TECHNICAL REVIEW ON THERMAL CONDUCTIVITY MEASUREMENT TECHNIQUES FOR THIN THERMAL INTERFACES

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Nomenclature

A	Area (in^2 or m^2)
k	Thermal conductivity (W/m↔K or W/m°C)
L	Thickness or length (in or m)
N	Calibration constant of heat flow transducer
Q	Heat flow (W)
R_o	Thermal contact resistance (m^2↔K/W)
R_s	Thermal resistance (m↔K/W or m°C/W)
T	Temperature (°C)
T_u	Temperature on upper heater (°C)
T_L	Temperature on lower heater (°C)
φ	Heat flow transducer output (mV)
ΔT	Temperature difference (°C or mV)
Δx	Sample thickness (m)

Subscriptions

r	Known calibration samples
s	Samples of unknown properties

I. Introduction

The ongoing need for miniaturization and speed in the electronics industry has brought about a requirement for better performing thermal management systems. Thermal management technology remains a vital part of electronics innovations for notebook computers, high-performing CPU chipsets, mobile electronic appliances, power conversion and amplification modules, and aerospace electronics.

A typical thermal management package consists of thermal interfaces, heat dissipaters, and external cooling systems. The main function of the thermal interface is to eliminate or minimize the thermal resistance across the interfacial barrier between two mating rigid surfaces. Thermal contact resistance between two metallic surfaces, such as aluminum or copper, can be as high as 1 in^2°C/W at the low compression loads used for most electronic components. The presence of suitable thermal interfaces between mating surfaces can drastically reduce the overall thermal resistance of the entire heat dissipation units. Thermal interfaces have been conventionally used for improving the effectiveness of the thermal management systems with heat sink or heat pipe modules as the primary heat dissipation means in laptop computers. Despite their critical role, the importance of the thermal interface is often overlooked in the design of thermal management systems. Moreover, fundamentally valid analytical tools for quantifying and characterizing the functional properties of a variety of thermal interface materials are scarce in the industry.

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Selecting an appropriate interface material for specific thermal management applications requires evaluating a number of physical properties. Such properties include material thermal conductivity, thermal contact resistance under applicable compression loads, mechanical strength, compressibility and resilience, thermal stability, coefficient of thermal expansion, dielectric strength, break-down voltage, etc. Among them, thermal conductivity and thermal contact resistance are the two most important properties that directly influence the performance of a thermal interface.

There have been a number of experimental techniques and procedures for estimating thermal conductivity of thin, compressible thermal interfaces. Most of these methods are rather over-simplified and empirical in nature. Quite often, these methods can only provide comparative results that cannot be compared across different methods or even under different testing conditions. Furthermore, the sensitivity of these measurement techniques may be relatively poor and conclusions based on testing data could result in under-design of thermal management systems. More importantly, thermal contact resistance is often ignored or combined into the thermal conductivity of the material. Such measurement results may critically mislead thermal system designers who specify thermal interface materials for specific applications.

This paper reviews several current methods for measuring thermal conductivity including ASTM E1530, D5470 and F433. The validity of the common assumptions associated with these protocols are highlighted and discussed. Requirements and criteria of an ideal and universal measurement protocol are addressed. A thermal analysis measurement system that incorporates these design considerations has recently been developed by the UCAR GRAPH-TECH INC. Thermal and Electrical Analysis Laboratory. Measurements carried out on a number of sample types are discussed. Future efforts on the development of reliable and systematic analytical tools for characterizing the functionality of thermal interfaces are suggested.

II. Fundamentals

The Fourier thermal conduction equation states that the conductive heat flux (Q/A) is proportional to the temperature gradient ($\partial T/\partial L$) in an object [1], i.e.,

$$\frac{Q}{A} \propto \frac{\partial T}{\partial L} \qquad \text{Eq. (1)}$$

When the heat flow is one-dimensional and at steady state, Eq. (1) can be rewritten in a finite form as:

$$Q = kA\frac{\Delta T}{\Delta L} \qquad \text{Eq. (2)}$$

The proportional constant, k, between the heat flux and the temperature gradient is the thermal conductivity of an object in the direction of heat flux. Equation (2) is commonly used to calculate the thermal conductivity given the temperature difference (ΔT) between two points separated by a known distance (ΔL). It should be noted that the thermal conductivity of almost all substances varies with temperature [1]. In principle, the calculated value of k is usually an average thermal conductivity for the temperature range of the test. Although calculating thermal conductivity is relatively straightforward, measuring the temperature and thickness of thin, resilient specimens can be prone to error.

Significant errors are often encountered and lead to inconsistent test results if measurement systems are perfunctorily designed and calculation algorithms are overly simplified. Especially important is the measurement of very small temperature differentials across thin test specimens. The accuracy and stability of temperature sensors such as thermocouples and thermistors critically influence the measurement sensitivity and repeatability.

Extreme caution must be taken to calibrate temperature sensors and to reduce the electrical drifting of sensors.

Factors that influence measurement accuracy and consistency include types and locations of thermal sensors, detection of heat flow, measurement of specimen thickness and compression stress, fixture alignment and quality, uniformity of controlled thermal and mechanical conditions, and other factors particular to specific fixture designs.

III. Technical Review on Common Thermal Measurement Protocols

Numerous test protocols exist for quantifying thermal conduction characteristics of thin materials. Many tests are carried out using ASTM Test Methods E1530, D5470 and F433. Quite often, researchers and system designers in the electronics thermal management industry develop simplified measurement techniques modified from these ASTM test methods. Often the rationale behind these individual in-house techniques is to minimize testing time and cost, or to obtain comparative results that can be used for commercial (rather than technical) purposes. The following sections briefly discuss the ASTM test methods. The test procedures, measurement assumptions, validation and limitations are addressed.

A. ASTM Test Method D5470 [2]

The test method was developed for measuring "apparent (or overall) thermal conductivity" of thin composite materials at the steady-state heat flux condition. The schematic diagram of the ASTM D5470 fixture is shown in Figure 1. Heat flow is measured using a reference calorimeter (Method A) or a heater-sensor element (Method B). Specimens are compressed at 3 MPa (435 psi) for reducing the contact resistance between the test specimen and fixture surfaces.

ASTM D5470 states that "the test methods assume that specimen layers coalesce and that there is no effective interfacial resistance between layers [2]." However, surface characteristics such as compressibility and conformability (and thus thickness) of the most commonly used thermal interfaces for electronics vary considerably with the compression load. Thermal conductivity and contact resistance of thermal interfaces change significantly since densities of specimens at the test condition are altered from the original state. Therefore, the test results from D5470 do not present the thermal functionality of tested specimens under actual service conditions and thus might be invalid for design purposes.

The measurement system consists of several components, each of which contributes to the measured overall thermal impedance and influences the heat flux and temperature profiles. The thermal impedance associated with the test specimen needs to be isolated. This is addressed by assembling several multiple-layer specimens and constructing a correlation between the overall thermal impedance and the thickness of the entire specimen stack. The correlation should be linear and can be represented by a straight line in a plot with thickness on the x-axis and thermal impedance on the y-axis.

The intercept at zero thickness (R_I) represents the thermal interfacial impedance of the system. It accounts for the impedances resulting from the contact between specimens and fixture. It can also come from the interfacial contact between specimens of multiple-layer stacks. The differential between R_I and the measured thermal impedance accounts for the thermal impedance of a specimen of a specific thickness. The slope of the line is the reciprocal of the apparent thermal conductivity of test specimens.

The ASTM D5470 methodology provides a convenient and consistent way of differentiating "interfacial thermal impedance" and "body thermal impedance" of test specimens. However, under certain situations the relationship between the overall thermal impedance and thickness may deviate from linearity due to, for example, inconsistent system errors, non-linear compression of specimens under load, and variation of specimen surface characteristics due to thermal degradation. Therefore, the value (R_i) of the intercept at zero thickness does not necessarily represent the thermal contact resistance and may substantially differ from the "real" thermal interfacial impedance. Additionally, the range of error for the calculated thermal conductivity is not proportional to errors in the thermal impedance measurement because of their reciprocal relationship. Nevertheless, ASTM D5470 represents a reasonably good protocol for estimating through-body thermal impedance (or intrinsic thermal conductivity), if specimens are well prepared, the test fixture is well maintained, and measurement conditions are well controlled.

B. ASTM Test Method E1530 [3]

ASTM E1530 describes a steady-state method of determining the thermal resistance of thin specimens, with thermal impedance ranging from 0.04 to10 m^2K/W. The method is modified from Method C518 [4] and uses a similar heater/sensor element as described in Method D5470 (Method B) for measuring heat flow through specimens.

Method E1530 is a comparative (or secondary) method. The method determines the thermal conductivity (and thermal impedance or resistance) by matching $(T_L-T_u)/Q$ of test specimens with that of calibrated samples of known thermal properties. The comparative method is based on the following equation:

$$R_s = \frac{N\,(T_L - T_u)}{Q} - R_o \qquad \text{Eq. (3)}$$

The tests are conducted at 0.28 MPa (40 psi) compression load.

Figure 2 shows the schematic diagram of the ASTM E1530 measurement system. The test method assumes that the heat flow transducer calibration constant (N) and thermal contact resistance (R_o) remains unchanged when different types of specimens are tested. However, the value of N might vary slightly as the temperature changes, depending on the thermal properties of the test specimen. More importantly, the value of R_o is a strong function of the material properties including the compression modulus, surface roughness and flatness, and hardness.

For a number of semi-rigid thermal interface materials, the thermal contact resistance could significantly outweigh the intrinsic thermal resistance, particularly when specimens are very thin. The value of R_o can also be overestimated for extremely soft materials, and thus the calculated thermal resistance underestimates the "true" value. Assuming constant contact resistance (R_o) can be problematic. This assumption of constant R_o has to be carefully confirmed to verify the validity of the method for specific types of specimens. A convenient way to verify the validity of the assumption is to follow the ASTM Test Method D5470 by using stacks of multiple layered specimens. The value of R_o is independently estimated and subtracted from the measured overall thermal resistance, i.e., $N(T_L-T_u)/Q$. After "filtering" out R_o, the thermal conductivity of the test specimens is then compared to those of the calibration standards.

C. ASTM Test Method F433 [5]

This method is also similar in concept to Method C518 and measures the heat flow using a heat flow transducer (HFT). Like Method E1530, Method F433 is a comparative method of measurement. Thermal conductivity of test specimens is calculated or interpolated in reference to the calibration standards of known properties by the following equation.

$$k_s = k_r \frac{\phi_s}{\phi_r} \frac{\Delta x_s}{\Delta x_r} \frac{\Delta T_r}{\Delta T_s} \qquad \text{Eq. (4)}$$

In Method F433, temperatures on the surfaces of the test specimen and heater plates are measured and used to calculate the thermal contact resistance based on reference samples of known thermal conductivity. It should be noted that calculated values of the contact resistance are often sensitive to the location and dimension of temperature sensors or probes. More importantly, the thermal contact resistance between the test specimen and heater plates depends on the surface characteristics of test specimens. Since most of the reference specimens are rigid solids, e.g., copper, aluminum and glass, the assumption of a constant contact resistance is not valid for the majority of thermal interface materials, e.g., silicones, epoxy, polyurethane and flexible graphite. Again, a procedure similar to Method 5370 should be performed to quantify the thermal contact resistance of individual specimens before using Eq. (4) to calculate the thermal conductivity of specimens in comparison to the calibration standards.

IV. Design Considerations for Measurement of Thermal Conductivity

Modifications to the ASTM methods have been used as a qualification and inspection tool by some interface manufacturers and designers, in order to fit with specific materials or service conditions. Because most commercial thermal interfaces are thin, flexible and compressible, development of analytical systems for measuring the "true" thermal conductivity is not only tedious but also expensive. For simplicity (and to mimic the end use), thermal interfaces are often evaluated together with the entire thermal management system for the purpose of material screening and comparison. However, measurement sensitivity suffers when combining the thermal performance and characteristic of multiple components. The poor sensitivity makes it impossible to recognize the critical role of the thermal interface in the overall thermal management system. More importantly, results and observations based on the quick-cheap-relative measurement philosophy cannot be "extrapolated" for systems of different configurations and requirements.

A fundamentally valid and feasible thermal conductivity testing system needs to comprise five essential components: (1) The first component is for the temperature measurement to provide reliable and consistent readings. The physical presence of temperature sensors (e.g., thermocouples and thermistors) critically interferes with heat flow and thus temperature profiles. Minimum intrusion of the temperature sensors is one of the most important design aspects of the measurement system. (2) The second component deals with the direct or indirect measurement of heat flux through specimens. Quite often indirect heat flux measurement is adopted for its simplicity. Calibration of the heat flux through the measurement system needs to be carefully performed to ensure the accuracy of results and for estimation of the error range. (3) The third component relates to the compression mechanisms of the test apparatus. Thermal conductive characteristics of most thermal interfaces, particularly silicone-based materials, change to a significant degree with the external compressive stress. A consistent and high-quality compression mechanism is needed to insure the repeatability and sensitivity of test protocols. (4) The fourth component is required for in-situ measurement of specimen thickness. Especially with thin thermal interface, errors arising from the thickness measurement can affect the validity of the final measurement results. In-situ thickness measurement can also be used to monitor the mechanical integrity of the test specimen under the thermal and mechanical stresses of the service conditions. (5) Last, but not least, the analysis should be performed at thermal steady state for several apparent reasons. A transient system, such as the Laser flash method and the "Fitch" calorimetry method, requires additional material properties including density and heat capacity that introduce additional sources of errors. The transient methods require more sophisticated detection equipment and calculation algorithms. Most of all, transient methods are not suitable for analyzing materials with temperature-sensitive thermal and mechanical properties.

V. Examples of Analytical Techniques for Thermal Interface Evaluation

A thermal analysis system integrating concepts of ASTM Methods D5470, E1530, and F433 has been developed by the UCAR GRAPH-TECH INC. Thermal and Electrical Analysis Laboratory. The technique utilizes a fixture similar to that of Method D5470 and combines the methodologies of Methods F433 and D5470 for measuring the surface contact resistance. The steady-state heat flow through a specimen is measured with a reference calorimeter and is confirmed by the energy removal through a cooling plate. Errors due to the heat loss through insulated enclosure and signal noises of thermocouples can thus be assessed.

Surface temperatures on specimens are measured using 10-gauge (0.001-inch thick) thermocouples located in the interfaces between a test specimen and fixture platens, equivalent to the set-up of ASTM F433. Several proprietary designs have been evaluated and proven successful. Surface temperatures of the fixture platens are calculated by the extrapolation method as described in ASTM D5470. Combining these two methods of measuring temperatures in the interface, the intrinsic thermal conductivity of thin specimens is obtained without the interference of the thermal contact impedance. Additionally, consistency and reproducibility of the measurement system are greatly improved over previous versions and other types of analytical devices.

The measurement system is calibrated with solids of known thermal properties, such as copper and aluminum. Sample result sheets are illustrated in Figures 3 and 4 representing the thermal properties of flexible graphite and silicone-based thermal interfaces, respectively. Result sheets record all the required variables and test conditions. The measurement results clearly demonstrate distinct characteristics of the two extremely different thermal interfaces. Thermal contact resistance of flexible graphite is extremely low at very low compression loads, i.e. 30 psi. For certain types of silicone-based materials, however, contact resistance could be as high as, or even higher than, that of the through-body thermal resistance. These results highlight the importance of the total thermal impedance (or resistance) and material thermal conductivity when selecting suitable materials for thermal interface applications, or designing thermal management systems.

VI. Concluding Remarks

Principles and techniques of three ASTM test methods of measuring thermal conductivity and thermal impedance of thin thermal interfaces are discussed. Although some measurement limitations exist in these ASTM methods, they provide an excellent starting point for the development of thermal property test protocols. The key issue is to minimize the error associated with the assumptions related to the thermal contact resistance. This paper presents a fundamental and philosophical view of an ideal thermal conductivity measurement system. A measurement device and technique were developed that incorporates these ideals by modifying existing ASTM test methods. The developed technique provides a systematic means of minimizing measurement errors and increasing measurement sensitivity.

References

(1) M.N. Ozisik, *Heat Conduction*, 1980.
(2) "Thermal Transmission Properties of Thin Thermally Conductive Solid Electrical Insulation Materials," ASTM Test Method D5470-93.
(3) "Evaluating the Resistance to Thermal Transmission of Thin Specimens of

Materials by the Guarded Heat Flow Meter Technique," ASTM Test Method E1530-93.

(4) "Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus," ASTM Test Method C518-91.

(5) "Evaluating Thermal Conductivity of Gasket Materials," ASTM Test Method F433-77.

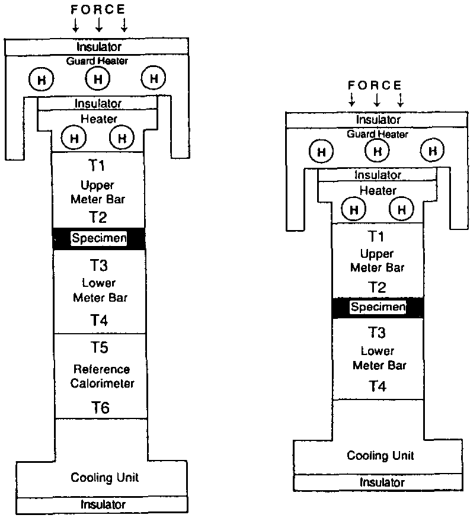


Figure 1. Schematic Diagram of ASTM D5470 Testing Fixture
Left: Method A; Right: Method B

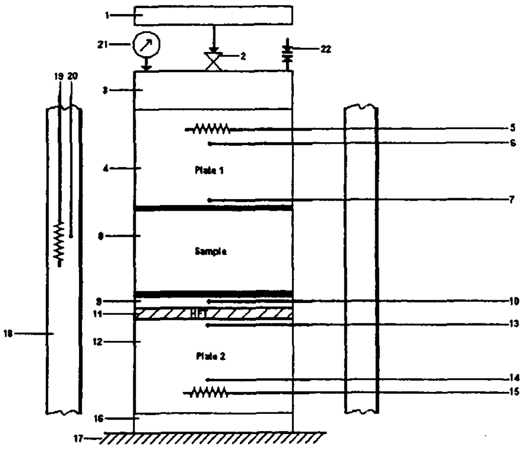
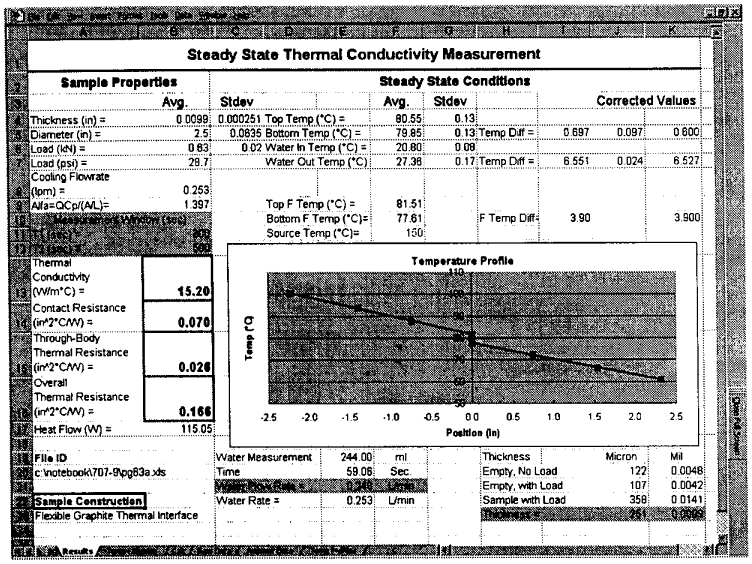


Figure 2. Schematic Diagram of ASTM D1530 Testing Fixture



Steady State Thermal Conductivity Measurement

Sample Properties	Avg.	Stdev	Steady State Conditions	Avg.	Stdev		Corrected Values		
Thickness (in) =	0.0099	0.000251	Top Temp (°C) =	80.55	0.13				
Diameter (in) =	2.5	0.0635	Bottom Temp (°C) =	79.85	0.13	Temp Diff =	0.697	0.097	0.600
Load (kN) =	0.63	0.02	Water In Temp (°C) =	20.80	0.08				
Load (psi) =	28.7		Water Out Temp (°C)	27.36	0.17	Temp Diff =	6.551	0.024	6.527
Cooling Flowrate (lpm) =	0.253								
Alfa=QCp/(A/L)=	1.397		Top F Temp (°C) =	81.51					
Measurement Window (sec)			Bottom F Temp (°C)=	77.61		F Temp Diff=	3.90		3.900
			Source Temp (°C)=	150					

Thermal Conductivity (W/m°C) =	15.20
Contact Resistance (in^2°C/W) =	0.070
Through-Body Thermal Resistance (in^2°C/W) =	0.026
Overall Thermal Resistance (in^2°C/W) =	0.166
Heat Flow (W) =	115.05

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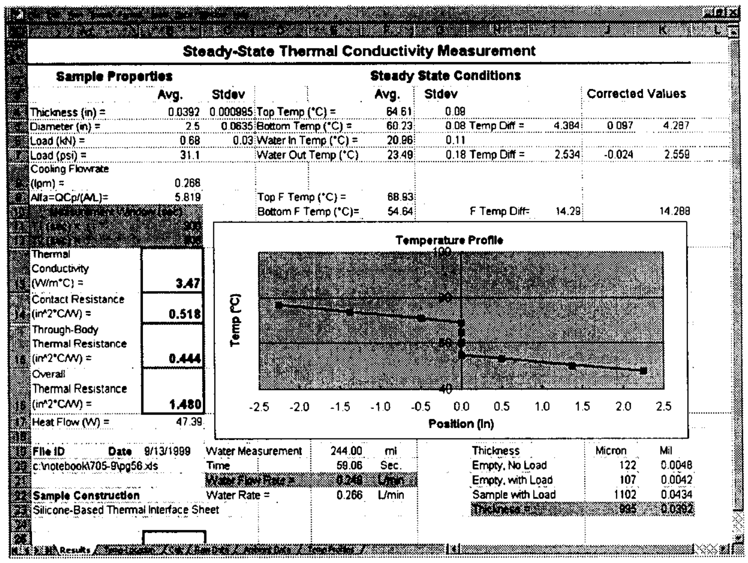
Sample Construction
Flexible Graphite Thermal Interface

Water Measurement	244.00	ml
Time	59.06	Sec.
Water Flow Rate =	0.248	L/min
Water Rate =	0.253	L/min

Thickness	Micron	Mil
Empty, No Load	122	0.0048
Empty, with Load	107	0.0042
Sample with Load	358	0.0141
Thickness =	251	0.0099

Figure 3. Sample Measurement Record Sheet for a Flexible Graphite Thermal Interface Sheet



Steady-State Thermal Conductivity Measurement

Sample Properties	Avg.	Stdev	Steady State Conditions	Avg.	Stdev		Corrected Values		
Thickness (in) =	0.0392	0.000985	Top Temp (°C) =	64.61	0.09				
Diameter (in) =	2.5	0.0635	Bottom Temp (°C) =	60.23	0.08	Temp Diff =	4.384	0.097	4.287
Load (kN) =	0.68	0.03	Water In Temp (°C) =	20.96	0.11				
Load (psi) =	31.1		Water Out Temp (°C)	23.49	0.18	Temp Diff =	2.534	-0.024	2.559
Cooling Flowrate (lpm) =	0.266								
Alfa=QCp/(A/L)=	5.819		Top F Temp (°C) =	68.93					
Measurement Window (sec)			Bottom F Temp (°C)=	54.64		F Temp Diff=	14.29		14.288

Thermal Conductivity (W/m°C) =	3.47
Contact Resistance (in^2°C/W) =	0.518
Through-Body Thermal Resistance (in^2°C/W) =	0.444
Overall Thermal Resistance (in^2°C/W) =	1.480
Heat Flow (W) =	47.39

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Sample Construction
Silicone-Based Thermal Interface Sheet

Water Measurement	244.00	ml
Time	59.06	Sec.
Water Flow Rate =	0.248	L/min
Water Rate =	0.266	L/min

Thickness	Micron	Mil
Empty, No Load	122	0.0048
Empty, with Load	107	0.0042
Sample with Load	1102	0.0434
Thickness =	995	0.0392

Figure 4. Sample Measurement Record Sheet for a Silicone-Based Thermal Interface Sheet