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Rapid thermal conductivity measurement with a hot disk sensor Part 2. Characterization of thermal greases

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Abstract

Thermal transport properties of several thermal greases with applications in electronic packaging have been characterized at room temperature using the hot disk technique, which is a transient plane source technique for rapid thermal conductivity and thermal diffusivity measurement. In this technique, the hot disk sensor serves as a heat source and a thermometer. During the measurement, the sensor is sandwiched between two halves of a sample, and a constant current is supplied to the sensor. The temperature increase at the sensor surface is strongly dependent on the thermal transport properties of the surrounding material. By monitoring the temperature increase as a function of time, one can determine the thermal conductivity and the thermal diffusivity of the surrounding material with high accuracy. The results on thermal greases are in good agreement with supplier data, which were obtained by another method.

Keywords: Thermal conductivity; Hot disk technique; Thermal interface material; Thermal grease

1. Introduction

The management of heat flow is a major concern for many industries. In semiconductor industry, as the speed of microprocessors increases continuously and the characteristic size of a transistor shrinks steadily, a key challenge facing microelectronic package design is thermal management, or how to remove heat away from the microprocessors. Without an efficient way to remove heat away from the microprocessor, package reliability and component service life will face serious degradation. It was estimated that for every 10 °C increase in the processor core temperature, its service life is reduced by 50% [1]. A common method to aid heat dissipation away from the silicon chip is to use heat spreaders and heat sinks [2], as illustrated in Fig. 1. Heat spreaders and heat sinks are made of highly conductive metals, alloys, and composites, such as Cu, Al, Cu–W, Al–SiC, and Ag–Cu [3], etc. To achieve effective heat dissipation, a low thermal interface resistance between the silicon chip and the heat spreader or the heat spreader and between the heat sink is also extremely important. For this purpose, highly conductive thermal interface materials (TIMs) must be applied to these interfaces. These materials are used to fill the voids and grooves in the interfaces, effectively reducing the thermal contact resistance of these interfaces, thus enhancing heat conduction between the silicon chip and the outside environment. A critical property of a TIM is its thermal conductivity.

There are several types of TIMs widely used in semiconductor industry. They can be classified as phase change materials, conductive elastomers, thermal gels, and thermal greases. Among them, thermal greases have several advantages: they usually have good wettability, do not require cure, they are easy to process and cost-effective, and they have excellent thermal performance. The main disadvantage of grease materials is that they tend to flow out or "pump-out" of the interfaces, especially when the package is under cyclic thermomechanical stresses during temperature cycling. Because of this reason, thermal greases are not used as the primary but as the secondary interface materials (Fig. 1). This is because the primary interface, which is the

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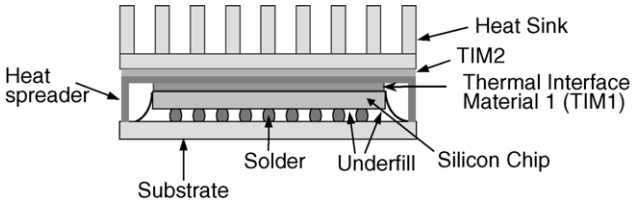

Fig. 1. Illustration of a microelectronic package. To assist heat conduction, primary thermal interface material is applied between the silicon chip and the heat spreader, and secondary thermal interface material is applied between the heat spreader and the heat sink.

interface between the silicon chip and the heat spreader, is usually a metal–Si interface. It suffers from higher stresses due to higher mismatch of the coefficients of thermal expansion (CTE) between the metal and silicon. On the other hand, the interface between the heat spreader and the heat sink, which is usually a metal–metal or metal–composite interface, will have lower stresses because of lower CTE mismatch. Therefore, thermal greases are good candidates as secondary thermal interface materials.

In this work, we examined the thermal transport properties of several thermal greases used in electronic packaging. These properties, together with thermal contact resistance, are critical in developing and screening new TIMs [4].

Although a variety of techniques are available for measuring thermal conductivity, many of them suffer from limitations such as time-consuming, expensive to set up, low accuracy, or limited measurement range of thermal conductivity. The hot disk technique [5–8], which is a transient plane source method, represents a metrology that can be used to measure thermal conductivity and thermal diffusivity of a wide variety of materials rapidly and with high accuracy. This technique is based on using a metal strip or disk as a continuous plane heat source as well as a temperature monitor. The sensor is sandwiched between two thin polyimide films for electrical insulation. During a hot disk measurement, the sensor is sandwiched between two halves of a sample. By supplying a constant electric current to the hot disk sensor, the output heating power is nearly constant. The temperature change in the sensor itself is highly dependent on the thermal transport properties of the material surrounding the sensor, and this temperature change can be accurately measured by measuring the electric resistance across the hot disk sensor. By monitoring the temperature increase over a short period of time after the start of the experiment, it is possible to obtain precise information on the thermal transport properties of the material surrounding the hot disk sensor.

It can be shown that during a hot disk measurement, the average temperature increase of the sensor can be expressed as [6,7,9,10]:

$$\Delta\bar{T}(\tau) = \frac{P_0}{\pi^{3/2} a K} D(\tau), \quad (1)$$

where P_0 is the output power to the sensor, a the sensor radius, K the thermal conductivity of the sample, and $\tau = \sqrt{\kappa t}/a$ is a dimensionless parameter called characteristic time ratio, κ the thermal diffusivity of the sample, and t the time. $D(\tau)$ is a rather complicated function, but can be evaluated numerically with high accuracy. If one knows the relationship between t and τ, one can plot $\Delta\bar{T}$ as a function of $D(\tau)$, and a straight line should be obtained. From the slope of this line, thermal conductivity K can be calculated. However, the proper value of τ is generally unknown, but it can be determined through a series of optimization process [6,7,9,10]. In practice, we can measure the density ρ and the specific heat C_p separately, so that between K and κ, there is only one independent parameter. Therefore, both thermal conductivity and thermal diffusivity of the sample can be determined.

The hot disk technique has proven to be a highly effective and accurate way to measure both thermal conductivity and thermal diffusivity of various materials. In our laboratory, this technique has been applied to measure the thermal conductivity of two materials with well-known thermal conductivity values, i.e. stainless steel and Pyrex® glass. Repeated measurements revealed that the results obtained by the hot disk technique are within 2% of the established literature values [11].

In this study, thermal transport properties of several thermal greases, which are used as secondary thermal interface materials in microelectronic packaging, were evaluated. The specific heat of each material was determined using differential scanning calorimetry (DSC). The thermal conductivity and thermal diffusivity of these greases materials were determined at room temperature (23 °C) using the hot disk technique. Thermal conductivity results obtained from the hot disk measurements are in good agreement with the results provided by the material supplier, which were obtained by a different method (hot wire technique).

2. Experimental

2.1. Materials

The thermal greases used in this study are code-named G9, G1, and S78, respectively. All three materials are classified as organopolysiloxane mixtures with high filler content. Upon heating, these greases are not curable and they remain in the paste form. Table 1 lists the general properties of these materials. The detailed chemistry and microstructure contain proprietary information and cannot be presented in this work.

Table 1
Supplier data on general properties of three thermal greases used in this study

Properties	G9	G1	S78
Filler content (wt.%)	89.5	90.7	88
Density, ρ (g/cm^3)	2.76	2.50	2.34
Specific heat, C_p (J/g K)	0.767	0.876	0.906

Density and specific heat were determined at room temperature.

2.2. Hot disk measurement

To prepare samples for the hot disk measurements, the grease material was dispensed into a cylindrical plastic container. In our experiments, we found that plastic bottle caps can be used as sample container. The depth and the diameter of the container are 10 and 40 mm, respectively. During the measurement, the hot disk sensor was sandwiched between two plastic caps filled with the grease material. Due to the high viscosity of these materials, the overflow of the material in the top container was minimal during the test. The applied output power to the hot disk sensor was 0.5 W and the typical measurement time was 2.5–5 s. The diameter of the hot disk sensor used in our measurements was 3.3 mm. The effects of possible temperature drift during the measurement, thermal contact resistance between the sensor and the sample (including the thermal resistance of the polyimide film), instrument time delay, and the specific heat of the sensor itself were all corrected when the thermal conductivity and the thermal diffusivity of the sample were calculated.

2.3. Density

Using buoyancy test results provided by the material supplier, the densities of G9, G1, and S78 were determined to be 2.76, 2.50, and 2.34 g/cm^3, respectively, as listed in Table 1.

2.4. Specific heat

The specific heat, C_p, of each material was measured using a Perkin-Elmer DSC7 system with a sapphire single crystal as the C_p standard. The principle of the C_p measurement using DSC has been described elsewhere [12,13]. The mass of the sapphire standard was 61.37 mg, and the typical sample mass was 60–90 mg. The thermal program used for the C_p measurement was:

1. Isothermally held at 25 °C for 5 min;
2. Ramp at 10 °C/min to 100 °C;
3. Isothermally held at 100 °C for 3 min.

To start with the specific heat measurement, a baseline heat flow curve was first measured with empty aluminum pans. Then, a sapphire standard was placed in the sample pan and the heat flow curve was measured again using the same set of DSC pans. Finally, the heat flow curve for the sample was measured using the same DSC sample pan. The specific heat of the material can then be calculated as [12,13]:

$$C_{p1} = \frac{m_2 y_1}{m_1 y_2} C_{p2}, \tag{2}$$

where C_{p1} and C_{p2} are specific heat of the sample and sapphire standard, respectively, m_1 and m_2 are the sample mass and the mass of the sapphire standard, y_1 and y_2 are the net heat flow of the sample and the sapphire, respectively.

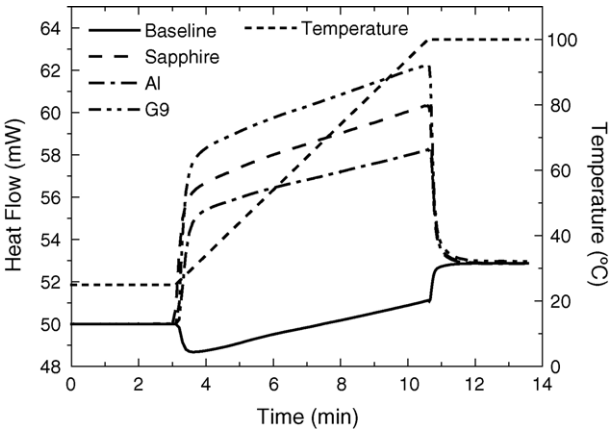

Fig. 2. DSC heat flow curves for the empty pan baseline, sapphire standard, pure Al, and G9 thermal grease. The sample weight for the sapphire, Al, and G9 were 61.37, 46.08, and 73.17 mg, respectively. The heating rate was 10 °C/min.

3. Results and discussion

3.1. G9 thermal grease

Fig. 2 shows the typical DSC heat flow curves obtained during the specific heat measurement of G9 grease. The empty pan baseline, sapphire single crystal standard, high purity aluminum, and G9 grease were measured using the same set of DSC pans. The specific heat of G9 was plotted as a function of temperature in Fig. 3. Based on the experimental data, the specific heat of G9 can be expressed by

$$C_p = 0.7442 + 0.0018\,T, \tag{3}$$

where C_p has the units of J/g K and T is in °C. Therefore, at room temperature of 23 °C, C_p = 0.786 J/g K, which is in excellent agreement with the supplier data listed in Table 1. Similar analysis revealed that for pure Al, the specific heat at 23 °C is 0.89 J/g K, which is in excellent agreement with

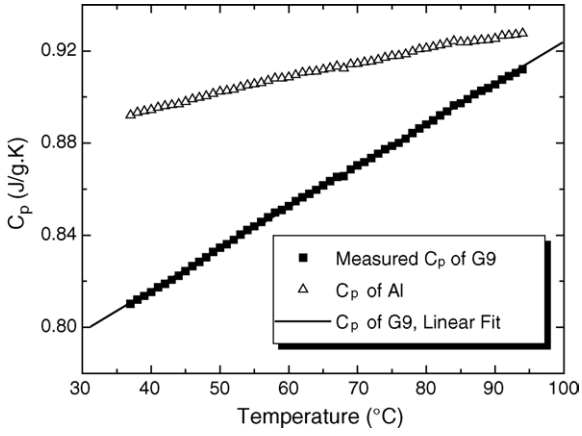

Fig. 3. Square symbols represent the measured specific heat of G9 thermal grease and the line is the linear fit of the experimental data. C_p of high purity Al was also plotted.

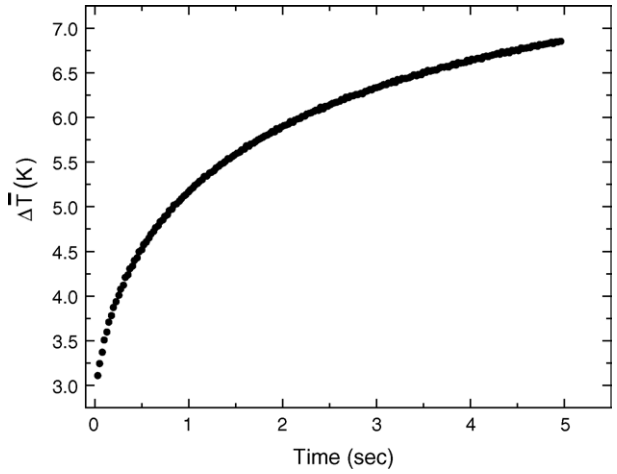

Fig. 4. Average increase of sensor temperature ($\Delta\bar{T}$) as a function of time during a typical hot disk measurement of the G9 thermal grease. The measurement was done at room temperature (23 °C) and the output power of the hot disk sensor was 0.5 W.

the literature data of 0.90 J/g K [14]. This indicates that the specific heat measurement by DSC is quite accurate.

Based on our data, the volumetric specific heat of this material was determined to be $\rho C_p = 2.17 \times 10^6$ J/m^3 K. This number will be used in calculating the bulk thermal conductivity of G9 grease.

Fig. 4 plots the average temperature increase of the hot disk sensor as a function of time during a typical thermal conductivity measurement for G9 grease. Fig. 5 shows the temperature increase as a function of $D(\tau)$ after necessary corrections. In generating this figure, the first 10 points from the raw data were excluded, so that the influence of the insulating layer of the sensor can be eliminated [15]. Based on Fig. 5, the bulk thermal conductivity of G9 was determined to be 2.85 W/m K, and the thermal diffusivity was 1.32 mm^2/s. During the measurement time selected to plot Fig. 5, the probing depth [6,7,9,10] was estimated to be 4.4 mm, well within the available probing depth of about 10 mm. Therefore, the influence of sample boundaries on the measurement results is negligible.

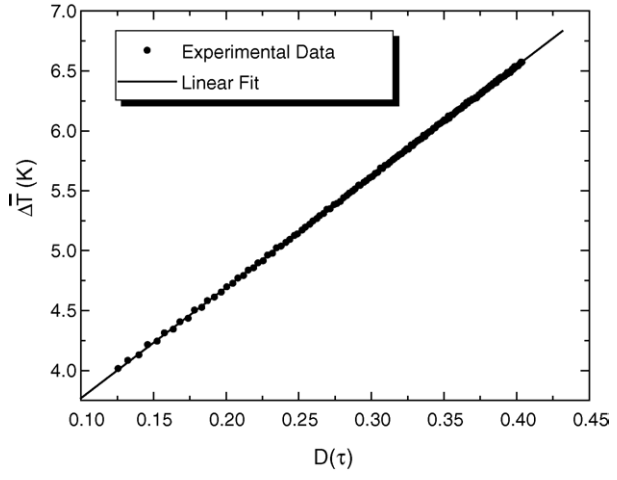

Fig. 5. Filled circles: experimentally determined $\Delta\bar{T}$ as a function of $D(\tau)$ for the G9 thermal grease based on the results from Fig. 4. The straight line is the linear fit. The correlation coefficient R for the fit is 0.9995, indicating that the fit is excellent.

Table 2
Room temperature (~23 °C) thermal conductivity and thermal diffusivity results of three thermal greases obtained from the hot disk measurements

	G9	G1	S78
K (W/m K)	2.87 ± 0.01	5.00 ± 0.03	3.08 ± 0.01
κ (mm^2/s)	1.32 ± 0.01	2.25 ± 0.01	1.38 ± 0.002
Supplier K (W/m K)	2.83	4.80	3.325

The mean values and standard deviations were calculated based on 10 repeated measurements on the same sample. Supplier data from the hot wire technique are also listed for comparison.

Based on 10 repeated measurements on the same sample, the average value of the thermal conductivity of G9 was 2.87 W/m K, and the average thermal diffusivity was 1.32 mm^2/s, as listed in Table 2. Our results are in excellent agreement with the one provided by the supplier.

3.2. G1 thermal grease

The resin chemistry of G1 thermal grease is similar to that of G9, but the filler content of G1 is about 1 wt.% higher. In addition, G1 uses a different material as one of the main filler. Because of these differences, the density and thermal conductivity of G1 are drastically different from that of G9.

Based on the DSC results, the temperature dependence of the specific heat of G1 can be expresses as

$$C_p = 0.8732 + 6.73 \times 10^{-4} T, \quad (4)$$

where C_p is in J/g K and T is in °C. The correlation between the experimental data and the linear fit was excellent, as indicated by a correlation coefficient of 0.99988. At 23 °C, C_p = 0.889 J/g K, again in excellent agreement with the supplier data listed in Table 1. Thus, the volumetric specific heat was 2.22×10^6 J/m^3 K, which is quite close to the value of G9.

Using the hot disk technique, the average sensor temperature increase, $\Delta\bar{T}$, was obtained as a function of measurement time in a similar way as described above. From this result, $\Delta\bar{T}$ versus $D(\tau)$ curve was plotted, from which the bulk thermal conductivity of G1 was determined to be 4.97 W/m K at room temperature, and the thermal diffusivity was 2.24 mm^2/s. The average values of K and κ based on 10 repeated measurements were listed in Table 2. Again, our result on K is in excellent agreement with the supplier data. Our results indicated that changing the filler composition and loading has a big impact on the bulk thermal conductivity of the thermal grease.

3.3. S78 grease

This thermal grease has approximately 88 wt.% of various fillers. Based on the DSC measurements, the specific heat of

S78 can be fitted as

$$C_p = 0.9054 + 1.17 \times 10^{-3} T, \quad (5)$$

where C_p is in J/g K and T in °C. The correlation coefficient for the fit is 0.9998. At 23 °C, C_p = 0.932 J/g K and the volumetric specific heat was 2.18×10^6 J/m^3 K, which will be used in calculating the bulk thermal conductivity of the material.

Similarly, based on the $\Delta\overline{T}$ versus $D(\tau)$ curve, the bulk thermal conductivity of S78 thermal grease was determined to be 3.02 W/m K, and the thermal diffusivity was 1.38 mm^2/s. Based on 10 repeated measurements on the same sample, the average room temperature values for K and κ were determined to be 3.08 W/m K and 1.38 mm^2/s, respectively, as listed in Table 2. Our measured thermal conductivity value is about 8% lower than the supplier data. The main reason for this discrepancy may be attributed to the presence of voids in the S78 hot disk sample. In the G9 and G1 greases, the presence of such voids was minimal and their effect negligible. In S78, a small number of voids can be observed by visual inspection. These voids were homogeneously dispersed in the sample, thus, the measured thermal conductivity was the effective thermal conductivity, which is lower than that of the true thermal conductivity of the grease.

4. Conclusions

Thermal transport properties of three thermal greases used as secondary thermal interface materials in microelectronic packaging were evaluated. Using DSC, the specific heat of each material was determined as a function of temperature. Thermal conductivity and thermal diffusivity of these materials were measured using the hot disk technique. The measured results are in good agreement with the supplier data, which were obtained using the hot wire technique. Among these greases, G1, which has more than 90 wt.% of highly conductive fillers, showed a thermal conductivity of 5.00 W/m K, which is rather high for thermal greases. The ability to provide quick thermal conductivity analysis of thermal interface materials has been very valuable in developing and screening new materials for thermal management applications in electronic packaging.

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