Thermal diffusivity of advanced composite materials of e-glass fiber reinforced plastic in the temperature range 5-120K

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Thermal diffusivity of plain-woven fabric composite in a closed cycle cryo-refrigerator (CCR) based setup in the temperature range 5-120K has been studied. The modified temperature wave method (Angstrom) is applied to measure the thermal diffusivity of glass fiber reinforced plastic (GFRP). The set up is a plug in type which can be used anywhere because of its portability and also works without use of any cryogenic fluid. The results show that there exists an inverse relation between the thermal diffusivity of e-glass fiber composite and temperature in very low temperature domain but decreases very slowly after 100K.

Keywords: Thermal diffusivity, Cryogenic temperature, Phase difference

1 Introduction

In recent years resin glass fiber composite material has been widely used in almost every field starting from small household products to manufacturing of products used in aerospace because of its low weight, high tensile strength and non-corrosive properties⁷. Among various types of fibers, the most widely used is the plain woven glass fiber due to its relatively high tensile strength. The successful and proficient design in the low temperature system demands the minimum cool down time and cool down losses and these two parameters are directly related to the thermal diffusivity of the materials. Many newly emerging materials such as fiber-reinforced plastic (FRP) are coming up with very attractive mechanical and thermal properties at cryogenic temperature. High strength, low density and low conductivity are among the few properties, which are improved by reinforcement of fiber in the resin. But the lack of such thermo physical data has hindered the full exploitation of composite at various low temperature applications. Thermal diffusivity of the composite plays a key role in the design of cryogenic dewar and storage vessel. Also, recently the composite cryotanks have been used in launch vehicle in order to increase the payload capability². Thus, the experimental data of thermal diffusivity of the composite materials is of considerable support to optimize the cryo-system performance, heat transfer and cool down process analysis in cryogenic transfer lines. Modified temperature wave method is used to measure the thermal diffusivity of solid composite materials from cryogenic temperature to higher range by many workers⁶-⁷. The literature review reveals that it was made by various workers for many materials but the studies on the variation of thermal diffusivity of FRPs with temperature are done by only few workers⁸-¹⁰,¹⁵. In most studies liquid cryostat has been used for the production of low temperature. The main drawbacks of those studies include: (a) not cost-effective in long term use, (b) continuous change of set point temperature is difficult. In the present study an attempt has been made to develop the experimental set-up, to evaluate the thermal performance of advanced composite materials down to the temperature 5K without use of any cryogen. Experimental studies have been carried out in such a way so as to overcome all the difficulties encountered in all earlier studies. The results obtained have been found to be in good agreement with the theoretical results.

2 Materials and Methods

2.1 Materials

Composite samples mainly have two parts matrix and reinforcement. In the present sample plain-woven e-glass fiber was used as reinforcement for its various qualities like low cost, easy availability and wide
The compositions of the e-glass fiber used are given in the Table 1. In the matrix part Epoxy resin (LY-556) along with hardener (HY-951) were used. Besides few limitations, like high cost, epoxy has several advantages over other resins. It gives the increased complexity of the polymer chain and the potential for a greater degree of control over the cross-linking process which gives a much improved matrix in terms of strength and ductility as well as high moduli. It is water resistant, temperature resistant and low shrinkage during curing. The samples are of 18 mm dia and 1 to 1.25 mm thickness with different volume fractions. For the preparation of the sample, compression molding process is used. The void less specimen is prepared by applying pressure of about 10 bar using hydraulic press to the mold area on the prepreg forces the resin through the reinforcement, pushing the air front to the pinch off area which is composed of 10-20 piles of prepreg depending upon thickness of the specimen. The pressure is applied on the dye during curing which helps the fabric in shape of the component during the transition from liquid to solid. The mold is heated to about 130-170°C by the heating coil attached to the plates of the hydraulic.

2.2 Theory

The essential feature of the modified temperature wave method is that if one end of the sample is heated periodically, a temperature wave is propagated along the length of the sample with the same period but with diminishing amplitude. Also there exists a definite phase relationship between any two points across the length of the sample and the measurement of phase difference enables the determination of thermal diffusivity. In the present paper thermal diffusivity is determined using the equation given below which is derived from the conservation of energy and Fourier’s equation of heat conduction:

\[
D = \frac{\pi l^2}{T (\Delta \phi)^2}
\]

where \(l\) is the length of the sample, \(T\) is the time period of the temperature wave and \(\Delta \phi\) is the phase difference suffered by the thermal wave in passing through the sample.

2.3 Experimental Details

A closed cycle cryo-refrigerator based experimental set-up, shown in Fig. 1, is developed and calibrated for measurement of thermal diffusivity of the composite sample in the temperature range 5 to 300 K. Figure 2 shows the sample holder used in the set-up developed specially for the study of disc shaped sample. To restrict different mode of heat leak the samples are of 18 mm dia and 1 to 1.25 mm thickness with different volume fractions. For the preparation of the sample, compression molding process is used. The void less specimen is prepared by applying pressure of about 10 bar using hydraulic press to the mold area on the prepreg forces the resin through the reinforcement, pushing the air front to the pinch off area which is composed of 10-20 piles of prepreg depending upon thickness of the specimen. The pressure is applied on the dye during curing which helps the fabric in shape of the component during the transition from liquid to solid. The mold is heated to about 130-170°C by the heating coil attached to the plates of the hydraulic.

### Table 1—Composition of e-glass fiber

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Quantity in %</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>55.2</td>
</tr>
<tr>
<td>Al&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>14.8</td>
</tr>
<tr>
<td>B&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>7.3</td>
</tr>
<tr>
<td>MgO</td>
<td>3.3</td>
</tr>
<tr>
<td>CaO</td>
<td>18.7</td>
</tr>
<tr>
<td>K&lt;sub&gt;2&lt;/sub&gt;O</td>
<td>0.2</td>
</tr>
<tr>
<td>Na&lt;sub&gt;2&lt;/sub&gt;O</td>
<td>0.2</td>
</tr>
<tr>
<td>Fe&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>0.2</td>
</tr>
<tr>
<td>F&lt;sub&gt;2&lt;/sub&gt;</td>
<td>0.1</td>
</tr>
</tbody>
</table>

![Fig. 1—Schematic diagram of experimental set-up for measuring thermal diffusivity](image)

![Fig. 2—Diagram of sample holder fixed on the cold head](image)
sample holder is insulated by vacuum at the order $10^{-6}$ mbar created by rotary and diffusion pump assembly and covered with stainless steel radiation shield. The cold head temperature is controlled by a 50 watt PID controlled heater (Lakeshore model-332) via a calibrated silicon diode sensor with an accuracy of 0.01K from 120 to 40K, and below 40K the fluctuation in cold head temperature is ±0.2 to 0.3K. This set-up takes around 40 to 50 min to reach new set temperature at loaded condition. The sample is sandwiched between heater-cum-sensor palate and another sensor palate as shown in Fig. 3. Sensor palate, heater and sample assembly each is clamped by pressure mounting screw to the base of the sample holder which is directly placed on the cold head as shown in Fig. 2. The linear distance between two sensors in the sensor palate is 6 mm approximately. Highly conducting Apizon-N grease is applied at every contact surface for good thermal contact. For better result three pairs of silicon diodes (placed in two sensors palate each containing three) are used to measure the phase difference suffered by the thermal wave propagating through the sample. The thermal wave is generated by resistance heater made up-of insulated copper wire of approximately 15 ohm resistances at 300 K. The current is fed to the heater from a programmable current source (Keithley Model-6221), the r.m.s value of the current varies from 70 mA (120K) to 90 mA (5K) with different frequencies. Phase differences of the temperature wave across the two sides of the sample was measured by a dual phase lock-in amplifier (SRS, Model-830). Since the length of the sample is fixed by the experimental set-up, frequencies are selected in such a way that the temperature wave is not reflected from the cold end. Again, the thermal diffusivity does not depend on how heat is lost from the sample. So the result would be expected to be the same as that of higher power input within the allowable range.

To reach the temperature down to 5K from 120K, a close cycled cryorefrigerator (Janis Research Co., Inc. Model No. SHI- 4-5) has been used. The temperature of the cold finger of the CCR may be varied by PID temperature controller. The temperature feedback needed for running the temperature controller is taken from silicon thermal sensor attached near the base of the sample holder as shown in Fig. 2.

### 3 Results and Discussion

The experimental set-up has been calibrated using Teflon (PTFE) as standard sample and these results (Fig. 4) are compared with the calculated values of thermal diffusivity using equation $D = \frac{K}{\rho c}$. These values are referred from reference 12 at the temperature range from 300 to 20K. The departure of theoretical values and experimental results from the calculated values are within 20%.

The experiment is repeated to verify the reproducibility of the results and found to be within ± 20% variation. The variation of thermal diffusivity of e-glass/epoxy plain-woven fabric composites having different volume fraction ($V_f$) of fiber with the temperature range of 5 to 120K is illustrated in the Fig. 5. It is observed that the thermal diffusivity decreases exponentially as the temperature increases, but increases with fiber concentration. Thermal diffusivity decreases rapidly up to 100K followed by slow decrease there after. It is observed that

![Fig. 3—Diagram of the heater-cum-sensor plate. Face that will be in-contact with sample heater wire is wound around this sensor plate and hence forming heater-cum-sensor plate (the dimension is given in mm)](image)

![Fig. 4—Variation of thermal diffusivity with temperature of Teflon](image)
According to the Hill, conductivity decreases and specific heat increases. Due to short circuit within the woven fabrics, thermal contact between the fiber increases with the increase in volume fraction.

The observed decrease in the thermal diffusivity with increase in temperature may be the effect of shorting of the mean free path of phonons with the increase in temperature. In other words, it may be said that as the temperature increases the mean free path of phonons in the material decreases and as a result the thermal conductivity decreases and specific heat increases.

**References**

11. Hand Book of Course Materials on FRP, CCIRI, Kolkata.