IOP Conf. Series: Materials Science and Engineering **278** (2017) 012158 doi:10.1088/1757-899X/278/1/012158

Optimization of epoxy-aluminium composites used in cryosorption pumps by thermal conductivity studies from 4.5 K to 300 K

R Verma[1,2], N C Shivaprakash[1], S Kasthurirengan[2] and U Behera[2]

[1]Instrumentation and Applied Physics, Indian Institute of Science, Bangalore 560012, India

[2]Centre for Cryogenic Technology, Indian Institute of Science, Bangalore 560012, India

E-mail: behera@ccf.iisc.ernet.in

Abstract. Cryosorption pump is a capture vacuum pump which retains gas molecules by chemical or physical interaction on their internal surfaces when cooled to cryogenic temperatures. Cryosorption pumps are the only solution in nuclear fusion systems to achieve high vacuum in the environment of hydrogen and helium. An important aspect of this development is the proper adhesion of the activated carbons on the metallic panels using a high thermal conductivity and high bonding strength adhesive. Typical adhesives used are epoxy based. The thermal conductivity of the adhesive can be improved by using fine aluminium powder as the filler in the base epoxy matrix. However, the thermal conductivity data of such epoxy-aluminium composites is not available in literature. Hence, we have measured the thermal conductivities of the above epoxy-aluminium composites (with varied volume fraction of aluminium in epoxy) in the temperature range from 4.5 K to 300 K using a G-M cryocooler based thermal conductivity experimental set-up. The experimental results are discussed in this paper which will be useful towards the development of cryosoprtion pumps with high pumping speeds.

1. Introduction

Thermal conductivity is defined as the rate at which heat passes through a specific material expressed as the amount of heat that flows per unit time through a unit area with a temperature gradient of one degree per unit distance. It is one of the fundamental thermal properties of the material. Thermal conductivity plays a vital role for design of various thermal components and systems. Thermal conductivity of material depends on temperature, crystal structure, density of states, Fermi energy, impurities etc.

In the field of science and technology where measurement of heat transfer plays an important part, the accurate values of thermal conductivity of associated materials must be known beforehand. With the advances in the technology, new compound materials for low temperature applications are being developed whose thermal conductivity data is still unknown. Therefore measurement of thermal conductivity of those materials fills the blank of low temperature thermal property data base. Epoxy and epoxy-aluminium composites are such materials whose low temperature thermal properties are unknown [1], [2].

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

Measurement of thermal conductivity values of epoxy and epoxy-aluminium composite is useful in development of cryosorption pump. They are one of the essential components in the design of high efficiency cryosorption pumps. Cryosorption pumps are only solution in nuclear fusion environment to achieve ultra high vacuum where hydrogen and helium are the byproducts. In the development of cryosorption pump, epoxy and epoxy-aluminium composite is used to adhere the adsorbing material onto the metallic panel. The photographic view of such panels with different adsorbing material is shown below. Figure 1(a) shows the photograph of the cryopanel with granular activated carbon used in a commercial cryopump (Varian model: SP8), while figure 1(b) shows an identically dimensioned cryopanel with Knitted Carbon Cloth developed at our laboratory (KCC/IIS01). Cooling of adsorbing material to the lowest possible temperature is important for optimum performance of cryosorption pump. Therefore epoxy and epoxy-aluminium composite must have high thermal conductivity along with high bonding strength to adhere the adsorbing material onto the panel at operating temperature range of cryosorption pump.

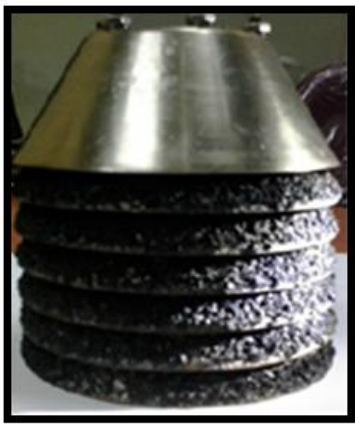

Figure 1(a). Cryopanel with granular activated carbon.



Figure 1(b). Cryopanel with Knitted Carbon Cloth.

Towards this we have developed a cryocooler based experimental setup to measure the thermal conductivity of various materials. The photographic view of the experimental setup and the cut-section view of the cryocooler cold head are shown in figure 2 and 3 respectively. Figure 4 shows the detailed arrangement of the test sample, the heater and the thermal link. Figure 5 shows the photographic view of the sample along with the temperature sensors, mounted on the second stage of the G-M cryocooler. Figure 6 shows the block diagram of the experimental system. The experimental setup has been standardized by measuring the thermal conductivity of Al-2024 T4 sample whose thermal conductivity data is known in the temperature range of 4.5 K to 300 K [3], [4]. The thermal conductivity of the developed epoxy and epoxy-aluminium composite is measured with the help of the developed experimental setup and compared with the commercially available epoxies such as "Stycast®" 2850 FT and G10 Cryocomp. These studies will be helpful towards the development of high efficiency cryosorption pump.



Figure 2. Cryocooler based experimental setup.

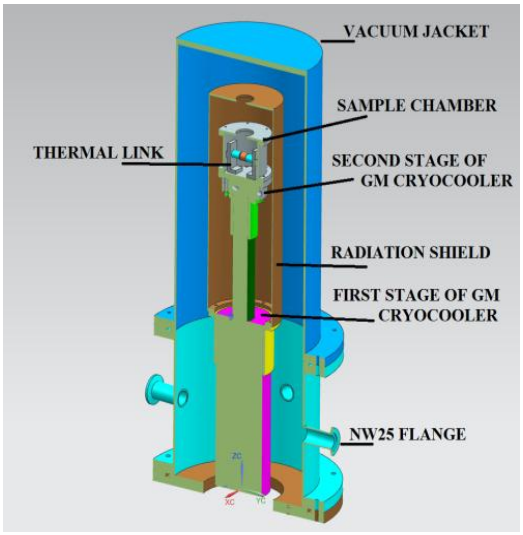

Figure 3. Cut-section view of experimental setup.

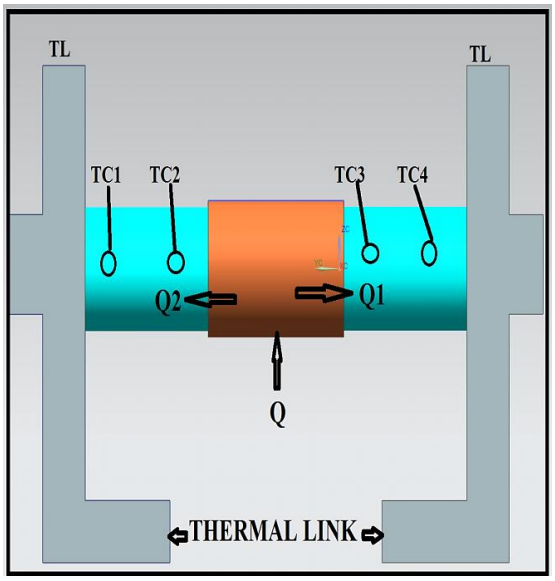

Figure 4. Detailed arrangement of the test sample, the heater and the thermal link.

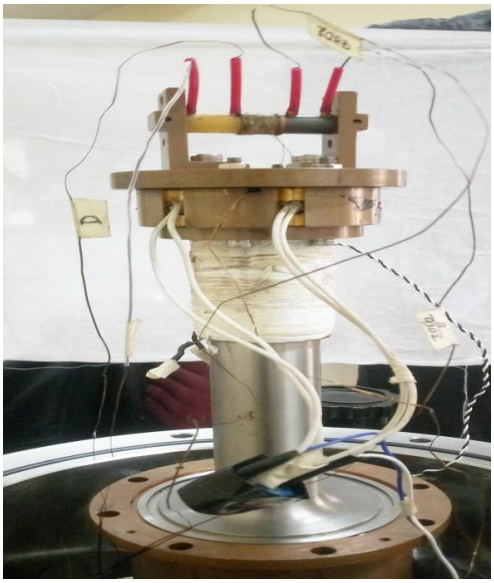

Figure 5. Photographic view of the sample along with temperature sensors.

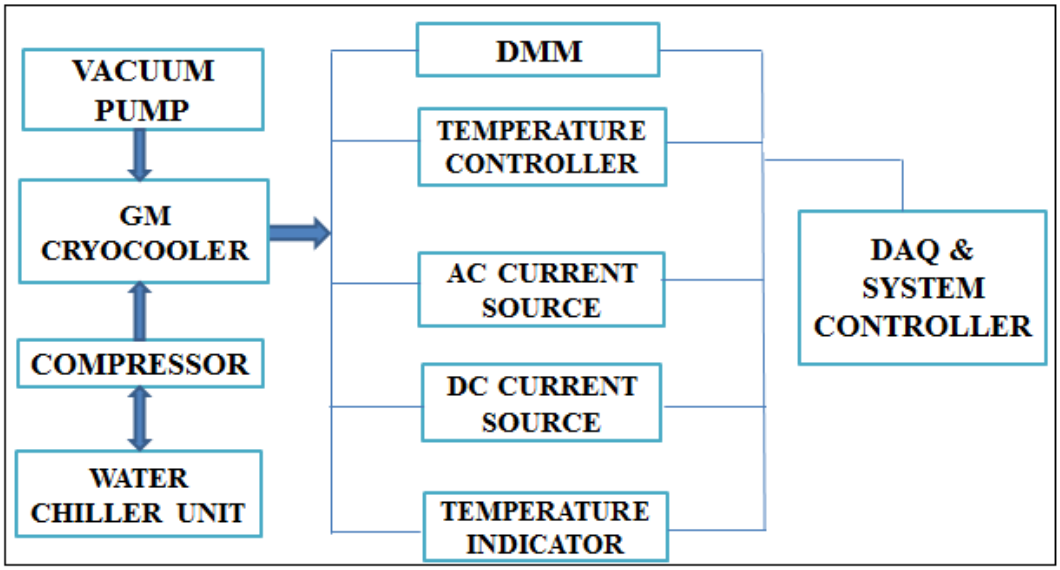

Figure 6. Block diagram of an experimental setup.

2. Experimental principle

Thermal conductivity is measured by longitudinal steady heat flow method on the basis of one dimensional Fourier heat conduction law [5], [6].

A heating power Q is supplied to the heater sandwiched between the two samples. At one side of the heater is kept a sample whose thermal conductivity is known. On the other side of the heater is kept the sample whose thermal conductivity is to be measured.

The Fourier heat conduction equation to measure the thermal conductivity is given by,

$$Q = k_1(T)A_1(\Delta T_1/\Delta x_1) + k_2(T)A_2(\Delta T_2/\Delta x_2) \quad \text{.....(1)}$$

Where;

Q = Heating power in Watt
$\mathbf{k_1(T)}$ = Thermal conductivity as a function of temperature of known sample in Watt per meter Kelvin
$\mathbf{A_1}$ = Cross sectional area of the known sample in square meter
$\mathbf{\Delta T_1}$ = Temperature gradient of the known sample in Kelvin
$\mathbf{\Delta x_1}$ = Effective length of the known sample across which temperature is measured in meter
$\mathbf{k_2(T)}$ = Thermal conductivity as a function of temperature of the unknown sample in Watt per meter Kelvin
$\mathbf{A_2}$ = Cross sectional area of the unknown sample in square meter
$\mathbf{\Delta T_2}$ = Temperature gradient of the unknown sample in Kelvin
$\mathbf{\Delta x_2}$ = Effective length of the unknown sample across which temperature is measured in meter

3. Experimental apparatus

A G-M cryocooler (Model: Oerlikon, Cool power 4.2 GM 1 Watt), with a refrigeration power of 1 Watt at 4.2 K based experimental setup has been developed for measurement of thermal conductivity of materials in the range of 4.5 K to 300 K. A turbo-molecular pump (Model: Varian-V301-AG), a vacuum jacket along with necessary instrumentation for data acquisition is used for performing the experiment. Special cryogenic temperature sensors such as Si410B & DT670 are used in the experimental setup for temperature measurement. Scientific instrument temperature indicator (Model: 9302) is used for indicating the temperature across the sample. Lakeshore PID controller (Model: 332) is used to maintain the temperature of sample chamber at different temperature. KEITHLEY DC

current source (Model: 6220) is used to supply current to the sample heater whereas KEITHLEY DMM (Model: 2000) is used to indicate voltage across the heater. LabVIEW software is used for data acquisition.

4. Experimental procedure

The test sample is fixed to the sample chamber and placed on the second stage of the G-M cryocooler. The Sample chamber is surrounded by Multi-Layer Insulation (MLI) along with radiation shield to avoid radiation heat load. Whole assembly is surrounded with vacuum jacket and the space inside vacuum jacket is evacuated up to 10^{-6} mbar with the help of turbo-molecular pump to avoid convection heat load. After achieving the desired vacuum, the cryocooler is turned on to cool the sample along with the sample chamber. The sample is maintained at the desired temperature with the help of Lakeshore temperature controller. When the sample is maintained at steady state temperature, the heater sandwiched between it is energized.

After achieving steady state condition, the temperature difference (ΔT) along the axial direction of the sample is measured with the help of temperature sensors. The heater voltage and the current are measured with the help of the DMM and the current source respectively.

The experimental setup is calibrated by measuring the thermal conductivity of some known sample like Al 2024 T4 whose thermal conductivity data are known in the temperature range of 4.5 K to 300 K. The experimental results are compared with NIST published data as shown in figure 7 and are within 10 % error of the NIST data.

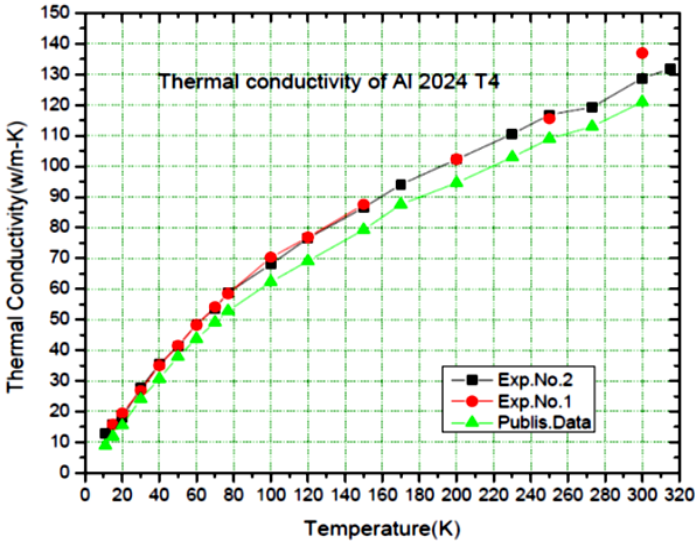

Figure 7. Comparison of thermal conductivity of Al 2024 T4 with NIST data.

5. Results and Discussions

Figure 8 shows the thermal conductivity of the developed epoxy in the range of 4.5 K to 300 K and figure 9 shows the comparison of thermal conductivity between developed epoxy and commercially available adhesives like "Stycast®" 2850 FT and G10 Cryocomp. It can be seen that the thermal conductivity of the developed epoxy is higher than the commercially available epoxy in the operating range of the cryosorption pump [6].

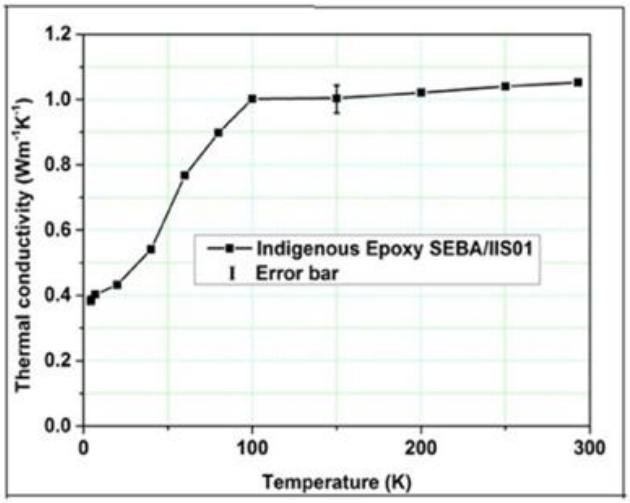

Figure 8. Thermal conductivity of pure epoxy from 4.5 K to 300 K.

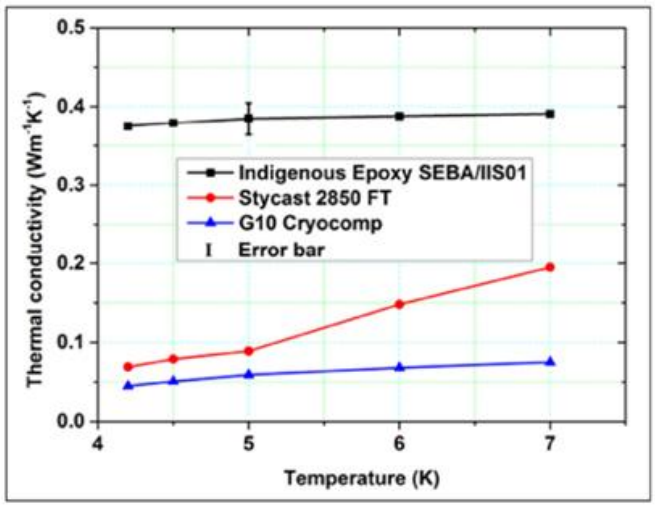

Figure 9. Comparison of thermal conductivity between developed epoxy and commercial epoxies from 4.5 K to 300 K.

Figure 10 shows the variation in thermal conductivity of epoxy-aluminium composite up to a critical volume fraction (~ 30 % in the present case) of aluminium in the base matrix of epoxy in the temperature range from 4.5 K to 300 K [7]. After the critical volume fraction, chain like heat transfer paths takes place by the aluminium particles in the base matrix of the epoxy, leading to sudden rise in thermal conductivity of the epoxy-aluminium composite. Figure 11 shows the thermal conductivity of epoxy-aluminium composite after the critical volume fraction of aluminium in the base matrix of epoxy. It can be seen that the thermal conductivity of the epoxy-aluminium composite increases with the increase of aluminium volume fraction in the base matrix of epoxy in the temperature range from 4.5 K to 300 K.

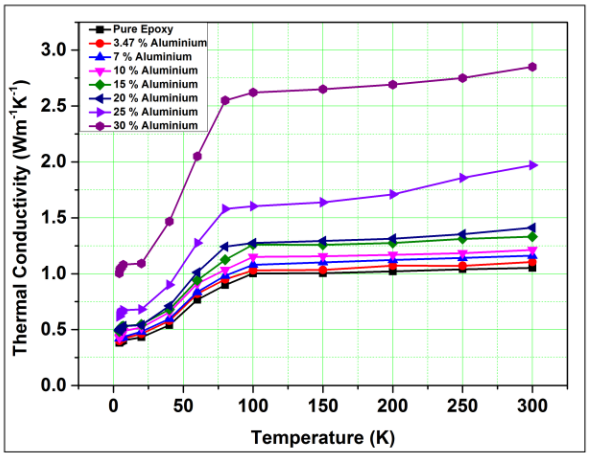

Figure 10. Varitaion in thermal conductivity of epoxy-aluminium composite for low volume fraction of aluminium in epoxy from 4.5 K to 300 K.

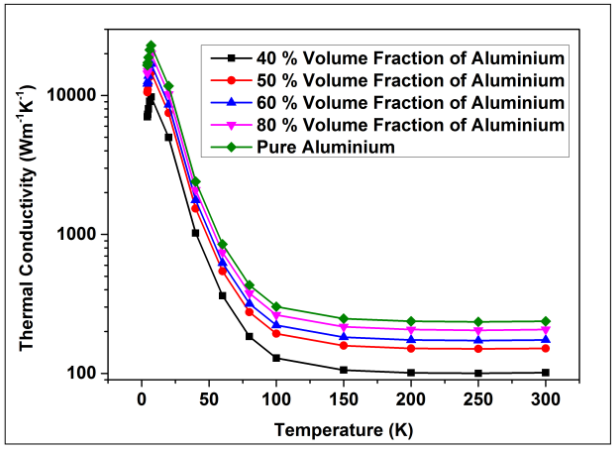

Figure 11. Varitaion in thermal conductivity of epoxy-aluminium composite for high volume fraction of aluminium in epoxy from 4.5 K to 300 K.

Figure 12 shows the variation in thermal conductivity of epoxy-aluminium composite with different volume fraction of aluminium in the base matrix of epoxy at 300 K. It corresponds to three stages; the first stage corresponds to the composite with low volume fraction (upto 30 %) of the filler aluminium particles that are randomly distributed in the base matrix of epoxy. In this range, the thermal conductivity increases slowly with increase in volume fraction of the filler aluminium particles. The abrupt increase of thermal conductivity of the second stage (from 30 to 40 % volume fraction) indicates the formation of the continuous chain like heat transfer paths by the filler aluminium particles in the base matrix of epoxy. In this range, as the aluminium content increases, the number of heat transfer paths increases leading to sudden rise in thermal conductivity due to the physical linking of aluminium particles in the base matrix of epoxy which provides the additional path for heat transfer as shown in figure 13.

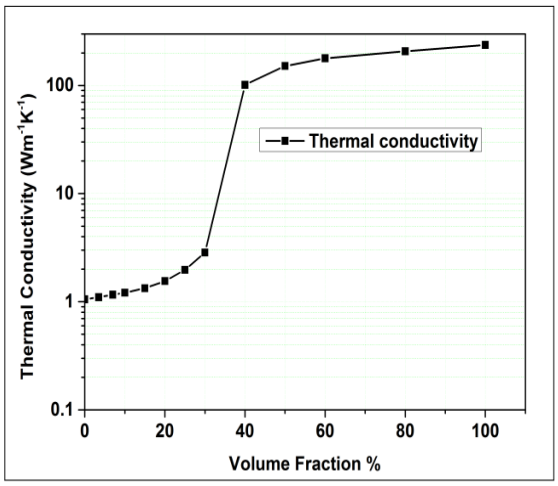

Figure 12. Varitaion in thermal conductivity of epoxy-aluminium composite with different volume fraction of aluminium in base matrix of epoxy at 300 K.

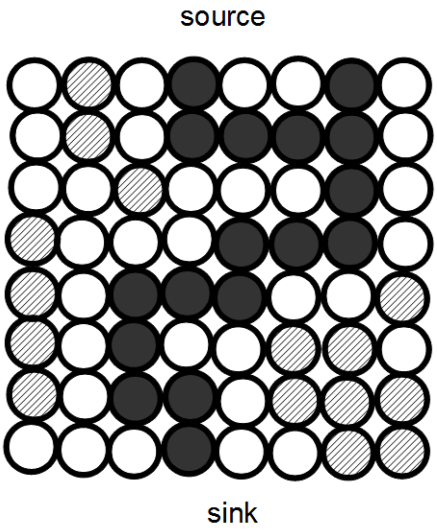

Figure 13. A scheme of heat transfer path formation due to percolation.

In the third and the final stage, with further increase of the filler particles in the epoxy matrix, the heat transfer paths gradually increases and become equal in all directions. Hence although there is an increase in thermal conductivity the rate of increase slows down and hence the observed thermal conductivity behaviour. The trend in increase of thermal conductivity with increase of aluminium particles in the base matrix of epoxy matches well with the published data [8], [9]. Studies are in progress to optimize the thermal conductivity of the epoxy-aluminium composite to obtain maximum thermal conductivity without hampering the bonding strength of the epoxy-aluminium composite to adhere the activated carbons on the cryopanel.

6. Conclusion

The thermal conductivity of developed epoxy and epoxy-aluminium composite is measured by the G-M cryocooler based experimental setup in the temperature range of 4.5 K to 300 K. It is observed that thermal conductivity of developed epoxy is better than the commercially available adhesives in the operating range of the cryosorption pump. The thermal conductivity of the epoxy-aluminium composites increases significantly with addition of aluminium particles in the base matrix of the epoxy. Enhanced thermal conductivity data of epoxy-aluminium composite will be very useful towards the development of the cryosorption pumps.

References

[1] Zhang P, Chen Y J, Ren X J, Wu A B and Zhao Y 2011 *Cryogenics* **51** 534-40.
[2] Verma R, Behera U, Kasthurirengan S, Shivaprakash N C, Udgata S S and Gangradey R 2017 *Conference Series: Materials Science and Engineering* **171** 1-7.
[3] Baudouy B 201 *Cryogenics* **51** 617-20.
[4] Woodcraft A L 2005 *Cryogenics* **45** 421-31.
[5] Choi Y S and Kim D L 2012 *Journal of Mechanical Science and Technology* **26** 2125-28.
[6] Hust J G 1975 *Cryogenics* **15** 8-11.
[7] Verma R, Chatterjee A, Mustafa A, Shivaprakash N C, Kasthurirengan S and Behera U 2017 *International Journal of Thermal Sciences* **118** 292-302.
[8] El-Tantawy F 2002 *European Polymer Journal* **38** 567-77.
[9] Nielsen L E 1977 *Journal of Applied Polymer Science* **21** 1579-84.

Acknowledgments

The authors greatly acknowledge the financial support provided by Board of Research in Fusion Science and Technology (BRFST) to undertake the project. The authors also acknowledge the support provided by CCT staff in the development of the experimental setup.