A Modified Steady State Method for Measurement of In-Plane Thermal Conductivity

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Abstract

A modified version of Parallel Thermal Conductance, Two-Length Method, for in-plane thermal conductivity measurement in slabs is presented. Two modifications have been applied to the currently available methods to improve the accuracy and repeatability of the results; first, to consider and calculate the heat loss resistance in the data analysis, and second, to design the tests in a way that can accommodate multiple samples instead of one to increase the accuracy and decrease the uncertainty of the data analysis. In the end, a semi-empirical technique, using a custom-designed test setup and an analytical solution for temperature distribution inside the anisotropic rectangular plates exposed to multiple hotspots, is proposed. In the introduced technique, using the experimental results, the in-plane thermal conductivity of the sample can be calculated with good accuracy.

Keywords

In-plane thermal conductivity, Experimental study, Anisotropic material, Custom-designed test

1. Introduction

To assist power dissipation in high-density electronic devices, there has been an increasing demand for materials that are characterized by high thermal conductivity in the given directions only. High in-plane thermal conductivity materials are of interest to many cooling mechanisms. Graphite-based materials are one of the well-known anisotropic materials which have in-plane thermal conductivities, up to 1500 $W.m^{-1}.K^{-1}$ [1,2]. This property is mainly due to their especial micro-structure (Figure 1). If properly utilized, these materials can mitigate the hot spot formations temperature build-up in different applications.

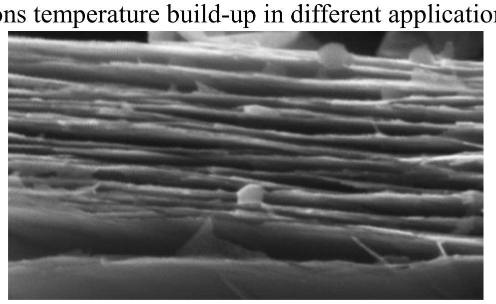

Figure 1: SEM image of compressed expanded graphite

Despite the wide employment of anisotropic materials in heat transfer and cooling industry, companies still are seeking accurate yet simple methods for the property measurement of their products. One of the key properties of such materials which need to be determined accurately is the in-plane thermal conductivity. Different approaches have been employed for this purpose. In a method developed by Wanager [3] in late 1990s, based on the method proposed by Angstrom in 1861, the propagation of a thermal wave along the axis of a constant cross section sample is exploited to measure the thermal diffusivity of the sample. It is calculated from the phase difference between harmonic components of temperatures measured at sample surfaces, perpendicular to the direction of thermal wave propagation. Several modifications of this approach has been proposed to enhance the accuracy and versatility of the method [4,5]. Another methodology, called Transient Plane Source (TPS) in which a hot-source is attached to the sample in proper orientations. The hot source which works both as heat source and temperature sensor experiences voltage variations as a single or multiple heat pulses are applied to the sample. Using the voltage feedback, the diffusivity along the given directions will be calculated [6,7]. Gustafsson [8,9] proposed a modified version of this method that is capable of accommodating the measurement of anisotropic materials. In his approach three hot-sources are attached in proper orientations, then using their feedback the diffusivity along the principle directions can be deduced. Laser flash method is another widely-used technique in which infrared images of evolving thermal patterns on the surface or rear face of the sample, injected with a laser beam, are analyzed and thermal diffusivities in in-plane as well as through-plane directions can be calculated [10–12]. There are also semi-analytical approaches such as Inverse method in which a sample is heated at one location and temperature increase is recorded at some predefined locations. Then using the analytical solution which approximates the heat transfer process, the directional thermal properties are back-calculated [6,13,14]. This method generally employs numerical tools for solving equations.

In addition to above mentioned methods there is another classic yet simple approach called steady state temperature gradient method which is very well-known for its simplicity, robustness and steady state nature. This method is mostly used for through-plane thermal conductivity measurement which is based on standard ASTM D5470. However it can be also employed to measure the in-plane properties in thin plate materials. Parallel thermal conductance (PTC) is a temperature gradient steady state technique which has been

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employed recently for in-plane conductivity measurement of films [15,16]. In this technique, the sample is placed in between two hot and cold sample holders which produce a 1-D temperature gradient along the longitudinal direction. The contribution of both sample holders and secondary heat paths are subtracted from the test results using the testbed characterization data. Knowing the amount of heat flux through the samples, the in-plane conductivity is simply calculated. The major drawback of this method is neglecting the effect of contact resistance which generally results in underestimation of thermal conductivity. To address this issue, Sadeghi *et al.* [17] measured the in-plane thermal conductivity of gas diffusion layer (GDL) used in fuel cells, by a novel temperature gradient approach. In their experimental method, using two different length of material exposed to same amount of heat flow from one end to the other, they managed to deconvolute the end effect resistances and reported the in-plane thermal conductivity within an acceptable range of accuracy; however they did not account for secondary heat losses through the test setup or radiation.

In this paper the modified version of the PTC, called Two-Length Method, is proposed in which the principles of both PTC and Sadeghi's method [17] are employed. The apparatus for the experimental setup is explained and the test results for three different graphite samples are presented. The multi-layer measurement capability is added to the testbed in order to mitigate the difficulties in measurement of low thermal conductivity materials and to decrease the uncertainty of the final results. As one of the essential requirements for temperature gradient method is to obtain a 1-D heat transfer along the longitudinal direction in the sample, an analytical criterion for the minimum allowable sample length based on a parametric study is presented.

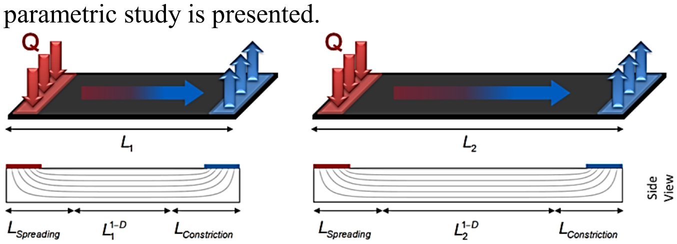

Figure 2: Concept of Two-Length Method and schematic of samples' three resistance sections

2. Methodology

Two-length method is an experimental method for measurement of in-plane thermal conductivity, which is based on obtaining the difference in 1-D thermal resistance of two identical test samples of different length. In this approach, two rectangular strips of the material with the same cross-section but different length are exposed to heat fluxes as shown in Figure 2. The ratio of samples' length to their thickness has to be chosen large enough (bigger than 20) in order for the heat flow-lines in the middle of the samples to become parallel to each other and form a one-dimensional heat transfer in the longitudinal direction. Since the experimental setup is identical for both sample lengths, and both samples are of the same material and cross-section areas, the thermal end-effects (including spreading/constriction and contact resistances) in both samples' ends can be assumed identical. Hence, it can be deduced that the difference between the measured thermal resistances of the two samples is due to the difference in bulk resistance of the 1-D heat transfer in the middle section of the samples (Eq. 1).

$$R_1 - R_2 = R_1^{1-D} - R_2^{1-D} = \frac{L_1 - L_2}{Ak_{in-plane}} \qquad (1)$$

The thermal resistance between the source and sink for each sample can be measured rather simply, i.e. the temperature difference between sink and source divided by the heat flow. So, after rearranging the Eq. 1 and rewriting the resistances in the form of $\Delta T/Q$, the following equation for calculating in-plane thermal conductivity will be derived.

$$k_{in-plane} = \frac{L_2 - L_1}{A}\left(\frac{\Delta T_2}{Q_2} - \frac{\Delta T_1}{Q_1}\right)^{-1} \qquad (2)$$

3. Experimental setup

The test setup consists of two aluminum end-holders (hot/cold) on an adjustable frame on which the distance of the end-holders can be set. On the back face of the hot end-holder, two 40-W cartridge heaters are sandwiched using a plate of the same material. This will help the heat flux to be distributed uniformly inside the end-holder. The same mechanism is employed on the cold end side, by sandwiching five 8-mm diameter and 20-cm length copper heat pipes (provided by Advanced Cooling Technology Inc.). The heat pipes transfer the incoming heat from the samples to the ice-water bath located underneath the test-bed. To measure the temperatures at the end holders two T-type thermocouples (Provided by Omega) are attached to each end. The temperature data is logged through a DAQ system (by National Instruments), and LABVIEW software. The testbed is designed to test four identical sample layers rather than only one. The use of four layers mitigates some of the experimental challenges and uncertainties. For example, using of a single layer of very low thermal conductivity materials between the sample holders leads to an excessive temperature drop across the sample holders, which in turn increases the temperature at the hot end tremendously, even for a small heat flux, and may damage the heater. The experimental apparatus and a schematic of the test setup for the in-plane thermal conductivity measurement are shown in Figure 3.

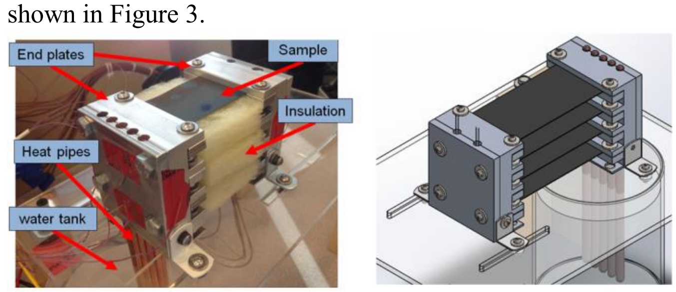

Figure 3: Two-Length Method experimental setup and schematic for measurement of in-plane thermal conductivity

After placing the samples, the whole test apparatus is insulated using three layers of insulating foam to minimize the heat dissipation to ambient throughout the experiment;

however, there still is a slight amount of heat transfer through the insulations and set-up to the ambient, which if not taken into account, will increase the error in final result. To account for the heat loss, the testbed can be considered as two parallel resistances (Figure 4.a), operating between the heat source and heat sink (in this case the ambient). The first resistance is the resistance of the samples and the second one represents the resistance of all other heat dissipation paths to the ambient. To calculate the exact amount of heat flow through the samples, Q_1 and Q_2 in Eq. 2, one has to know the value of the loss resistance of the test-bed. The loss resistance for the test bed is mainly the resistances of the insulation and the bypass paths from the source to sink such as convection of the entrapped air and conduction through the testbed frame. The simplest technique to account for this resistance, is running a no-sample test while having all the insulation on. By applying a known amount of heat flow and reading the steady state temperature difference between heat source and the ambient (Figure 4.b), the loss resistance can be calculated rather simply using $\Delta T/Q$. This resistance hardly changes for regular tests, as it is only a function of insulation and distance between source and sink. The test results of the no-sample experiment for testbed of this study is presented in Figure 5.

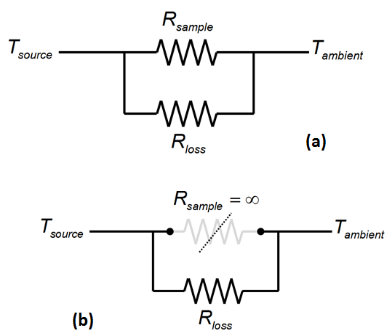

Figure 4: Resistance network of Two-Length Method experiments, a) with sample, and b) no-sample test

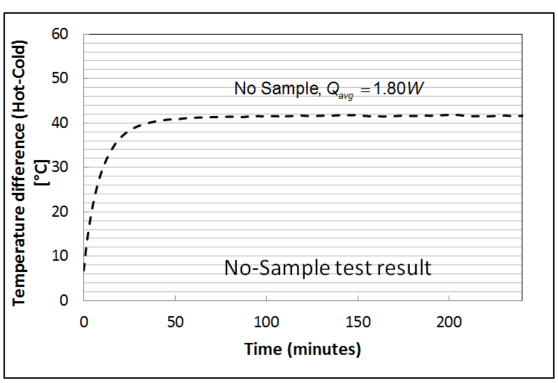

Figure 5: The experimental test-result of the no-sample test for 100 mm span between hot and cold sides

3.1. Uncertainty of the tests

As Two-Length Method involves measuring different parameters including heat flux, temperature, dimensions and etc. the uncertainty of the derived in-plane thermal conductivity can be calculated using the general uncertainty analysis introduced in [18].

The main uncertainty in this experimental setup was due to errors in determining the heat flux through the sample holders which led to a maximum error of 6.2%. The maximum uncertainties for the thermocouples and the data acquisition readings are ±1°C which introduces a maximum error of 1.8% between two sample holders. Other uncertainties including those associated with the width, thickness, and length measurements are 0.3%, 0.3%, and 0.9%, respectively. Considering this individual uncertainties, the uncertainty of the calculated thermal conductivity is between 6-9% depending on the parameters values.

Before conducting the experiments on anisotropic samples, the testbed is calibrated using aluminum sheets. Since aluminum is an isotropic material, its thermal conductivity can be measured rather easily using "*guided heat thermal conductivity measurement*" method, as per ASTM E1225 standard. This system is the most precise available method for thermal conductivity measurement; however, it is limited to isotropic materials only. The guided heat test is conducted in vacuum condition to ensure minimizing the effects of convective heat transfer. More details on the guided heat thermal conductivity measurement method can be found in [19]. The results obtained from our custom-made testbed are compared to the data given by the guided heat tests, and the maximum relative difference of 1.6% is reported.

3.2. Test results

Using the custom-designed testbed explained in section 3, the in-plane conductivity of four graphite samples of different thickness (Provided by Terrella Energy), and two graphite-based electrodes used in a prismatic Lithium-ion batteries (provided by EIG and ChangHong Energy) were measured and the results are presented in Table 1. The criterion for steady state detection was to reach a point where the rate of temperature change at all thermocouples reduces to less than 0.1°C/hr. To satisfy this criterion, each test took approximately three to four hours to complete. A sample of the test results for samples Graphite1 and Graphite3 are presented in Figure 6.

Sample	Dimensions [mm]			Power [W]	ΔT [°C] (short , long)	Thermal Cond. [$W.m^{-1}.K^{-1}$]	Uncertainty [%]
	Lengths	Width	Thickness				
Graphite1	100/150	40	0.57	8.0	36.4, 49.4	340	7.7
Graphite2	100/150	40	1.17	14.0	23.8, 36.4	345	6.9
Graphite3	100/150	40	0.38	6.0	41.1, 57.7	600	6.4
Graphite4	100/150	40	0.80	5.0	25.3, 37.6	505	7.0
Electrode1	60/80	40	0.18	4.0	58.0, 65.0	21	8.2
Electrode2	60/80	40	0.25	4.0	48.0, 58.0	35	7.7

Table 1: Results of the in-plane thermal conductivity measurement for four different graphite samples and two battery membranes using the Two Length Method

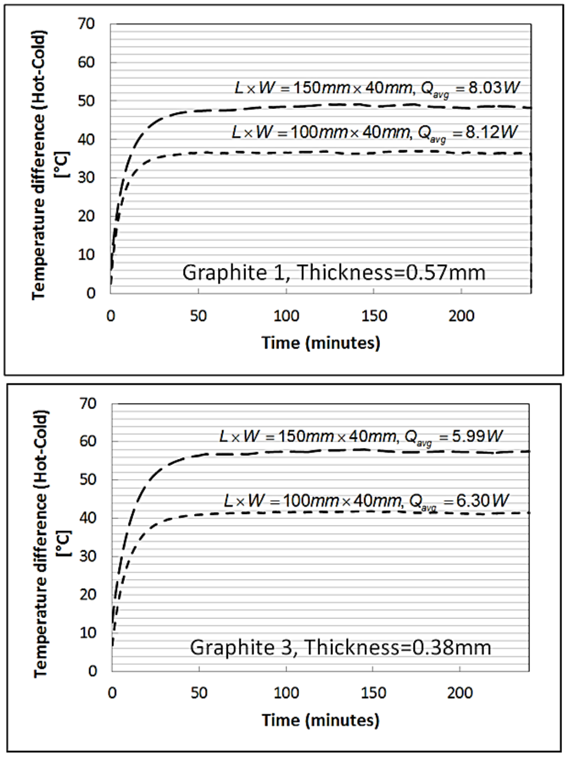

Figure 6: Experimental results of in-plane thermal conductivity measurement for samples Graphite1 and Graphite3

4. The semi-analytical method for measurement of in-plane thermal conductivity

In-plane thermal conductivity measurement of thin plates due to their low thickness and fragility, in some cases, is not as convenient as measuring through-plane conductivity. Besides, equipment that measure conductivity in in-plane direction are not as prevalent and standardized as the ones measuring this property in through-plane direction. As such, an algorithm is presented through which by knowing the through-plane thermal conductivity of a material and performing the experiments, as explained in the previous section, the in-plane thermal conductivity can be calculated iteratively using the analytical model presented in [20]. The analytical model is a solution for temperature distribution inside anisotropic slab exposed to multiple arbitrary-located hotspots. In the following the detailed steps of the algorithm are explained. Knowing the through-plane thermal conductivity of the sample, an in-plane thermal conductivity value for the plate is assumed in the first step. In the next step, a temperature difference along the sample is measured using the experimental setup, explained above. The temperatures along the sample can also be calculated analytically, using the equation introduced in [20]. If $\Delta T_{\text{model}}<\Delta T_{\text{experiment}}$, then the assumed in-plane thermal conductivity is decreased by ε, where ε is an arbitrary adjustment value, and if $\Delta T_{\text{model}}>\Delta T_{\text{experiment}}$, then the assumed in-plane thermal conductivity is increased by ε. The loop starts over from this step until the desired accuracy is achieved. The flowchart in Figure 7 demonstrates the above-mentioned algorithm.

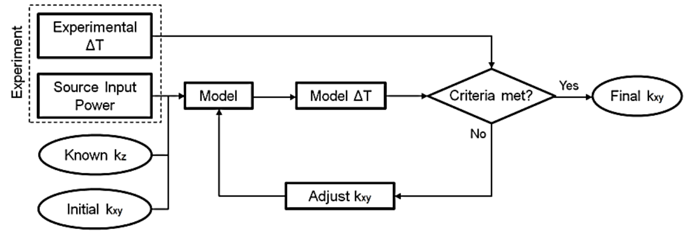

Figure 7: Semi-experimental method for in-plane thermal conductivity measurement (flowchart)

5. Conclusion

Characterizing thermal properties of anisotropic materials has become a vital necessity for industries that are majorly using these materials as the base component of their cooling systems. In this paper, a modified version of temperature gradient method for measuring in-plane thermal conductivity of slabs is presented. Testing multiple instead of single sample and implementing an innovative approach to account for energy loss in this method has increased the accuracy and repeatability and decreased the uncertainty of the results. The most important advantages of this technique over the existing method such as Angstrom, laser flash or transient plane source methods is its simplicity, low cost, minimized operational errors and considerably improved accuracy. At the end of this study, a semi analytical method for measuring the above-mentioned property is also introduced. In this method by employing an analytical model and the results of a straight-forward thermal experiment the sample's in-plane thermal conductivity is iteratively calculated.

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