2023

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2023 22nd IEEE Intersociety Conference on Thermal and Thermomechanical Phenomena in Electronic Systems (ITherm) | 979-8-3503-2166-1/23/$31.00 ©2023 IEEE | DOI: 10.1109/ITHERM55368.2023.10177650

In situ Optical Observations of Degradation of Thermal Greases with Thermal Cycling

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***Abstract*—Power requirements and cycling requirements of electronic devices have increased significantly over the past several years. Within electronics packages, thermal greases or thermal interface materials (TIMs) are extensively used to minimize contact resistance and enable effective heat flow paths for heat dissipation. However, due to time-varying heat loads from semiconductor chips as system use varies, thermal greases undergo thermal (and thermomechanical) cycling and often experience deterioration over their lifetime. The degradation can appear as regions with a deficit of the material or the separation of the matrix polymer from particles that give the material high thermal conductivity. In either case, this can lead to local hotspots with excessive junction temperatures. Past work on reliability of thermal greases documents a range of methods to accelerate the deterioration process but general focus on analyzing the system after failure. Here we develop a system for observing the redistribution of the thermal grease during thermal cycling with fixed bondline thickness (BLT). Briefly, the thermal grease is squeezed between a heater and an optically transparent substrate with shims to define the BLT during cycling. In this work, we evaluate degradation of two TIMs, differing in viscosity and thermal conductivity, for four fixed BLTs and two power cycle times. Periodically throughout the cycling process optical images are taken to observe the evolution of the thermal grease. Metrics for analyzing pump-out data include analysis of the boundary migration, as well as the evolution of the void fraction and TIM area. This *in situ* analysis of TIM deterioration will help to identify the governing factors for TIM pump-out and potential mitigation strategies.**

***Index Terms*—Thermal Interface Materials (TIMs), pump-out, power cycling, degradation, electronic packaging**

NOMENCLATURE

μ	Micro
BLT	Bondline Thickness
CTE	Coefficient of Thermal Expansion
PC	Power Cycling test
TC	Thermal Cycling test
THT	Temperature and Humidity Stress test
TIM	Thermal Interface Material

I. INTRODUCTION

The recent decade experienced an increase in demand for semiconductor applications such as electronic packages, automotive systems, quantum computing, and many other areas employing silicon chips. Along with increasing the computational power and capacity, device miniaturization gained priority and led to the need for high performance thermal management solutions. A typical electronic package comprises various layers of silicon chips, heat spreaders, and the heat sink and, thus, the numerous interfaces generally limit heat dissipation due to contact resistances. In order to minimize the contact resistance, thermal interface materials (TIMs) are employed that increase the heat transfer across the interface. Depending on the application, TIMs are available in different forms, such as phase change material-based TIMs, thermal greases or pastes, thermal adhesives, and thermal pads.

Thermal greases have gained attention in recent years due to their ability to conform to different mating surfaces, irrespective of the material and surface roughness. Thermal greases generally contain thermally conductive (often metallic) particles suspended in a polymer matrix. Polysiloxanes or silicone oils are typically used as base polymers due to its excellent wetting characteristics and thermal stability. In addition, applying thermal grease between the surfaces reduces the air pockets within interstitial voids when two surfaces are pressed together. Compared to some other thermal interface materials, thermal greases also have the advantage of being reworkable: the TIM can be cleaned from the interface and new material applied. But thermal greases suffer from two key degradation mechanisms over time: pump-out and dry-out.

An electronic package experiences cycles of power intensities of different frequencies and intensities. The cycling thermal loading and the coefficient of thermal mismatch (CTE) of different materials lead to package warping. Based on the temperatures, duration of the cycle and displacement from warping, thermal greases often degrade and create local hotspots. The degradation can be in two forms: pump-out and dry-out. Pump-out is a phenomenon where thermal greases shift from their initial location due to warping and lead to TIM-free regions where hotspots are generated [1], while dry-out is caused when the polymer matrix drains from the regions, leaving dried residues on the surface [2], [3]. The disintegration causes local temperatures to increase significantly.

Degradation of thermal grease in electronic packages necessitates reliability analysis and various tests to assess long-term thermal stability. The assessments are aimed toward the ability to maintain the beginning of life thermal resistance [4]. Some tests include temperature and humidity stress tests (THT), temperature or power cycling tests (TC/PC), and high-temperature bake tests. Temperature and humidity stress tests aim to induce moisture in the TIM to understand their degradation. In this test, TIM is kept under a constant temperature and humidity ratio for a longer duration [4]. High temperature storage tests use the JESD22-A103 standard to observe TIM degradation at high temperatures [5]. Temperature cycling or power cycling tests evaluate TIM reliability with repeated cycles either between fixed temperatures or varying power levels. The cycling tests aim to observe the evolution of the TIM due to thermomechanical stresses similar to those experienced during application [6]. Typically these tests conform to the JESD22-A104D or JESD-A105C standards [4].

Although the standardized tests provide useful information, they take significant time (on the order of thousands of hours), so there is a clear need to develop accelerated tests. Chiu *et al.* [1] induced rapid mechanical oscillations while holding the system at operating device temperatures and performed nearly 6000 cycles to observe TIM degradation in less than an hour. Wunderle [7] performed experiments under similar conditions with a capability to observe *in situ* behavior. A common finding amongst these and similar tests is the dependence of TIM degradation on a combination of different parameters such as clamping pressure, power cycle duration, and rheological properties of the thermal grease.

There is also a link between the structural changes in the TIM configuration within the interface and its thermal performance. In one study, thermal resistance was observed to increase with decreasing coverage area of the TIM [8]. In contrast, pump-out studies from Yang *et al.* [9] showed an initial increase and then a slight decrease in thermal resistance over 500 cycles and Gowda *et al.* [2] found that the thermal resistance decreased after 1000 power cycles. They used laser flash diffusivity method to characterize the thermal performance and acoustic microscopy to visualize the degradation pattern of the TIM configuration. The decreasing trend in resistance with cycling was attributed to the greater wetting capacity of the TIM with lower viscosity at higher temperatures and smaller BLTs. In addition, the void fraction, representing the structural degradation, was most significant for the TIM with lower viscosity at higher pressures (45 psi) and relatively higher surface roughness (1000 nm). The differing trends could be related to variation in the tested materials. For instance, Kusuma *et al.* [10] found the combination of sodium silicate matrix and zinc oxide to have lower thermal resistance and greater thermal stability than the conventional silicone matrix and metal-filled thermal grease. While low viscosity is necessary for filling gaps in the interfacial contact, for TIMs with a high thixotropic index value (that is, better dispensability), the grease is prone to move out of the die area during operation [11]. Similarly, Carlton *et al.* [12] experimented with two greases (silicone and non-silicone oils, with the former having a lower viscosity). For constant temperature mechanical cycling tests (up to 1000 cycles), for the silicone oil at 50°C and 80°C, the normalized thermal resistance at end of cycling increased with increasing strain rate. The *in situ* photos revealed that the silicone oil was more prone to void formation than the non-silicone oil. These works illustrate the variability in response of thermal greases to cycling under different conditions and motivates the need for simultaneous characterization of the structural configuration of the TIM and metrics of the thermal performance throughout the cycling process.

Much of the past literature evaluates TIM degradation through the evolution of thermal resistance trends over power-cycling tests. Despite numerous data on thermal resistance trends, only a few show images of the TIM before and after cycling and fewer discuss *in situ* observation of the thermal grease behavior. In addition, there is a need to evaluate the dependence of TIM pump-out on parameters such as BLT, cycle time and material properties of the grease. Moreover, the studies in literature generally evaluate the combined effect of thermomechanical stresses on TIM performance. In order to further understand the pump-out behavior, isolating the effect of temperature cycling alone (minimal mechanical movement) is necessary, along with *in situ* imaging of the motion of the thermal grease.

This paper investigates the degradation behavior of two thermal greases with control over BLT and cycle time. Samples are power cycled at fixed BLTs with multiple cycle times (4 and 10 min duration with 50% duty cycle, representing frequent and moderate chip usage in electronic packages). The power level is chosen to ensure the grease experiences temperatures from 20-100°C. The BLT is varied from 20 μm to 100 μm, a range which is relevant to industrial applications. Two greases are chosen based on their contrasting thermal conductivity and viscosity: DOWSIL TC-5622 Thermally Conductive Compound (DOWSIL 5622 in upcoming text) has low viscosity and high thermal conductivity, and DOWSIL 340 Heat Sink Compound (DOWSIL 340 in the upcoming text) possesses a high viscosity and low thermal conductivity. We first discuss the experimental methodology and cycling parameters, then evaluate and compare the behavior of the two thermal greases to understand the pump-out phenomenon.

II. Methodology

Two thermal greases are power cycled to observe the degradation behavior and understand key parameters affecting TIM degradation. The workflow is presented in Fig. 1. Briefly, the experiment begins with initially dispensing the grease on the thermal cycling test rig. The TIM is then compressed with an optically-transparent wafer to the desired BLT. Shims define the BLT to primarily focus on the effect of temperature, rather than the combined effect of temperature and thermally-driven mechanical oscillations. An optical image of the grease is taken just after dispensing the TIM and before the power cycling. The heater is then power cycled for 3 days and optical images are taken after every 24 hours. The images are then processed to estimate the overall TIM area and void fraction to understand the evolution of the TIM with cycling. The following sections further describe the experimental procedure with a brief description of the selected thermal greases, details of the experiment setup and procedure, and details of image processing.

In this study, we evaluate two thermal greases (DOWSIL 340 and DOWSIL 5622), two cycling times (4 and 10 mins), and 4 BLTs (20, 46, 84, and 100 μm). Table I shows the combinations of experimental parameters evaluated in this work.

A. Thermal Grease Selection

Careful selection of the thermal greases is required to understand which parameters impact degradation. In this investigation, two TIMs are chosen with different viscosity and thermal

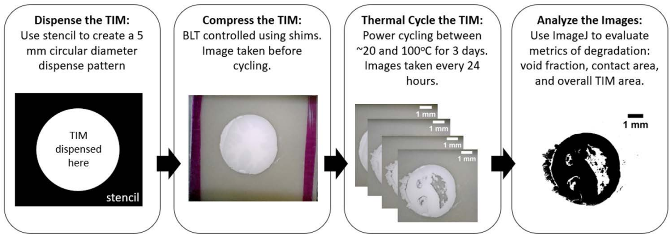


Fig. 1. Overview of the experimental workflow. First, the TIM is dispensed in a 5 mm circular pattern using a stencil. Then, the sample is compressed with shims in place to maintain the desired BLT. Next, images are taken every 24 hours during thermal cycling. Finally, the images are processed and analyzed using ImageJ.

TABLE I
LIST OF EXPERIMENTS

Test	Thermal Grease (Name)	Cycle Time (min)	BLT (μm)
1	DOWSIL 5622	4	20
2	DOWSIL 5622	4	46
3	DOWSIL 5622	4	84
4	DOWSIL 5622	4	100
5	DOWSIL 5622	10	20
6	DOWSIL 5622	10	46
7	DOWSIL 5622	10	84
8	DOWSIL 5622	10	100
9	DOWSIL 340	4	20
10	DOWSIL 340	4	46
11	DOWSIL 340	4	84
12	DOWSIL 340	4	100
13	DOWSIL 340	10	20
14	DOWSIL 340	10	46
15	DOWSIL 340	10	84
16	DOWSIL 340	10	100

conductivity: DOWSIL 5622 and DOWSIL 340. Table II lists the viscosity, thermal conductivity, and yield stress values of the two TIMs. The table lists the viscosity values from the datasheet and their corresponding shear rate from internal rheology experiments. While the viscosities are reported for higher shear rates like stencil printing or dispensing, the study aims to base its conclusions on the relative viscosities between the TIMs. DOWSIL 5622 has a lower yield stress and viscosity, and is a ready-to-flow material. DOWSIL 340, on the other hand, is more resistant to flow, more viscous, and has a lower thermal conductivity than DOWSIL 5622. Thus, we expect different degradation behavior for the two TIMs. Future studies will expand the range and combinations of properties evaluated.

B. Experiment Setup and Procedure

The test fixture (see Fig. 2) comprises stacked layer including a ceramic heater (Watlow Heaters, Ultramic 600, CER-1-01-00333) inset into a Teflon insulation housing, the dispensed thermal grease maintained at a constant BLT using shims, and a optically-transparent cover glass wafer held in place with clamps. At the beginning of each test, we dispense the thermal grease on the heater using a stencil and a squeegee to form a 5mm diameter circular patch. The heater is connected to a DC power source, a relay, and a function generator to automate the power cycling of the heater. An optically-transparent cover glass compresses the TIM down to the desired BLT (maintained with shims) and allows visual observation of the behavior of the TIM during power cycling. A blower forces airflow over the glass to improve the heat through the grease (as opposed to the through the Teflon insulation). The fixture is placed under an optical microscope and helps to record the evolution of grease parameters.

Before cycling tests, the power level is determined based on the magnitude needed such that when heated the heater temperature sensor is $\sim$100^{o}C. The heater is powered on and off with either 4 min or 10 min cycle time at 50% duty cycle (*i.e.*, 2 mins on/2 mins off or 5 mins on/5 mins off) using the function generator and relay system. The fan runs continuously and aids during heating in establishing a temperature gradient across the TIM similar to the application where a heat sink would be attached on the top side.

TABLE II
TIM PROPERTIES

TIM Name	Thermal Conductivity (W/(m K))	Dynamic Viscosity (Pa s)	Yield Stress (Pa)
DOWSIL 5622	4.3	95 @ 0.6 s^{-1}	42
DOWSIL 340	0.67	542 @ 0.15 s^{-1}	70

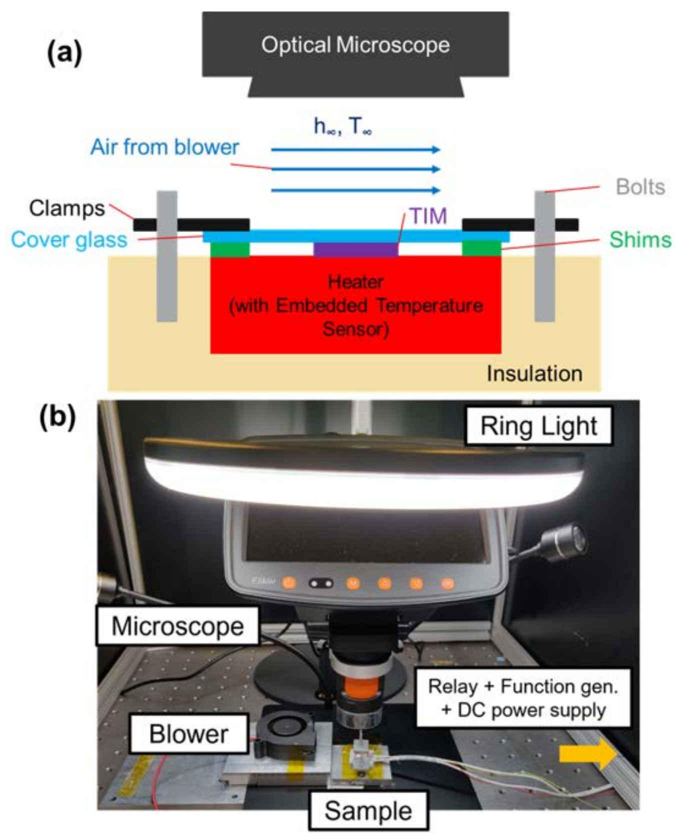


Fig. 2. (a) Schematic and (b) photo of the experiment setup: Heat generated from the heater flows through TIM to cover glass, with its top surface being cooled by blower. Clamps are used to hold the assembly in place with a constant BLT using shims. Insulation around the heater minimizes the parallel heat loss pathway. A digital optical microscope images the TIM through the coverglass from the top.

C. Image Processing

Optical images of the grease are taken every 24 hours, as well as prior to the first power cycle. From initial observation it was clear that when the TIM moves, in addition to voids (no TIM present), there are also portions of the of the TIM only in contact with the bottom surface of the fixture and regions where the TIM appears to still span the gap from the bottom heater surface to the cover glass. The optical images are processed in ImageJ to provide quantifiable information including:

- the area of the voids,
- the area of the grease in contact with both the top and bottom surfaces, and
- the area of the grease in contact with just the bottom surface

Several image processing steps are required to quantify these parameters. First, the image is converted into an 8-bit grayscale image. Then, the image is converted to black and white using a 'default' threshold and an inverting threshold to identify the voids. Figure 1 presents before and after images of image processing for one of the experiment conditions.

To identify the TIM contact area with just the bottom surface, for each image, a boundary is manually traced around the outermost perimeter of the grease. Then, a boundary is traced within the TIM at the interface where contact with the top surface is lost. Several image processing algorithms were evaluated, but due to the inconsistent geometries, a manual approach at identifying the boundaries is employed here.

III. Results

The qualitative images and quantitative metrics for each of the two thermal greases with different BLTs and cycle times provide insight into the degradation of these TIMs. The series of optical images for all the experiment conditions are presented for DOWSIL 340 and DOWSIL 5622 in Tables III and IV, respectively. For the the results shown here, "As Dispensed" refers to the image taken just after the grease is dispensed with the heater switched off, while "Day 0" is the image taken after the first power cycle. The labels Days 1-3 refer to images taken 24, 48, and 72 hours after Day 0.

Based on the processed versions of the optical images, the quantitative data for the grease contact area with the top and bottom surface, contact area with the bottom surface, and the void area are reported in Figures 3 and 4 for DOWSIL 340 and DOWSIL 5622, respectively. The evolution of the area of the voids are also separately presented in Figures 5 and 6 for DOWSIL 340 and DOWSIL 5622, respectively.

As the TIM is cycled, the grease moves outward (overall area increases), but it loses contact with the top surface. While DOWSIL 5622 expands, DOWSIL 340 is found to disintegrate. The following sub-sections consider the degradation of the thermal greases in more detail based on the BLT, cycle time, and rheological properties of the TIM.

A. Effect of the Bondline Thickness

The bondline thickness significantly impacts the thermal degradation behavior of the thermal greases. For DOWSIL

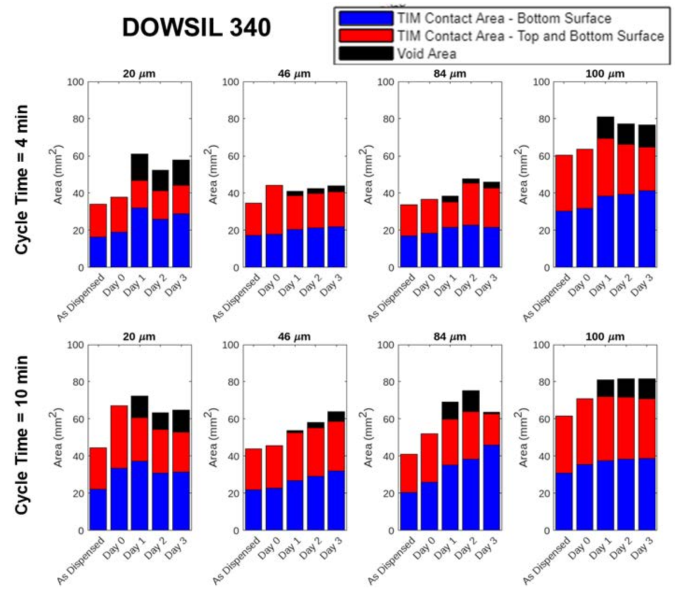


Fig. 3. Breakdown of the TIM Areas for DOWSIL 340 comprising the area of the TIM in contact with both top and bottom surface (blue), the area in contact with just the bottom surface only (red), and area where voids have formed (black).

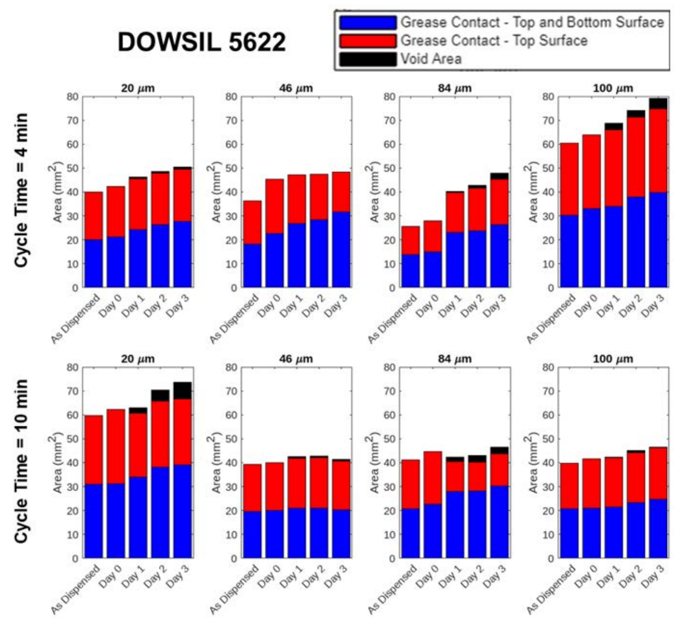
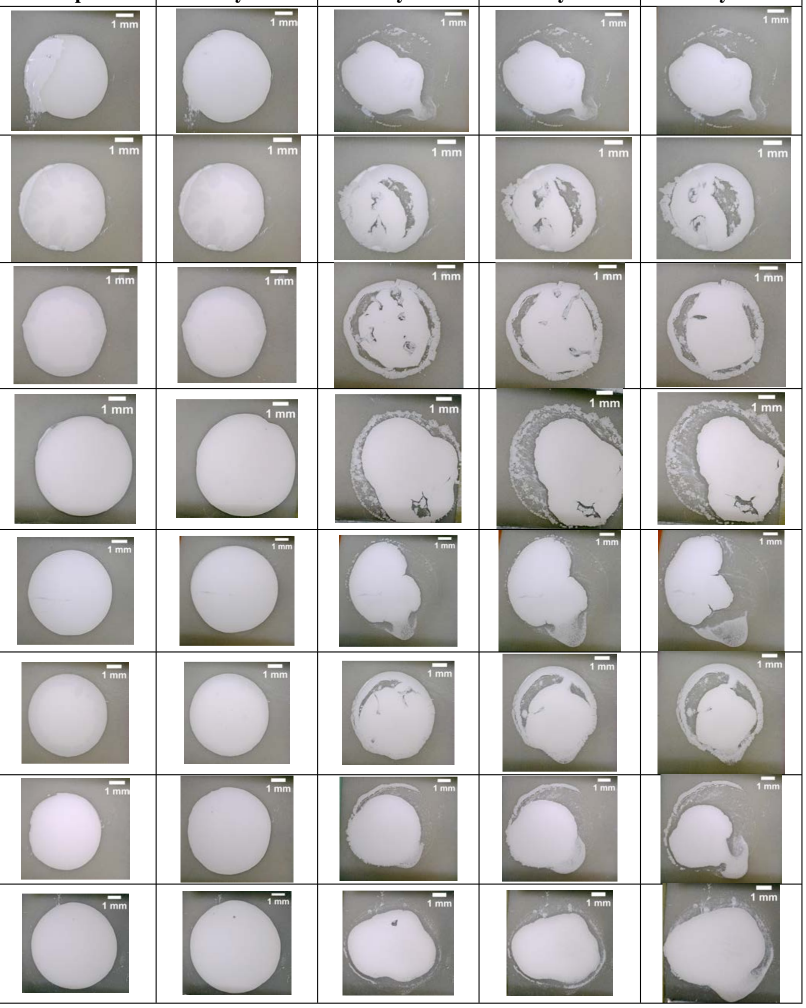


Fig. 4. Breakdown of the TIM Areas for DOWSIL 5622 comprising the area of the TIM in contact with both top and bottom surface (blue), the area in contact with just the bottom surface only (red), and area where voids have formed (black).

5622, at thicker BLTs, void formation and propagation is more prevalent. On the other hand, DOWSIL 340 disintegrates due to polymer matrix separation at higher BLTs. At lower BLTs, void generation or disintegration is less significant and the grease appears to be present throughout the thickness of the interface in much of the overall TIM area. This is also reflected with same areas for top and bottom and just the bottom. Although thinner BLTs help reduce the TIM degradation, we note that it could be challenging to achieve thin BLTs in high

TABLE III
Optical Images of TIM Evolution - DOWSIL 340

Cycle Time	BLT	As Dispensed	Day 0	Day 1	Day 2	Day 3
4 min	23 μm					
	46 μm					
	84 μm					
	100 μm					
10 min	23 μm					
	46 μm					
	84 μm					
	100 μm					



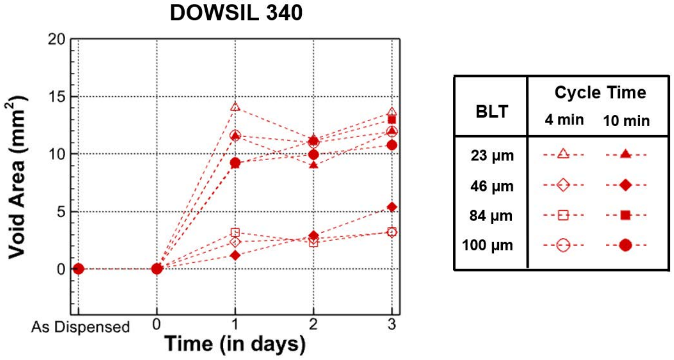


Fig. 5. Evolution of the void area for DOWSIL 340 with varying BLT (varying symbol type) and cycle duration (hollow symbols = 4 mins and filled symbols = 10 mins).

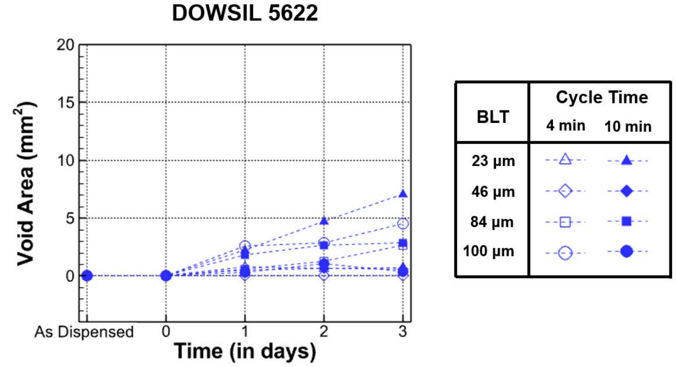


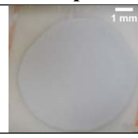
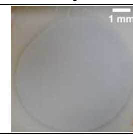
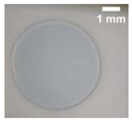
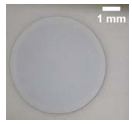
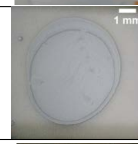
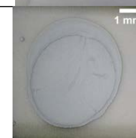
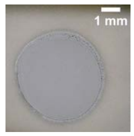
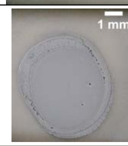
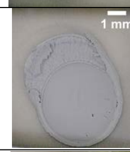
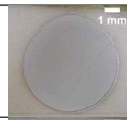
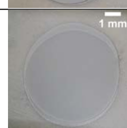
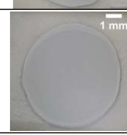
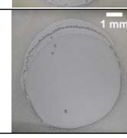
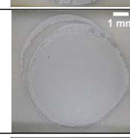
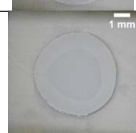
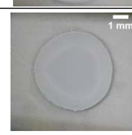
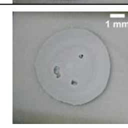
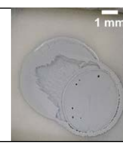
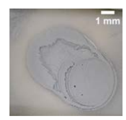
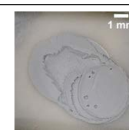
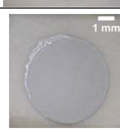
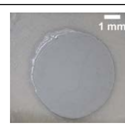
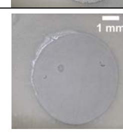
Fig. 6. Evolution of the void area for DOWSIL 5622 with varying BLT (varying symbol type) and cycle duration (hollow symbols = 4 mins and filled symbols = 10 mins).

volume manufacturing. Overall, it appears that thicker BLT appear to accelerate degradation.

B. Effect of Cycle Time

The cycle time is the duration of heating and cooling of the grease. Both greases are observed to expand in within the first day. The behavior of the greases after Day 1 depends on the TIM. It appears that the low viscosity of the DOWSIL

TABLE IV
OPTICAL IMAGES OF TIM EVOLUTION - DOWSIL 5622

Cycle Time	BLT	As Dispensed	Day 0	Day 1	Day 2	Day 3
4 min	23 μm					
	46 μm					
	84 μm					
	100 μm					
10 min	23 μm					
	46 μm					
	84 μm					
	100 μm					

5622 allows it to continue to expand, while DOWSIL 340 disintegrates leaving behind a residue. Longer duration of heating promotes grease expansion in DOWSIL 5622 and retention of the same contact area (from the bottom to top surfaces) in DOWSIL 340. Shorter cycles limits the expansion and is seen to cause more significant disintegration within DOWSIL 340, based on the decreasing contact area with top surface.

C. *Effect of TIM Rheological Properties*

The rheological properties of the thermal greases likely play an important role in determining the degradation behavior. DOWSIL 5622 is relatively a low viscosity thermal grease, which enables it to flow to adjust its area before voiding within the material. On the other hand, DOWSIL 340 is more viscous and expands relatively lesser than DOWSIL 5622. Moreover, the polymer matrix separates during power cycling, drains away from its original position, and leaves behind some residues. This can be observed as decreasing the area of the grease in contact with both the bottom and top surfaces. DOWSIL 5622 is also observed to have greater differences between the areas in contact with both top and bottom compared to just the bottom over the 3 days, while DOW 340 have nearly same areas.

D. *Statistical Analysis*

The metrics from the optical images during the thermal cycling of the TIM help in understanding the impact of each parameter on the thermal degradation. However, distinguishing the effect of each experimental parameter (*i.e.,* BLT, Cycle Time, TIM) from the plots alone is challenging. Therefore, an Analysis of Variance (ANOVA) test is carried out with the split-split plot design (see Figure 7) considering 95% confidence level([13]). The split-split block design is implemented with three parameters (BLT, Cycle Time, TIM) that can impact the evolution of the void areas over the 4 days. Table V illustrates the effects of each parameter and interaction between each parameter. In particular, the p-value indicate if a parameter or interaction is important. Specifically, a factor is statistically significant if the p-value is less than 0.05 and

TABLE V
STATISTICAL ANALYSIS

Factor	Degree of Freedom	Sum of Squares	Mean Square	F-value	p-value	Significant?
BLT	3	172.8	57.6	4.77	0.0055	Yes
Cycle Time	1	9.0	9.0	0.75	0.39	No
TIM	1	365.3	365.3	30.24	1.45×10^{-6}	Yes
BLT $\times$ Cycle Time	3	51.4	17.1	1.42	0.25	No
BLT $\times$ TIM	3	64.5	21.5	1.778	0.16	No
TIM $\times$ Cycle Time	1	0.5	0.5	0.039	0.84	No
TIM $\times$ Cycle Time $\times$ BLT	3	52.8	17.6	1.455	0.24	No
Residual	48	579.9	12.1			

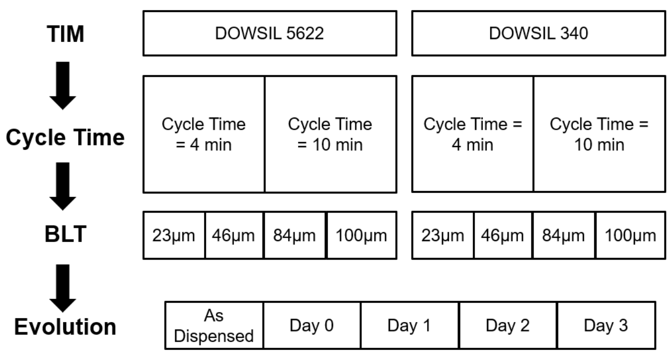


Fig. 7. Overview of the split-split plot design for the thermal grease degradation experiment with TIM, cycle time, and BLT as blocks with evolution days as treatment.

here the BLT and TIM are both significant. This implies both of these factors play a crucial in increasing void areas during cycling, while cycle time and interactions between the factors are not statistically significant. This indicates the factors can each be considered independent.

IV. CONCLUSIONS

The study presents an *in situ* observation of thermal grease degradation with power cycling. Two thermal greases of different viscosities and thermal conductivities were power cycled for 3 days with two different cycle times with a fixed BLT of 20 μm, 46 μm, 84 μm, and 100 μm. Optical images illustrate the evolution of the grease and enable quantification of the area of the grease in contact with both the top and bottom surface, in contact with just the bottom surface, and where contact was completely lost (voids). Both thermal greases expand within the first day and their evolution thereafter varied. DOWSIL 5622 expanded (likely due to its low viscosity), while the high viscosity of DOWSIL 340 resisted expansion, but ultimately the TIM failed due to disintegration or separation of the polymer. Results from statistical analysis align with the qualitative evaluation that BLT and the choice of TIM significantly affect the thermal degradation. Specifically, higher BLT generally promotes degradation, perhaps due to the non-uniformity within the thickness of the grease or in-plane shearing of the grease. With the objective of minimizing grease degradation, these preliminary results suggest it is preferable to have lower viscosity. Viscosity enables flow of the grease and helps to refill the voids formed during cycling. Overall, the impact of varying cycle time (4 vs. 10 mins) was statistically insignificant. This analysis of grease deterioration helps in understanding the effects of different parameters and suggests strategies to focus on for minimizing degradation.

ACKNOWLEDGMENT

Financial support for this work provided by members of the Cooling Technologies Research Center, a graduated National Science Foundation Industry/University Cooperative Research Center at Purdue University, is gratefully acknowledged.

We thank Prof. Ivan Christov for helpful discussions regarding the impact of viscosity and temperature-dependent viscosity on pump-out.

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