Effects of Interface Resistance on Measurements of Thermal Conductivity of Composites and Polymers

Andrzej Brzezinski, Akhan Tleoubaev
LaserComp, Inc., 20 Spring St., Saugus, MA 01906 USA
atleoubaev@lasercomp.com

(Presented at the 30th Annual Conference on Thermal Analysis and Applications Sept. 23-25, 2002 Pittsburgh, PA, USA

ABSTRACT

When testing samples of moderate thermal conductivity ($\lambda \sim 0.3$-10 W/mK) using steady state methods the thermal contact (or interface) resistance $R$ must be addressed, otherwise significant errors will result. It can be taken into account by instrumenting the sample - that is taking the temperature measurement of the sample surfaces with thermocouples placed directly on the sample.

Another effective means of accounting for the thermal contact (or interface) resistance is the Two Thickness Analysis. By using at least two samples of the same material with different thicknesses $\Delta x_1$ and $\Delta x_2$ a system of two equations containing two unknown values can be solved:

$$Q_1 = \frac{\Delta T}{\left(\frac{\Delta x_1}{\lambda} + 2R\right) S_{cal}}$$

$$Q_2 = \frac{\Delta T}{\left(\frac{\Delta x_2}{\lambda} + 2R\right) S_{cal}}$$

where $\Delta T$ is the plates’ temperature difference, $\lambda$ is thermal conductivity, $2R$ is sum of two thermal contact resistances, $Q_1$ and $Q_2$ are signals from the heat flow transducers, and $S_{cal}$ is their calibration factor. We assume that the thermal contact resistances at the two surfaces and for both samples are equal.

If the sample is instrumented and the temperature drop across it is measured directly only one equation is necessary. However it is frequently not practical and/or easy to do.

In case of thin samples and/or of moderate thermal conductivity the thermal contact resistance $2R$ may even exceed the sample’s thermal resistance $\Delta T / \lambda$. For example ¼” thick Pyroceram has thermal resistance $\Delta T / \lambda=1.59*10^{-3}$. The thermal contact resistance $2R$ is about $3*10^{-3}$ – or almost 2 times bigger.

This paper will present test data on polymers and composites used as reference materials exhibiting a range of thermal conductivities to illustrate the effects of not having accounted for the interface resistance.
INTRODUCTION

The general principle of the heat flow meter instruments (see ASTM C518 and E1530 Standards) is based on one-dimensional equation for Fourier-Biot law:

\[ q = - \lambda \frac{dT}{dx} \]

where \( q \) is heat flux \( (\text{W/m}^2) \) flowing through the sample, \( \lambda \) is its thermal conductivity \( (\text{W m}^{-1} \text{K}^{-1}) \) of the sample, \( dT/dx \) is temperature gradient \( (\text{K m}^{-1}) \) on the isotherm flat surface in the sample.

If a flat sample is placed between two flat isothermal plates maintained at different constant temperatures, eventually a uniform one-dimensional temperature field establishes within all the sample’s volume (size of the plates is supposed to be much larger than thickness of the sample). The temperature gradient within the sample is equal to the difference between temperatures of its surfaces \( \Delta T \) \( (\Delta T = T_{\text{hot surface}} - T_{\text{cold surface}}) \) divided by its thickness \( \Delta x \), because in this case average temperature gradient \( dT/dx \) is equal to \(-\Delta T/\Delta x\).

Thermal resistance of the flat sample \( R_{\text{sample}} \) is equal to its thickness \( \Delta x \) [m] divided by its thermal conductivity \( \lambda \) [W m\(^{-1}\) K\(^{-1}\)]:

\[ R_{\text{sample}} = \frac{\Delta x}{\lambda} \quad [\text{m}^2 \text{K W}^{-1}] \quad (2) \]

The thermal contact resistance \( R \) is equal to temperature difference \( \delta T \) [K] between two contacting surfaces divided by heat flux \( q \) [W/m\(^2\)]:

\[ R = \frac{\delta T}{q} \quad [\text{m}^2 \text{K W}^{-1}] \quad (3) \]

and depends on the types of adjoining materials, their surface roughness, and the interface pressure. Although the subject has been studied for a long time, still very little is known about the complex mechanism of heat transfer at the contact between two bodies [1-3].

In case of moderate and high thermal conductivity samples the temperatures of their surfaces are not equal to the temperatures of the instrument’s plates, so:

\[ T_{\text{cold plate}} < T_{\text{cold surface}} < T_{\text{hot surface}} < T_{\text{hot plate}} \quad (4a) \]

\[ \delta T + \Delta T_{\text{sample}} + \delta T = \Delta T_{\text{plates}} \quad (4b) \]

Temperature difference \( T_{\text{hot surface}} - T_{\text{cold surface}} \) can be measured directly using thin thermocouples installed in the grooves on the sample’s surfaces, however it is not easy to do.

Electric signal \( Q \) [\( \mu \text{V} \)] in heat flow meter instruments is proportional to the heat flux \( q \) [W/m\(^2\)], which is equal to temperature difference \( \Delta T \) divided by
the total thermal resistance - sum of thermal resistance of the sample \( \Delta x/\lambda \) and two thermal contact resistances \( 2R \) – see Eqs. (1a) and (1b). The calibration factor \( S_{\text{cal}} \), physically, is the heat flow (W/m\(^2\)) necessary to create 1 micro Volt signal on the heat flow transducer.

**EXPERIMENTAL**

Calibration of the Heat Flow Meter instruments has to be done using materials with known thermal conductivity [4-8] like Pyrex 7740, Pyroceram 9606 and Vespel® DuPont™ (accuracy of the values is assumed to be about 5%):

**Table 1.** Thermal conductivity (W/mK)

<table>
<thead>
<tr>
<th>T, °C</th>
<th>Vespel® DuPont™</th>
<th>Pyrex 7740</th>
<th>Pyroceram 9606</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.365</td>
<td>1.063</td>
<td>4.15</td>
</tr>
<tr>
<td>20</td>
<td>0.371</td>
<td>1.086</td>
<td>4.04</td>
</tr>
<tr>
<td>40</td>
<td>0.377</td>
<td>1.115</td>
<td>3.94</td>
</tr>
<tr>
<td>60</td>
<td>0.386</td>
<td>1.145</td>
<td>3.85</td>
</tr>
<tr>
<td>80</td>
<td>0.389</td>
<td>1.175</td>
<td>3.78</td>
</tr>
<tr>
<td>100</td>
<td>0.396</td>
<td>1.203</td>
<td>3.71</td>
</tr>
</tbody>
</table>

To obtain the correct value of the calibration factor \( S_{\text{cal}} \) either direct measurement of the samples’ surfaces temperatures should be done, or Two-Thickness calibration procedure should be used using solution of the system of two Eqs (1a) and (1b):

\[
S_{\text{cal}} = \frac{\Delta T}{\lambda_{\text{cal}} (Q_1-Q_2)}/[(Q_1Q_2(\Delta x_2 - \Delta x_1)]
\]  

(5)

\[
2R_{\text{cal}} = (\Delta x_2Q_2 - \Delta x_1Q_1)/[\lambda_{\text{cal}} (Q_1-Q_2)]
\]  

(6)

Ideally, all the calibration runs should give the same values of the calibration factor no matter what method or what calibration standard is used.

If the thermal contact resistance \( 2R \) is not taken into account then the samples of larger thickness give larger calculated value of thermal conductivity simply because the heat flow meter instruments are able to measure only total thermal resistance between their plates:

\[
R_{\text{total}} = R_{\text{sample}} + 2R_{\text{thermal contact}} = \frac{\Delta x}{\lambda} + 2R_{\text{thermal contact}} = \frac{\Delta T}{(Q S_{\text{cal}})}
\]  

(7)

If we plot the total thermal resistance for several (at least two) samples against thickness and linearly extrapolate it down to zero thickness, we will get the value of thermal contact resistance \( 2R \) (see Fig.1).

The correct value of the sample’s thermal conductivity is equal to its thickness divided by the sample’s thermal resistance, which is equal to total thermal resistance minus thermal contact resistance:
\[
\lambda = \frac{\Delta \nu}{R_{\text{sample}}} = \frac{\Delta \nu}{(R_{\text{total}} - 2R_{\text{thermal contact}})} 
\]

If we calculate thermal conductivity of different thickness samples taking into account the thermal contact resistance \(2R\) we get the same and correct value of thermal conductivity for all the samples of different thickness.

### Table 2. Total thermal resistance against thickness at 20°C

<table>
<thead>
<tr>
<th>Thickness (mm)</th>
<th>R total (Pyrex)</th>
<th>Thickness (mm)</th>
<th>R total (Vespel®)</th>
<th>Thickness (mm)</th>
<th>R total (Pyroceram)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.28</td>
<td>0.005784</td>
<td>6.68</td>
<td>0.022152</td>
<td>6.40</td>
<td>0.004575</td>
</tr>
<tr>
<td>6.50</td>
<td>0.008742</td>
<td>12.70</td>
<td>0.037505</td>
<td>12.73</td>
<td>0.006198</td>
</tr>
<tr>
<td>12.88</td>
<td>0.014690</td>
<td>19.13</td>
<td>0.054324</td>
<td>15.88</td>
<td>0.007006</td>
</tr>
</tbody>
</table>

**Th.Cond.:**

- 1.094
- 0.387
- 3.90

**2R**

- 0.00282
- 0.00482
- 0.00293

**Figure 1** – Total thermal resistance (m² K/W) against thickness in millimeters. Extrapolations down to zero thickness give values of the thermal contact resistance \(2R\). Reciprocal of slope gives value of thermal conductivity \(\lambda\). 

\[
y = 0.0025845x + 0.0048179 \\
y = 0.0009138x + 0.0028166 \\
y = 0.0002564x + 0.0029338 \\
\]
Correct value of thermal conductivity can be calculated using the Two-Thickness procedure and following formula – solution of the system of the two Eqs. (1a) and (1b):

\[
\lambda = S_{cal} Q_1 Q_2 (\Delta x_2 - \Delta x_1)/[\Delta T (Q_1 - Q_2)]
\] (9)

\[
2R= (\Delta x_2 Q_2 - \Delta x_1 Q_1) \Delta T/[Q_1 Q_2 S_{cal} (\Delta x_2 - \Delta x_1)]
\] (10)

It can be calculated from the slope (reciprocal value) of the graph of the total thermal resistance against thickness of the samples as well (see Fig.1).

Recently developed and tested new Guarded Hot Plate system (also using the Two-Thickness formulas) can obtain accurate absolute thermal conductivity values without using calibration standards (soon to be published). This is an additional proof of the validity of the Two-Thickness formulas and procedure.

**RESULTS AND DISCUSSION**

Single-thickness tests of the same materials may give different thermal conductivity values because the heat flow meter instruments are sensitive only to the total thermal resistance. One-thickness procedure of calculations does not take into account the difference of thermal contact resistance of the calibration standard and one of the tested sample. Two-thickness procedure does – so the result is the thermal conductivity of the tested material itself, without the interface resistance (see Fig.2).
In case of 1 cm and 2 cm thick Pyrex (thermal conductivity is ~1 W/mK), thermal resistances of the samples are 0.01 and 0.02. If thermal contact resistance is equal to, say 0.003, then thermal resistances measured by the heat flow meter instrument are 0.013 and 0.023 for these two samples. Thermal conductivity values calculated supposing that thermal contact resistance is zero will give us 0.769 W/mK for 1 cm sample (23% error), and 0.869 W/mK for 2 cm sample (13% error).

Typical value of $2R$ is about 0.002-0.005 m$^2$K/W, and is comparable to samples’ thermal resistance: 1 cm-thick Pyrex has thermal resistance ~0.01, and 1 cm-thick Pyroceram – ~0.0025. Actually, the value of $2R$ includes also thermal resistance between temperature sensor and sample’s surface (of both sides), and is not only the thermal contact resistances on the sample’s surfaces.

**CONCLUSIONS**

The Two-Thickness procedure of calibrations and tests provides significantly improved accuracy of thermal conductivity measurements compared to regular tests and procedures. ASTM E1530 presents only one-digit accuracy
(e.g. Pyroceram’s thermal conductivity is 4 W/mK), whereas the Two-thickness tests give almost three-digit accuracy (reliable 2 digits).

Lasercomp’s FOX50 instrument with “WinTherm50” software enables the User to conveniently do External Temperature measurements with the external thermocouples provided by simply plugging them into the outlets in the back of the FOX50. Their readings are factored directly into the calculation.

The FOX50 Two Thickness Analysis enables the User to perform this procedure simply and easily by merely entering a second sample of the same material at a different thickness into the FOX50 when the first sample’s test has finished. LaserComp has performed hundreds of Two Thickness Analyses and our data has shown the very real and significant advantages of this Procedure.
REFERENCES