International Journal of Heat and Mass Transfer 116 (2018) 1064–1073

Contents lists available at ScienceDirect

International Journal of Heat and Mass Transfer

journal homepage: www.elsevier.com/locate/ijhmt

A new device for measuring the thermal conductivity of heterogeneous multicomponent thin samples: Development and application to polymer composites

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ARTICLE INFO

Article history:
Received 22 May 2017
Received in revised form 26 September 2017
Accepted 26 September 2017

Keywords:
Thermal conductivity
New device
Measurement technique
Disc samples
Composite materials

ABSTRACT

A novel device for measuring the thermal conductivity of polymeric materials (pure polymers or nanocomposites) is presented here. Its design is based on two cylindrical tanks containing cold and hot water. Each tank wets one side of a disc-shaped polymeric sample. The proper selection of construction materials (aluminum body, Teflon fittings, etc.) and geometry (shape, size, etc.) of the two tanks and of the sample (diameter, thickness) renders the accurate measurement of the low thermal conductivity of several polymeric materials possible. The disc-shaped sample is placed in-between the two cylindrical tanks of the device, filled with cold and hot water. The water temperature in the two tanks is an adjustable operational parameter of the device. Both tank temperatures are recorded and fitted by a proper mathematical model in order to estimate the thermal conductivity of the sample. Extensive experimentation shows a very small scatter of the estimated conductivity values. The device was used to measure thermal conductivity of several polymers such as silicone rubber and epoxy resin prepared with a variety of cross-linkers and cross-linker blends. Moreover, the above polymers are used as matrices for the incorporation of various (nano)additives, like mesoporous silica foams (MCF) and organoclays (I.30E), for the preparation of the relative polymer nanocomposites whose thermal conductivity is measured.

1. Introduction

Knowledge of thermal conductivity of new materials is very important to establish their suitability for specific uses. Unlike volumetric properties, such as thermal capacity, thermal conductivity is a structural property, so its estimation in the case of multicomponent materials, is not reliable if based only on the composition. Despite the huge effort devoted to the estimation of thermal conductivity of composites, up to present there is not a generally accepted method for this purpose [1]. This is especially true in case of components with very different thermal conductivities where even slight structural variations of the composition can lead to a large variation of thermal conductivity [2]. In such a case, one should rely exclusively on the experimental determination of thermal conductivity.

There are many experimental techniques for measuring thermal conductivity of solids. All of them are based on a direct measurement of the solid temperature [3–8]. Existing techniques suffer from certain drawbacks, such as, locality of measurement which in the case of multicomponent heterogeneous samples can be critical, poor contact between solid material and temperature sensor, etc.

A new idea for a thermal conductivity measurement technique of polymeric materials (pure polymers or nanocomposites) is presented here which circumvents the above problems and, in addition, requires a small amount of material for the tests. Not only the technical details but also the basic principle of the technique is different from the existing ones. The central idea of the technique is to use the polymeric sample as a thin wall that separates two tanks filled with liquid at different temperature. The liquids in the two tanks are well agitated and their temperature is recorded. The mathematical analysis with all the details that has led to the specific design concept of the technique will be presented elsewhere.

The three main features of the new technique are: (i) it needs a test sample in the form of a thin slice of material with thickness of few millimetres and surface area of a few square centimetres. This peculiarity is significant when thermal conductivity measurements are used to distinguish among materials produced by different production recipes, where minimizing the material wasted for the measurement is of concern. (ii) it is a quasi-steady state technique

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https://doi.org/10.1016/j.ijheatmasstransfer.2017.09.107
0017-9310/

leading to direct computation of thermal conductivity and not of thermal diffusivity as in the case of transient techniques. Moreover, the technique is better than other fully steady state ones because estimation of thermal conductivity is derived from a whole experimental curve and not from a single data point. The quasi-steady state character is achieved by the small thickness of the test sample and by the agitation of the liquid in the two tanks (ensured by appropriate stirring). (iii) The temperature sensors (thermocouples) are not in contact with the solid but they are submerged in well agitated liquids. The above features yield a more reliable and accurate temperature measurement. It is noted that in the present version the technique has been adapted to measure thermal conductivities in the range 0.1–1 W/mK because the current interest is in low conductivity polymeric materials.

2. Implementation and construction of the device

Fig. 1, shows a schematic representation of the proposed device where the two liquid filled tanks (hot tank/cold tank) and the thin sample between them can be seen. At the top of each tank there is an inlet port close to its open end (where the sample is positioned) and an outlet port at its opposite closed end. The inlet port is sealed with adjustable fittings which on one hand allow filling the tank with liquid and on the other hand serves to insert and fix stably a thermocouple measuring the liquid temperature. The outlet port at the other end of the tank acts as gas purging/relief valve controlled with a small adjustable screw. This relief valve is a critical item having the aim to discard bubbles formed inside the tanks when filled with liquid. The thermocouples -one at the hot tank and one at the cold tank- are placed at a distance of 2 mm away from the surface of the sample in order to record the liquid temperature during the measurement period.

The sample has the shape of a disk placed between the two tanks like a lid that seals and separates them. At the rim of the open end of each tank, at the side where the sample is placed, there is a sealing flange. The two tanks with the sample disk placed between them are stably fixed tightening both flanges against the sample with screws. Due to the small diameter of the sample and the requirement to place it accurately between the tanks, four small metallic holders (guides) are constructed. The flanges are machined in order to obtain four fins, 90° spaced each other (See Fig. 2). Two O-rings are located between the flanges and the sample and pressed during the sealing procedure to prevent leakage of the liquid from the tanks. All holes and inlets are lined with Teflon foil to ensure proper sealing. To suppress heat losses to the environment through the inlet ports, apart from the glasswool sheets used to wrap the device externally, two custom-made Teflon fittings are used to hold the thermocouples. In addition, Teflon foil covers all four holes on each flange where the screws fit into suppress heat conduction between flanges. Fig. 1 displays all the aforementioned parts.

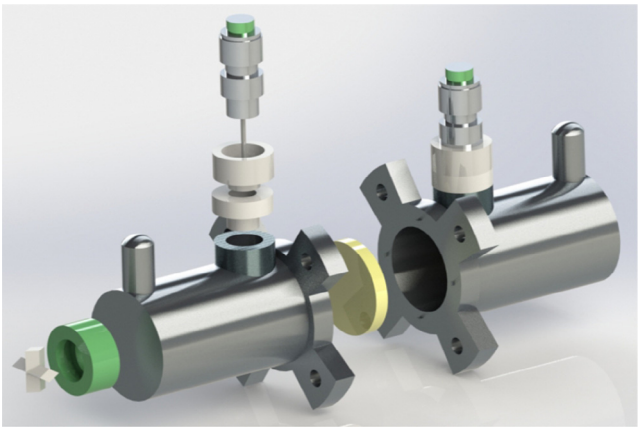

Fig. 2. Expansion of a 3D sketch of the device representing all the parts with the trimmed flanges.

Fig. 2 is a 3D expanded illustration of the device assembly showing the trimmed flanges. The flanges are trimmed in order to decrease their mass and so improve the overall accuracy of measurements and to prevent radiation heat exchange between flanges since they are positioned only few millimetres apart from each other.

A good stirring system has a noticeable impact on the accuracy of results as it ensures a uniform liquid temperature in the tanks, in order to fulfil the hypotheses of the data processing procedure. Due to the small size of the apparatus the stirring system is based on a miniature magnetic spin bar (1.5 cm long) at the flat closed bottom of each tank. In order to keep the magnetic bar in place and at the same time minimize the friction between the bar and the bottom of each tank, an aluminium cap (ring) is constructed to house the bar, Fig. 3. Spin bars are made to rotate at 700 rpm using two powerful magnetic stirrers (MR 3001, Heidolph) that are strong enough to rotate the spin bars although the magnet is placed 2 cm from the tank bottom because of a glasswool sheet that insulates externally each tank. The particular rotation speed is selected after long experimentation with the target to take temperature recordings independent on the rotation speed. This implies a negligible contribution of liquid-solid heat transfer resistance and of viscous dissipation heat release. In order to reduce losses from the tanks to the environment, several insulating materials were tested, and glasswool was chosen to give the best performances. However the need of magnetic stirring prevents the use of unlimited thickness of insulating material. The strategy was to minimize the losses to the environment primarily by a proper construction of the device

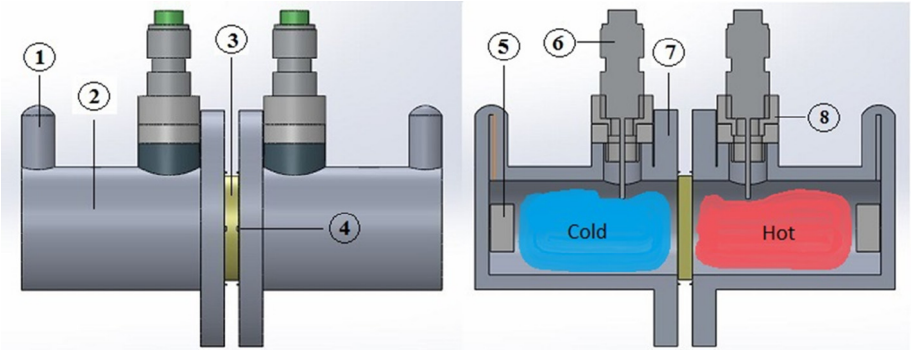

Fig. 1. Sketch of the device, 1. Gas purging/Relief valve, 2. Tank, 3. Sample, 4. Sample holders, 5. Magnetic spin bar, 6. Thermocouple 7. Flange, 8. Liquid Inlet port.