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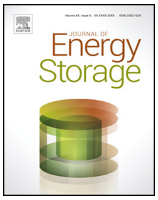

# Novel method for thermal conductivity measurement through flux signal deconvolution

Heinrich Badenhorst*, Carl Sandrock

*SARChI Chair in Carbon Materials and Technology, Department of Chemical Engineering, University of Pretoria, Lynnwood Road, Pretoria 0002, South Africa*

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ABSTRACT

Rapid thermal conductivity measurement of porous solids and composites remains a challenge. A modified steady state technique has been proposed which uses two heat flux sensors instead of one. The parameter estimation is achieved through the deconvolution of these signals and the identification of the system impulse response. A detailed derivation of the theoretically expected behaviour has been done, which provides a basis for fitting the measured impulse response. A six term expansion is required for the theoretical model to achieve full convergence. The unit requires a calibration step to measure the convective boundary condition. A signal validity check has been built into the approach through the use of the energy balance which detects any drift due to ambient losses or other factors. Through suitable choice of the mathematical algorithm rapid convergence of the non-linear fitting procedure is achieved. The parameter estimates of the standard test samples are excellent, with average errors of 2.3% for brass and 6.3% for aluminium. The system has several advantages in addition to the short measurement time, including low cost and no guard furnace or insulation requirement for room temperature measurements. The approach is suitable for measuring the overall behaviour of practical, composite systems.

## 1. Introduction

High thermal conductivity materials are used in countless applications from thermal management to energy storage. Accurate knowledge of the thermal transport properties is critical for design activities as well as the rapid development of new materials. Recently novel, highly conductive, graphitic foams have been developed for these applications [1,2]. These materials have extensive macro and micro porosity and correspondingly low densities ($\sim 0.3\,\mathrm{g\,cm^{-3}}$). The open pore structure makes them ideal for passive heat dissipation applications due to the ease of convective heat transfer. Alternatively these foams may be employed for property enhancement of substances having high energy capacity but low thermal conductivity. Such composites are ideal for thermal energy storage applications.

Many techniques have been developed over the years to measure the thermal properties of different materials, each with its advantages and disadvantages. These may be broadly classified into two main categories: transient and steady state. Due to their short measurement time transient methods such as the line source [3–5], hot strip [6–8], plane source [9–11] and laser flash [12,13] methods have been widely used. A closely related method to the hot-wire and hot-strip techniques is the 3ω method [14] which uses a frequency based analysis to measure thermal conductivity rather than a temporal approach.

However, these methods do suffer from practical issues when measuring the properties of porous solids. This is especially true for composite or multi-layer materials which are more representative of practical arrangements [15]. These may include contact issues, due to the rough surface of porous solids the number of contact points are reduced leading to inaccurate results unless large sample sizes are used. Sample penetration may also be limited [16,17] which is a problem for composites which are inhomogeneous and often have different surface properties due to anisotropic processing. The laser flash method for example is only applicable to suitably thin discs or films as stipulated by the ISO standard 22007-4:2008. For some composites such as foams which are very brittle, it is difficult to obtain very thin slices.

In addition, the transient hot strip method for example also suffers from the use of an approximate relationship for the property estimation rather than a rigorous theoretical solution [18]. Steady state methods on the other hand have a sound theoretical basis and many have been developed according to the American Society for Testing and Materials (ASTM) standards, for example C177 (standard test method for steady-state heat flux measurements) and C518 (standard test method for steady-state

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* Corresponding author. Fax: +27 12 420 2516.
*E-mail address:* carbon@up.ac.za (H. Badenhorst).

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**Nomenclature**

| | |
|---|---|
| $u$ | Temperature |
| $y$ | Position |
| $\alpha$ | Thermal diffusivity |
| $t$ | Time |
| $h$ | Convective transfer coefficient |
| $k$ | Thermal conductivity |
| $L$ | Total path length |
| $\lambda$ | Eigenvalue |

thermal transmission properties). One of these, ASTM E1530 (guarded heat flow meter method), involves the use of plates with fixed temperatures in conjunction with a heat flux sensor. The primary problem with a steady state method is the time required to reach steady state [19]. For the aforementioned method a waiting time of at least a few hours is recommended and as such a guard furnace is required to minimize any heat loss during the measurement. In addition to this, steady state methods are generally not advocated for materials that have a high thermal conductivity. This is because of the small temperature gradients developed and the ASTM E1530 is not recommended for materials having a thermal resistance lower than $1\times10^{-3}\,\mathrm{m^2\,K\,W^{-1}}$.

To overcome these limitations, a novel modification of the ASTM 1530 technique is proposed in this paper which involves the use of two heat flux sensors rather than one. This method retains the robustness of the steady state technique, making it possible to measure highly conductive, porous solids and composites, whilst significantly reducing the measurement time. What makes the approach unique is the use of flux signal deconvolution to find the impulse response of the system, which is in turn used to determine the thermal conductivity. In process identification terminology a transient system model may be represented as shown in Fig. 1.

In this case the input signal is a step and the output signal is a simple first order response. However, the input signal is arbitrary and can be an impulse function in which case the output signal is termed the impulse response of the system. If the system model is unknown it can be obtained from the input and output signals. However, this is not a straightforward division of the time based signals. In real measurement systems the input signal is not a continuous measurement as shown in Fig. 1 but is instead comprised of discrete values sampled at a given frequency. For this reason the input signal may be viewed as a set of impulses and the impulse response of the system may be obtained by the frequency domain decomposition and deconvolution of input and output signals [20]. This is true irrespective of the overall observed shape of the input signal which still remains arbitrary. In the case of this investigation the input and output signals of the system are the flux measurements.

To avoid the use of an approximate relationship a fundamental model was first developed for the proposed measurement technique. This enables a detailed analysis of the expected impulse response of the system. Unfortunately, this function is very non-linear and cannot be cast into a form which would explicitly give the desired parameter estimate. For this reason it is necessary to employ an optimization approach to find the appropriate parametric value which fits the impulse response of the experimental data. The objective of this investigation is to develop a suitable methodology for parameter estimation using the new technique. This includes signal validation, model formulation and the choice of optimization algorithm. The approach is validated using two, high thermal conductivity, metallic samples with known thermal properties. This work represents the first step in the development of a technique for the overall heat transfer coefficient measurement of practical, composite systems.

## 2. Experimental

In essence the sample is sandwiched between two heat flux sensors with a hot source and cold sink above and below respectively. The experimental setup is shown schematically in complete detail, in Fig. 2 below.

The assembly is clamped using steel bolts between a steel top and copper base plate to ensure good contact. The entire system is placed in a circulating water bath at a constant temperature which acts as the cold sink. The PVC pipe open spaces are filled with glass fibre wool for additional insulation. The heating element is linked to a controlled power source (maximum power 1 kW) and is shielded from the steel plate using a ceramic insulator. The element is a tungsten wire ($L = 10$ cm, $D = 100\,\mu$m) placed in a zigzag arrangement on top of a very thin, highly conductive graphite foil. The graphite foil has comparatively low thermal conductivity through the plane but very high in plane conductivity ($\sim 1500\,\mathrm{W\,m^{-1}\,K^{-1}}$). This ensures a very low thermal gradient with a virtually homogenous temperature distribution and hence flux across the sample top surface. The graphite foil (SS1500 eGRAF$^{®}$ Spreadershield$^{TM}$) was obtained from a commercial supplier, GrafTech International (U.S.A.). Since the input flux signal is measured, there is no requirement for an exact, controlled shape. For a single experimental run the power source is set to maximum output and is turned on only for a few seconds before being switched off.

The heat flux sensors (HFS-4) were obtained from OMEGA Engineering, Inc (U.S.A.). The sensor is a thin film comprised of a 50 + junction thermopile bonded to either side of a Kapton barrier, which has known thermal characteristics. Since the heat transfer rate is directly proportional to the temperature difference across the thermal barrier, the exact rate of transfer can be calculated by measuring this difference. The sensor is 35 mm by 28.5 mm with a reported sensitivity of $2\,\mu\mathrm{V}\,(\mathrm{W\,m^{-2}})^{-1}$ and has an extremely low thermal resistance. All interfaces are coated with a thin film of silicon heat transfer compound (Unick Chemical Corp, Taiwan) to ensure surface homogeneity. For testing, brass (CDA 385) and aluminium (6082) rods (diameter = 20 mm) of different lengths (25, 50, 80, 100 mm) were obtained from Non-Ferrous Metal

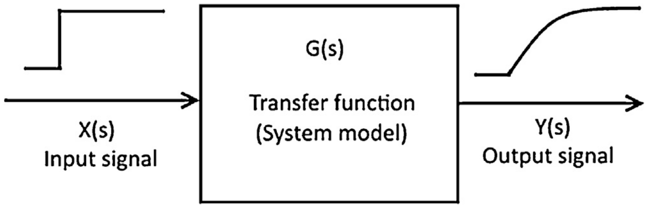

**Fig. 1.** System model.