

The Effect of Test Conditions on HAST and THB Reliability Testing of Thermal Interface Material, and the Introduction of an Improved Thermal Interface Material

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Abstract

As the world becomes more and more dependent on electronic devices the reliability of these devices increases in importance. Reliability testing of electronic components has been reduced to a rigorous practice, and it is well defined in the literature [12-15]. Of all the materials utilized by electronic devices, Thermal Interface Materials (TIMs) are particularly important to reliability. Since the TIM enables proper thermal management of the electronic components, if the TIM fails, the entire device fails.

The testing of the individual materials utilized in a specific electronic device represents a particular challenge above and beyond that of testing the entire device, and TIMs are no exception. During the development of TIMs, the actual electronic devices are typically unavailable so the TIM designers must replicate the expected conditions of the actual electronic device (i.e. surface materials, clamping pressure, and geometry). Furthermore, in most electronic devices TIMs are utilized for multiple conditions (e.g. TIM1 for silicon / heat spreader interface, TIM2 for heat spreader / heat sink interface), therefore, unique fixtures must be employed for each condition, requiring a separate reliability test for each condition.

Most modern TIMs display adequate performance just after assembly, but after reliability testing only the truly reliable TIMs maintain their performance. Furthermore, while a TIM may show excellent reliability performance in a fixture designed to replicate one condition of the electronic device, it may show marginal to poor reliability performance when tested for another condition. This paper will discuss the importance of test conditions during the THB and HAST reliability testing of TIMs, and introduce an improved TIM that was designed based upon these findings.

Keywords: Thermal Interface Material, HAST, THB, Phase Change Material, Thermal Test Vehicle

Introduction:

Thermal Interface Materials (TIMs) provide significant enhancement for heat transfer between two independent surfaces, and their reliable performance is critical for high-power microelectronic devices. During TIM development, these materials undergo significant reliability testing during which they are typically fixtured in a manner that allows for testing using the ASTM D5470 method [10]. After development, the TIM is further tested using a Thermal Test Vehicle (TTV) to more closely replicate the actual physical conditions of the application. Occasionally, this change of test conditions has a significant effect on the materials reliability performance. Unfortunately, successful reliability performance from the ASTM D5470 test conditions does not always translate into successful TTV performance.

Nevertheless, reliable TIM performance under TTV conditions is ultimately a requirement for many TIM applications. In order to develop these materials, TIM designers need an economical test that accurately replicates the TIM's ultimate operating conditions. TTV's accurately represent these ultimate conditions; however, TTV's can vary greatly with application and are relatively expensive. Therefore, determining a low-cost general technique for replicating TTV results is important.

TTVs closely represent the device and accurately replicate its important properties. In order to replicate TTV results, one must replicate the key properties of TTV behavior that affect the TIM. Furthermore, these properties need to be replicated in a manner that allows the TIM's performance to be measured, and with materials that can withstand the appropriate reliability tests.

For device suppliers, TTV's are a part of the design cycle and are therefore budgeted for and available. They then select an appropriate TIM and test it. If the TIM fails, they often have multiple alternative options. Conversely, for TIM suppliers, TTV's are expensive and diverse so it is difficult to incorporate TTV's into their design cycle. TIM suppliers typically rely on their customers for TTV testing. If their TIM fails a customer's TTV qualification, they may not have another chance. Furthermore, they may not receive any information for corrective action.

In this study, TIM HAST performance from ASTM D5470 conditions and TTV's were compared and found to differ drastically. The TIM material passed HAST under the D5470 conditions but failed under the TTV conditions. Therefore a new approach was

developed using flash diffusivity to measure TIM HAST performance. This allowed for a setup with conditions that more closely matched the TTV conditions. With this new setup, TTV HAST performance was replicated. Finally, this new approach was used to further develop the TIM so as to improve its HAST performance, and this improvement was also verified on the TTV.

Background:

The concept of Contact Resistance and its minimization through the use of interfacial materials is introduced in undergraduate heat transfer textbooks [1]. Phase Change Thermal Interface Materials (PCM TIMs) have gained industry approval and are certainly considered a reliable option for minimizing contact resistance in electronic systems [2,3]. PCM TIMs are typically composed of relatively high-conductivity particles (filler) suspended in a matrix that exhibits phase change near the operational temperature of the electronic device. The phase change property allows for good handleability at temperatures below phase change while enabling excellent compliance/wetability at temperatures above phase change.

This ability to comply to and wet non-uniform surfaces reduces the contact resistances into and out of the PCM TIM. One measurement of performance for a PCM TIM is the thermal resistance ($^{\circ}\text{C}\cdot\text{cm}^2/\text{W}$), and this is typically measured with a device that utilizes the ASTM D5470 standard [10]. The ASTM D5470 method allows for the measurement of bulk thermal resistance of a material and the contact resistances entering and exiting the material (see Figure 1). To accomplish this, a material of known conductivity (e.g. copper) is machined to a uniform cross section and outfitted with thermocouples at two or more known locations (see Figure 1). The material of interest, in this case TIM, is positioned between two of the previously-mentioned machined blocks, and heat is transferred axially through the assembly (see Figure 1).

Once the system reaches steady state thermal equilibrium, the values of the positioned thermocouples are used to calculate the heat flow and predict the temperature at the surfaces of the machined blocks. The ratio of this temperature difference to the heat flow is the thermal resistance (Equation 1). Equation 1 is a linear equation with a slope of $1/k_{\text{TIM}}$ and a y-intercept equal to the sum of the contact resistances (see Figure 2). By measuring the total thermal resistance for several different thicknesses of TIM, one can further clarify the

resistance of the bulk from the sum of the contact resistances.

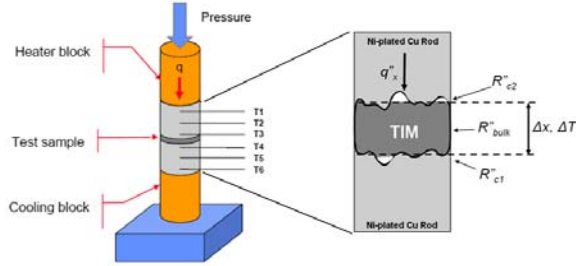


Figure 1: TIM thermal resistance measurement

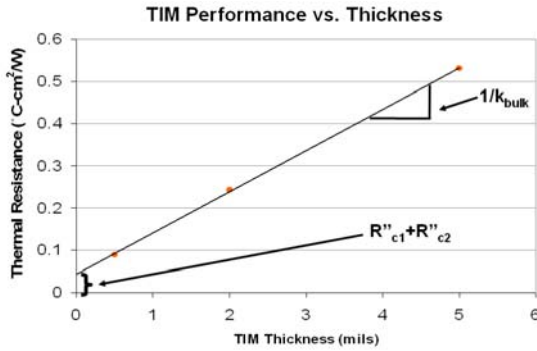


Figure 2: TIM thermal performance

$$R''_{TIM} = \frac{\Delta T}{q''_x} = \frac{\Delta x}{k_{bulk}} + R''_{c1} + R''_{c2} \quad (1)$$

Another way to determine the thermal resistance of a TIM is with the Flash method (also known as the Flash Diffusivity method and described in ASTM E1461-01 [11]). This method is based upon one dimensional transient conduction described by Equation (2) [9].

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha} \cdot \frac{\partial T}{\partial t} \quad (2)$$

Equation 2 is governed by a combined bulk material property called the thermal diffusivity, Equation (3).

$$\alpha = \frac{k}{\rho \cdot c_p} \quad (3)$$

The Flash method consists of a radiant heat source and a precise infrared temperature detector. The sample of interest is flashed with radiant energy on one side and its temperature is monitored on the opposite side (see Figure 3). This data set is then fitted to the solution of Equation (2) using the thermal diffusivity. Once the thermal diffusivity is known, the thermal conductivity can be calculated using Equation (3) (density and specific heat must also be determined in separate tests).

This method can also be extended to multiple stacked samples [9]. For TIM measurement, the TIM is assembled between two layers of known material (i.e. silicon and copper) to create a 3-layer sandwich (see Figure 3). This sandwich is then measured using the previously-described technique. If the properties of the two outer layers are known, the thermal diffusivity of the TIM (plus contact resistances) can be calculated. Further clarification of the data can be obtained by measuring samples with different TIM thickness (as previously described).

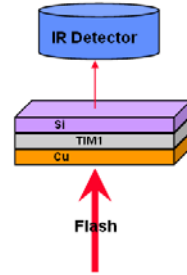


Figure 3: The Flash method

Next to testing TIM in its actual application, testing TIM using a thermal test vehicle (TTV) provides the best representation of TIM performance. TTV's are typically designed to represent an actual processor, but they can also be generic. For the case of a flip-chip processor, the TTV will look something like Figure 4.



Figure 4: A typical thermal test vehicle (TTV)

The TTV's processor is a piece of silicon that has been fabricated with resistors for heat generation and temperature sensors. These heaters and sensors can be arrayed to provide non-uniform heating and multiple temperature sense points. The silicon itself is a mechanical replica of the processor it was designed to represent, including any metallization schemes on the backside. TTV's can be used to test multiple TIM applications. Figure 5 shows a cross section of a FCBGA with a heat spreader (the cross section of a TTV would look identical). In this case the TTV could be used to test the TIM1 (TIM

between the die and the heat spreader) and/or the TIM2 (TIM between the heat spreader and the heat sink). For the work presented here, the TTV was utilized to test the TIM1.

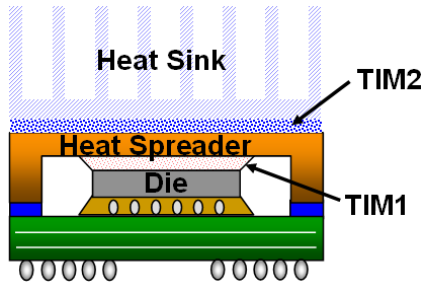


Figure 5: FCBGA cross-section

At least as important as handleability and initial thermal performance is reliable performance over the life of the device. Since PCM TIMs have been in use for well over 10 years, time itself has proven the reliability of these materials for their existing applications; however, this was not by chance. Typically these materials are put through multiple reliability tests to ensure their sustained performance [2]. In particular, two related reliability tests are Temperature Humidity (TH) and Highly Accelerated Stress Test (HAST).

In preparation for these reliability tests, the PCM TIM material must be fixtured in manner that suitably replicates its actual working environment while still allowing for measurement of thermal performance. If performance is to be measured using the ASTM D5470 method, then one technique for fixturing is to devote a set of machined copper blocks to each TIM sample (see Figure 1). The TIM is assembled between the blocks, appropriately conditioned (in the case of a PCM, it is thermally cycled one time through phase change), and its thermal performance is measured. This block-TIM assembly may then be maintained with a spring/clamp mechanism to replicate the constant pressure environment experienced by most TIMs and placed in a reliability chamber for testing. After reliability testing, the block-TIM assembly can once again be thermally tested and compared with previous thermal performance data. This is one of the fixturing techniques used to obtain the data presented here.

TH and HAST

The TH reliability test was designed to test a material's resistance to moisture-related degradation and failure. JEDEC test method A101-B calls for test conditions of 85 °C and 85% relative humidity for

1000 hours [12]. The 85 °C, 85% RH condition allows for some acceleration with little risk of saturated moisture. It is important to avoid saturated conditions during these tests because condensed water contacting the parts introduces a change in failure mechanism [4].

While the TH reliability test does represent an accelerated test, 1000 hours (42 days) is a relatively long time so a means of further acceleration was desired. In order to increase the temperature above 100 °C while maintaining relative humidity of 85%, the pressure inside the test chamber must increase from sea level conditions. Therefore, in order to further accelerate TH testing by increasing temperature, a special version of the test was introduced by F. N. Sinnadurai and originally named the "non-saturating autoclave" [5]. Sinnadurai attributes the acronym "HAST" to "imaginative reliability engineers" [6].

Perhaps the most familiar version of HAST is described in JESD22-A118 test condition A as 130 °C, 85% RH, for 96 hours, however, there is also test condition B (110 °C, 85% RH, 264 hours), and Sinnadurai defines HAST as anything above 100 °C (any TH test requiring above atmospheric pressures). Sinnadurai et al at BT Labs performed extensive testing on integrated circuits, hybrid microcircuits, and thick film circuits between 1968 and 1990. Using their experimental data, they developed the S-H expression [7]:

$$\frac{t_s}{t_{ambient}} = e^{\left(-0.00044[RH_s^2 - RH_{ambient}^2] + 7000 \left[\frac{1}{T_{ambient}} - \frac{1}{T_s} \right] \right)} \quad (4)$$

In Equation (4), the "s" subscript indicates the accelerated condition while the "ambient" subscript represents the condition one is accelerating to. The units for Equation 4 are hours for time (t), % for RH, and K for temperature (T). It is important to note that Equation 4 is only valid for temperatures up to 130 °C and unsaturated vapor up to 99% [7]. Sinnadurai lists the following three regions as "sheltered but otherwise uncontrolled climates in the temperate and tropical regions averaged over 10 years" [4].

Region	Climate
Temperate (Europe, North America)	12 °C & 72% RH
Sub-tropical India (99% of locations)	29 °C & 86% RH
Full Tropical (Rainforest regions)	35 °C & 90% RH

Table 1: Sinnadurai's three regions

Sinnadurai states that the stress condition for the most comprehensive humidity and HAST tests, conducted over 20 years at BT Labs, was 108 °C, 90% RH, 2 atm. However, Sinnadurai performed tests from as low as 40 °C and 50% RH up to 150 °C and 95% RH. From this work he asserts that the limit of validity of genuine acceleration of aging was found to be 130 °C and 95% RH. Furthermore, he states that above 130 °C, polymers exposed to high humidities are found to de-bond. Finally, Sinnadurai also recommends 50 hours as minimum test duration (to allow for the induction time for moisture permeation required for the failure mechanisms may occur).

Experimental Procedures Part I:

In the first part of this work, a prototype PCM material, PCM45G, was tested first with a setup that allowed measurement via the ASTM D5470 method [10]. Samples were constructed, measured at time zero, subjected to JEDEC HAST A (130 °C, 85 % RH, 96 hours), and then measured again. In addition, 2 TTV's were assembled with PCM45G, measured, subjected to JEDEC HAST A, measured again, and disassembled for failure analysis [13].

Condition 1:

For the setup measured with the ASTM D5470 method, the PCM45G was assembled between a pair of 25.4 mm diameter copper blocks with 3 uniformly spaced thermocouples at known positions. A 50.8 µm wire was used to maintain the thickness of the PCM45G between the blocks. The initial performance was then measured using the ASTM D5470 method. Immediately afterwards each pair of blocks was clamped with a devoted clamping fixture that maintained 40 psi of pressure at all times. The assembled pair of copper blocks maintained with the clamping fixture was then subjected to JEDEC HAST A [13]. After HAST, the assembled copper-block pair was carefully removed from the clamping fixture, and the TIM performance was again measured using the ASTM D5470 method.

Condition 2:

In addition the PCM45G was tested as a TIM1. Two generic FCBGA TTV's were assembled with PCM45G and appropriate nickel-plated copper heat spreaders in a manner in which the TIM thickness was maintained at 50.8 µm. The performance of the TIM was measured by first assembling the TTV into a fixture that enabled the TTV to be powered and cooled. The TTV was then powered and allowed to reach steady state equilibrium. Finally, the power input to the TTV, temperature of the TTV's junction and the temperature of the surface of the heat spreader were measured. The TTV's were then subjected to JEDEC HAST A and their thermal performance was measured again [13].

Experimental Procedures Part II:

Condition 3:

As will be explained later in the Results section, the HAST results from Condition 1 did not correctly predict the HAST results from Condition 2. Therefore a different approach was developed in an attempt to create a test condition that would correctly predict TTV HAST performance. To accomplish this, the TIM thermal performance was instead measured using the Flash Diffusivity Method and a 3-layer sandwich [8, 9]. A 12.7 mm x 12.7 mm x 0.8 mm thick piece of silicon was used to represent the processor, and a 12.7 mm x 12.7 mm x 1.0 mm thick piece of nickel plated copper was used to represent the heat spreader.

The TIM was assembled between these and cycled through phase change using an assembly fixture that ensured a uniform thickness of 50.8 µm. The sandwich assembly was then coated uniformly with graphite to ensure predictable surface optical properties (for absorption and emission of radiation during Flash testing). Next the sandwich was placed in the Netzsch NanoFlash™ and measured at 60 °C. After measuring the initial performance of all the sandwiches for the test, the samples were either clamped with a clamping force of either 6 lbf or left unclamped (0 lbf) and run through JEDEC HAST A [13]. Finally upon completing HAST, the samples were inspected to ensure the graphite coating was intact and once again measured using the Netzsch NanoFlash.

Results and Discussion:

Figure 6 shows the thermal performance of PCM45G tested under test condition 1 before and after JEDEC HAST A [13]. The TIM performance actually improves slightly through HAST. It is hypothesized that the contact resistances are reduced during

prolonged exposure to high temperature and clamping force.

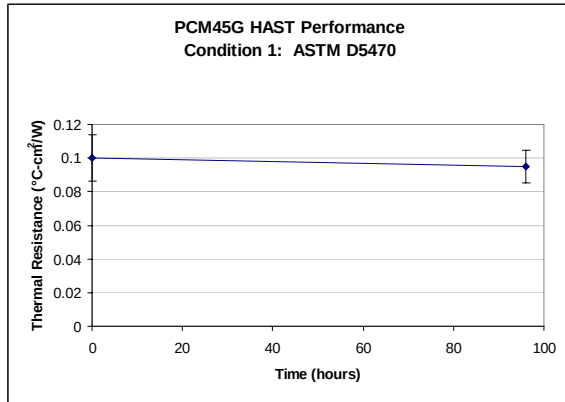


Figure 6: HAST performance for condition 1

Figure 7 shows the thermal performance of PCM45G tested in the two TTVs (test condition 2) before and after JEDEC HAST A [13]. The TIM performance degrades by nearly 75%. The TTVs were carefully disassembled after the HAST test and the TIM was observed to have degraded during the test (see Figure 8). The mechanism of this degradation will not be discussed in this paper.

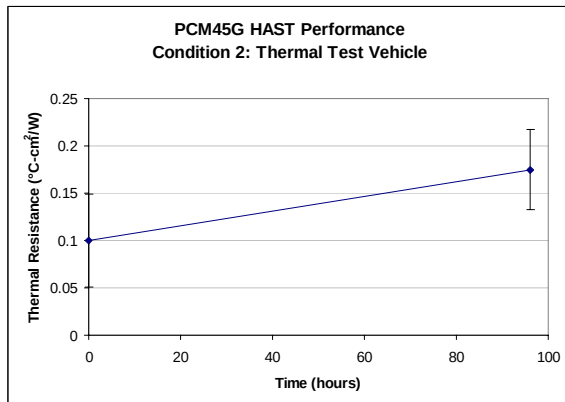


Figure 7: HAST performance for condition 2



Figure 8: TTV TIM at die and heat spreader surfaces after HAST testing

Figure 9 shows the thermal performance of PCM45G tested under test condition 3 before and after JEDEC HAST A [13]. For the unclamped parts, there is significant degradation after 48 hours and catastrophic degradation after 96 hours of HAST. For the clamped parts the degradation was still

significant after 96 hours of HAST, but not nearly as drastic as the unclamped parts. It is expected that increased clamping would further improve HAST performance, however, 6 lbf was the maximum possible for our setup (4, 1.5 lbf Conner clips are all that fit on the 12.7 mm x 12.7 mm parts).

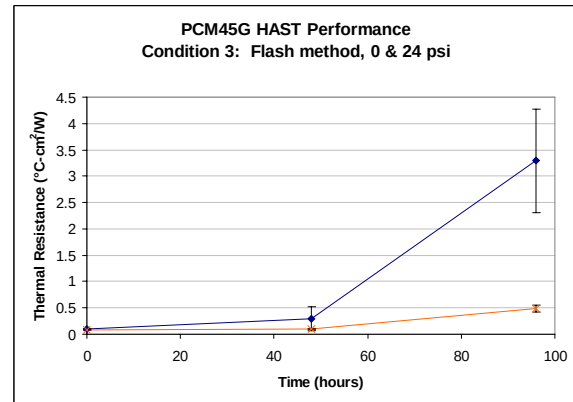


Figure 9: HAST performance for condition 3, Flash method at 0 and 24 psi

While the results of Condition 3 don't match the results of Condition 2 exactly, the trend is similar. Therefore, Condition 3 was utilized to test the HAST performance of new formulations of TIM. Eventually a formulation was discovered that provided excellent HAST performance even at 0 psi (unclamped) (see Figure 10). This new formulation, called PTM3180¹, was also tested using TTVs (condition 2) where the improvement in HAST performance was verified (see Figure 11).

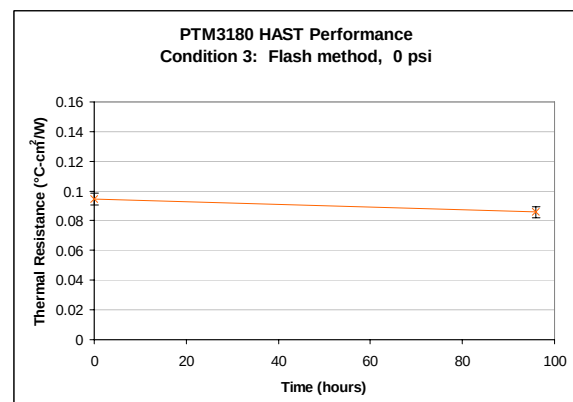


Figure 10: PTM3180¹ HAST performance for condition 3, Flash method

¹ Honeywell (March 17, 2009) "Honeywell Develops New High-Performance Thermal Management Material for Semiconductors". Press release.

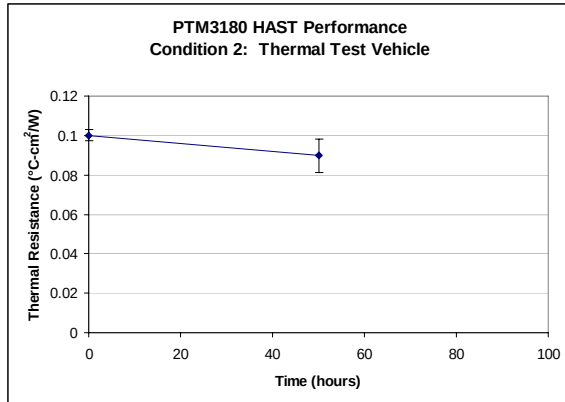


Figure 11: PTM3180¹ HAST performance for condition 2, Thermal Test Vehicle

During the development of PTM3180¹, it was noted that JEDEC standard 22-A110b considers THB (85 °C, 85 % RH, 1000 hours) to be usually equivalent to HAST A and B [13]. However, as previously discussed, Sinnadurai noted that above 130 °C, polymers exposed to high humidities are found to debond [4]. Prasher shows the onset of oxidation for a PCM TIM to be 125 °C [3]. Therefore it was decided to test one of the PCM TIMs in THB using condition 3 unclamped. As seen in Figure 12, the unclamped samples degraded significantly after only 48 hours of HAST A testing. However, the unclamped samples exposed to THB showed no degradation after 1000 hours.

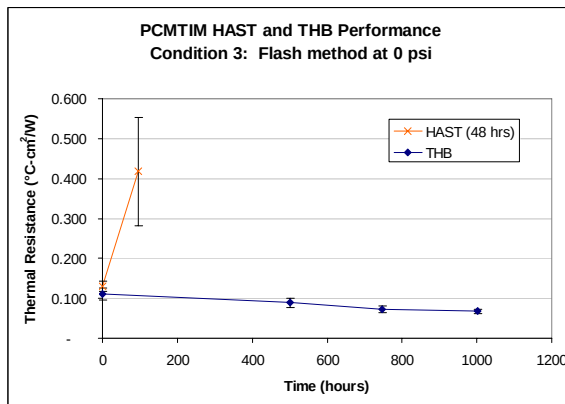


Figure 12: HAST THB Comparison

Ramaswamy et. al. also measured performance differences between HAST and THB while testing PCM TIM performance on single chip modules [2]. In their testing, HAST produced mild degradation in TIM performance while THB produced measurable improvement in TIM performance. Interestingly, Ramaswamy used 121 °C as the temperature for his test. While he does not explicitly mention the duration of his HAST test, a close examination of his graph of HAST performance yields a time of 143

hours. This agrees quite well with Equation (4) given by Sinnadurai (121 °C 85 % RH for 143 hours is equivalent to 130 °C, 85% RH, 96 hours).

Most device suppliers require HAST A performance for their products; therefore, TIMs must pass the HAST A test if only for the purpose of assisting accelerated testing efforts. Fortunately acceptable HAST performance can be obtained with a formulation change. However, the results shown in Figure 12 provoke an interesting discussion that is likely not historically new. One item that receives little attention is the ambient conditions the reliability tests are attempting to accelerate to. Sinnadurai is very clear on this topic in his work and provides Equation (4) as a means for calculating the acceleration factor for a given set of test conditions and ambient conditions. Using this equation, the HAST A test represents 4.25 years of exposure to Sinnadurai's subtropical India region (Table 1). Also according to Equation (4), the equivalent THB test would last 856 hours, about 6 days less than the currently accepted 1000 hours.

Conclusions

The manner in which a TIM is fixtured for a reliability test significantly influences the effect of the test on the TIM. The thermal performance of a TIM that is clamped at 40 psi between two copper blocks (similar to a TIM2 environment) is hardly affected by HAST and THB while the thermal performance of a TIM that is assembled on a TTV or lightly clamped between silicon and nickel degrades significantly. Therefore, when testing a TIM for reliability performance, it is important to replicate the conditions the TIM will encounter in its final application. For a TIM2, testing between copper blocks may well represent that application. However, for a TIM1, a test with more realistic conditions is required.

To replicate TTV-like conditions for TIM1 reliability testing, the Flash method allows for a wider selection of materials on either side of the TIM1. It was shown that unclamped and moderately clamped 3-layer sandwiches of silicon, TIM, and nickel-plated copper show degradation trends similar to thermal test vehicles. This technique was utilized to formulate a new TIM1, PTM3180¹, with improved HAST A performance and these results were verified on a thermal test vehicle.

Finally, it is important to remember that accelerated reliability tests are carefully developed based on many years of actual data. While certain standard versions of these tests exist, the experimenter is still

free to adjust the testing parameters. In the case of some polymeric materials, it is possible to over accelerate reliability testing. By choosing a test with a lower acceleration factor (lower temperature and hence longer test time), the experimenter may find that certain materials are in fact adequate for their application. While the testing time may be slightly longer, the choice of lower-cost materials may result in a competitive advantage.

References

- [1] F. P. Incropera, D. P. DeWitt, "Fundamentals of Heat and Mass Transfer", John Wiley & Sons, third edition, New York, Chapter 3, pp. 86-92, 1990.
- [2] C. Ramaswamy, S. Shinde, F. Pompeo, W. Sablinski, S. Bradley, "Phase Change Material as a Viable Thermal Interface Material for High-Power Electronic Applications", Proceedings of the 2004 Inter Society Conference on Thermal Phenomena (ITherm), Las Vegas, Nevada, June 1-4, pp. 687-691, 2004.
- [3] R. Prasher, "Thermal Interface Materials: Historical Perspective, Status, and Future Directions", Proceedings of the IEEE, Vol. 94, No. 8, pp. 1571-1596, August 2006.
- [4] N. Sinnadurai, "The Use and Abuse of HAST", Proceedings of the Third ESA Electronic Components Conference (ESTEC), Noordwijk, April 22-25, pp. 375-379, 1997.
- [5] N. Sinnadurai, "The Accelerated Ageing of Plastic Encapsulated Semiconductor Devices in Environments Containing a High Vapour Pressure of Water", Microelectronics and Reliability, Vol. 13, 23-27, 1974.
- [6] N. Sinnadurai, "More than a Decade of the Non-saturating Autoclave as a Highly Accelerated Stress Technique for Evaluating the Reliability of Nonhermetic Microelectronic Components", Microelectronics Reliability, Vol. 23, No. 5, pp. 833-836, 1983.
- [7] N. Sinnadurai, "Plastic Packaging is Highly Reliable", IEEE Transaction on Reliability, Vol. 45, No. 2, June, 1996.
- [8] Lee, T.R., Purdue University Ph.D. Thesis, "Thermal Diffusivity of Dispersed and Layered Composites", University Microfilms International, 1977.
- [9] Campbell, R. and Smith, S., "Flash Diffusivity Method: A Survey of Capabilities", Electronics Cooling, Vol. 8, No. 2, 2002. http://electronics-cooling.com/articles/2002/2002_may_a4.php
- [10] Test Method D5470-01, "Standard Test Methods for Thermal Transmission Properties of Thin Thermally Conductive Solid Electrical Insulation Materials", Annual Book of ASTM Standards, Vol. 10.02, 2001.
- [11] Test Method E1461-01, "Standard Test Method for Thermal Diffusivity of Solids by the Flash Method", American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 14.02, 2001.
- [12] Test Method JESD22-A110-B, "Highly-Accelerated Temperature and Humidity Stress Test (HAST)", JEDEC Standard 1999.
- [13] Test Method JESD22-A101-C, "Steady State Temperature Humidity Bias Life Test", JEDEC Standard 2009.
- [14] Test Method JESD22-A104D, "Temperature Cycling", JEDEC Standard 2009.
- [15] Test Method JESD22-A103C, "High Temperature Storage Life", JEDEC Standard 2004.

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