the heat sink thus heat conducted along the wire in intercepted at the sink. In sample holder assembly, an additional resistance by the contact surfaces and the differential thermal contraction between the surfaces increase the contact resistance. These are nullified by applying Apiezon-N grease at the contacting surfaces. The contraction of length and diameter of the sample due to cooling would result in a measurement error and it is reduced by perfectly clamping the samples.

The total uncertainties for thermal conductivity values consist of contributions from the systematic bias, experimental imprecision, material variability and calculation error. The main error lies in the measurement of the temperature difference across the sample and the heat input to the sample. In order to reduce the conduction losses from the heaters, thin phosphor-bronze wire is used as connecting leads because of its high electrical and low

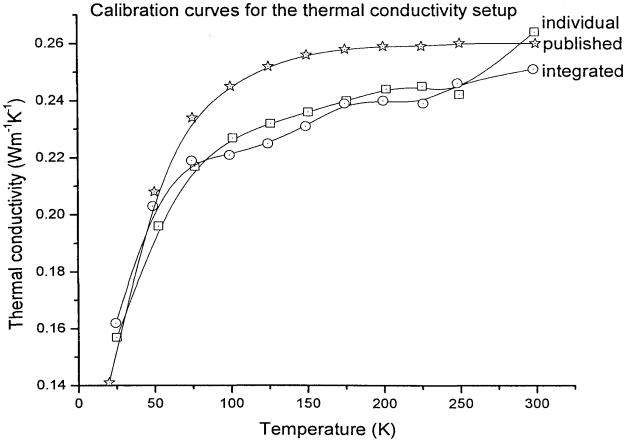

FIGURE 3. Comparison of experimental and published values of thermal conductivity (Teflon)

thermal conductivity. The measurement error of voltage and current is found less than 0.1 %. Although the accuracy of the form factor (l/A) is limited by an error of 1 to 2 %, it is constant for one set of samples. This does not affect the relative values of the measurements taken at different temperatures. The inaccuracy of the measurement of thermal conductivity of the insulating materials is difficult to reduce because the maintaining of the thermal losses in the level of microwatts is more complicated. The inaccuracy due to non-unidirectional heat flow through the sample is prevented by using a guard heater. The relative error in the measurement of thermal conductivity is the sum of the relative errors as that of applied power, thermal losses, temperature difference, length and area of the sample. It is estimated to be about 9 % resulting from the summation of the individual errors. The desired set point temperature of the sample holder is maintained by the temperature controller. Even when it has stabilized, there is a small temperature difference across the sample. This is measured and subtracted from the temperature difference created by main heater. The temperature of the sample is measured with time from the start of stabilization to the end of it. Heat leak by convection is minimized using high vacuum and thermal anchoring of lead wires to restrict the conduction loss. Similarly, the radiation loss is eliminated using number of mylar layers followed by a highly polished stainless steel shield chamber. Thus the heat leak by all possible means is reduced to a negligible value.

In the measurement of thermal expansion, the distance between the capacitor plates is kept at minimum in order to reduce the fringing effect. The guard electrode is used to control the fringing flux and to avoid the electrical field. Thus, capacitor plates are free from the edge effects. To reduce the thermal noise in the measurement of capacitance, the heaters are kept away from capacitor cell. As temperature decreases, the area of the capacitor plates also decreases inducing a change of capacitance. Non-parallelism of the capacitor plates is also checked by light and images and this error comes about 1%. The expansion of the sample holder is determined during the calibration of the thermal expansion measurement setup. The relative error in the measurement of coefficient of thermal expansion is the sum of square of measurement of length, measurement of increment of length, temperature measurement and the temperature distribution in the sample, which is a common source of error in various types of dilatometer [17]. These considerations have substantially reduced the total error [12]. The relative error in the measurement of thermal expansion coefficient is within 8 %.

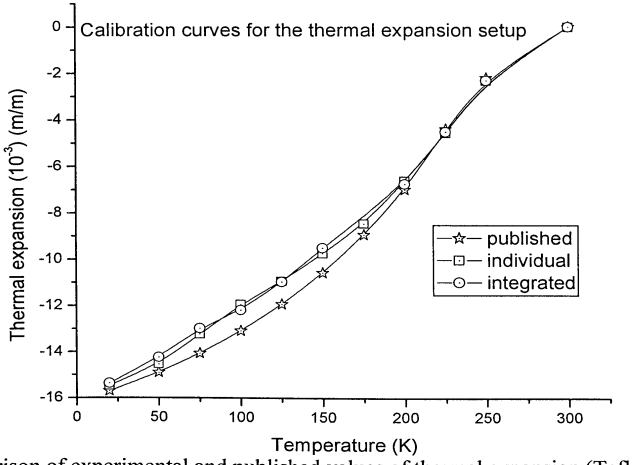

FIGURE 4. Comparison of experimental and published values of thermal expansion (Teflon)

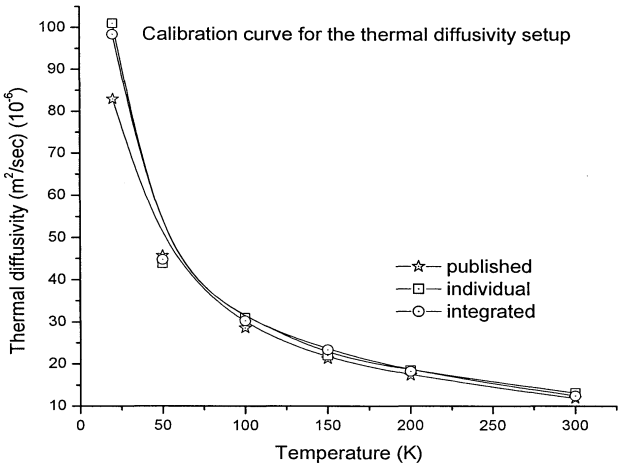

FIGURE 5. Comparison of experimental and published values of thermal diffusivity (Teflon)

The thermal diffusivity setup requires a voltage pulse while raising the temperature of the sample. Thus it is difficult to keep the sample and radiation shield of the sample holder at the same temperature. The frequency of the pulsating voltage is selected in such a way that the thermal wave is attenuated at the other end of the sample so effects of finite length are nullified. The relative error in the measurement of thermal diffusivity is the sum of the relative errors in the measurement of phase difference, sample thickness and period of the wave. The results are found to be within 10 % of the published value in the literature.

The preceding error analyze explains the systematic errors in this measurement. Experiments have been carried out to calculate the errors in repeatability.

CALIBRATION

Material variability is so extensive that only standard specimens are required to be used for comparison of the performance of the setup. The availability of the standard reference materials makes the results in more accurate data for solids. The experimental setup is calibrated for the measurement of thermal conductivity, thermal expansion and thermal diffusivity using stainless steel, copper and stainless steel respectively. Teflon as an insulating material is also used as a sample to calibrate the developed setup. It is observed that the experimental results are always within 10% of the literature values. The calibration curves for thermal conductivity, thermal expansion and thermal diffusivity are shown in FIGURE 3, 4 and 5 respectively.

SALIENT FEATURES

The salient features of this setup are: a) no need of cryogenic fluids to generate cryogenic temperature, b) simultaneous measurement of thermal conductivity, thermal expansion and thermal diffusivity under identical conditions, c) saving of experimental time, d) easy and simple operation, e) portable, f) any material can be tested with minor

modification of the setup and g) specific heat of the materials may be calculated from the measured experimental values of thermal conductivity and thermal diffusivity thus enthalpy and entropy could also be calculated. The simultaneous measurement gives the advantage of rapidity of execution, the reduced sample handling time and the uniformity about same physical conditions during measurements.

CONCLUSION

It is concluded that simultaneous measurements of thermal conductivity, thermal expansion and thermal diffusivity of the composites at temperatures down to 30 K under identical environment is possible using this integrated experimental setup.

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